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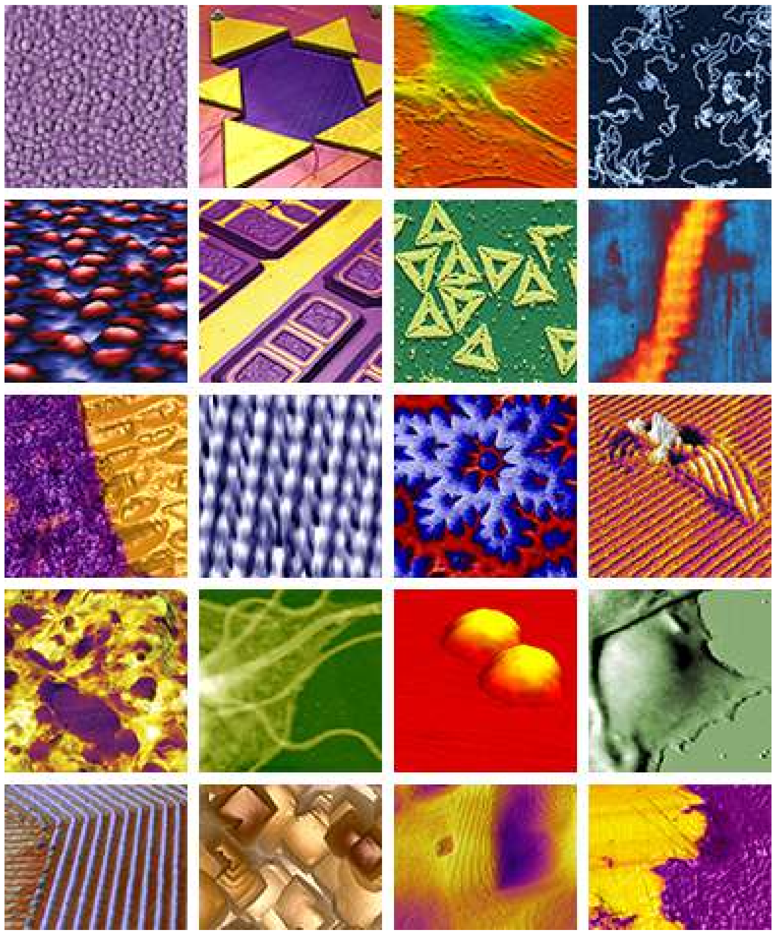
SPM Applications Guide

USER GUIDE 3

Applications Guide



Including beta (complete, reviewed) chapters.

Version 18, Revision: A-2437

Dated 09/04/2021

Asylum Research

an Oxford Instruments company

Contents

Introduction

Volumes of the AR Imaging and Spectroscopy Applications Guide

The Asylum Research Scanning Probe Microscope (SPM) Software manual comes in volumes. To date, these volumes include:

Part I *SPM Imaging Techniques.* Step by step instructions for various imaging techniques. One chapter per imaging Mode (*e.g.* Contact Mode, AC Mode, Conductive AFM, *etc.*) In some cases an additional chapter for extensive theoretical background.

Part II *SPM Non-Imaging Techniques.* Step by step instructions for various non-imaging techniques. One chapter per Mode (*e.g.* Force Spectroscopy, Force Volume Mapping, Nanolithography, Nanomanipulation, *etc.*)

Part III *Spring Constant Calibration and Thermals.* Step by step instructions for cantilever calibration techniques. Background and theory are devoted when necessary.

Part IV *Supplemental Information.* Extra applications background information.

Part V *Bibliography, Glossary, and Index* . Self explanatory. Covering all parts of this user guide.

AR SPM Software version It is assumed that AR SPM Software version 14 or later is installed on your system. The current AR SPM Software release is version 18.

Getting Help For additional help with your Asylum Research instrument, including software support, refer to: https://afm.oxinst.com/Support-US

Updates to the Manual Bundled with the software updates.

Send Feedback Send e-mail to sba.manuals@oxinst.com (clickable link) and mention which version of the user guide you are using and the chapter and section you are commenting on.

Prerequisites

We recommend that you have a running AFM or at least a functioning copy of the Asylum Research SPM software installed on your computer. For an overview of a properly set up MFP-3D AFM, please refer to *MFP-3D User Guide, Chapter: Installation*. Likewise, for the Cypher AFM, a properly operating AFM system includes a PC with the AR SPM software installed.

Part I

SPM Imaging Techniques

Part I: Who is it for? Succinct step-by-step instructions for various imaging techniques. Light on theory and gets to the point.

Part Contents

1. Contact Mode

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

1.1. Preparation

1.1.1. Cantilever Selection

Any standard contact cantilever will work in this mode. A list of contact cantilevers we offer can be found here: Contact Cantilevers.

1.1.2. Preparation and Tutorial

Contact Mode imaging is sufficiently commonplace that a tutorial has been developed for it. If you own an MFP-3D family AFM, please follow *MFP-3D User Guide, Chapter: Tutorial: Contact Mode Imaging in Air*. If you own a Cypher AFM, please follow *Cypher User Guide, Chapter: Tutorial: Contact Mode in Air*. These will familiarize you with the basics of loading the cantilever, sample, etc. Once everything is adjusted and the cantilever is ready to engage above the sample surface, you can switch over to the sections included here to create high quality images by adjusting parameters in the software. Software differences among the instruments will be covered, as necessary.

1.2. Parameter Selection

This section describes how to select parameters for Contact Mode Imaging.

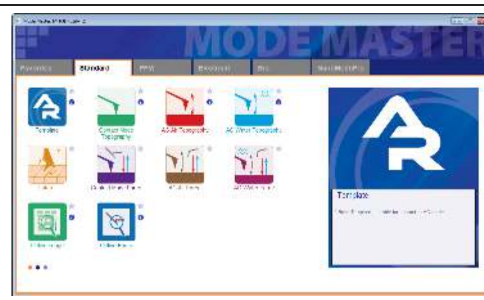
Based on the previous section, it is assumed that:

- The cantilever is close enough to the surface so that the Z piezo actuator can bring the tip and the sample into contact.
- The laser is aligned on the cantilever, and the photodetector difference (deflection) signal has been zeroed.

1.

The Mode Master window:

- The software should now be showing the Mode Master screen.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.

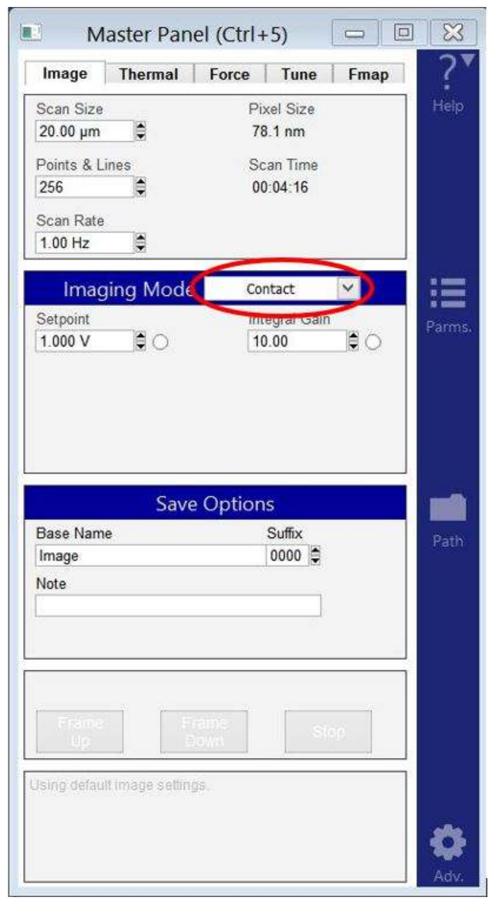
Select Mode:

- Select *Standard tab > Contact Mode Topography*
- The screen now presents all the controls necessary for this type of AFM imaging.

3.

Set up Master Panel:

- In the Main tab of the Master Panel, select *Contact* from the *Imaging Mode* pull-down menu.
- *Setpoint*: 1.0 V.
- *Integral Gain* (I):
  - for the MFP-3D: 8 - 10
  - for the MFP-3D Extended Z: 2-3
  - for the Cypher: 30
- *Scan Rate*: 1 Hz; for softer samples, choose a slower scan rate, perhaps 0.5 or 0.2 Hz.
- Scan angle, resolution (scan "Points & Lines"), and image size is up to you. These are found in the Parms. tab, on the right side of the Master Panel.

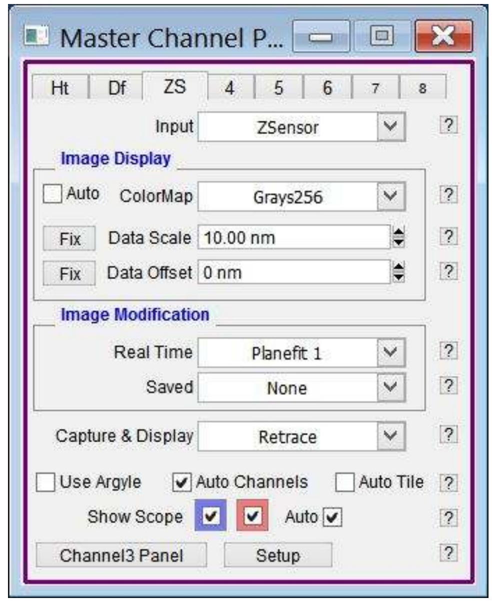


4.

Image channel selection:

- Go to the Master Channel panel.
- Select the leftmost tab and select "Height" under the *Input* pull-down menu. Also set the *Saved* pull-down to "None".
- For the next two tabs, do the same for *Zsensor* and *Deflection*.

Note The *Real Time* and *Saved* pull-downs should be reviewed before scanning begins. It is important that no effect is applied to the saved data in order to preserve at least one unaltered copy of original data. An effect may be selected for scanning, but this setting could also cause image artifacts depending on the topography.



A brief summary of these data channels:

Height: The voltage applied to the Z piezo to maintain defined positive deflection per X,Y scan point. This signal is a linear approximation to a non-linear signal, meaning it is inaccurate at larger scales; for larger scales, you should use the Z sensor. The advantage of the height channel is that it has less noise than the Z sensor channel.

Deflection: The error signal of the feedback loop used to maintain the user setpoint deflection.

Z sensor: The movement of the optical lever detection assembly as monitored by a closed loop sensor. It is recommended to always activate the Z sensor channel when imaging, especially when sample features are larger than a few tens of nanometers; the Z LVDT sensors are more linear than the piezos, and thus give a more precise Z measurement.

Mode Master: Most of what was described in this section is automatically set by the Mode Master when *Contact Mode* is selected from the standard modes.

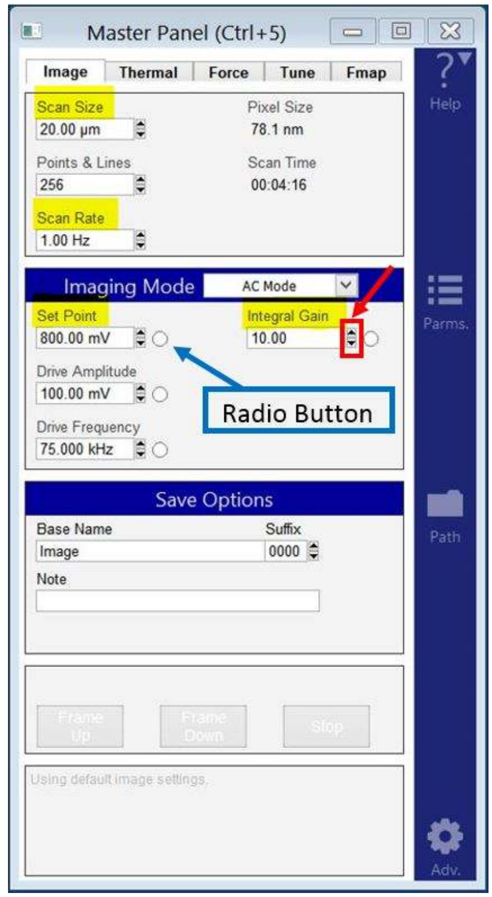
1.3. Optimize Imaging Parameters

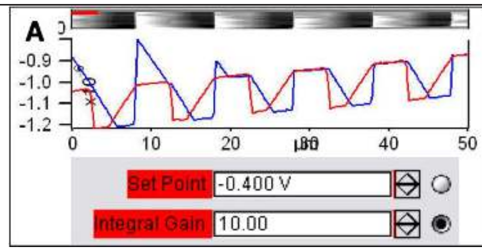
1. Click the 'Frame Up' button on the Image tab. After a moment, imaging will begin.

2.

Fine-tuning the parameters:

- Tune the parameters with *Set Point*, *Integral Gain*, and *Scan Rate*.
- Use the arrow clickers to right of setvar windows to adjust parameters.
- Alternately, you can fine-tune the parameters using the Hamster Wheel on the front of the controller. The Hamster Wheel on the first-generation controller, ARC1 black, is permanently attached to the controller; whereas on the second-generation controller, ARC2 silver, it is detachable.
- Any parameter with a “radio” button next to it can be changed during a scan when it is activated by the Hamster Wheel. The Hamster gives “digital control with analog feel.”
- For the ARC1, there is a toggle switch that allows you to scroll through the parameters. For the ARC2, the outer ring allows you to select the parameter whereas the inner ring changes the value. This is a GREAT feature for tuning on the fly and eliminates the need for the mouse.





3.

Determining image quality:

- Image quality can be monitored by image resolution and by the amount of noise in the line traces (located below images).
- Start the learning process on a sample with a known topography, like an Asylum Research Calibration Grating (basically a matrix of square pits). You then know immediately if the image looks sub-optimal.
- Look at the Scope Trace below the image. This graph represents the most recent line of the image. **Blue** indicates the tip moving left to right (**trace**) and **Red** indicates tip returning from right to left (**retrace**).

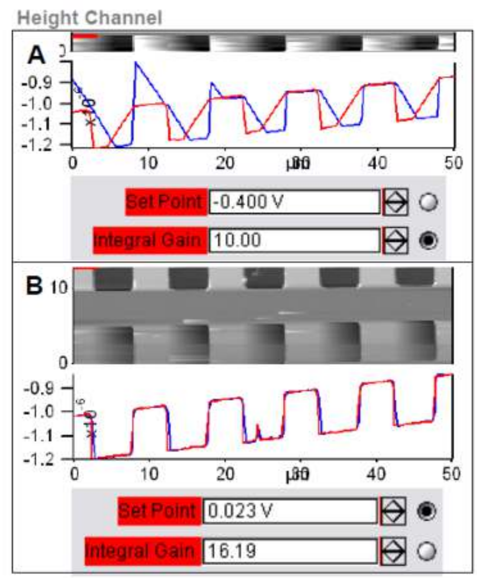
On most samples with relatively slowly changing features, trace and retrace should look the same. In other words, the landscape should look the same if you are flying the exact same route one way or the other. In the image above, the two are quite different; this is an indication that imaging parameters need to be adjusted.

Nomenclature In the previous image, the tip is not following the surface. As the blue trace shows (left to right), the tip seems to climb up out of the pits of the calibration grating quite nicely, leaving a sharp edge along the left side of each pit; but then it descends back into the next pit along a relatively gentle slope. During this descent, the tip actually flies through the air while it is completely undeflected, a bit like a hang glider running off a cliff. The lateral motion of the tip simply marches on as dictated by the XY scan pattern. The feedback control algorithm is simply not aggressive enough to bring the tip back down to the bottom of the pit. Such behavior is commonly called **parachuting** or **poor tracking**.

4.

Adjusting Set Point and Integral Gain

- The *Set Point* voltage and *Integral Gain* should first be adjusted to achieve good tracking. The image to the right shows an example of imaging a calibration grid, with corresponding parameters from the Main tab below.
- Panel A shows the initial tracking of the tip upon engagement with a *Set Point* voltage of -0.4V when the free air voltage was about -0.45V.
- Panel B shows that with an increase in the *Set Point* force and a slight increase in the Integral gain, tracking is improved greatly.



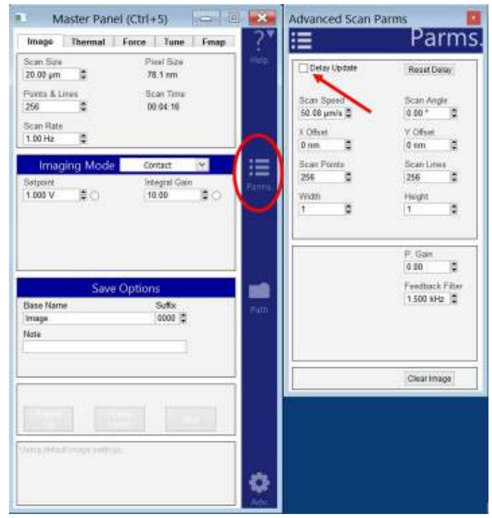
Note Do not be alarmed if you have to crank up the *Integral Gain* when using long, floppy cantilevers – the gain is related to the optical lever sensitivity (more gain for less sensitivity).

5. The second set of parameters to adjust are the *Scan Rate* and *Scan Angle*. Having a vague idea of the orientation of the tip on the cantilever can make scanning at 90 degrees more advantageous than at 0 degrees because of the shape of the tip at the end of the cantilever. Some cantilever manufacturers compensate for the slight angle that the cantilever is mounted relative to the surface by having the front and back angles of the pyramid at different angles.

Delay Update

6.

- The *Delay Update* checkbox allows you to change the parameters during a scan, which will take effect at the end of that scan.
- During the period before update, any parameters changed will be highlighted in orange.
- Notice that there are setvars without radio labels. Highlights occur here because these values only take effect when the frame is finished. (e.g., *Scan Rate*)
- The *Delay Update* checkbox can be found by clicking on the Parms. Tab to the right of the Master Panel, as shown on the image at right.



Q When I make changes to scanning parameters, when do the changes take effect in the scanned image?

A Most imaging parameters in the main tab of the main panel (See 3) update as soon as you make a change. Points, Lines, and Scan Rate do not change mid-image; those parameters will only change at the next frame.

If you check the *Delay Update* box just above the *Setpoint* parameter, any changes you make to parameters above that box will update only when a new image is started. Until the image is complete, the changed variables are highlighted in orange.

You can always force a new image by clicking 'Frame Up' or 'Frame Down'.

A good way to see the effect of changing imaging parameters can be as follows:

- Check the *Delay Update* box as described above.
- Click 'Frame Up' and collect a dozen scan lines. Observe the image quality.
- Make some changes to the scan parameters (number of points, rate, gains, setpoint).
- Click 'Frame Up' again.
- Observe as the exact same scan region is painted over with new data taken with your new parameter choices.

2. Contact Mode Imaging: Scientific Background and Theory

CHAPTER REV. 2397, DATED 06/26/2021, 17:54. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

Contact Mode AFM, also known as Constant Force Mode, is one of the more commonly used imaging modes in AFM. It is often used in imaging hard materials, in some electrical techniques, and in imaging biological materials, such as cells, under low setpoint forces and scan rates. (Because cells are big bags of water, they can be easily oscillated if AC Mode is used on them. Contact Mode works better, and the Deflection images shows a great deal of detail.) It is also among the simplest methods to explain, and therefore, leads the chapters on AFM imaging modes. The typical user will do more AC Mode imaging than Contact Mode imaging but should know Contact Mode imaging as well.

2.1. Theory and Background

An AFM imaging technique typically requires a method by which the tip can track the topography of the sample. A relatively simple method of doing this is to monitor the cantilever deflection. Please refer to Figure 2.2 on page 16.

For the purposes of this discussion, we will assume the tip hovers above the sample surface by a few microns with the Z feedback turned off and the Z actuator fully retracted. The lever is now in its relaxed position. The photodetector is aligned so that the laser beam is centered and the difference output from the photo detector is zero. Any deflection of the lever away from the relaxed position registers as a non-zero voltage: negative for bending toward the sample and positive for bending away. Next, a setpoint voltage for this output is chosen, corresponding to a certain deflection. Usually, the quantity of interest is the force exerted on the sample by the lever. Chapter 20 on page 281 shows how to calibrate detector output voltage in terms of the force on the sample. Once you are familiar with using a particular type of cantilever, you will become accustomed to the forces involved and will probably skip the step of calibrating the force.

To conceptually aid the process of selecting of a setpoint voltage, a qualitative depiction of cantilever deflection vs. Z Sensor is shown in Figure 2.1 on page 15. At the far right of the graph, the tip is above the surface. Further left, the tip is approaching the surface, but since it is not yet touching, the deflection stays the same. At the contact point the tip becomes fixed against the surface, and the deflection voltage starts the increase. The more the Z actuator extends, the more the lever bends, and the bigger the deflection signal gets.

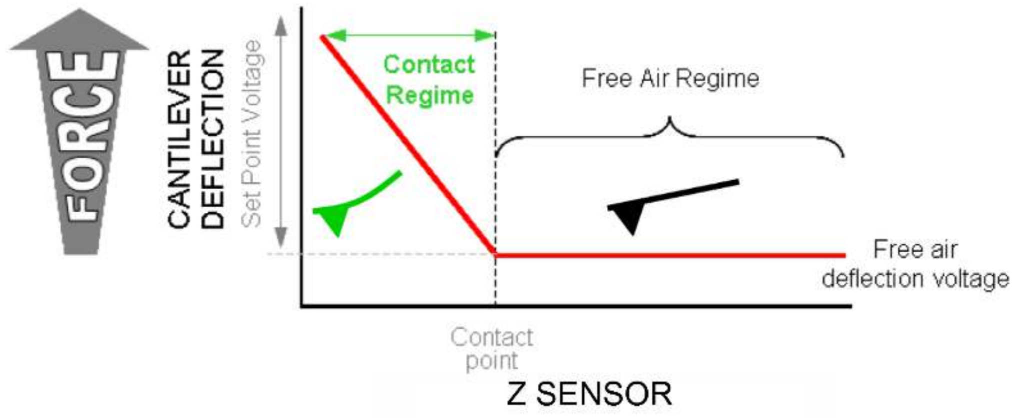


Figure 2.1.: Qualitative depiction of Cantilever Deflection vs. Z Sensor.

The Z feedback loop is what controls the Z actuator. It tries to maintain the specified setpoint voltage (see Figure 2.2 on page 16). If the measured deflection voltage is less than the setpoint voltage, it extends the Z actuator. If the measured deflection is greater than the setpoint voltage, it retracts the Z actuator. When the Z voltage is railed at 150V it implies that the Z actuator is fully extended, and the deflection is still below the required setpoint. What this means is that, physically, the cantilever is not in contact enough or at all with the surface. Likewise, when the Z voltage is railed at -10V, it means that the Z actuator is fully retracted, yet the cantilever is still so bent against the sample the deflection remains higher than the setpoint.

While the Z feedback keeps the deflection constant, the scanner is rastered in X and Y. (See Chapter 22 on page 303.) As the cantilever goes over the sample, voltage to the Z actuator changes due to the feedback. The Z actuator is made from piezoelectric material, which is inherently non-linear over large distances; over small ranges, it acts very linearly, and the voltage sent to the Z actuator is an accurate measurement of the sample topography. In the software, this voltage scaled is the *Height* channel. For large ranges, it is more appropriate to use the *Z sensor* channel. This sensor is much more accurate but introduces some noise to the measurement. Over large ranges, this noise is negligible compared to the range itself. If your sample features are around 1um or greater, it is good idea to use *Z sensor* rather than *Height*.

If the Z feedback worked perfectly, the deflection would never change. In reality, the Z feedback is close to maintaining a constant deflection but not perfect. Sudden changes in the sample topography cause the Z feedback to either under or overcompensate. The *Deflection* channel is therefore a measurement of the error signal. This channel can be useful for detecting features that do not show up well on the other channels.

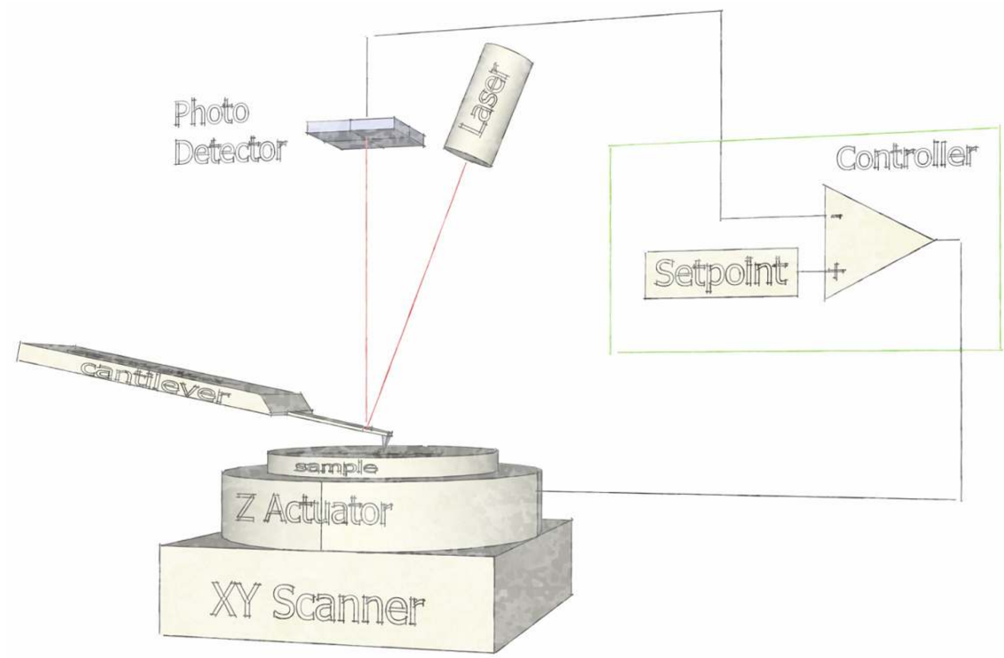


Figure 2.2.: Contact Mode AFM. Z actuator feeds back to keep cantilever deflection at a given setpoint.

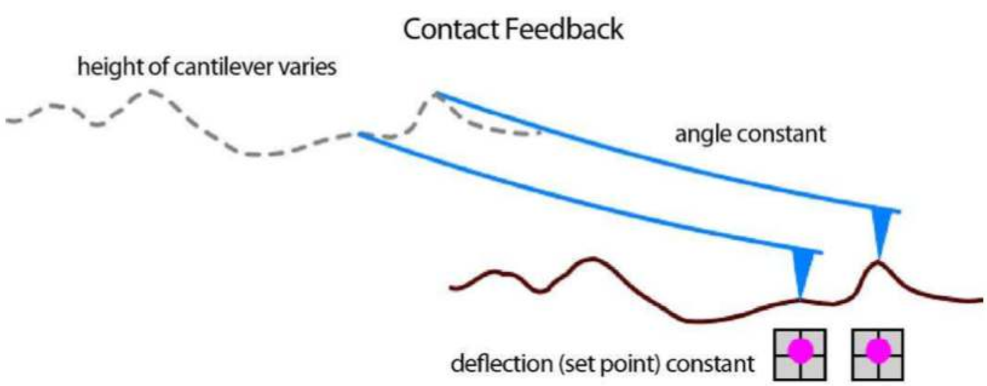
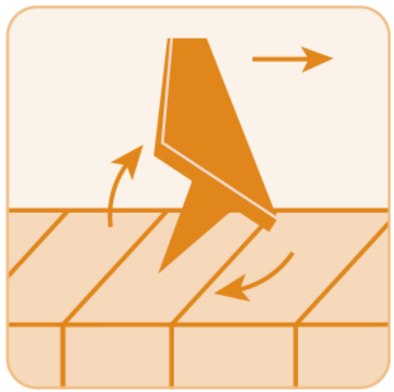


Figure 2.3.: Contact Mode tracking of the surface. Feedback keeps the cantilever deflection constant.

3. Lateral Force Microscopy

CHAPTER REV. 2014, DATED 05/09/2018, 18:29. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

3.1. Lateral Force Imaging

3.1.1. Introduction

Lateral Force Microscopy (LFM) can easily be performed on both the MFP-3D and Cypher family systems. It is a close cousin of Contact Mode; however, it is instead performed with the "fast scanning" direction orthogonal to the length of the cantilever. Contact mode AFM is typically performed by scanning the tip (or sample) back and forth in the manner of sawing a piece of wood. The cantilever (saw) moves along a path that is parallel to its own length. For LFM, it moves more like a blind man's cane, in a path perpendicular to its own length. This is referred to as the "*Scan Angle*".

LFM fundamentally performs Contact Mode AFM but also monitors an extra piece of information, how much the cantilever twists from the friction experienced by the tip during scanning. This signal is measured by the same quadrant photodetector that measures the cantilever deflection, except that "left" and "right" halves of the photodetector are differenced to measure the lateral deflection (twist) of the cantilever.

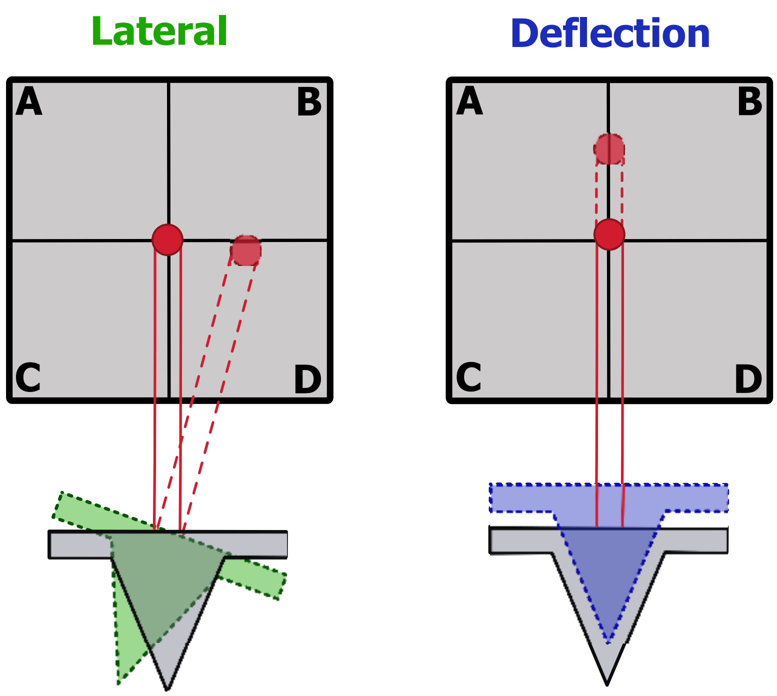


Figure 3.1.: The Lateral signal versus the Deflection signal on the photodetector.

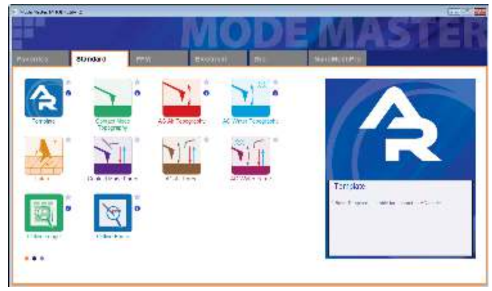
3.1.2. Tutorial: Lateral Force Imaging

It is assumed the user is already familiar with Contact Mode AFM (Chapter 1 on page 6). If you own an MFP-3D family AFM, please follow *MFP-3D User Guide, Chapter: Tutorial: Contact Mode Imaging in Air*. If you own a Cypher AFM, please follow *Cypher User Guide, Chapter: Tutorial: Contact Mode in Air*. This will familiarize you with the basics of loading the cantilever, sample, etc.

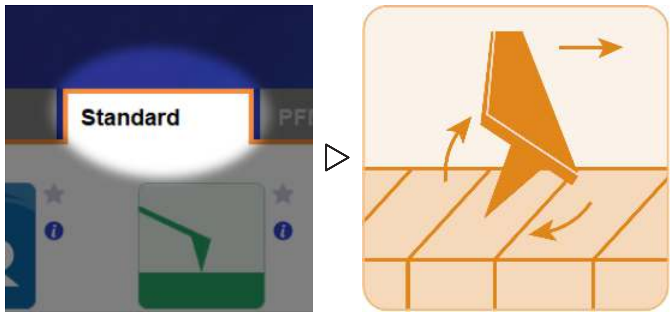
1.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.



Select Mode:

- Select *Standard tab > Lateral*.
- The screen will rearrange and present all the controls necessary for this type of AFM imaging.

Note Loading *Lateral* mode will also load the Friction Loop panel automatically. You may not need to perform this for LFM imaging, but we cover this technique separately. (See Section 3.2 on page 22)

3.1.3. Zeroing the Lateral Signal

Note	LFM produces the best results if performed on either a **Cypher** family AFM or **MFP-3D Infinity** AFM, as the electronic gain for the Lateral signal on the photodetector is higher and, therefore, more sensitive.

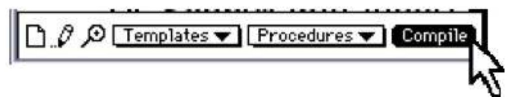
Just the same as the Deflection signal, the Lateral signal should be centered/zeroed on the photodetector during LFM as well. Please consult the manual of your particular AFM on how to do this. However, the three options are covered in the following sections.

3.1.3.1. MFP-3D Classic/Origin/Origin+

A standard MFP-3D Head does not have a mechanical means to zero the Lateral signal on the photodetector, for reasons not to be discussed herein. There is, however, an electronic way to zero the Lateral, in the form of an Igor procedure file (.ipf) called "**LFM Rocks**." Please contact support.asylumresearch.com for a copy of this file.

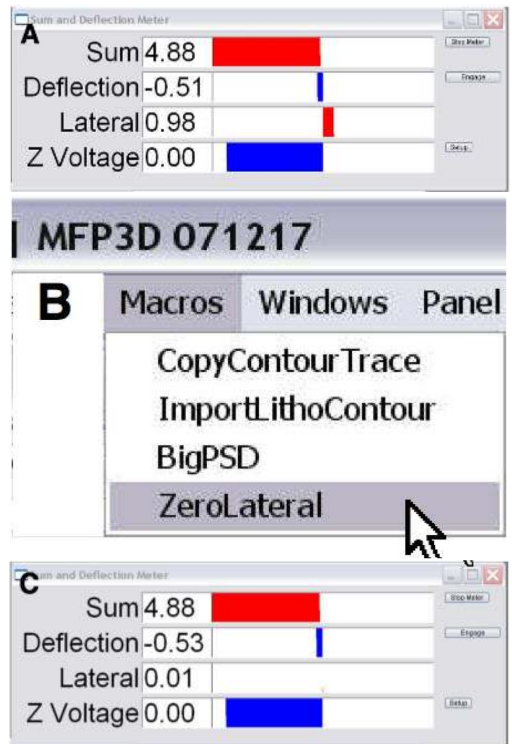
1. Load the **LFM Rocks.ipf** - Go to *File>Open File>Procedure*; load **LFM Rocks.ipf** from where it was saved.

2. Compile the .ipf by clicking the ‘Compile’ button at the lower left of the .ipf window. Once compiled, this button will no longer be visible. To learn more about compiling Igor procedure files, read the Igor manual.



3. To zero the lateral signal, go to *Macros > ZeroLateral*, and the lateral signal will be electronically zeroed.

- The top image shows a typical lateral signal from a given cantilever.
- With LFM Rocks compiled, select *ZeroLateral* from *Macros* in AFM software menu bar, as seen in the middle photo.
- Image C, at right, demonstrates the result: electronic zeroing of the *Lateral* signal.



3.1.3.2. MFP-3D Infinity or MFP-3D with LFM Option

The MFP-3D Infinity or MFP-3D Classic/Origin/Origin+ with LFM option has a special adjustment mechanism built into the Head to mechanically center the laser spot on the photodetector.

3.1.3.3. Cypher S/ES/VRS

Thec Cypher has built-in motorized zeroing of the deflection and lateral signal; it all happens automatically. When the Cypher is prepared to do Contact Mode imaging, it is also ready to do LFM.

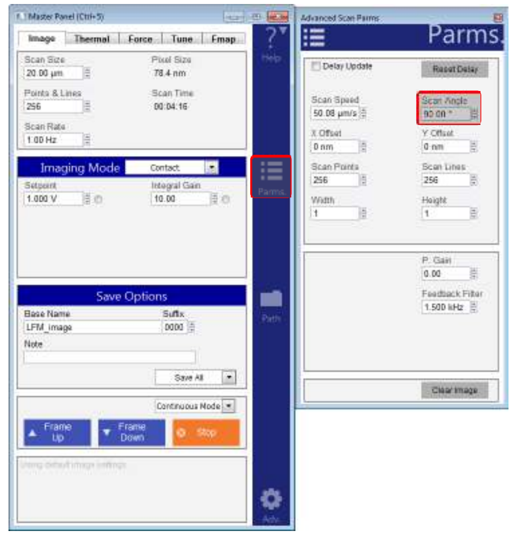
3.1.4. Setting the Parameters

1.

Scan Angle:

- Loading *Lateral* mode automatically sets the *Scan Angle*to 90°.
- To check this, click the 'Parms.' icon in the Master Panel.

Note It is also possible to set the *Scan Angle* to 270° instead during LFM. This will essentially switch the Trace and Retrace with respect to 90°.

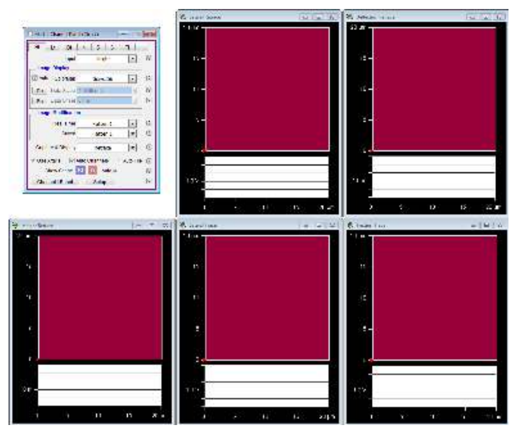


2.

Channel Configuration

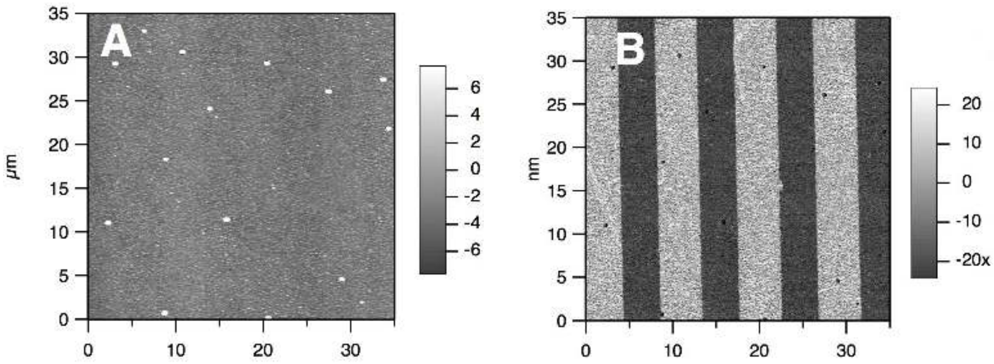
- Loading *Lateral* mode automatically configures the Lateral channel in the Master Channel Panel.
- 5 channels are automatically displayed: **Height**, **Deflection**, **Lateral Trace**, **Lateral Retrace**, and **Friction Trace**

Note The **Friction Trace** channel is a "User Calculated" channel. It displays the measured **(Lateral Trace–Lateral Retrace)/2** channel in real-time during the scan.



3.1.5. Example of LFM Imaging

Figure 3.2 on page 22 shows images from a sample that has bands of alternating molecules adhered to its surface. This was achieved by micro-contact printing. Note that Height image in Figure 3.2a on page 22 shows nearly imperceptible evidence of these two molecular species. Since only their termination differs, they are the very nearly the same length. The Lateral image in Figure 3.2b on page 22, however, shows great contrast, indicating a difference in tip-sample frictional forces between these two molecular species.



(a) Height Image **(b)** Lateral Image

Figure 3.2.: Micro-contact printed alkanethiols on polycrystalline gold. The bright areas of the LFM images are terminated with a carboxylic acid, while the dark areas are a methyl terminus.

3.2. Friction Loops

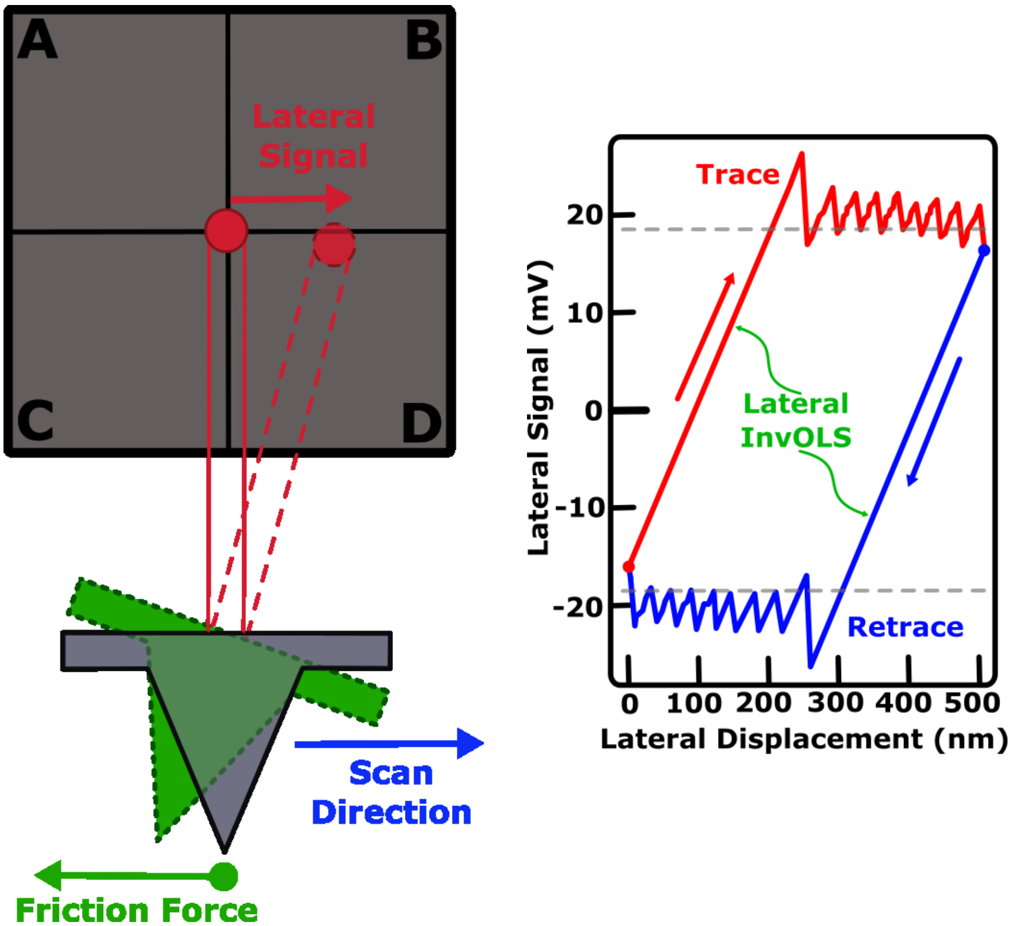
3.2.1. Introduction

Another major method of performing LFM measurements is through something called a "Friction Loop". This is a non-imaging technique where the "slow axis" of the scan is disabled, and the cantilever is simply rastered back and forth over the same line on the sample. This Friction Loop measurement can be used in a variety of ways to calculate/calibrate the Lateral Optical Lever Sensitivity (Lateral InvOLS) in order to quantify the friction measurements, somewhat like a Deflection force curve might be used. (For an example of a standard Friction Loop measurements, see Figure 3.3 on page 23.)

The Friction Loop Panel will ramp the Y-axis of the AFM Scanner to move orthogonal to the long axis of the cantilever, while the tip is engaged in contact on the surface. It will record the Lateral signal as a function of the Y-sensor position during this. The software will aim to fit the Static "turn around" regions to a straight line to obtain the Lateral InvOLS value for the cantilever; however, this can be problematic if the data is not very clear. If there is a particularly jagged turn around, it can produce inaccurate values. The main goal of this panel is to provide a means to collect the data; the automated analysis is provided as a starting point, but you need to confirm its validity before fully trusting it.

In Figure 3.3b on page 23, note that in the forward Trace (red), the lateral signal is about +20 mV, while in the backwards Retrace (blue), the Lateral signal is about -20 mV. The "Average Friction" signal is taken to be the difference of the Trace (forward) and Retrace (backward) path signals divided by 2 (e.g., 20 mV).

One aspect to note is that, if the cantilever, laser, and detector are all perfectly aligned, and the sample is perfectly flat, the Friction Loop will appear centered and symmetric about zero. This is most often not the case. By taking the difference of the Trace and Retrace of the Lateral signal, most offsets can be eliminated due to "Optical Beam" misalignments and sample slope.



(a) A diagram of LFM **(b)** A typical Friction Loop

Figure 3.3.: An example of a standard Friction Loop measurement.

3.2.2. Lateral calibration

One way to calibrate the Lateral InvOLS is to run a Friction Loop on a stiff, flat, very high friction surface, such as mica or glass. At the beginning of the loop (Trace) and at the point the cantilever turns around (Retrace), the tip actually sticks to the surface before it starts sliding. This sticking portion is the Static Friction portion of the Friction Loop (red arrow and blue arrow shown in Figure 3.3b on page 23).

The slope (k_{tot}) of this represents the lateral stiffness of the lever (k_{lat}), the stiffness of the tip (k_{tip}), and stiffness of the contact (k_{cont}):

$$\frac{1}{k_{tot}} = \frac{1}{k_{lat}} + \frac{1}{k_{tip}} + \frac{1}{k_{cont}}$$

Note that, if the stiffness of the tip is very high (e.g., a silica ball), and the stiffness of the contact is also high (e.g. a silica ball on a silica surface), then these two terms approach zero, and the total stiffness just approximates the lateral stiffness of the lever.

3.2.2.1. The Wedge Calibration Method

A method that bypasses the separate determination of the lateral optical sensitivity and the lateral force constant is named the "Wedge Calibration Method". With this method, the Deflection and Lateral signals are monitored while scanning on a sloped surface (e.g., reconstructed SrTiO3 or a silicon grating.) This method is based on the geometric relationship between the components of the normal and lateral forces as the probe slides over a substrate with a *known* slope. Experimentally, what one measures are the load dependence of the half-width (W) and the offset (Δ) of the Friction Loops, representing the cantilever's torsional sensitivity due to the frictional force and the slope, respectively. Static force analysis of the sliding, assuming JKR contact conditions for adhesive friction, leads to the instrument-dependent lateral force calibration factor α (in N/V units).

For more on this topic, see references [1,2]

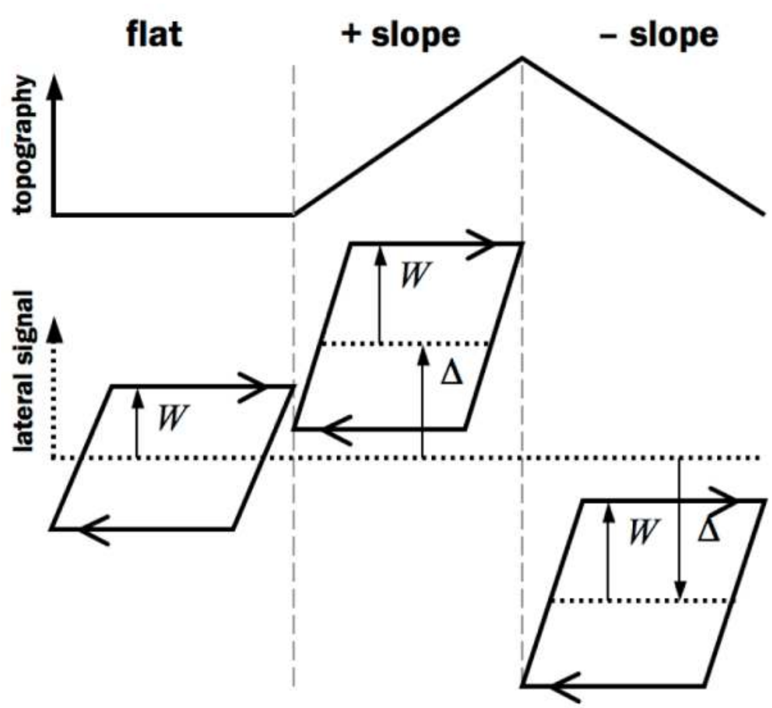


Figure 3.4.: Friction loops on flat, inclined, and declined surfaces at a given applied load. The half-width (W), representing the frictional response, only slightly varies; while the offset (Δ), due to the surface tilt, varies substantially. The load dependence of W and Δ are used to determine the lateral force sensitivity of the cantilever.

[1] Ogletree, D.F., Carpick, R.W., Salmerson, M.: Calibration of frictional forces in atomic force microscopy. Rev. Sci. Instrum. 67, 3298-3306 (1996)

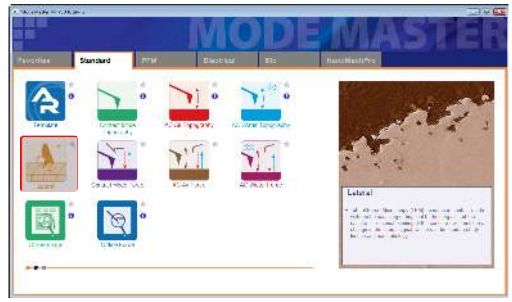
[2] Varenberg, M., Etsion, I., Halperin, G.: An improved wedge calibration method for lateral force microscopy. Rev. Sci. Instrum. 74, 3362-3367 (2003)

3.2.3. Tutorial: Friction Loops

1.

Mode Master:

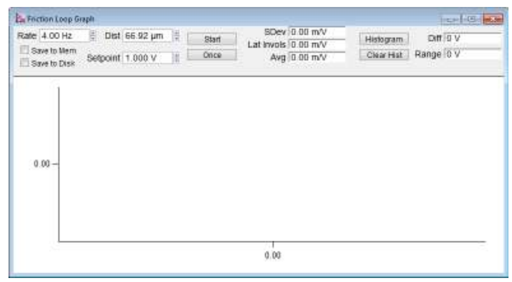
- When the Mode Master window appears, choose the *Standard* tab.
- Select the 'Lateral' mode icon.
- All the necessary panels for Friction Loop operation automatically load and rearrange on the desktop.



2.

Friction Loop panel:

- The Friction Loop Graph has a few parameters you can control:
  - *Rate* - Tip scan rate
  - *Dist* **-** Distance the Y-piezo will ramp
  - *Setpoint* - Tip deflection/normal force



3.3. Friction-Load Maps

3.3.1. Introduction

The **Friction-Load Map** is a plot of the friction (Lateral) force versus the normal load (Deflection). The slope of this curve, assuming that it is linear, is therefore the Coefficient of Friction (COF). This plot is generally linear at high loads but typically becomes less linear at low loads. It can better understand and interpret with the appropriate contact model (Hertz, JKR, DMT). Without explicitly calibrating the Deflection and Lateral Spring Constants and the respective Optical Leer Sensitivities, you can still plot the Lateral versus Normal signals (see Figure 3.5 on page 26) and make relative comparisons of changes in friction between tip and sample.

Everything considered, a Friction-Load Map is essentially a Friction Loop (see Section 3.2 on page 22) in combination with a Force Curve (see Chapter 18 on page 228). In this technique, the cantilever is rastered back and forth across a single line as in a Friction Loop. However, the normal force is also incremented after each completed line up to some maximum value ("loading") and then decremented in the same manner ("unloading"). Both the Lateral signal and Normal signal are monitored simultaneously, which ultimately constitutes the data for a Friction-Load Map.

Note that the Deflection signal in Figure 3.6 on page 27 looks similar to a Force Curve, in that it starts out flat away from the surface at the beginning of the loading segment, suddenly snaps to the surface when it comes very near, increases as the tip pressed in to the surface ("loading"), decreases as you start to move away ("unloading"), and then finally snaps off the surface. You can create a similar Friction-Load Map by collecting individual Friction Loops at different deflection setpoints while engaged on the surface; but you will not see the data at the point of contact, at pull off, and at negative loads.

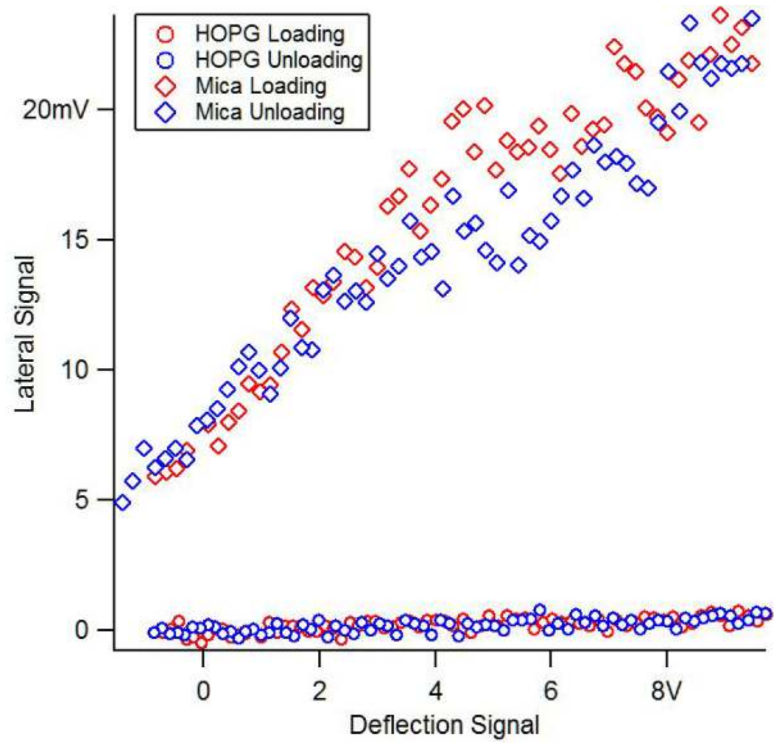


Figure 3.5.: Uncalibrated, relative comparison of the COF for HOPF and Mica

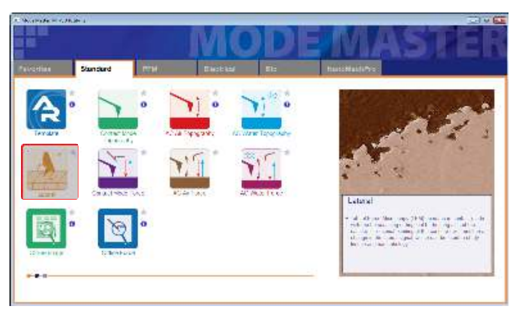
3.3.2. Tutorial: Friction-Load Maps

Note	Performing a Friction-Load Map requires an Igor procedure file (.ipf) called **RampZDuringNap_stepwise.ipf.** Please contact Support@AsylumResearch.com for a copy of this file.

1.

Mode Master:

- When the Mode Master window appears, choose the *Standard* tab.
- Select the 'Lateral' mode icon. All the necessary panels for Friction Loop operation automatically load and rearrange on the desktop.



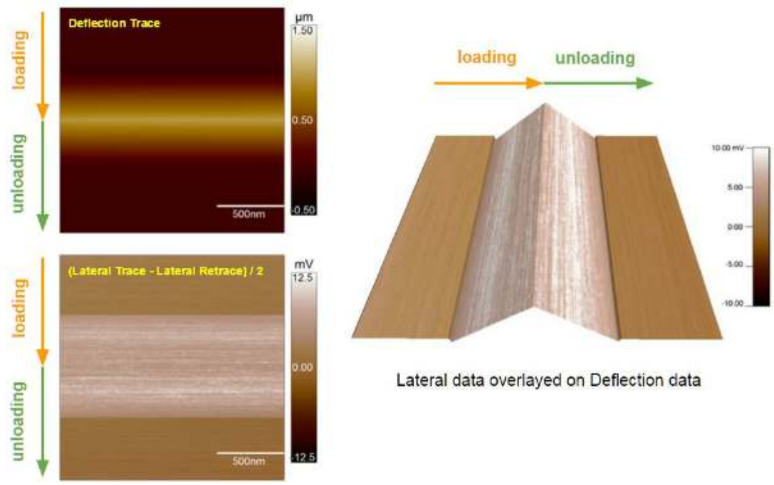
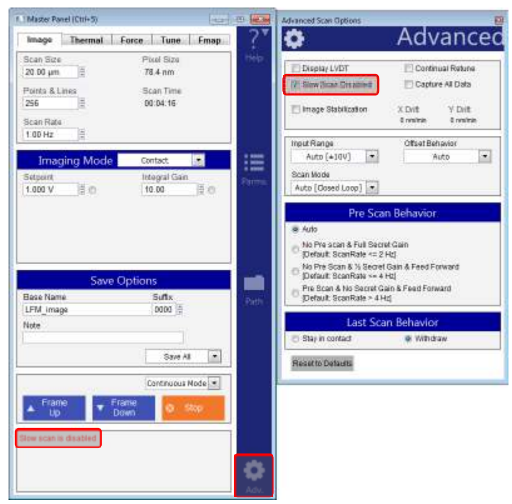


Figure 3.6.: An example of a typical Friction-Load Map dataset

2.

Disable Slow Scan:

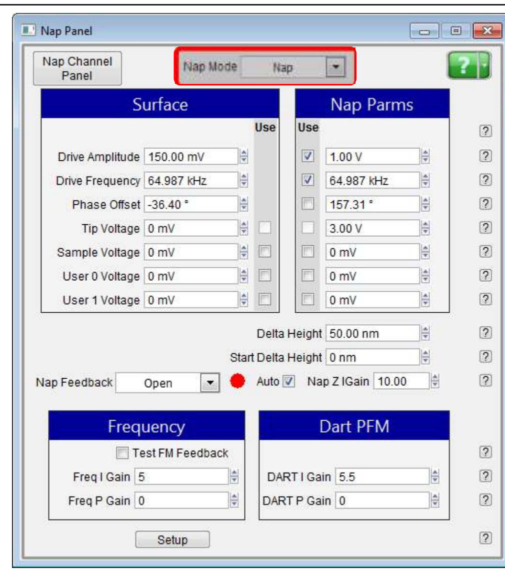
- In the Master Panel, select the *Adv. tab.*
- Select the checkbox for *Slow Scan Disabled*. This will cause the AFM to scan the same line repeatedly when the AFM scan is begun.



3.

Nap Panel:

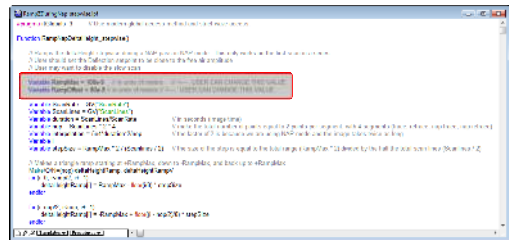
- In the *AFM Controls >Nap Panel.*
- Set the *Nap Mode* in the **Nap Panel** pulldown menu to "Nap". This enables standard Nap Mode.



4.

Launch the .ipf:

- Launch the **RampZDuringNap_stepwise.ipf** by double-clicking the file icon.
- Adjust the two variables: `RampMax` and `Offset`.

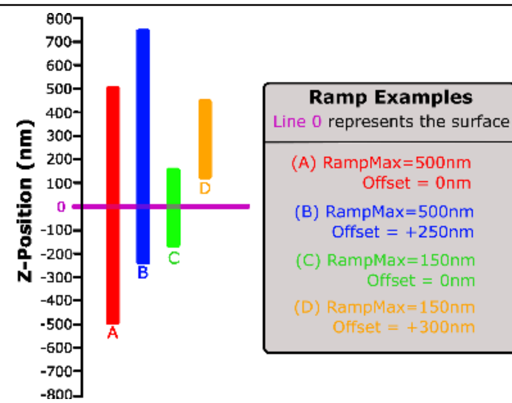


Note This file hijacks what ‘Do Scan’/ ‘Frame Up’/ ‘Frame Down’ does. Instead of topographic imaging, it will lift the Z-piezo away from the surface to a defined initial position, ramp a defined stepwise amount towards the surface while scanning a line, then retract to the initial defined position.

5.

Adjusting the variables:

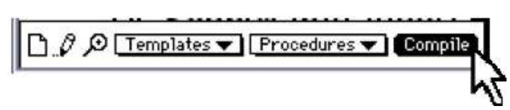
- `RampMax` adjusts the magnitude of the stepwise loading/unloading.
- `Offset` adjusts the offset of the `RampMax` relative to sample surface.



6.

Compile the file:

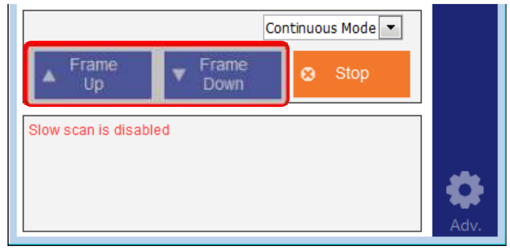
- Once you have set these two variables, you must compile the **RampZDuringNap_stepwise.ipf** by clicking the ‘Compile’ button at the lower left of the .ipf procedure window.



Note Once compiled, this button will no longer be visible. To learn more about compiling Igor procedure files, please read the Igor manual.

7. **Begin the Friction-Load Map:**

- Select 'Frame Up' or 'Frame Down' in the Master Panel.

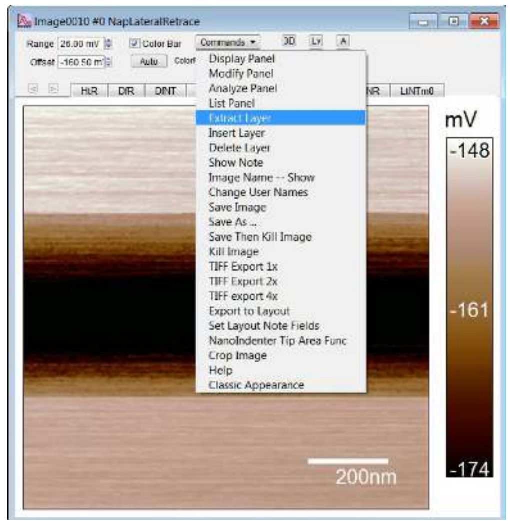


3.3.3. Tutorial: Data Processing

Friction-Load Map data generally requires a bit of processing to understand well. This section steps you through a typical procedure.

1. **Retrace layer:**

- Open Friction-Load Map .ibw file and select the *Nap Lateral Retrace* image tab.
- Navigate to *Commands > Extract Layer* at the top of the image window. This will copy the **Nap Lateral Retrace** image to an Igor wave called *LayerData*.

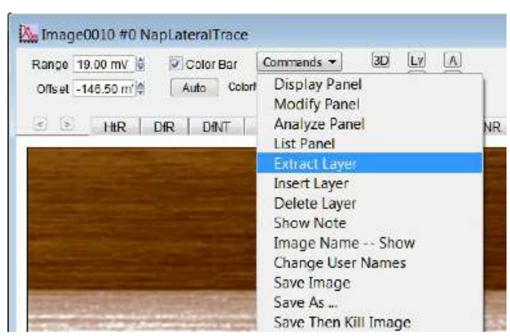


2.

- In the Igor command line (CTRL+J), copy the *LayerData* wave to a new wave using the `duplicate` command.
- In the example here, that new wave is given the name "*latretrace0010*".

3. **Trace layer:**

- Go back to the Friction-Load Map .ibw file and select the *Nap Lateral Trace* image tab.
- Navigate to *Commands > Extract Layer* at the top of the image window. This will copy the **Nap Lateral Trace** image to an Igor wave called *LayerData*. This will overwrite the data of the previous *LayerData.*

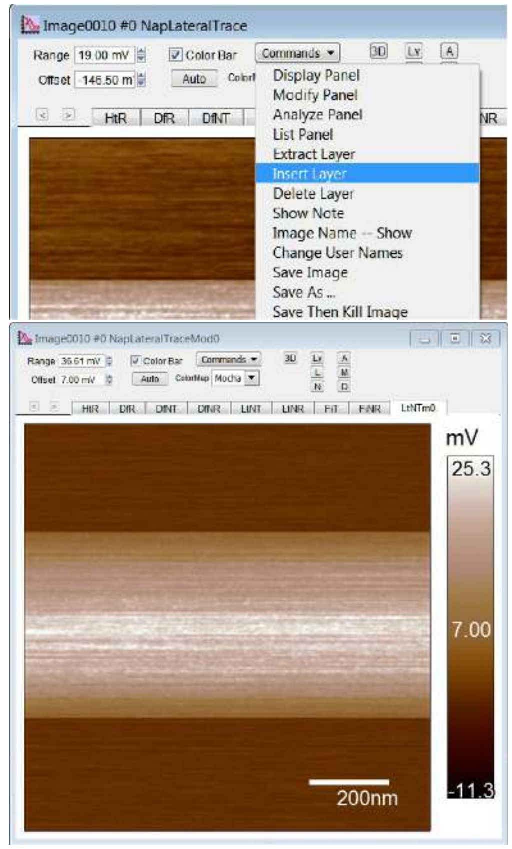


4. In the Igor command line (CTRL+J), subtract the **Nap Lateral Trace** wave from the **Nap Lateral Retrace** wave and divide that result by 2.

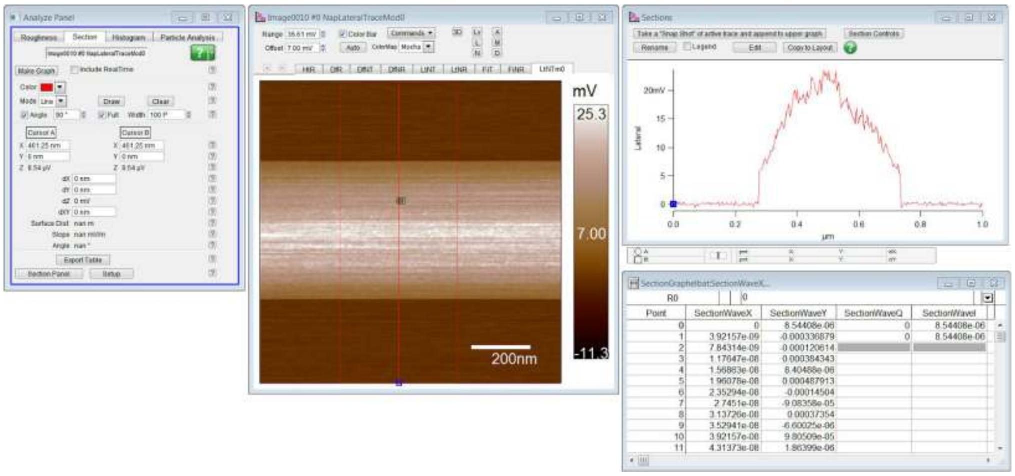
5. **Insert the layer:**

- Navigate to *Commands > Insert Layer* at the top of the image window.
- This inserts the result of that subtract (*LayerData* wave) as a new layer into the Friction-Load Map **.ibw** file.
- In this example, the inserted layer is named **NapLateralTraceMod0** or **LtNTm0** in the tab.

Note This is the **Average Friction** image and is actually the same data as the **NapFrictionTrace**.



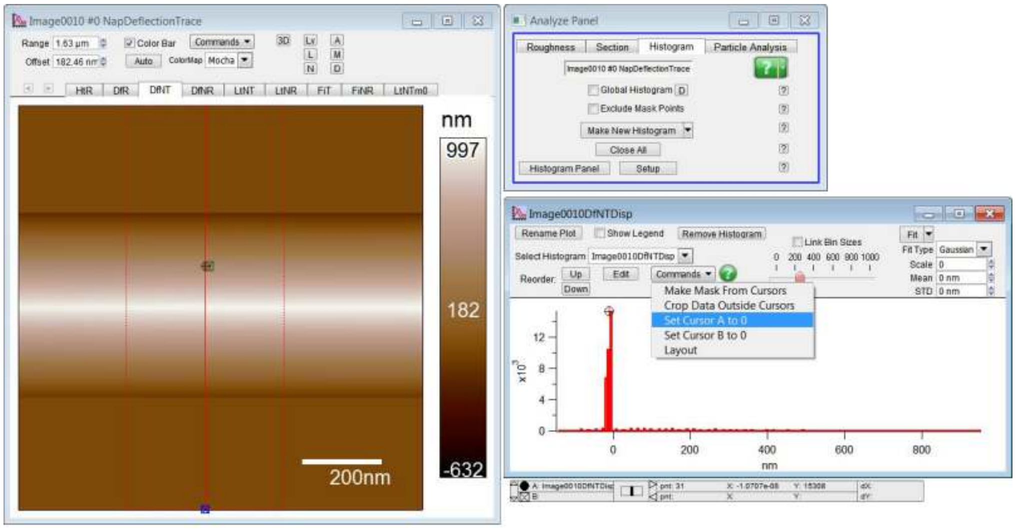
6.



Average the Friction Data:

- Use the *Section* tool in the *Commands > Analyze Panel* in order to average the data over the “slow scan” axis for the inserted layer (**NapLateralTraceMod0**).
- Increase the width of the section to the number of pixels you want to average. In the example here, it is averaged across 100 pixels.
- Copy the section data to another wave and separate the “loading” and “unloading” segments.

7.

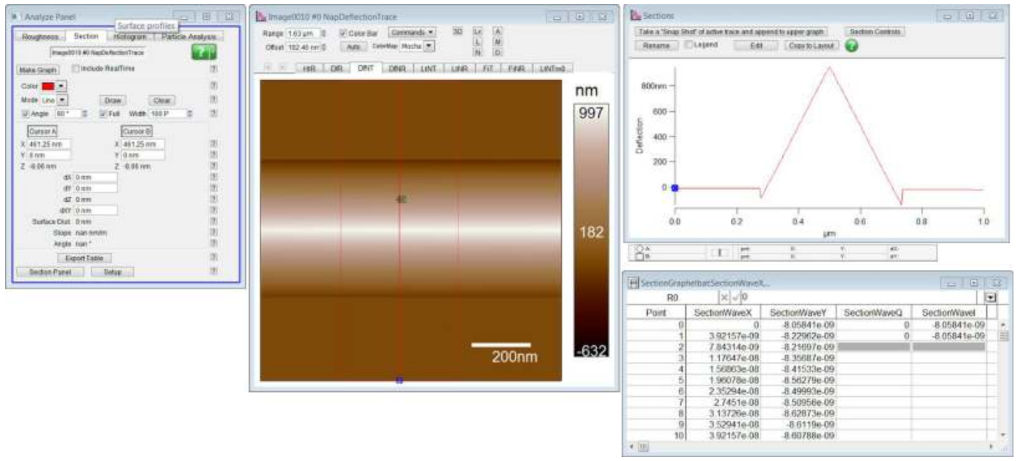


Deflection Offsets:

- Open the tab for either the **Nap Deflection Trace** or **Nap Deflection Retrace** channel.
- Create a histogram of the channel using the *Histogram* tool in the *Commands > Analyze Panel*.
- Put **Cursor A** on the tallest peak of the histogram and in the *Commands* menu of the histogram, select *Set Cursor A to 0*.

Note This removes any offsets in the **Deflection** image and set the flat regions of the **Deflection** image to be zero.

8.



Average the Deflection Data:

- Use the *Section* tool in the *Commands>Analyze Panel* in order to average the data over the “slow scan” axis for the offset **Deflection** layer.
- Increase the width of the section to the number of pixels you wish to average. In the example here, it is averaged across 100 pixels.
- Copy the section data to another wave and separate the “loading” and “unloading” segments.

9.

Apply the Calibration

- You may wish to convert the **Deflection** data to a Voltage signal or Force units using the *Deflection InvOLS* or the *Spring Constant (Bending)*, respectively.
- You may also wish to convert the **Lateral/Friction** data to Force units used the *Lateral InvOLS* and *Spring Constant (torsional)*.

Note This step is necessary only if you wish to perform quantitative comparisons.

10.

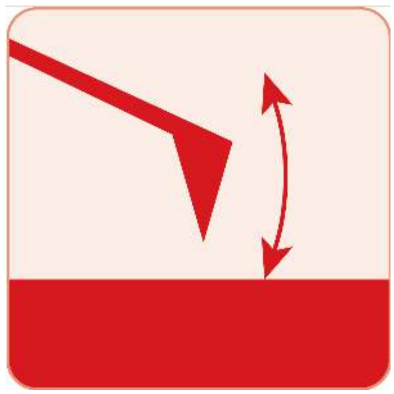
Graph the Data

- Graph the **Lateral/Friction** data versus the **Deflection** data to get the finished Friction-Load Map, as seen in Figure 3.5 on page 26.

Note You may want to remove the regions where the tip is away from the surface, where the **Deflection** signal is flat or where the **Lateral** signal is zero.

4. AC Mode in Air

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

4.1. Introduction

This section assumes you are already capable of starting the imaging process. If this is new to you then:

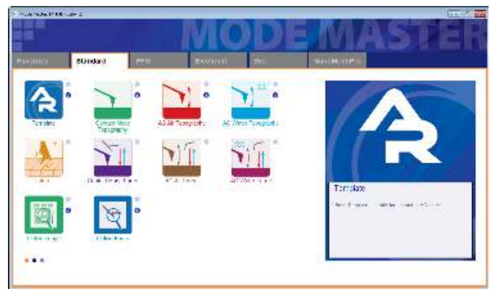
- For MFP-3D family AFMs, follow this tutorial:[1]
- For the Cypher family AFMs, follow this tutorial:[2]

The following sections assume the cantilever is ready to engage above the sample surface, meaning the tip is within piezo range of the sample. At that point, from a software perspective, the particular AFM you are using does not make much difference. Any differences in how the software interacts with our different instruments will be addressed as applicable.

1.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.

Select the Mode:

- Select *Standard tab > AC Air Topography*
- The screen will rearrange and present all the controls necessary for this type of AFM imaging.

4.2. Video Tutorial

Consider watching this introductory video tutorial: AC (Tapping) Mode Imaging (internet connection required).

[1] *MFP-3D User Guide, Chapter: Tutorial: AC Mode Imaging in Air..*
[2] *Cypher User Guide, Chapter: Tutorial: AC Mode in Air, Std. Scanner..*

4.3. Auto Tuning

The tutorials recommended above briefly touch upon the business of tuning the cantilever. Since this is at the heart of AC imaging, we'll talk about that now in a little more detail. If you just completed your AC mode imaging tutorial, you may be in a state of collecting an image. In this case, please halt the scan by clicking the 'Stop!!!' button in the *Main* tab of the Master Panel. This will retract the tip from the surface and stop the XY scanning of the sample. If you left your system not scanning, but with the tip simply engaged on the surface, click the 'Withdraw' Button on the Sum and Deflection Meter Panel. This pulls the tip from the sample.

Note These instructions are for repulsive mode AC imaging.

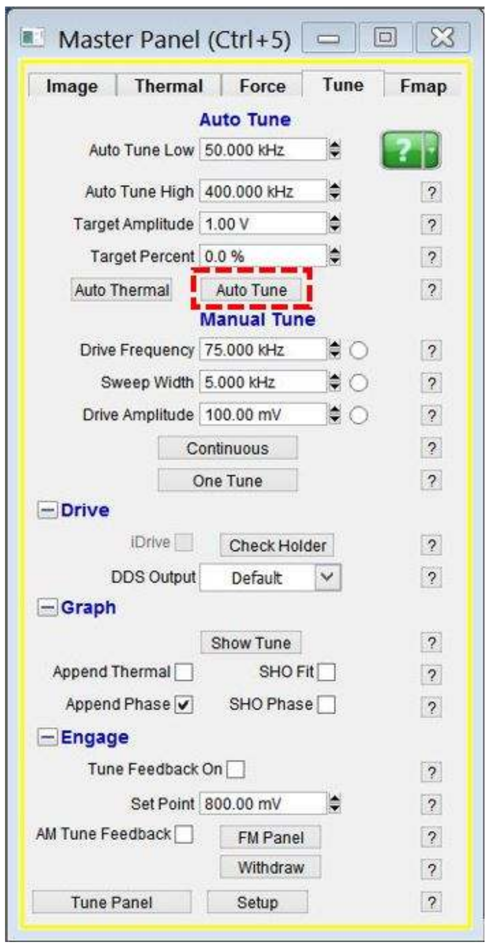


Figure 4.1.: Tune tab

1. Open the *Tune* tab of the Master Panel.
2. Choose a *Target Amplitude* of 1.0V. This will be the free air amplitude voltage. This is the peak-to-peak voltage of the oscillating cantilever.
3. Change the *Target Percent* to -5.0%. The minus sign indicates that the drive frequency will be on the left side of the resonant peak, which helps ensure the tip will remain in NET repulsive

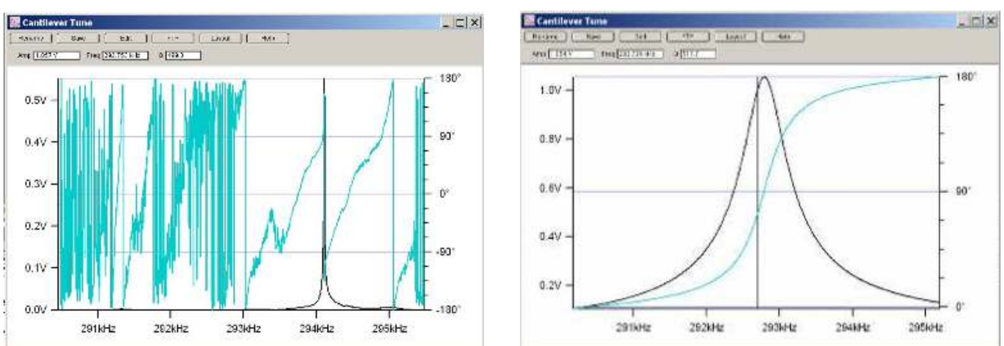
mode when engaged and scanning. For more detailed information about repulsive mode, see Section 5.3 on page 48.

4. The default *Auto Tune Low* and *Auto Tune High* values for air imaging are typically 50 kHz to 400 kHz, respectively, accommodating most fundamental drive frequencies for common commercially available AC Mode cantilevers.
5. Click the 'Auto Tune' button for the frequency sweep to commence. The shake piezo applies a frequency ramp through the Auto Tune low to high frequencies. The cantilever will give the greatest oscillation amplitude at its resonant frequency, allowing the tune algorithm to locate it and determine the Q factor (quality) of the peak.
6. The *Drive Frequency* value will automatically update in the *Tune* and *Main* tabs of the Master Panel, and the Q factor of the cantilever will be determined and displayed at the top of the Cantilever Tune graph.

The Cantilever Tune graph, similar to the one in Figure 4.2 on page 37, will appear. The first tune will likely appear similar to Figure 4.2a on page 37, which then updates to one similar to Figure 4.2b on page 37.

The software automatically does the following:

- Picks a *Drive Frequency* at the specified *Target Percent* (default of -5%).
- Adjusts the *Drive Amplitude* applied to the shake piezo needed to make a 1.0 V (peak to peak) amplitude voltage on the photo diode, as displayed in the Sum and Deflection Meter.
- Adjusts the *Phase Offset* to have the phase signal at 90° on resonance.



(a) Early stages of Auto Tune

(b) Final result of Auto Tune

Figure 4.2.: Auto Tune of an Olympus AC160 Si (f 300kHz; k= 40N/m; nominal values) cantilever in air

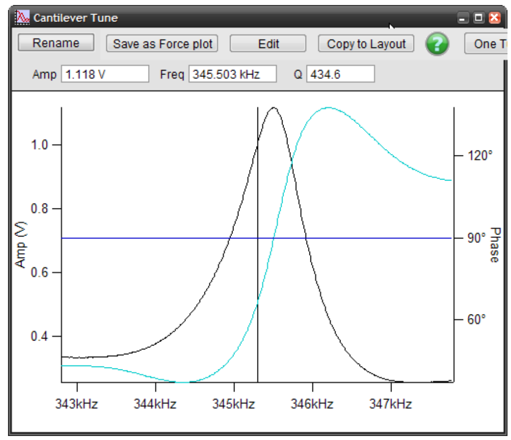
4.3.1. Auto Tune Troubleshooting

If you do not know the resonant frequency of your cantilever, see Chapter 21 on page 293.

4.4. Saving Tune Data

Tip How to Save Tune plots?

- Click 'Save as Force Plot'. The saved tune can then be reviewed in the Master Force Panel.
- Click 'Rename'. The graph is saved in a separate window. Subsequent tunes can be overlaid on top of each other if they are given the same name or saved in a new graph when given a new name.
- Click 'FTP'. This saves the experiment on the computer in a Temp Folder. This allows you to upload the file to the Asylum Research FTP site for discussion with Asylum Research technical staff.
- Click 'Layout'. This appends the graph to a layout.



4.5. Optimizing Imaging Parameters

Please refer to the tutorial[3]. It has a very good explanation of how to get going in AC mode, adjust gains, interpret the images, and figure out what is going wrong. It may be written to be MFP3D centric, but it covers the basics. Even if you have a Cypher, a lot of what is in there is still very useful; you may only need to adjust the values of the gains and rates for your Cypher.

4.6. Net Attractive and Repulsive AC Modes

Repulsive AC mode is the most common AC mode. It is where the tip is in repulsive (hard) contact with the surface in some fraction of its oscillation. It is much easier to perform but is rougher on the tip and sample.

Attractive AC mode attempts to image while sensing only the very weak attractive forces by gently oscillating the tip just above the surface. In this way the tip will experience attractive forces over a broad range of its oscillation. This is much more difficult to do and cannot be done with all levers and samples. But it is much gentler on the samples and is sometimes required for soft biological materials.

See also 5.3.2 for a good summary of attractive and repulsive modes.

[3] *MFP-3D User Guide, Chapter: Tutorial: AC Mode Imaging in Air.*.

4.6.1. Net Repulsive Mode Imaging

Imaging in Repulsive mode was used in the example tutorial from the *ARMFP3DUserGuide.pdf*. It is much easier and tends to give better phase contrast. You can tell you are in repulsive mode when your phase signal is < 90°.

4.6.1.1. Use a new cantilever

If all else fails, the tip apex may be too blunt to stay in Repulsive Mode. (Yes, this is actually true.) Large radius, blunt tips can experience too much attractive force approaching the surface and get stuck in attractive mode. Put in a new cantilever and start over.

4.6.2. Net Attractive AC Mode

Imaging in Attractive mode is great for soft samples that can be damaged or situations where you want your AFM tip to stay sharp for a long time. You can tell you are in attractive mode when your phase signal is > 90°.

As it can be tricky when you want attractive mode imaging to occur, some simple steps to help keep the tip in attractive mode are included here.

4.6.2.1. Chose the right probe

Softer, longer levers with relatively low resonance frequencies are a good choice. We always have good luck with the AC240 or AC160. Also see Asylum Research Probe Store.

4.6.2.2. Gently drive the probe above resonance

Smaller amplitudes with drive frequencies above resonance improve Attractive mode imaging stability.

For an Olympus AC240 probe, the process is as follows:

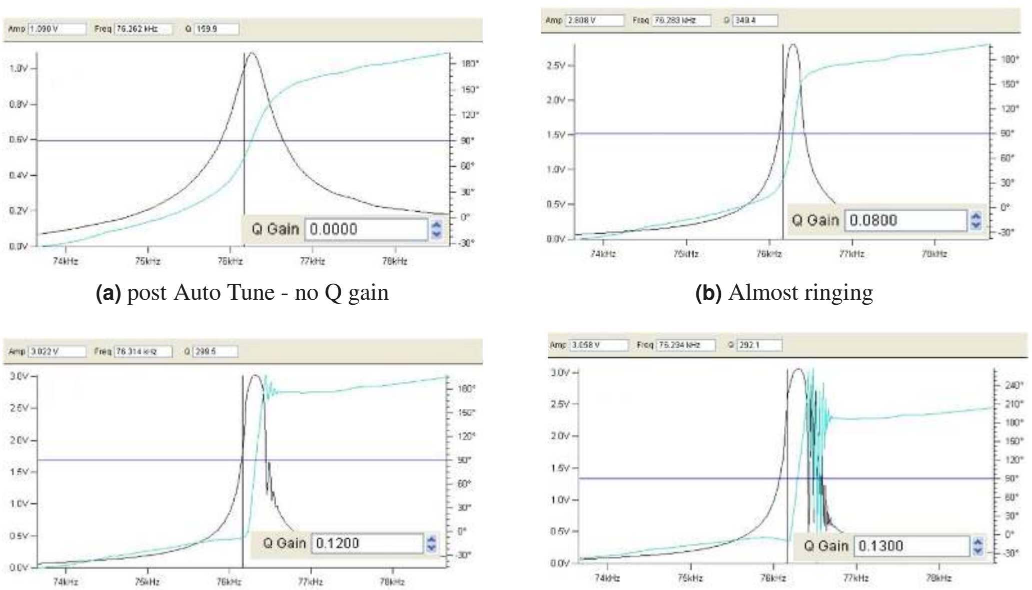
1. In the *Tune* tab of the Main panel, set the *Target Amplitude* to 250mV.
2. Set the *Target Percent* to +10 to +20%; this will set the *Drive Frequency* on the higher (right) side of the cantilever's resonant peak, a position more likely to keep the tip in Attractive mode while imaging.
3. Click the 'Auto Tune' button. If this does not do well, try again with a *Target Amplitude* of 500 mV.

4.6.2.3. Try Q control with positive Q gain

Higher Q resonance peaks favor Attractive mode imaging. Q control gives some control of this.

Figure 4.3 on page 40 shows this process with an AC240 (~76kHz Si lever) with some common visuals to look for, although every lever shows different degrees of response relative to the amount of Q gain.

- Figure (a) shows the last tune of an Auto Tune; no Q gain is added.
- Figure (b) shows the response after a slight increase in Q gain. Notice the top of the peak is starting to look more round than sharp; this means the ringing is about to start.
- Figure (c) Ringing shows up to the right of the frequency peak; at this point, you should decrease the Q gain until the ringing goes away.
- Figure (d) is an example of it going even higher, showing even more ringing in the amplitude and phase data.
- Notice with additional Q gain, the amplitude response goes up. You may need to lower the *Drive Amplitude* to get back to the desired free air amplitude.



(a) post Auto Tune - no Q gain

(b) Almost ringing

(c) Ringing

(d) Freakout

Figure 4.3.

4.6.3. Preventing Mode Hopping

Mode Hopping is where the tip will jump between Repulsive and Attractive mode while scanning. While it is good to stay in one mode or the other for imaging, the uncontrolled switching between them makes analysis difficult. The images in Figure 4.4 on page 41 show red regions, where phase is much smaller than 90° (Section 4.6.1 on page 39), and blue, where phase is much larger than 90° (attractive behavior). During each scan line, the probe is switching back and forth between Attractive and Repulsive mode depending on what features of the sample it is interacting with.

The first thing to do to prevent Mode Hopping is to go more repulsive. Repulsive mode is easier to achieve. Decrease the *Drive Frequency*, increase the *Drive Amplitude*, and possibly decrease your setpoint.

If Mode Hopping is occurring and you want to be in Attractive mode, reduce the Drive Amplitude and then the *Set Point*. This should work most of the time, but if it does not, retune and select a *Drive Frequency* slightly more to the right of the peak.

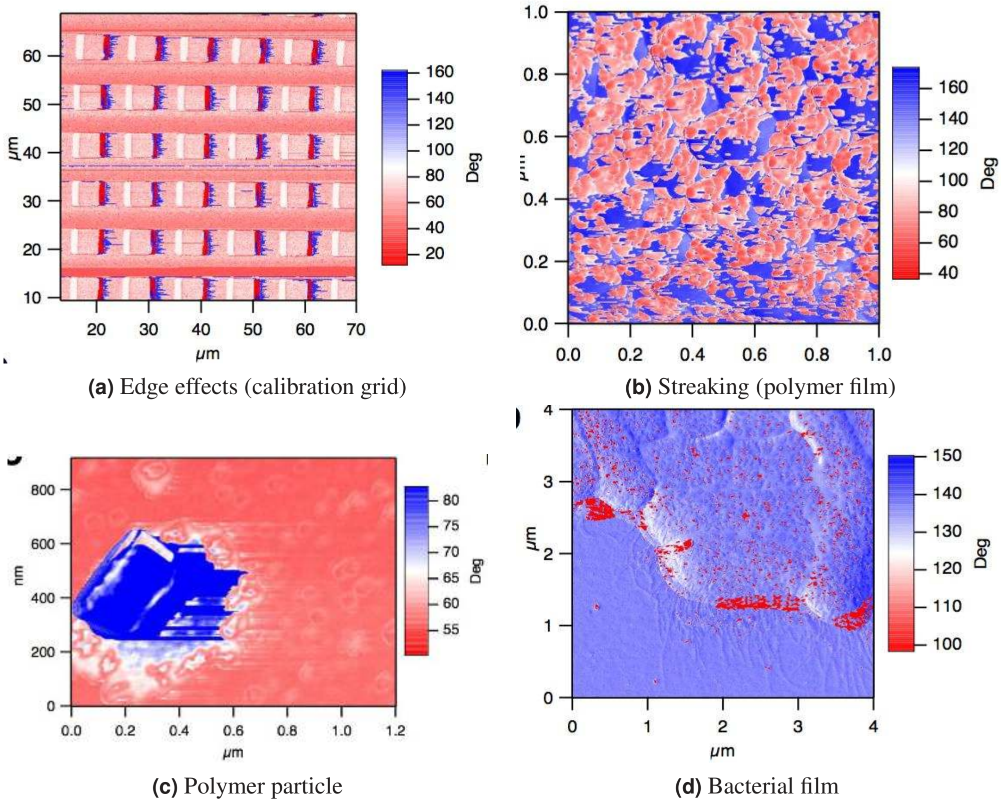


Figure 4.4.: Examples of mode hopping in the Phase Channel

4.6.3.1. Make the Sample Less Attractive

If Mode Hopping is still problematic, it could be due to the sample. The following situations may be contributing to it:

- Surface charges:
  - Use the Static Master device to ionize the air around the sample and make it slightly conductive; this dissipates surface charges.
  - Place the Static Master in the vicinity of the sample (see Figure 4.5 on page 42).
  - Sometimes the glass slide the sample is glued to is the culprit of the excess charge. In these cases, mounting the sample on a magnetic puck and placing it on a metal sample holder can help quell this charge.
- Tip sticking to the sample: Our findings have shown that a Pt-coated Si cantilever can sometimes work well with sticky samples. We use Electrilevers (Olympus AC 240s coated with Pt).

4.7. Setpoint-Based Imaging Method

Setpoint-based imaging is an iterative method of determining scanning parameters. Formerly, the procedure involved setting an arbitrary free air amplitude and adjusting the set point accordingly. In this alternative method, a setpoint is chosen based on the estimated roughness of the sample

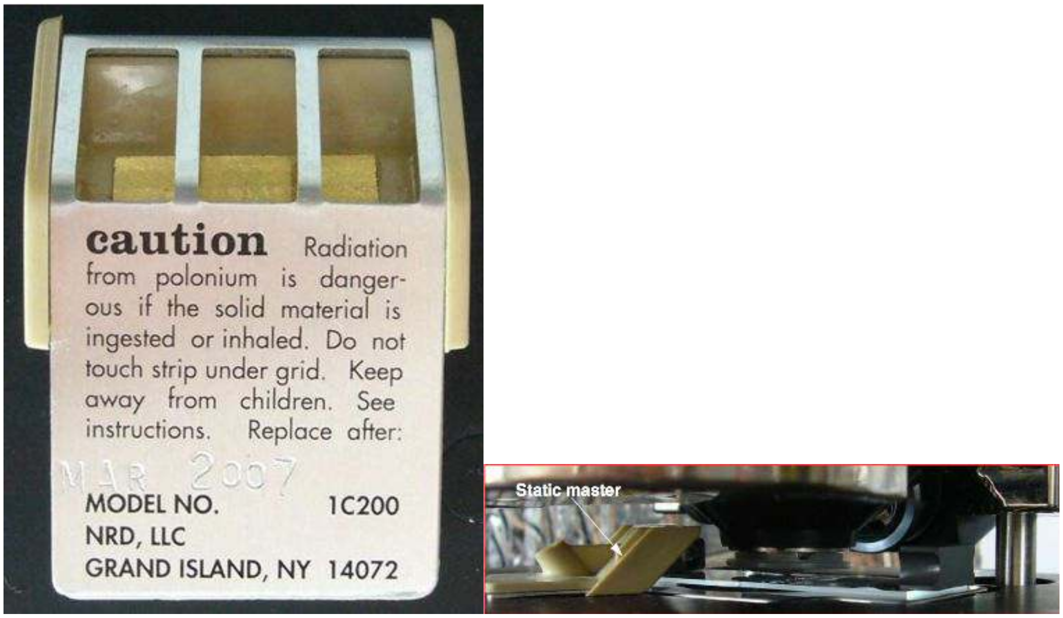


Figure 4.5.: Placing static master near sample on an MFP-3D AFM to dissipate charge

surface and the InvOLS of the cantilever. Once this has been determined, the setpoint is left as is, and the drive amplitude is instead adjusted to maintain either attractive or repulsive mode imaging. Overall, the goal is to establish the minimum setpoint to maintain a stable image.

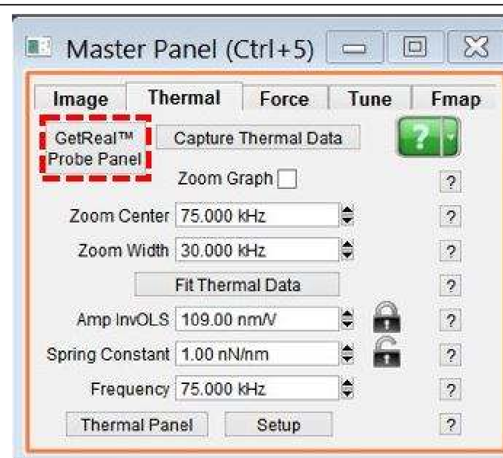
This is accomplished by first determining the optical sensitivity of the cantilever. In a physical sense, this refers to how much the laser point on the photodetector moves relative to the vertical motion of the cantilever.

To perform setpoint-based imaging:

1.

Getting started:

- Make sure the cantilever is engaged on the surface of the sample.
- Select the *Thermal* tab on the Master Panel (Ctrl+5).
- Click 'GetReal Probe Panel'. The panel opens.



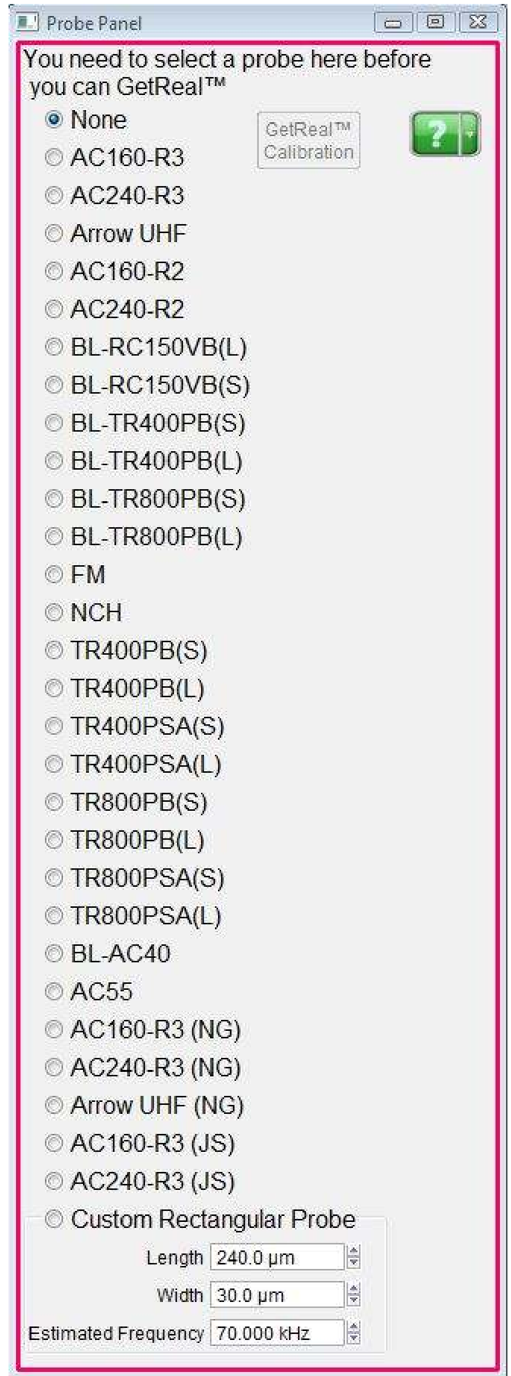
2.

Initiating cantilever calibration:

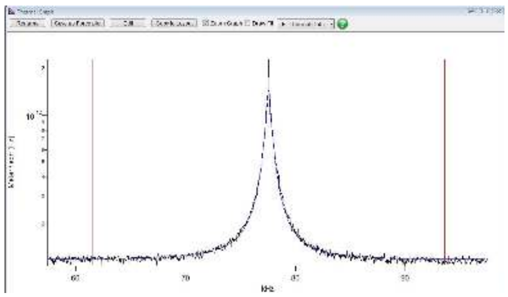
- On the Probe Panel, you will see a list of different cantilevers. Select the cantilever you are using.

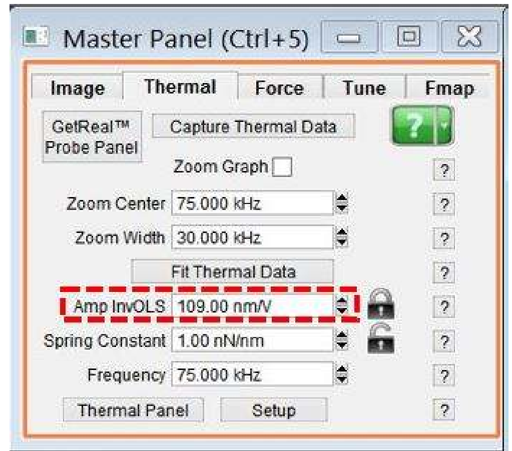
Notes

- It is possible to select a similar cantilever, though the results will be slightly inaccurate.
- If you are using a cantilever not on the list, there is a link on the Asylum User Forum that allows you to request information about special cantilevers. It also includes instructions for performing your own calibrations (with reference to the John Sader paper on the subject).



3.





Calibration results:

- Click ‘GetReal Calibration’. After a few moments, a graph of the calibration will display. It should resemble the figure shown, above left.
- Once the cantilever has been calibrated, a value appears in the *Amp InvOLS* field. This represents the amplitude in nm/V.

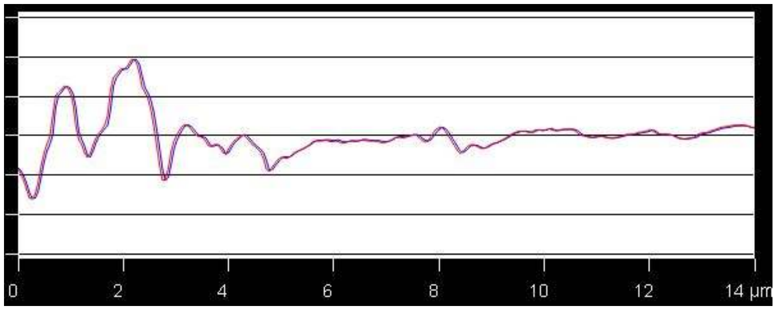
Note “InvOLS” stands for “Inverse Optical Lever Sensitivity.” However, the number is the reciprocal of the optical lever sensitivity rather than its inverse.

4.

Engaging on the surface:

- Choose a set point that is slightly higher than the free air amplitude. This will cause the cantilever to retract.
- Increase the *Drive Amplitude* until the tip is just on the surface. The Z voltage will stop changing at this point.
- Start imaging.

Note The set point can never be lower than the sample height. If the sample height is unknown, determine it before proceeding.



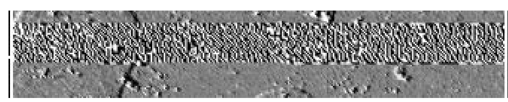
5. **Attractive or Repulsive Mode**

- For repulsive mode, increase the drive amplitude until the phase drops below 90 degrees. If this doesn't seem to work, it might be necessary to restart scanning with a higher set point.

  -OR-

- For Attractive mode, keep the same set point but adjust the drive amplitude for closer tracking of the trace and retrace lines. See above image with an example of closely tracked trace and retrace lines.

Tip For better tracking in Attractive mode, increase the *Integral Gain*. Increasing the integral gain too much may cause feedback oscillations, pictured at right; aim for a gain that minimizes amplitude error without introducing feedback oscillations.



5. AC Mode Imaging: Scientific Background and Theory

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

5.1. Feedback

In AC mode, the cantilever is typically oscillated mechanically by a small piezo electric actuator very near the cantilever chip. Prior to imaging, the Drive Frequency is swept over a broad range to locate the first resonance of the cantilever. The Drive Frequency is then set at or near that Resonance Frequency. The name “AC mode” stems from the tip oscillation: Think of AC current vs. DC current. The optical detector senses the oscillatory motion of the cantilever, and the electronics inside the controller measure the amplitude of this oscillation and also the phase with respect to the drive signal.

When thinking about AC mode, it is a useful experiment to dissect an AC mode force plot. An AC mode force plot collects the cantilever amplitude as the oscillating tip is moved towards the surface and away again. This is shown in Figure 5.3 on page 48. On the right side of the graph, the cantilever is oscillating 100 nm above a flat sample surface. In this force plot, the cantilever was oscillating at resonance with an amplitude of 60 nm. That means the tip swings sinusoidally from 60 nm below the rest to a position 60 nm above. Now we move the base of the lever (as well as the resting point of the lever) towards the surface, and we follow on the graph from right to left (Figure 5.3 on page 48). The amplitude remains constant until the resting position of the cantilever gets within 60 nm of the surface, then the bottom of the oscillation is just barely touching the surface. As we bring the cantilever closer to the surface, the tip oscillation must be reduced. You may think that the tip motion will now have the form of a truncated sine wave, but in reality, the equations of

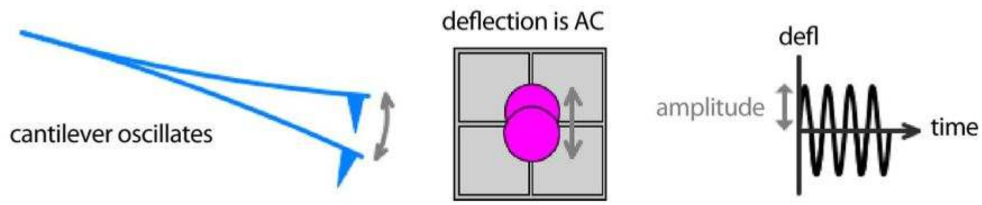


Figure 5.1.: An oscillating cantilever leads to an oscillating signal on the photodetector

Pause to consider the absolute magnitude of these oscillations. Picture a 12 μm tall cone. The absolute motion is twice the amplitude (120nm) or only 1% of the entire conical tip structure. Clearly, this is quite exaggerated as it shows an oscillation amplitude a thousand times larger than reality. Keep in mind that the oscillation amplitudes are quite small and that nearly any figure you see in any paper, book, or manual, will greatly exaggerate the amplitude of oscillation.

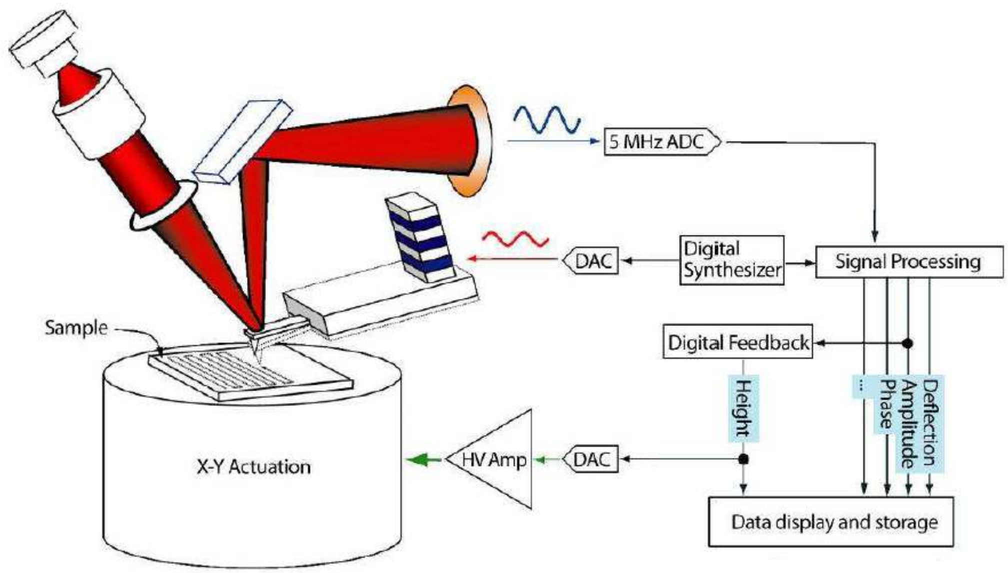


Figure 5.2.: Basic diagram of AC mode imaging.

motion produce something that is very nearly a sine wave with a reduced amplitude. You can see this in the plot; the amplitude is linearly decreasing as the surface inhibits the oscillation.

This linearly sloping amplitude with Z is well suited for feedback signal. In continuing our example, an amplitude setpoint of 40 nm will cause the feedback to reduce the average tip sample separation (z-position) to 40 nm. If, during scanning, the surface suddenly slopes up, the oscillation amplitude will decrease, and the feedback will drive the z piezo up until the amplitude is back at its setpoint.

5.2. Phase Image

A Phase image has a wealth of information; it is equivalent to a map of dissipation. Polymer samples may show little of interest in terms of topography but contain a lot of phase contrast in terms of nano variations in sample mechanical properties. While previous sections explain a lot

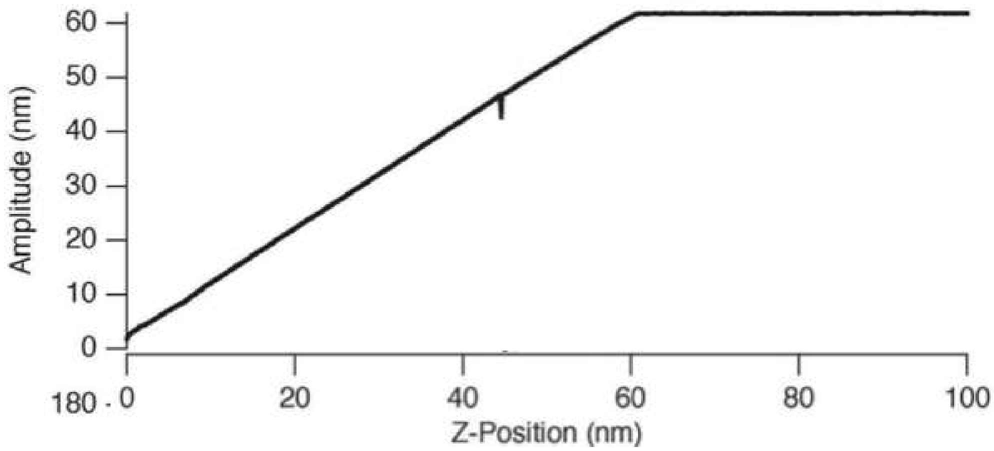


Figure 5.3.: Tip oscillation amplitude as it approaches the surface. The small glitch in amplitude is explained later. See 5.4.

about AC mode imaging in terms of amplitude, it is only half the story, as any driven oscillator has both amplitude *and* phase. Phase is expressed in degrees. A cantilever driven a good deal below resonance is said to be in phase (0°), i.e., while the base of the cantilever is slowly driven up, the entire lever moves up at the same speed. A cantilever driven a good deal above resonance will do the opposite at its tip and base. This condition is called “out of phase” (180°). At resonance, it is neither in-phase nor out, but in between. We can think about this with a human scale example, a swing. When swinging, you change the sign of the drive when the amplitude of the swing is at a maximum, meaning the drive signal is 90° behind the amplitude signal.

5.3. Attractive and Repulsive Behavior

Let’s revisit Figure 5.3 on page 48. This time we will show the phase signal as well.

Again, we’ll start at the right side of the graph. The cantilever is driven nearly at resonance. The Amplitude is 60 nm, and the Z-Position is 100 nm. The tip does not touch the surface, even at its lowest point. The Phase is nearly at 90°, slightly larger, indicating that the cantilever is almost being driven at resonance.

As soon as Z-Position decreases to the point where the tip starts interacting with the surface, the Phase starts to grow larger than 90°. This behavior can be explained by the attractive force the tip experiences from the sample, during the brief time of each oscillatory cycle that it spends near the surface. The closer we move the cantilever to the sample, the more attractive force experienced, and the larger the phase grows. But as the tip gets closer to the surface, there is also repulsive forces, damping the oscillation. At some point during the decreasing of z-position, the repulsive forces are greater than the attractive forces, and the phase shifts to below 90°. The attractive forces are pulling down on the lever at the bottom of the cycle, and the repulsive forces are pushing at the bottom of the cycle. The attractive forces will shift the resonance frequency to lower frequencies, so if you started driving on resonance, the shift of the resonance to lower frequency means your phase is > 90°. Then when the repulsive forces exceed the attractive forces, it is net repulsive. That repulsive pushing at the bottom of the oscillation shifts the resonance frequency to higher

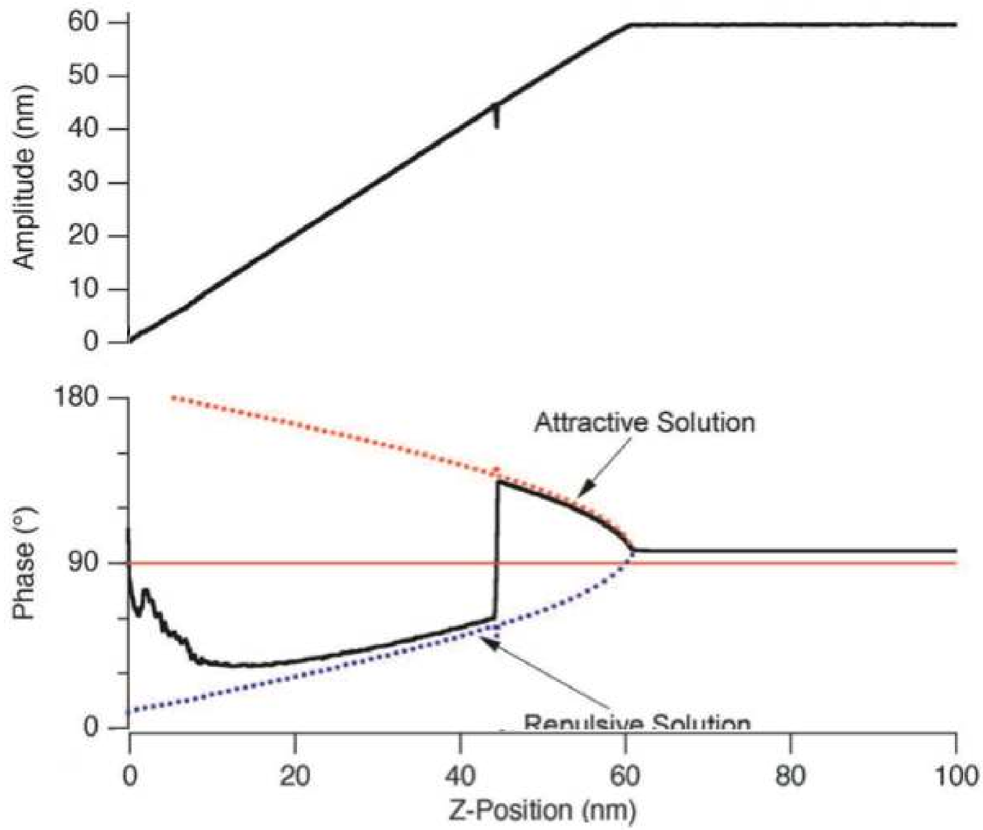


Figure 5.4.: Cantilever amplitude and phase as a function of average tip-sample separation

frequencies, meaning the drive frequency is below resonance, and you see a phase < 90°. This shift in resonance frequency is further illustrated in Figure 5.6 on page 51.

Notice from Figure 5.6 on page 51 that if you choose a negative Target Percent amplitude during an Auto Tune (Section 4.3 on page 36), it helps keep the tip in Repulsive mode during AC mode imaging (black vertical line crosses blue dotted line in Section 4.6.1 on page 39). Likewise, positive target percent amplitudes help the tip stay in Attractive mode (black vertical line crosses the red dotted line; see Section 4.6.2 on page 39).

For more on this topic, see references[1,2,3,4].

5.3.1. Selecting Attractive vs. Repulsive

In practice, it can be quite difficult to select between Attractive mode and Repulsive mode imaging. For the specific case shown in Figure 5.5 on page 50, a setpoint chosen too close to 60nm will lead to unstable imaging. A setpoint chosen too close to 43 nm will cause the phase to constantly flip between attractive and repulsive. Luckily, we have not explored the option of choosing a different free amplitude. The 60 nm free amplitude used in the examples so far was arbitrary.

[1] J. Tamayo, R. Garcia, Appl. Phys. Lett., 1998 73(20), p2926.

[2] J.P. Cleveland, B. Anczykowski, A.E. Schmid, V.B. Elings, Appl. Phys. Lett., 1998 72(20) , p2613

[3] A. San Paulo, R. Garcia, Biophys. Journ., 2000 78, p1559.

[4] R. Garcia, R. Perez, Surface Science Reports, 2002 47 p197-301.

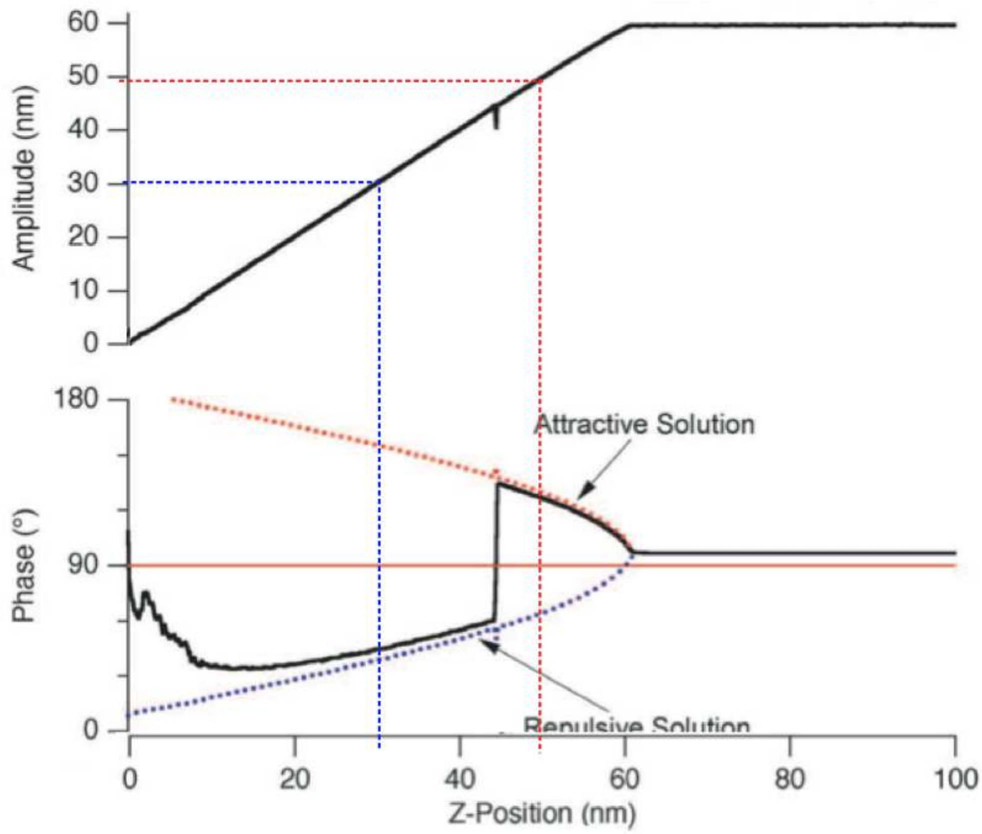


Figure 5.5.: AC mode feedback setpoint of 50nm (red dashed lines) gives attractive mode imaging, a setpoint of 30nm (blue dashed lines) gives repulsive mode imaging.

Take a look at Figure 5.7 on page 52. This is a busy graph, but to get some frame of reference, the red curves on the graph are the same as we have been looking at in the few previous figures. The other curves are just repetitions but with higher or lower free amplitudes. For instance, for the top black curve, the cantilever is oscillating with an Amplitude of nearly 300 nm when it is well above the surface. Note that from the corresponding phase curve, the cantilever only oscillates in Attractive mode (phase > 90°) for a very small range of setpoints very close to that free amplitude and then stays repulsive (phase < 90°) for lower setpoints.

The green, blue, purple, and pink curves are for successively lower and lower cantilever drive amplitudes. The behavior is similar, but there is a growing trend of attractive behavior with setpoints near the free amplitude.

For the blue curve with a 50 nm free amplitude and the black curve with 20 nm free amplitude, there is a remarkable change of general behavior. No matter what setpoint you choose (below the free amplitude), the cantilever always oscillates in the attractive regime. Now we can make some fairly good rules of thumb for imaging in purely Attractive mode, purely Repulsive mode, or something prone to switching between the two.

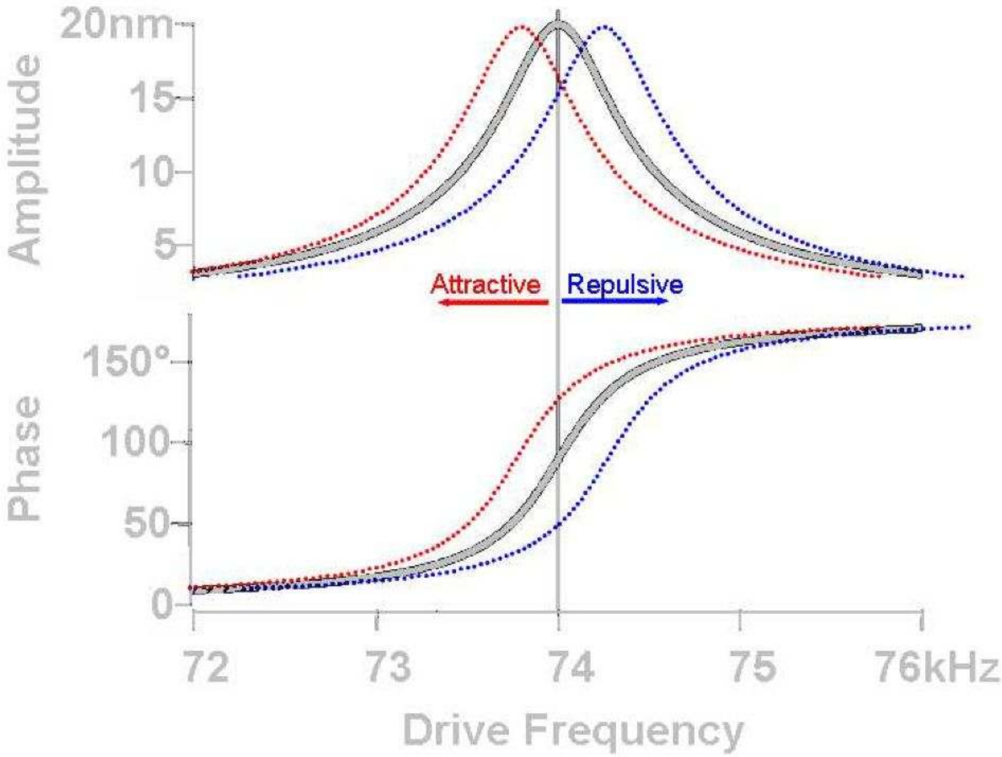


Figure 5.6.: A shift in resonance frequency, f_o (at constant amplitude) as the tip interacts with surface results in phase shift of the AC signal: Decrease in f_o results in phase increase (attractive mode), while increase in f_o results in phase decrease (repulsive mode). The colors represent the color of the phase on the Sum and Deflection Meter Panel.

5.3.2. Rules of Thumb for Attractive or Repulsive Mode Imaging

Repulsive Mode More Likely	Attractive Mode More Likely
Bigger free amplitude	Smaller free amplitude
Stiffer cantilever	Softer cantilever
Drive frequency lower than resonance	Drive frequency higher than resonance
Lower Q, Negative Q gain	Higher Q, Positive Q gain
Sample with smaller attractive forces	Sample with bigger attractive forces
Sharp tip	Dull tip

Table 5.1.: Attractive vs. Repulsive AC mode imaging “rules of thumb”

5.3.2.1. Free amplitude

Bigger or smaller free amplitude are relative terms. Only once you perform a series of AC force curves with a particular cantilever and a particular sample, you will be able to quantify what bigger and smaller mean. For instance, the experiments in Figure 5.7 on page 52 show for that cantilever and sample bigger means > 150 nm and smaller means < 50nm.

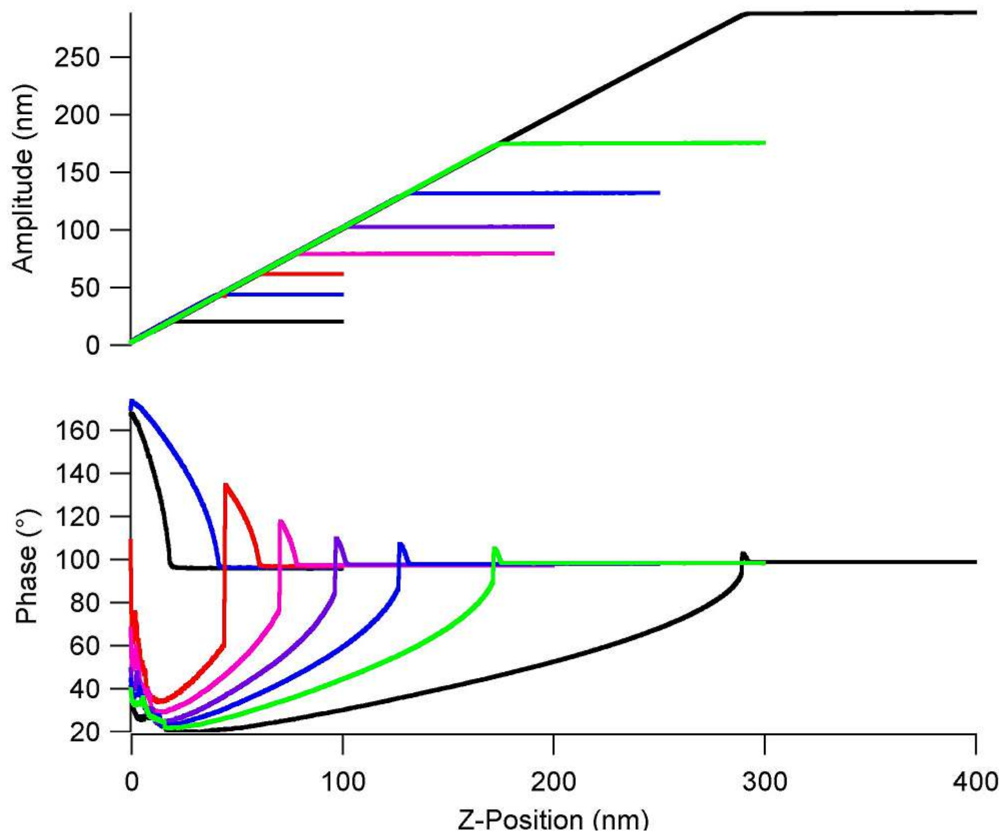


Figure 5.7.: A family of AC force curves for different free amplitudes

5.3.2.2. Cantilever choice

Stiffer and softer are relative terms, but for the sake of simplicity we will consider the two most common AC mode levers. On the Asylum Research Probe Store some general use AC mode probes are listed. The first choice for general AC mode imaging should probably be the Olympus AC160. With a spring constant of around 40 N/m it is considered a relatively stiff probe good for repulsive mode imaging. The next most commonly used AC mode lever is the AC240, it is 20 times softer, and considered a good probe for attractive or repulsive mode imaging. Note that the data presented in earlier in this section were for a probe similar to the AC240.

5.3.2.3. Drive frequency above or below resonance

In practice, the AR software makes it very easy to tune the cantilever slightly above or below resonance. Under the *Tune* tab of the Master Panel, you can select 'Target Percent'. A value of -10% is good to promote Repulsive mode imaging, and +10% helps promote Attractive mode imaging. Alternatively, you can right-click on the resonance curve after tuning and manually select the drive frequency.

5.3.2.4. Q (Quality Factor)

The Q factor of the cantilever is a result of both the lever and the environment (liquid or gas). If you have a relativity low Q (<30), you may find it difficult to stay in Attractive mode. In such cases, consider using the Q control feature of the AR SPM software. By means of digital processing in the SPM controller, it allows you to increase or decrease the Q. You can read more in Section 4.6.2.3 on page 39.

5.3.2.5. Sample attractive forces

You typically don't have much control over sample attractive forces; but if you find it particularly difficult to achieve attractive or repulsive mode imaging, it may be due to your sample. See Section 4.6.3.1 on page 41 for ways to combat this issue.

5.3.2.6. Tip sharpness

A dull tip has more contact area and is more strongly attracted to a surface than a sharp tip and increases the probability of Attractive mode imaging.

6. Piezo Force Microscopy: Scientific Background and Theory

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

by Roger Proksch[1] *and Sergei Kalinin*[2]

Chapter Contents

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6.1. Summary

Electromechanical coupling is one of the fundamental mechanisms underlying the functionality of many materials. These include inorganic macro-molecular materials, such as piezoelectrics and ferroelectrics, as well as many biological systems. This application note discusses the background, techniques, problems, and solutions to Piezoresponse Force Microscopy (PFM) measurements using the MFP-3D™ and Cypher™.

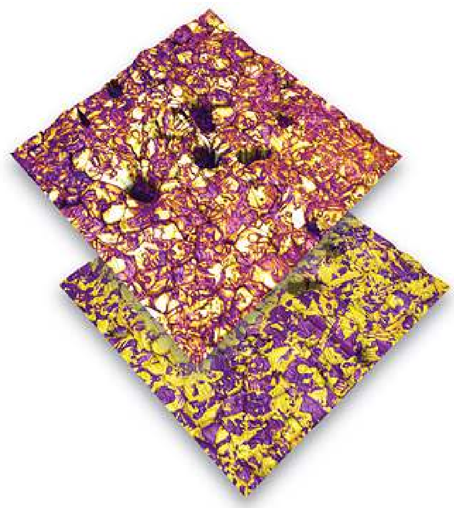

Figure 6.1.: PFM amplitude channel overlaid on AFM height (top) and phase image overlaid on height (bottom) of lead zirconium titanate (PZT), 20 µm scan

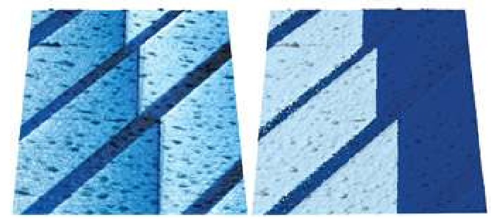

Figure 6.2.: PFM amplitude overlaid on AFM topography (left) and PFM phase overlaid on topography (right) on (100) oriented BaTiO3 single crystal (from Castech Crystals). The amplitude and phase image show 90° and 180° domain walls in BaTiO3. (10 µm scan courtesy of V. R. Aravind, K. Seal, S. Kalinin, ORNL, and V. Gopalan, Pennsylvania State University.)

6.2. Background

The functionality of systems ranging from non-volatile computer memories and micro-electromechanical systems to electromotor proteins and cellular membranes are ultimately based on the intricate coupling between electrical and mechanical phenomena[3]. The applications of electromechanically active materials include sonar, ultrasonic and medical imaging, sensors, actuators, and energy harvesting technologies. In the realm of electronic devices, piezoelectrics are used as components of RF filters and surface-acoustic wave (SAW) devices[4]. The ability of ferroelectric materials to switch polarization orientation – and maintain polarization state in a zero electric field – has led to emergence of concepts of non-volatile ferroelectric memories and data storage devices[5]. Electromechanical coupling is the basis of many biological systems, from hearing to cardiac activity. The future will undoubtedly see the emergence, first in research labs and later in industrial settings,

[3] Kalinin, S/Gruverman, A, editors, *Scanning probe microscopy : electrical and electromechanical phenomena at the nanoscale*. Springer, New York, 2007.
[4] Uchino, K., *Ferroelectric Devices*. Marcel Dekker, 2005.
[5] Scott, J., *Ferroelectric Memories*. Berlin: Springer Verlag, 2006.

of the broad arrays of piezoelectric, biological, and molecular-based electromechanical systems. Progress along this path requires the ability to image and quantify electromechanical functionalities on the nanometer and molecular scale (Figure 6.1 on page 55 and Figure 6.2 on page 55). Areas such as nanomechanics and single-molecule imaging and force measurements have been enabled by the emergence of microscopic tools such as nanoindentation and protein unfolding spectroscopy.

Similarly, the necessity for probing electromechanical functionalities has led to the development of PFM as a tool for local nanoscale imaging, spectroscopy, and manipulation of piezoelectric and ferroelectric materials[6].

6.3. Principles of PFM

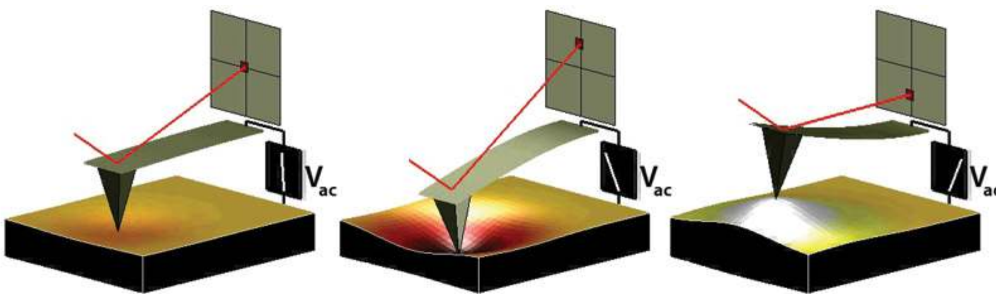

Figure 6.3.: Depiction of PFM operation. The sample deforms in response to the applied voltage, which in turn causes the cantilever to deflect, which can then be measured and interpreted in terms of the piezoelectric properties of the sample. (Image courtesy of S. Jesse, ORNL.)

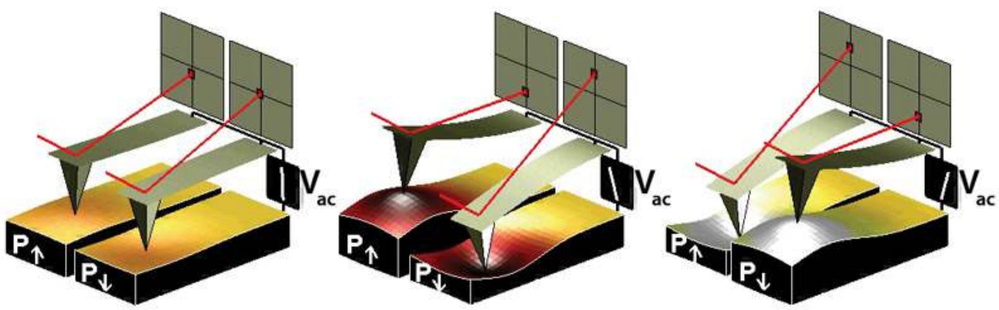

Figure 6.4.: Sign dependence of the sample strain. When the domains have a vertical polarization that is pointed downwards and a positive voltage is applied to the tip, the sample will locally expand. If the polarization is pointed up, the sample will locally contract. The phase of the measured response is thus proportional to the direction of the domain polarization. (Image courtesy of S. Jesse, ORNL.)

6.3.1. Basics

PFM measures the mechanical response when an electrical voltage is applied to the sample surface with a conductive tip of an AFM. In response to the electrical stimulus, the sample then locally

[6] Jesse, S/Baddorf, AP/Kalinin, SV, Dynamic behaviour in piezoresponse force microscopy. NANOTECHNOLOGY, 17 MAR 28 2006, Nr. 6, ISSN 0957–4484.

expands or contracts as shown in Figure 6.3 on page 56

When the tip is in contact with the surface and the local piezoelectric response is detected as the first harmonic component of the tip deflection, the phase φ, of the electromechanical response of the surface yields information on the polarization direction below the tip. For c- domains (polarization vector oriented normal to the surface and pointing downward), the application of a positive tip bias results in the expansion of the sample, and surface oscillations are in phase with the tip voltage, $\varphi = 0$. For c+ domains, the response is opposite and $\varphi = 180^\circ$. More details are given in Section 6.3.2 (below).

Detection of the lateral components of tip vibrations provides information on the in-plane surface displacement, known as lateral PFM. A third component of the displacement vector can be determined by imaging the same region of the sample after rotation by 90°.[7] Provided that the vertical and lateral PFM signals are properly calibrated, the complete electromechanical response vector can be determined, an approach referred to as "vector PFM"[8]. Finally, electromechanical response can be probed as a function of DC bias of the tip, providing information on polarization switching in ferroelectrics, as well as more complex electrochemical and electrocapillary processes[9,10].

PFM requires detection of small tip displacements induced by relatively high amplitude, high frequency voltages measured at the same frequency as the drive. Any instrumental crosstalk between the drive and the response will result in a virtual PFM background that can easily be larger than the PFM response itself, especially for weak piezo materials. Minimizing crosstalk between the driving voltage and the response imposes a number of serious engineering limitations on the microscope mechanics and electronics. In the past, significant post-factory modifications were required to decouple the drive and response signals. Asylum's PFM uses a unique proprietary design of the head and the high voltage sample holder to eliminate drive crosstalk (see below) .

6.3.2. Piezo Effect

The relationship between the strain and the applied electric field (often referred to as the "inverse piezo effect") in piezoelectric materials is described by a rank-3 tensor. The most important component of this tensor for typical "vertical" PFM is the d_{33} component[11] as it couples directly into the vertical motion of the cantilever. The voltage applied to the tip is

$$V_{tip} = V_{dc} + V_{ac}\cos(\omega t) \tag{6.1}$$

resulting in piezoelectric strain in the material that causes cantilever displacement

$$z = z_{dc} + A(\omega, V_{ac}, V_{dc})\cos(\omega t + \varphi) \tag{6.2}$$

[7] Eng, LM et al., Nanoscale reconstruction of surface crystallography from three-dimensional polarization distribution in ferroelectric barium-titanate ceramics. Applied Physics Letters, 74 JAN 11 1999, Nr. 2, ISSN 0003–6951.

[8] Kalinin, Sergei V. et al., Vector piezoresponse force microscopy. Microscopy and Microanalysis, 12 JUN 2006, Nr. 3, ISSN 1431–9276.

[9] Verdaguer, A et al., Molecular structure of water at interfaces: Wetting at the nanometer scale. Chemical Reviews, 106 APR 2006, Nr. 4, ISSN 0009–2665.

[10] Sacha, G. M./Verdaguer, A./Salmeron, M., Induced water condensation and bridge formation by electric fields in atomic force microscopy. Journal of Physical Chemistry B, 110 AUG 3 2006, Nr. 30, ISSN 1520–6106.

[11] Eliseev, Eugene A. et al., Electromechanical detection in scanning probe microscopy: Tip models and materials contrast. Journal of Applied Physics 102 JUL 1 2007, Nr. 1, ISSN 0021–8979.

Table 6.1.: Some Properties of common piezoelectric materials.

Material	Application	$d_{33'}$ pm/V†	Coercive bias (for local switching)††	Breakdown voltage / onset of conductivity†††
Bulk Materials				
PZT ceramics	Actuators & transducers	100-500	10V-1kV	N/A
$L_iN_bO_3$ single crystals	Electro-optical devices	10-20	10V-1kV	N/A
Quartz	Balances, frequency standards	3	N/A	N/A
Polar Semiconductors	RF devices, switches	0.1-0.2	N/A	N/A
Calcified Tissues		0.5-3	N/A	N/A
Collagen		0.5-3	N/A	N/A
Thin Films and Capacitor Structures				
1-5 micron PZT	Capacitors	10-30	1-100	100
100-300nm PZT	FeRAM elements	3-10	1-10	10-20
30-100 nm $B_iF_eO_3$	FeRAM	3-10	1-10	10-20
Ultrathin Films				
1-5nm $B_iF_eO_3$	Tunneling Barriers	1-10	1-5	10 (can be below switching voltage in air)
10nm PVDF	Actuators	20	2-5	10

† The PFM signal is given by equation6.6 A=d_{33}VacQ where d_{33} is material property, Vac is driving voltage, and Q is the quality factor. Q=1 for low frequency PFM, and Q = 20-100 if resonance enhancement (DART or BE) method is used. Vac is limited by material stability and polarization switching. The microscope photodetector sensitivity, thermal noise and shot noise impose the limit A > 30pm. The ultimate limit is A = thermal noise.
††Quantitative spectroscopic measurements require probing bias to be one to two orders of magnitude smaller than coercive bias, limiting the voltage amplitude.
††† Measurements are not always possible due to sample and tip degradation.

due to piezoelectric effect[12]. When the voltage is driven at a frequency well below that of the contact resonance of the cantilever, this expression becomes

$$z = d_{33}V_{dc} + d_{33}V_{ac}\cos(\omega t + \varphi) \tag{6.3}$$

where we have implicitly assumed d_{33} depends on the polarization state of the material. From this last equation, and from Figure 6.3 on page 56, the magnitude of the oscillating response is a measure of the magnitude of d_{33}, and the phase is sensitive to the polarization direction of the sample.

Note In reality, the d_{33} component in equation 6.3 is an "effective" d_{33} that depends on the contribution from other tensor elements and on the crystallographic and real space orientation of the piezo material, as well as details of the tip-sample contact.

[12] Jesse, S/Baddorf, AP/Kalinin, SV, Dynamic behaviour in piezoresponse force microscopy. NANOTECHNOLOGY, 17 MAR 28 2006, Nr. 6, ISSN 0957–4484.

Typical values for d_{33} range from 0.1 pm/V for weak piezo materials to 500pm/V for the strongest. Table 1 shows a listing of representative values.

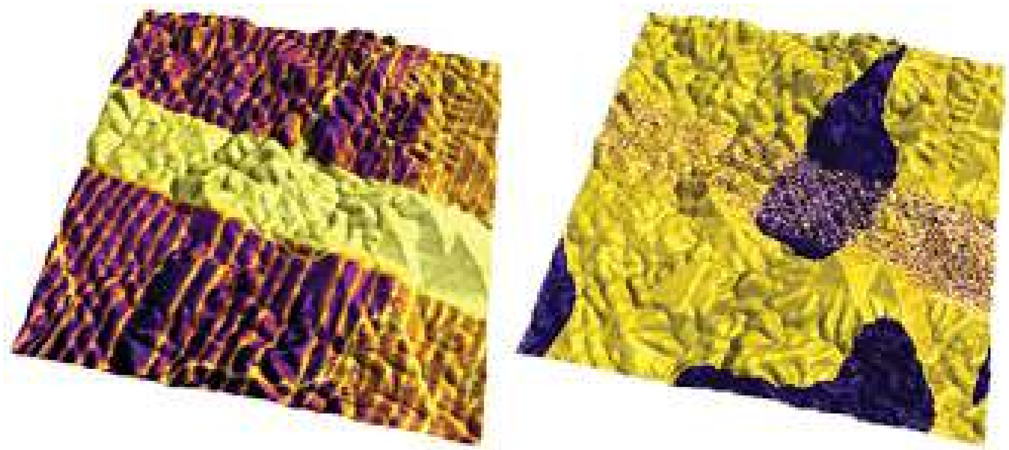

Figure 6.5.: Vertical PFM amplitude overlaid on AFM topography (left) and PFM phase overlaid on AFM topography (right) images of lead titanate film, 5 µm scan. (Images courtesy of A. Gruverman and D. Wu, UNL. Sample courtesy H. Funakubo.)

As mentioned above, the direction of sample polarization determines the sign of the response. Figure 6.4 on page 56 demonstrates this idea. If the polarization is parallel and aligned with the applied electric field, the piezo effect will be positive, and the sample will locally expand. If the local sample polarization is anti-parallel with the applied electric field, the sample will locally shrink. This sign-dependent behavior means that the phase of the cantilever provides an indication of the polarization orientation of the sample when an oscillating voltage is applied to the sample.

The relationship in Equation 6.1 and the values for d_{33} in Table 6.1 on page 58 suggest that typical deflections for a PFM cantilever are on the order of picometers. While the sensitivity of AFM cantilevers is quite impressive–on the order of a fraction of an angstrom (or tens of pm) in a 1kHz bandwidth, it also implies a very small signal-to-noise ratio (SNR) for all but the strongest piezo materials.

Because of this small SNR, piezoelectricity is most frequently detected by a lock-in amplifier connected to the deflection of the AFM cantilever. By employing an oscillating electric field, low-frequency noise and drift can be eliminated from the measurement. Until recently, PFM was usually accomplished by researchers who modified a commercial SPM system with an external function generator/lock in setup. As a result, in most cases, the operation frequency was limited to <100kHz. This, and the lack of sophisticated control options, precluded the use of resonance enhancement in PFM since typical contact resonance frequencies are >300kHz.

6.3.3. PFM Imaging Modes

The three typical PFM imaging modes and piezoelectric lithography are briefly described in the following sections.

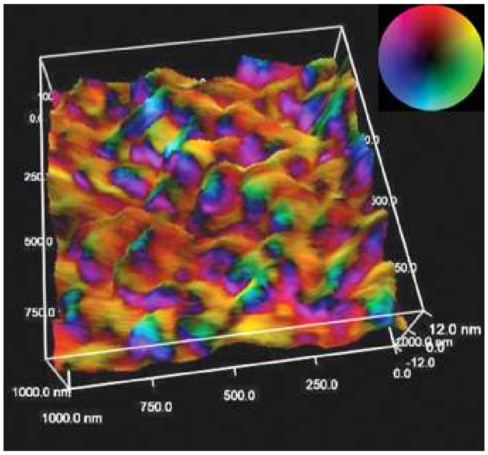

Figure 6.6.: BST film with vector PFM overlaid on AFM topography, 1µm scan. (Image courtesy of C. Weiss and P. Alpay, Univ. of Conn., and O. Leaffer, J. Spanier, and S. Nonnenmann, Drexel University.) Color wheel indicates PFM vector orientation.

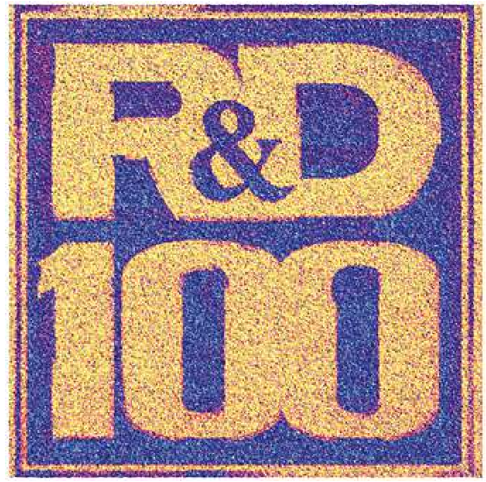

Figure 6.7.: R&D 100 logo written on a sol-gel PZT thin film by PFM lithography. PFM phase is overlaid on top of the rendered topography, 25 µm scan. Oak Ridge and Asylum Research were awarded an R&D100 award for Band Excitation in 2008.

6.3.3.1. Vertical PFM

In Vertical PFM imaging, out-of-plane polarization is measured by recording the tip-deflection signal at the frequency of modulation. Figure 6.5 on page 59 shows an example image of Vertical PFM for a lead titanate film. Antiparallel domains with out-of-plane polarization can be seen in the PFM phase image, while in-plane domains are seen in the PFM amplitude image as yellow stripes due to the weak vertical piezoresponse signal.

6.3.3.2. Lateral PFM

Lateral PFM is a technique where the in-plane component of polarization is detected as lateral motion of the cantilever due to bias-induced surface shearing. Eng et al.[13], Abplanalp et al.[14], and Eng et al.[15], have recently shown that the in-plane component of the polarization can be observed by following the lateral deflection of the AFM cantilever, and have applied this technique to reconstruct the three-dimensional distribution of polarization within domains of ferroelectric single crystals. Roelofs et al. applied this method to differentiate 90° and 180° domain switching in

[13] Eng, LM et al., Nondestructive imaging and characterization of ferroelectric domains in periodically poled crystals. Journal of Applied Physics, 83 JUN 1 1998, Nr. 11, Part 1, ISSN 0021–8979.

[14] Abplanalp, M/Eng, LM/Gunter, P, Mapping the domain distribution at ferroelectric surfaces by scanning force microscopy. APPLIED PHYSICS A-MATERIALS SCIENCE & PROCESSING, 66 MAR 1998, Nr. Part 1 Suppl. S, ISSN 0947–8396.

[15] Eng, LM/Abplanalp, M/Gunter, P, Ferroelectric domain switching in tri-glycine sulphate and barium-titanate bulk single crystals by scanning force microscopy. APPLIED PHYSICS A-MATERIALS SCIENCE & PROCESSING, 66 MAR 1998, Nr. Part 2 Suppl. S, ISSN 0947–8396.

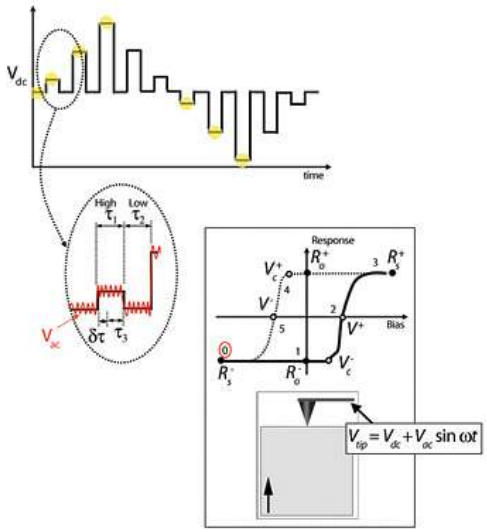

Figure 6.8.: Switching spectroscopy PFM diagram (see text for discussion). (Reused with permission from Jesse, Baddorf, and Kalinin, Applied Physics Letters, 88, 062908 (2006). Copyright 2006, American Institute of Physics.)

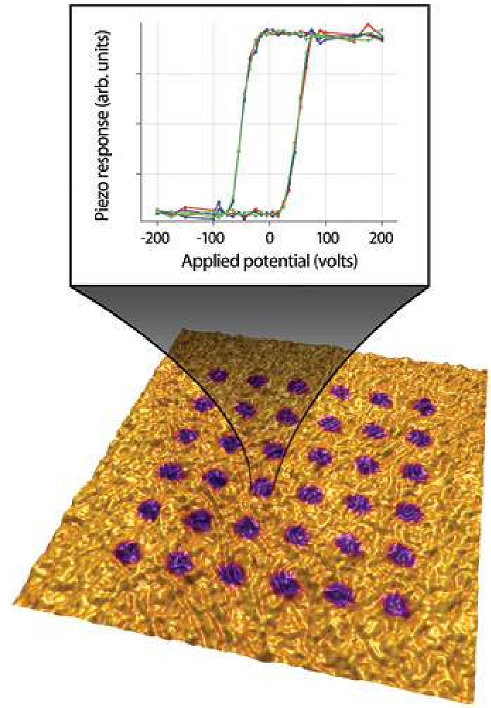

Figure 6.9.: Rendered topography of a $LiNbO_3$ sample with the PFM signal overlaid on top, 4µm scan.

$PbTiO_3$ thin films[16].

6.3.3.3. Vector PFM

In Vector PFM, the real space reconstruction of polarization orientation comes from three components of piezoresponse: Vertical PFM plus at least two orthogonal lateral PFM. Figure 6.6 on page 60 shows an example of a vector PFM image of a barium strontium titanate film (BST), permitting qualitative inspection of the correlation of grain size, shape, and location with local polarization orientation and domain wall character. Here, the color wheel permits identification of the local orientation of the polarization. Regions colored as cyan (darker blue/green) possess polarizations that are oriented predominantly normal to the plane of the film, whereas regions that appear magenta-blue or light green possess polarizations that are oriented predominantly within the plane of the film. The intensity of the color map denotes the magnitude of the response.

6.3.3.4. Lithography

For ferroelectric applications, PFM can be used to modify the ferroelectric polarization of the sample through the application of a bias. When the applied field is large enough (e.g., greater than

[16] Roelofs, A et al., Differentiating 180 degrees and 90 degrees switching of ferroelectric domains with three-dimensional piezoresponse force microscopy. Applied Physics Letters, 77 NOV 20 2000, Nr. 21, ISSN 0003–6951.

the local coercive field), it can induce ferroelectric polarization reversal. This technique can be used to 'write' single domains, domain arrays, and complex patterns without changing the surface topography.

Figure 6.7 on page 60 shows an example of PFM bit-mapped lithography where the color scale of a black and white photo was used to control the bias voltage of the tip as it rastered over the surface and then re-imaged in PFM mode.

6.3.3.5. Spectroscopy modes

PFM spectroscopy refers to locally generating hysteresis loops in ferroelectric materials. From these hysteresis loops, information on local ferroelectric behavior, such as imprint, local work of switching, and nucleation biases, can be obtained.

Understanding the switching behavior in ferroelectrics on the nanometer scale is directly relevant to the development and optimization of applications such as ferroelectric non-volatile random-access memory (FRAM) and high-density data storage. Multiple studies have addressed the role of defects and grain boundaries on domain nucleation and growth, domain wall pinning, illumination effects on the built-in potential, and domain behavior during fatigue. The origins of the field date back to the seminal work by Landauer, who demonstrated that the experimentally observed switching fields correspond to impossibly large (~103 - 105kT) values for the nucleation activation energy in polarization switching. Resolving this 'Landauer paradox' requires the presence of discrete switching centers that initiate low-field nucleation and control macroscopic polarization switching[17]. However, difficulties related to positioning of the tip at a specific location on the surface (due in part to microscope drift), as well as time constraints related to hysteresis loop acquisition, limit these studies to only a few points on the sample surface, thus precluding correlation between the material's microstructure and local switching characteristics.

Switching Spectroscopy Mapping A new spectroscopy technique, Switching Spectroscopy PFM (SS-PFM), has demonstrated real-space imaging of the energy distribution of nucleation centres in ferroelectrics, thus resolving the structural origins of the Landauer paradox[18]. These maps can be readily correlated with surface topography or other microscopic techniques to provide relationships between micro- and nanostructures and local switching behavior of ferroelectric materials and nanostructures. Figure 6.8 on page 61 shows how it works. In SS-PFM, a sine wave is carried by a square wave that steps in magnitude with time. Between each ever-increasing voltage step, the offset is stepped back to zero with the AC bias still applied to determine the bias-induced change in polarization distribution (e.g., the size of the switched domain). It is then possible to see the hysteresis curve of the switching of the polarization of the surface (bottom diagram). If the measurements are performed over a rectangular grid, a map of the switching spectra of that surface can be obtained. Figure 6.9 on page 61 shows an example image of a $LiNbO_3$ sample with the PFM signal overlaid on top. The image was taken after switching spectroscopy. The graph shows the hysteresis loops measured at one individual point.

As additional examples, Figure 6.10 on page 63 shows a sol gel PZT sample where the local switching fields were measured. After the switching spectroscopy, the area was re-imaged. The

[17] Jesse, S/Baddorf, AP/Kalinin, SV, Dynamic behaviour in piezoresponse force microscopy. NANOTECHNOLOGY, 17 MAR 28 2006, Nr. 6, ISSN 0957–4484.

[18] Jesse, Stephen et al., Direct imaging of the spatial and energy distribution of nucleation centres in ferroelectric materials. Nature Materials, 7 MAR 2008, Nr. 3, ISSN 1476–1122.

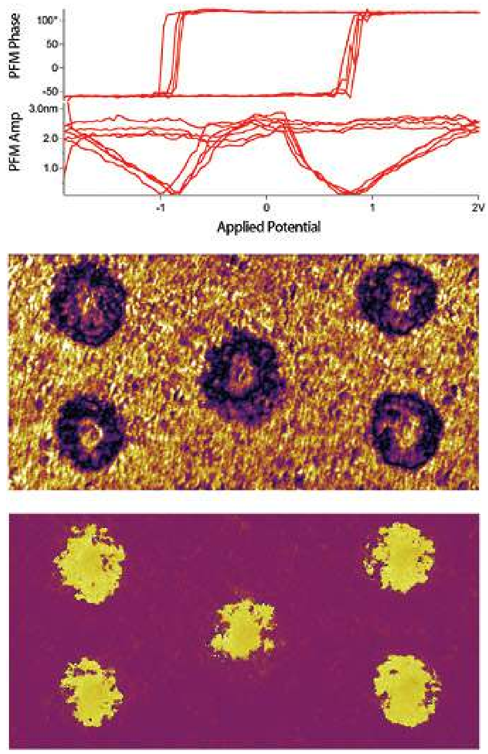

Figure 6.10.: Sol gel PZT sample where local hysteresis loops were measured and displayed (representative phase and amplitude loops shown at top). After the switching spectroscopy measurements, the area was imaged, the DART amplitude (middle) and phase (bottom) are shown, 3.5µm scan.

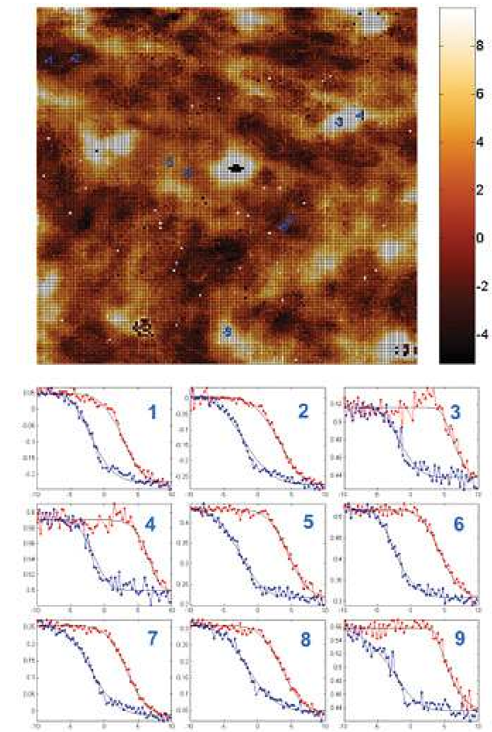

Figure 6.11.: SS-PFM and hysteresis loops of capacitor structures. (Data courtesy K. Seal and S.V. Kalinin, ORNL. Sample courtesy P. Bintacchit and S. Trolier-McKinstry, Penn State Univ.)

PFM signal clearly shows five dots in the phase signal denoting portions of the sample where the polarization was reversed during the hysteresis measurements. Figure 6.11 on page 63 shows SSM-PFM of capacitor structures and Figure 6.12 on page 64 shows an image of phase and amplitude hysteresis loops measured at five different locations on a lead zinc niobate - lead titanate (PZN-PTi) thin film.

6.4. Limitations of Conventional PFM Methodologies

In this section we will discuss how conventional PFM imaging and the contact resonance, we are left with the situation where we need to choose between two sub-optimal alternatives:

- Operate on resonance to benefit from the boosted signal but have complicated artifacts that do not allow unambiguous determination of the sample domain structure,

  OR

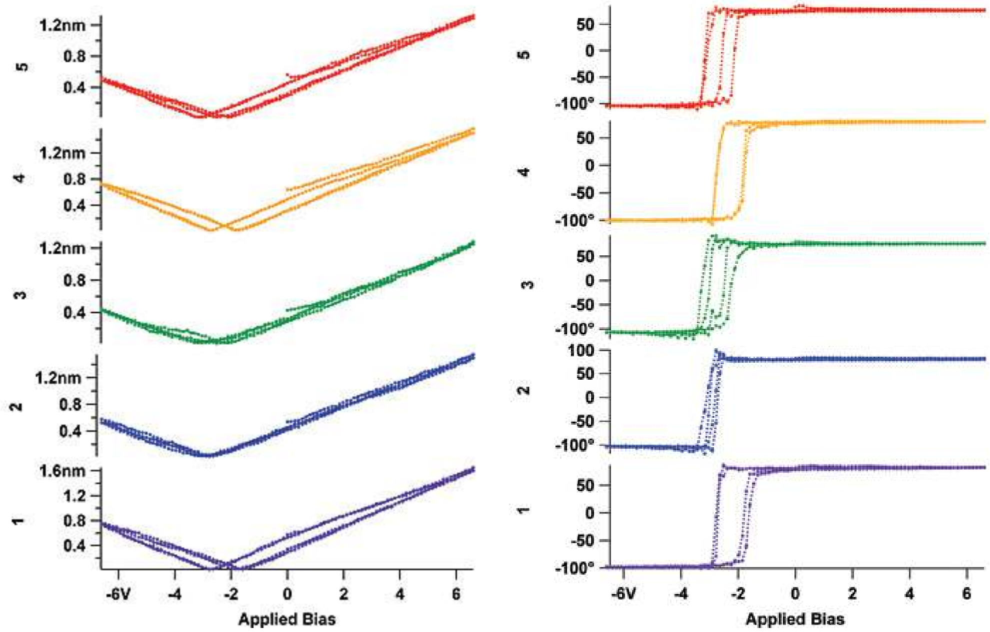

Figure 6.12.: Amplitude (left) and phase (right) hysteresis loops measured at five different locations on a PZN-PTi thin film.

- Avoid resonance to minimize topographic crosstalk, but suffer from the small signals inherent in piezo materials.

6.4.1. High Voltage Limitations

Traditionally, the use of 1-10Vpp driving amplitude on materials with strong electromechanical responses (e.g., $d_{33}\approx100$ pm/V for PZT, 10 pm/V for $LiNbO_3$) allowed direct imaging and spectroscopy of ferroelectric materials sufficient for applications corresponding to a detection limit of 50pm at ~100 kHz. Measurements of lower sensitivity materials require the use of higher voltages or the use of contact resonance.

6.4.2. Imaging at Contact Resonance

For some samples, using a higher drive voltage is undesirable. High drive voltages will result in polarization switching or even damage to the sample. Recent advances in theoretical understanding of the PFM imaging mechanism illustrate that the primary limitation of previous commercial and home built SPMs is their inability to effectively use resonance enhancement.

Probe polarization dynamics in commercial low-voltage ferroelectric capacitors is optimal for driving amplitudes of 30-100mV (to avoid bias-induced changes in domain structures), which is 1-2 times below the magnitude of standard, low-frequency PFM capabilities. Finally, the use of PFM as an electrophysiological tool necessitates operation in the mV regime, required to prevent damage to biological systems as well as stray electrochemical reactions[19].

[19] Frederix, PLTM et al., Assessment of insulated conductive cantilevers for biology and electrochemistry. NANOTECHNOLOGY, 16 AUG 2005, Nr. 8, ISSN 0957–4484.

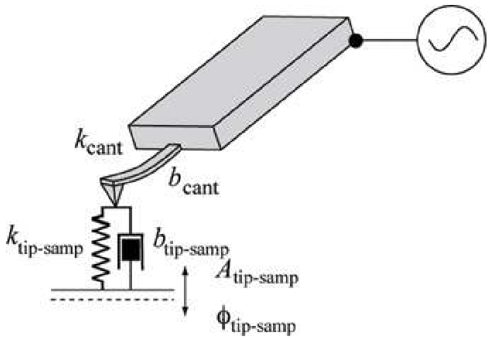

Figure 6.13.: In PFM, the cantilever voltage is modulated, usually at some fixed frequency. This causes the sample to distort at some amplitude and phase. Mediated by the contact mechanics, this drives the tip which, in turn, is monitored by the AFM sensor.

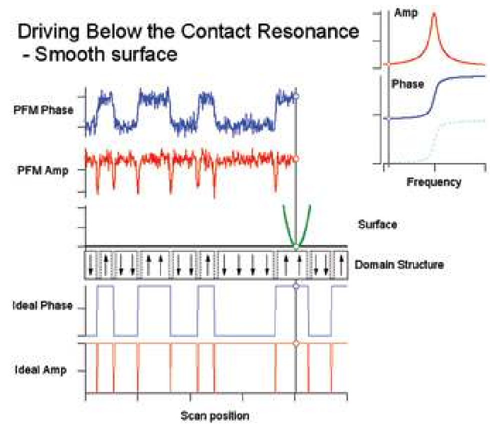

Figure 6.14.: This figure shows the ideal and measured PFM response of an idealized tip (green) scanning over a smooth surface (black line below the "tip"). The domain structure of the ferroelectric sample is shown below the surface where the arrows correspond to the sample polarization direction. The gray hatched regions between the domains are representative of the domain walls. The "ideal phase" (blue, thin curve) and "ideal amp" (red, thin curve) show the idealized response of a probe that measures the piezoelectric response over the domains. The measured PFM amplitude (red, thick curve) and phase (blue, thick curve) channels appear above the scanning tip. Because these measurements are made below the resonant frequency where there is no resonance enhancement of the PFM signal, the signal to noise is relatively small for the measured signal.

The resonant frequencies are determined only by the weak voltage-dependent mechanical properties of the system and are independent of the relative contributions of the electrostatic and electromechanical interactions. As shown by Sader[20], in the vicinity of a resonance for small damping ($Q > 10$), the amplitude and phase frequency response can be described using the harmonic oscillator model[21] as

$$A(\omega) = \frac{A_{max}\omega_0^2/Q}{\sqrt{(\omega_0^2 - \omega^2)^2 + (\omega_0\omega/Q)^2}} \tag{6.4}$$

[20] Sader, JE, Frequency response of cantilever beams immersed in viscous fluids with applications to the atomic force microscope. Journal of Applied Physics, 84 JUL 1 1998, Nr. 1, ISSN 0021–8979.

[21] Garcia, R/Perez, R, Dynamic atomic force microscopy methods. Surface Science Reports, 47 2002, Nr. 6-8, ISSN 0167–5729.

$$\tan \varphi(\omega) = \frac{\omega_0 \omega}{Q(\omega_0^2 - \omega^2)} \tag{6.5}$$

where A_{max} is the amplitude at the resonanceω_0 and Q and is the quality factor that describes energy losses in the system. Resonance is a phenomenon used in many SPM techniques. The cantilever response at resonance is essentially multiplied by the quality factor (Q) of the cantilever

$$A = d_{33} V_{ac} Q \tag{6.6}$$

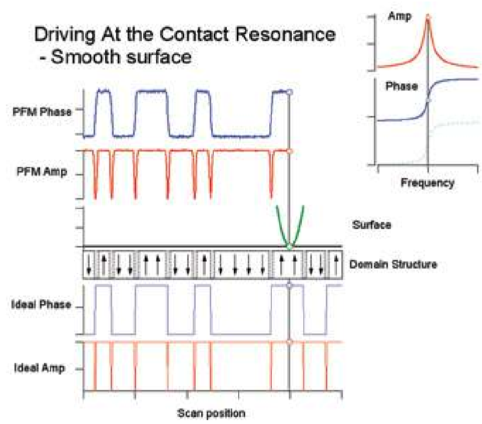

Figure 6.15.: This figure shows the same situation as described in Figure 6.14 on page 65, except that here we are using resonance enhancement to boost the small PFM signal. The inset frequency tune in the upper right corner shows the drive frequency. In this case, since the Q-factor of the resonance is 100, the SNR of the measured PFM amplitude (red, thick curve) and phase (blue, thick curve) has dramatically improved.

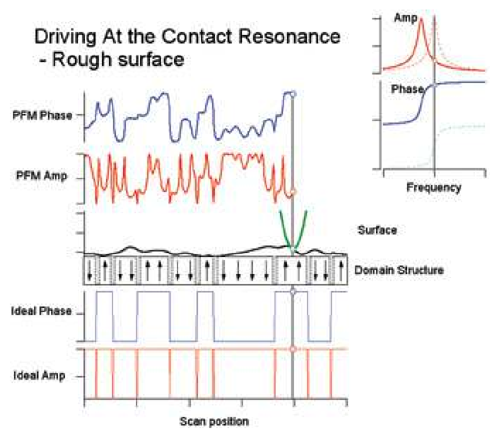

Figure 6.16.: This figure shows a practical limitation of using the contact resonance as the drive frequency. In conventional PFM systems, the contact resonance can change by 10-30 kHz over the course of imaging a rough sample. Typical cantilevers have a full-width half max of 4-10 kHz meaning the phase shift due to the changing contact resonances will easily be near 180° over the scan. The PFM phase shift will be added to the phase of the cantilever contact resonance, yielding a convolution that makes practical interpretation of domain structures very difficult. This is clear in comparing the PFM phase signal to the sample domain structure. In contrast to the off-resonance smooth sample, it is quite difficult to correlate the domain structure with the PFM phase.

Typical Q factors in air for PFM cantilevers range from 10-100. This implies that one can amplify a weak PFM signal by a factor of 10-100 by simply driving the tip voltage at the contact resonant frequency, as shown in figure 6.15.

Figure 6.13 on page 65 shows a representative cantilever in contact with a surface. The potential of the cantilever is being oscillated, which in turn induces a piezoresponse in the sample surface

($A_{tip-samp}$, $\varphi_{tip-samp}$). The cantilever in contact with the surface has a resonance defined by the mechanical properties of the cantilever and the stiffness of the tip-sample contact. This resonance can have a high quality factor (Q) for typical PFM samples that effectively amplifies the piezo signal by a factor of $\sim Q$ near the resonance. For samples with small piezo coefficients, this is potentially a very important effect and could mean the difference between only noise or a measurable signal. Unfortunately, because the cantilever resonance frequency depends on the tip-sample contact stiffness, the resonance frequency is very unstable. As the tip scans over the sample topography, the stiffness of the mechanical contact ($k_{tip-samp}$) will typically change significantly, which in turn affects the resonance frequency.

To understand how resonance is affected in PFM, we first describe an "ideal" situation, as illustrated in Figure 6.14 on page 65. This shows a numerical simulation of the cantilever response using realistic cantilever parameters (Olympus AC240 cantilever with a 320 kHz contact resonant frequency, 2 N/m spring constant) and sample parameters (d_{33}≈100 pm/V). The noise visible in the PFM amplitude and phase curves were calculated to be the ideal thermal (Brownian motion) noise of a cantilever at typical room temperature (300K). Here, the domain structure is shown in the middle of the image with purely vertical polarization vectors. The sample is treated as perfectly smooth, meaning that the contact stiffness remains constant as a function of position. The simulation reproduces many of the features present in a real scan where the measured phase reproduces a map of the domain structure, and the amplitude goes to zero at the domain boundaries. This occurs as the tip is being driven by two oppositely oriented domains, each canceling the other as they are 180° out of phase. However, these experimental conditions are very rare.

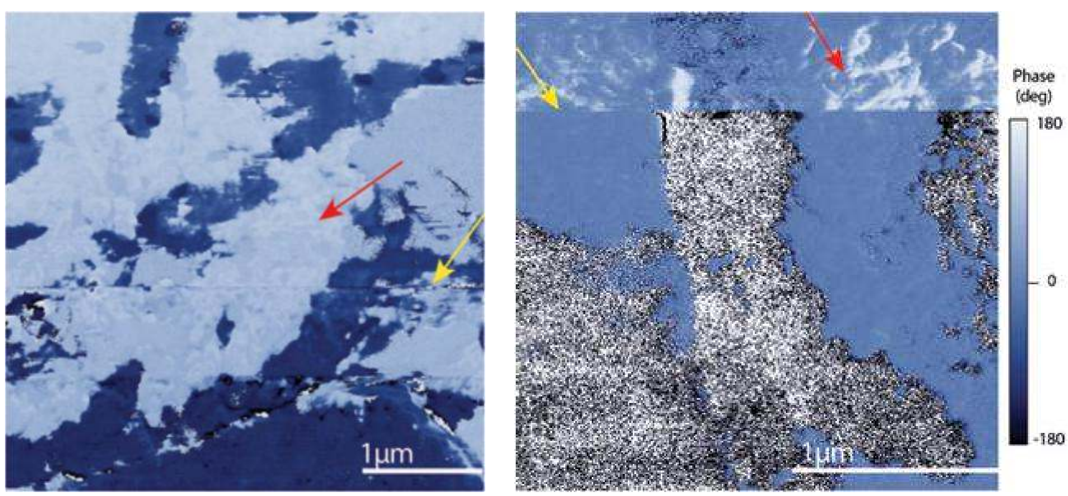

Figure 6.17.: PFM phase channel on a polished PZT sample. The cantilever was driven near the contact resonance to enhance the SNR. There is significant crosstalk between the sample topography and the PFM signal. Red arrows indicate "roughness" where the contact stiffness changes the measured phase. The yellow arrows indicate a sudden tip change causing a change in the contact resonance, consequently causing significant phase shifts. 4 µm scan (left image), 2 µm scan (right image).

Usually, the sample will have some roughness, which will lead to position-dependent changes in the contact resonant frequency. The effects of this resonant frequency variation on PFM contrast can easily completely mask the desired PFM signal. Figure 6.19 on page 68, Figure 6.17 on page 67,

and Figure 6.18 on page 68 illustrate this.

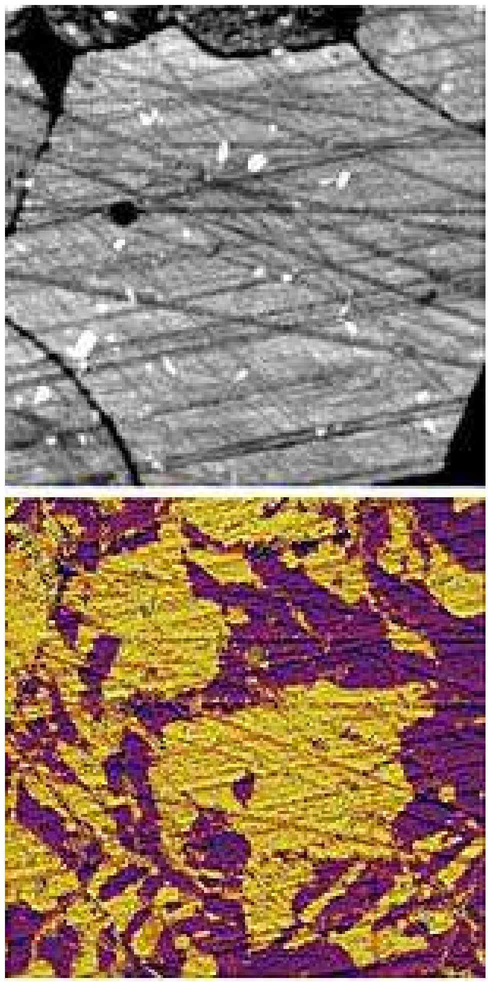

Figure 6.18.: PZT showing crosstalk, 14 µm scan.

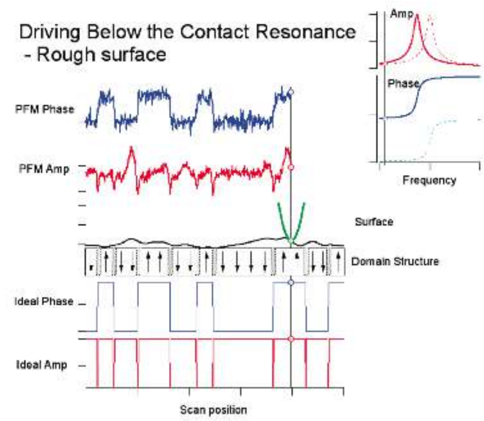

Figure 6.19.: Driving below contact resonance with conventional PFM. Here, the cantilever is driven well below the contact resonant frequency. The effects of surface roughness are minimized, though still visible in the measured PFM amplitude. However, this reduction in crosstalk comes at the high price of severely reduced sensitivity. Thus, for weak piezo materials, this operational mode is undesirable. The improved topographic crosstalk rejection results in an immeasurably small signal with conventional PFM.

If we return to our idealized sample and add roughness to the surface, we can see that it modulates the contact resonance. For example, if the tip is on a tall part of the sample, it is in contact with a relatively compliant part of the sample. Sharp points are, after all, relatively easy to blunt. Because the contact stiffness is small, the contact resonance frequency will drop. If the cantilever is being driven at a fixed frequency, the phase will increase as the resonance moves to lower values. Conversely, if the tip is in a valley, the contact stiffness will be increased, raising the resonant frequency, and the phase measured at a fixed frequency will drop. Phase shifts associated with changes in the contact resonance sum with phase shifts due to domain structures of the piezo material. As a consequence, interpretation of the domain structure becomes much more difficult, and in many cases, impossible. Figure 6.16 on page 66 shows a case where the domains are completely masked by the large phase shifts originating with the moving contact resonance.

Another source of phase shifts can come from irreversible changes to the cantilever itself. PFM is a Contact mode technique and can therefore exert large forces on the tip. If the tip fractures or picks up a contaminant, the contact resonance can experience a sudden jump, usually positive, as tip wear tends to blunt the tip. The resonance jumps are typically of the order of a few kHz. This causes large, discontinuous changes in the measured phase. Figure 6.17 on page 67 and Figure 6.18 on page 68 show PFM data taken on a rough PZT surface. A number of successive tip changes caused the contact resonance to change, resulting in an irreversible change in the overall measured phase. Note that, in addition to these jumps, there is significant "roughness" in the phase signals, probably originating with topographic contact resonance crosstalk.

By avoiding the resonance, the topographic crosstalk on rough samples can be reduced, as shown in Figure 6.19 on page 68. When the cantilever is driven well below resonance, the domain structure is reproduced quite accurately. However, this comes at the high price of a poor SNR. In practice, the reduced SNR (see the PFM phase trace) may obviate imaging of a large number of weak piezo materials with conventional PFM.

In the following sections we discuss new solutions for improving PFM signals.

6.5. Solutions to Limits of Conventional PFM

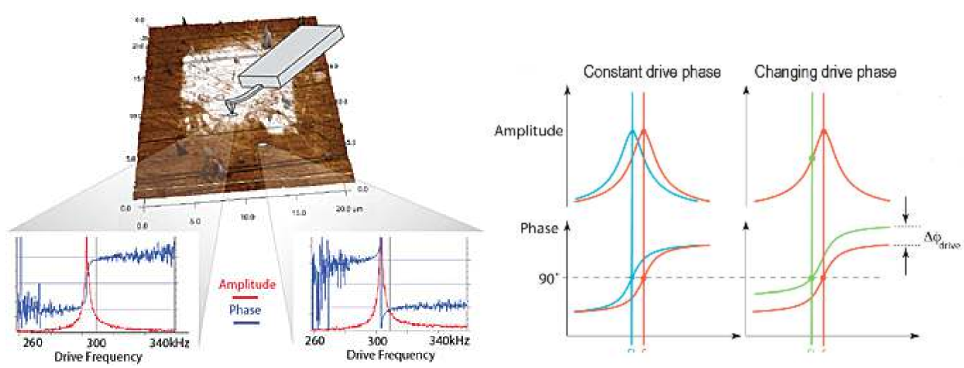

Figure 6.20.: For domains with an antiparallel (180°) orientation, conventional PLLs drive the PFM frequency away from resonance. (Top) Amplitude, red, and phase, blue, cantilever response over antiparallel domains. In the measurement, phase is offset by 180° over anti-parallel domains (see curves on the right). (Bottom) PFM phase signal driving the cantilever off resonance. Note the increased noise in the phase signal away from the resonant frequency. This increased noise would be apparent in an image as well, similar to the PZT image in Figure 6.17 on page 67 and Figure 6.18 on page 68. (Reprinted with permission[22].)

6.5.1. Increasing the Drive Voltage

Perhaps the most obvious option for improving the response of PFM is to simply increase the drive amplitude. The signal is usually proportional to the drive voltage, so increasing the drive voltage by 10x will result in a 10x improvement in the SNR. A more powerful drive amplifier also enables operation at higher frequencies (see section 6.6).

6.5.2. Using Contact Resonance as a PFM Amplifier

Sometimes increasing the SNR by simply increasing the drive voltage is not an option. In some ferroelectric samples, the polarization might be reversed by too large a PFM drive voltage. On others, the sample might actually breakdown, leading to large current flow, sample damage or even destruction. Another effective way to increase the SNR in PFM imaging and other measurements is to make use of the contact resonance. Resonance enhances the signal by the natural gain of the cantilever – by roughly the factor Q of the cantilever.

As noted in 6.4, driving near the contact resonance at a fixed frequency can sometimes lead to enormous topographic cross-coupling. To avoid this, and to maintain the advantages of resonance, you must continually adjust the drive frequency to keep it at the contact resonance. If you can remain on resonance despite changes in the contact resonance frequency, then the artifacts present in the above examples would not be present, while still reaping the resonance amplification.

The most common kind of resonance-tracking feedback loop is called a Phase-Locked Loop (PLL). It utilizes the phase-sensitive signal of a lock-in amplifier to maintain the system at a specific phase value, typically 90°. The PLL is generally limited to techniques where the phase and amplitude of the driving force is constant (e.g., the mechanical excitation of a cantilever resonance using an external actuator). This is manifestly not the case in PFM, where the relationship between the phase of the excitation force and driving voltage strongly depends on material properties[23,24]. The amplitude and phase of the local response are a convolution of material response to the external field and cantilever response to the material-dependent local force, which cannot be separated unambiguously. Figure 6.20 on page 69 is an example where, for antiparallel domains, a conventional PLL will actually drive a PFM away from resonance.

[23] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007, Nr. 47, ISSN 0957–4484.

[24] Kalinin, Sergei/Jesse, Stephen/Proksch, Roger, Information acquisition & processing in scanning probe microscopy. R&D MAGAZINE, 50 AUG 2008, Nr. 4, ISSN 0746–9179.

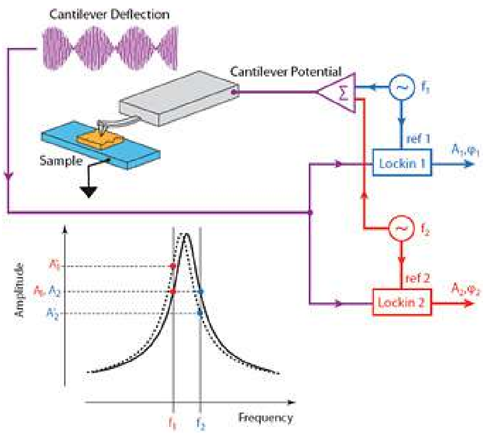

Figure 6.21.: Schematic diagram of DART, showing a drive phase independent feedback signal

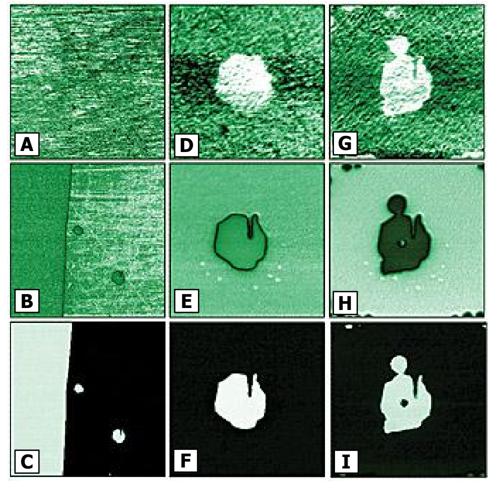

Figure 6.22.: (A), (D), (G) Resonance frequency, (B), (E), (H) piezoresponse amplitude, and (C), (F), (I) piezoresponse phase images of antiparallel domains in lithium niobate. Shown are images of the (A)–(C) native domain structure, (D)–(F) an intrinsic domain and (G)–(I) domains switched by ±176V (locations marked in (E)). The images are obtained at wf = 4kHz and Vac = 66V. The frequency images have been flattened to account for minute changes of contact radius from line to line.

Figures 6.21 and 6.22 reprinted with permission[25].

6.5.3. Dual AC Resonance Tracking (DART)

This patent pending dual-excitation method allows the cantilever to be operated at or near resonance for techniques where conventional PLLs are not stable. Figure 6.21 on page 71 shows how DART works. The potential of the conductive cantilever is the sum of two oscillating voltages with frequencies at or near the same resonance. The resulting cantilever deflection is digitized and then sent to two separate lock-in amplifiers, each referenced to one of the drive signals. By measuring the amplitudes at these two frequencies, it is possible to measure changes in the resonance behavior and, furthermore, to track the resonant frequency. Specifically, by driving at one frequency below resonance (A1), and another above (A2), A2-A1 gives an error signal that the ARC2™ controller uses to track the resonance frequency changes[26].

DART-PFM studies of polarization switching are illustrated in Figure 6.22 on page 71, where the resonant frequency (A), amplitude (B), and phase (C) images of a lithium niobate surface are shown

[25] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007, Nr. 47, ISSN 0957–4484.

[26] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007, Nr. 47, ISSN 0957–4484.

Figure 6.22A. The PFM amplitude and phase images show a macroscopic 180° domain wall and two inversion domains that are typical for this material. Higher resolution DART-PFM images of pre-existing domains (D-F) illustrate strong frequency contrast, and nearly constant PFM amplitudes within and outside the domain. In comparison, Figures 6.22 (G-I) are DART-PFM images of domains switched by the application of three 176V magnitude pulses for ~10 seconds in three adjacent locations. Note the significant change of resonant frequency and the strong amplitude depression in the newly fabricated domain[27].

Additional DART images of ferroelectric materials are shown in Figure 6.23 on page 73 and Figure 6.24 on page 73. Figure 6.23 on page 73 shows a series of images of PFM on multiferroic $BiFeO_3$ nanofibers. Figure 6.24 on page 73 shows a short relaxation study on a sol-gel sample. Regions of the sol-gel PZT were reversed by applying a 15V bias to the tip. These regions gradually relaxed over a 1.5-hour period. DART allowed stable, reproducible imaging over an extended period of time.

6.5.4. Band Excitation (BE)

Band Excitation (BE) is an option that can be utilized with PFM. The technology is exclusively available with Asylum Research SPMs under license from Oak Ridge National Laboratory[28] and has received the R&D 100 award for 2008. The BE controller and software extend the capabilities of Asylum's AFMs to probe local amplitude vs. frequency curves and transfer functions and map local energy dissipation on the nanoscale.

The applicability of SPM for mapping energy transformations and dissipation has previously been limited by the fundamental operation mechanism employed in nearly all conventional SPMs, i.e., the response was measured at a single frequency. Determining dissipation with a single frequency measurement required time-consuming multiple measurements. Simply put, there were more uncertainties than there were measured quantities (see Equations 6.4 and 6.5)[29]. BE surmounts this difficulty by detecting responses at all frequencies simultaneously. BE introduces a synthesized digital signal that spans a continuous band of frequencies and monitors the response within the same frequency band. This allows ~100x improvement in data acquisition speed compared to other commercially available technologies.

The immediate benefit of this approach is that a full response spectrum can be collected (with insignificant [30-50%] decrease in signal to noise ratio) in the amount of time required for obtaining a single pixel in conventional single-frequency SPM. BE allows quantitative mapping of local energy dissipation in materials on the nanoscale[31]. Figure 6.26 on page 75 shows an example image of an amyloid fibril (bovine insulin) on mica imaged in water using the BE-PFM technique. The image size 250nm x 250nm.

In summary, both DART and BE modes have numerous advantages for PFM measurements, including:

[27] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007, Nr. 47, ISSN 0957–4484.

[28] Jesse, Stephen et al., The band excitation method in scanning probe microscopy for rapid mapping of energy dissipation on the nanoscale. NANOTECHNOLOGY 18 OCT 31 2007, Nr. 43, ISSN 0957–4484.

[29] Jesse, Stephen et al., The band excitation method in scanning probe microscopy for rapid mapping of energy dissipation on the nanoscale. NANOTECHNOLOGY 18 OCT 31 2007, Nr. 43, ISSN 0957–4484.

[31] Jesse, Stephen et al., The band excitation method in scanning probe microscopy for rapid mapping of energy dissipation on the nanoscale. NANOTECHNOLOGY 18 OCT 31 2007, Nr. 43, ISSN 0957–4484.

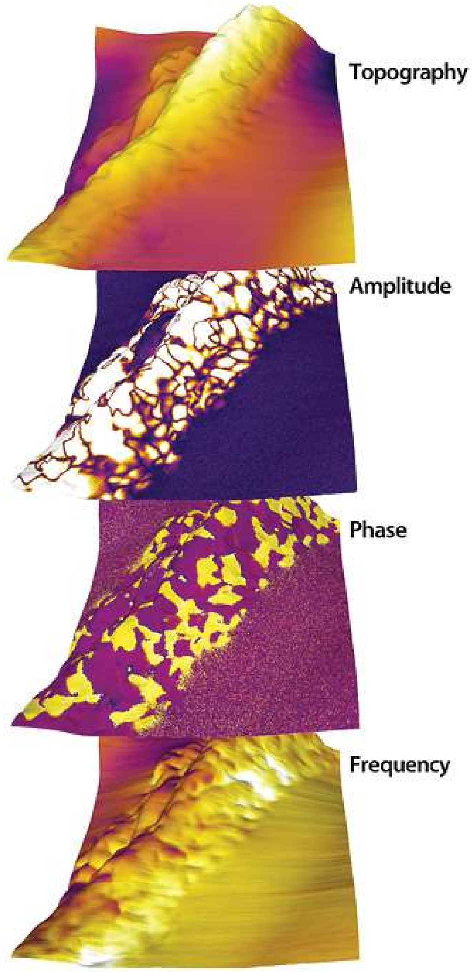

Figure 6.23.: PFM of multiferroic $BiFeO_3$ nanofibers, 1 µm scan. (Collaboration with Shuhong Xie, Xiangtan University, China and JiangYu Li, University of Washington.)

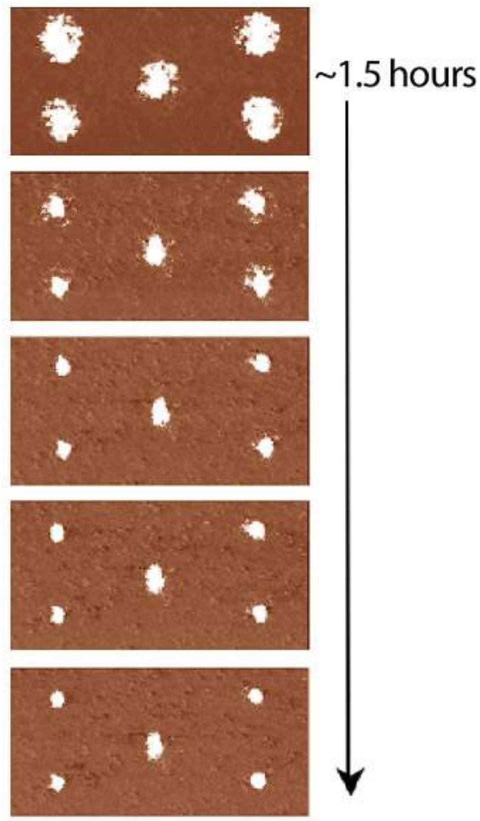

Figure 6.24.: Stable imaging using DART allows relaxation studies. This series of images shows the relaxation of sol-gel taken at different intervals for approximately 1.5 hours. 3.5 µm scan.

- SNR is increased by a factor of 100, eliminating crosstalk issues by using, rather than avoiding, resonance.
- Problems with PLL stability are eliminated.
- For BE, data acquisition is improved by ~100x compared to other commercially available swept frequency technologies.
- Imaging modes and hardware are fully integrated.

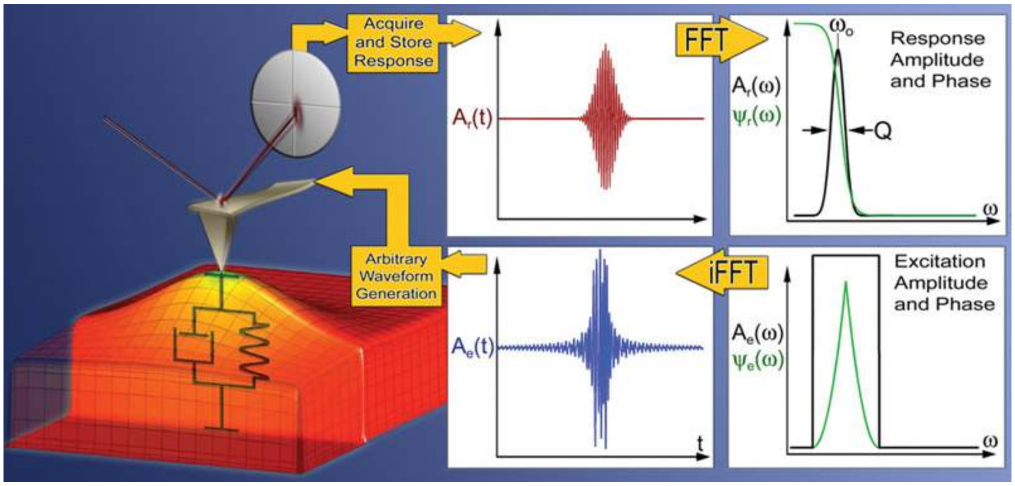

Figure 6.25.: Operational principle of the BE method in SPM. The excitation signal is digitally synthesized to have a predefined amplitude and phase in the given frequency window. The cantilever response is detected, and Fourier transformed at each pixel in an image. The ratio of the fast Fourier transform (FFT) of response and excitation signals yields the cantilever response (transfer function). Fitting the response to the simple harmonic oscillator yields amplitude, resonance frequency, and Q-factor, which are plotted to yield 2D images or used as feedback signals. (Reprinted with permission[30].)

6.6. Emerging Applications for PFM

6.6.1. High Frequency PFM

High-frequency imaging allows for an improved SNR by avoiding 1/f noise. Furthermore, inertial stiffening of the cantilever improves contact conditions. By probing the PFM signal with higher resonances, topographic imaging is performed with a soft cantilever, while PFM is performed with a higher mode where the dynamic stiffness is much greater. This both reduces the electrostatic contribution to the signal and improves the tip-surface electrical contact through effective penetration of the contamination layer. Finally, resonance enhancement using the higher mode amplifies weak PFM signals. It should be noted that in this regime, the response is strongly dependent on the local mechanical contact conditions, and hence, an appropriate frequency tracking method is required to avoid PFM/topography crosstalk, e.g., using DART or BE as described above.

The limiting factors for high-frequency PFM include inertial cantilever stiffening, laser spot effects, and the photodiode bandwidth. Inertial stiffening is expected to become a problem for resonances n>4-5, independent of cantilever parameters. This consideration suggests that the use of high-frequency detector electronics, shorter levers with high resonance frequencies, and improved laser focusing will allow the extension of high-frequency PFM imaging to the 10-100 MHz range. Asylum's microscopes allow cut-off at ~2-8MHz and potentially higher, opening a pathway for high frequency studies of polarization dynamics. Figure 6.27 on page 76 illustrates the different information that is revealed by imaging a ceramic PZT material at various frequencies.

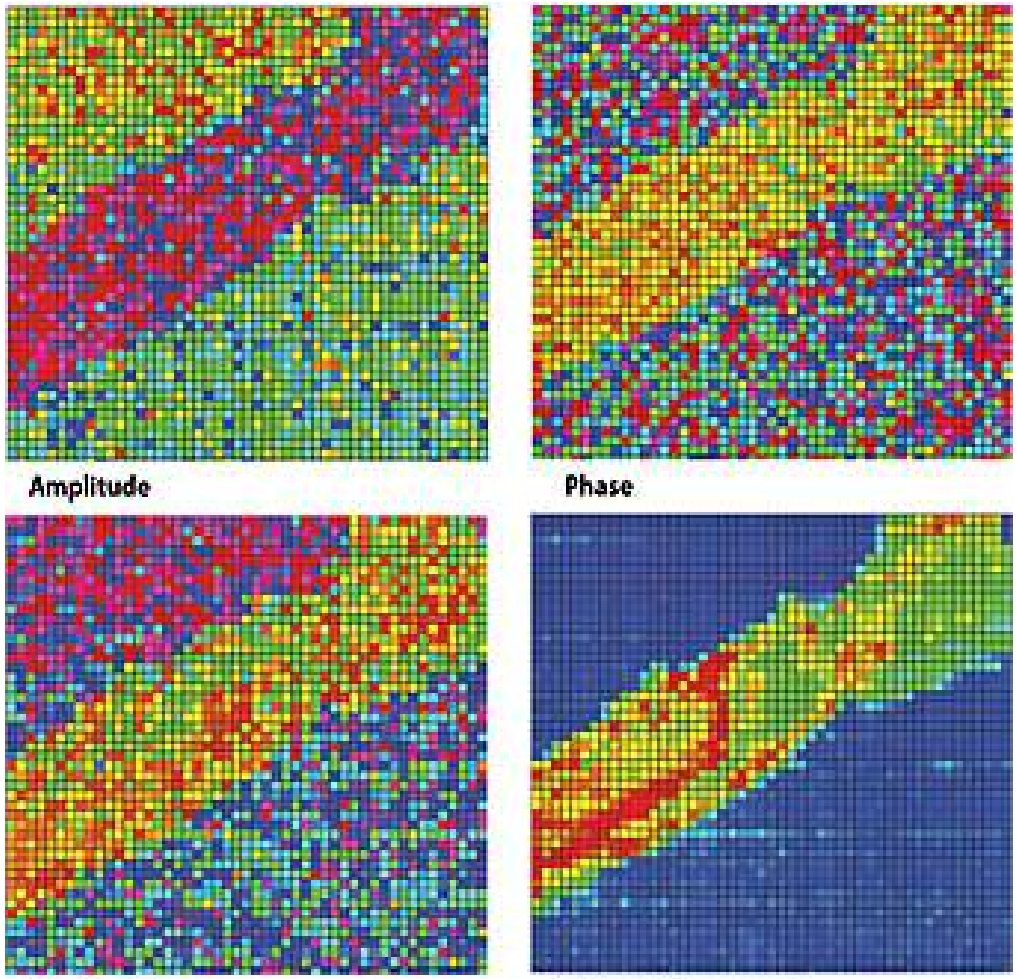

Figure 6.26.: Amyloid fibril (bovine insulin) on mica imaged in water using BE-PFM technique, 250nm x 250nm. (Image courtesy of G. L. Thompson, V. V. Reukov, A. A. Vertegel, M. P. Nikiforov, Clemson University, Dept. Bioengineering, and S. Jesse, S. V. Kalinin, Oak Ridge National Lab.)

6.6.2. High-Speed PFM (HSPFM)

High-Speed PFM (HSPFM) utilizes high speed data acquisition and sample actuation to significantly enhance imaging speeds by increasing line rates from roughly 1Hz to well above 100 Hz. The strong amplitude and phase contrast achievable in PFM, as well as the resolution enhancement provided by this Contact mode-based method, have allowed 10nm spatial resolution even at image rates of up to 10 frames per second[32].

In addition to higher throughput, the primary benefit of this advance is dynamic measurements, for example tracking the evolution of ferroelectric domains during switching, exposure to light, changing temperature, and other effects Figure 6.28 on page 76 and Figure 6.29 on page 77.

The more general High Speed Scanning Property Mapping (HSSPM) allows rapid measurements

[32] Nath, Ramesh et al., High speed piezoresponse force microscopy: < 1 frame per second nanoscale imaging. Applied Physics Letters 93 AUG 18 2008, Nr. 7, ISSN 0003–6951.

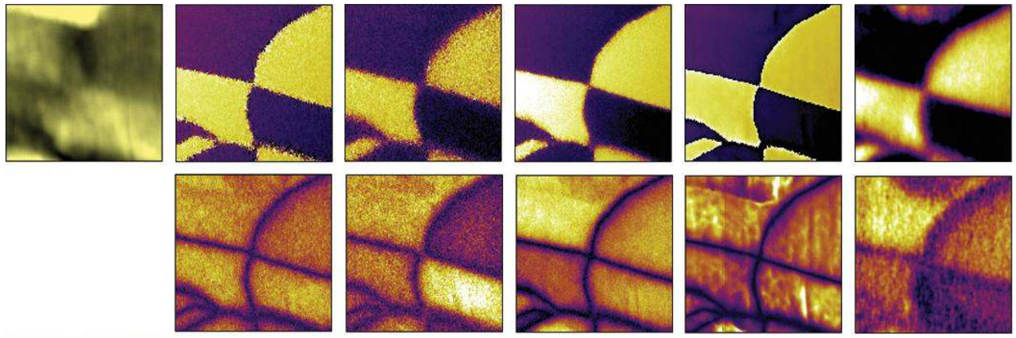

Figure 6.27.: High Frequency PFM using Asylum's fast photodiode on a ceramic PZT sample at different frequencies (phase left, amplitude right) – below first resonance (top row) and at cantilever resonances (all others) using a MikroMasch NSC 35B cantilever. 1µm scans. (Image courtesy of K. Seal, S. Kalinin, S. Jesse, and B. Rodriguez, Center for Nanophase Materials Science, ORNL.)

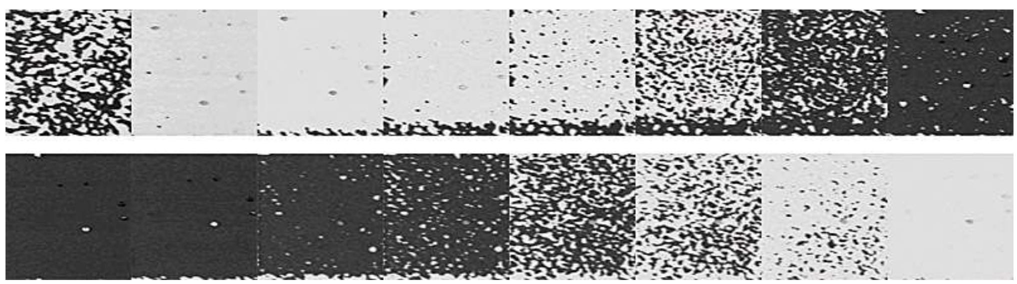

Figure 6.28.: This image sequence (left to right, top to bottom) is excerpted from a movie of 244 consecutive High Speed PFM images (4 µm scans) depicting in situ ferroelectric memory switching. For the first half of the movie, the tip is biased with a positive DC offset throughout the measurements. By monitoring the phase of the piezoresponse, this allows direct nanoscale observation of ferroelectric poling, in this case from white to black contrast (a 180° polarization reversal). The second half of the movie is then obtained with a continuous negative DC bias, causing a black to white contrast shift. The switching mechanism is clearly nucleation dominated for this sample and experimental conditions. Each image is acquired in just 6 seconds. (The PZT film is courtesy of R. Ramesh, UC Berkeley, and the HSPFM measurements were performed by N. Polomoff, HueyAFMLabs, UConn.)

of mechanical compliance, electric fields, magnetic fields, friction, etc., with similar benefits for novel dynamic measurements of surfaces[33].

6.6.3. PFM Nanoindenting

For quantitative materials properties measurements, AFMs have a few well-known shortcomings. One is that the shape of the tip is usually ill-defined. Forces between the tip and sample have a strong dependence on this tip shape; therefore, extracting materials properties, such as the Young's modulus, can be problematic. Another issue is that the cantilever geometry means that the motion of the cantilever tip is not well-defined. Specifically, when the cantilever deflects, there is motion along the vertical axis (z-axis) that is well defined, but there is also motion parallel to the sample surface. This motion is not well characterized and, in most cases, is not even measured.

[33] Huey, Bryan D., AFM and acoustics: Fast, quantitative nanomechanical mapping. Annual Review of Materials Research, 37 2007, ISSN 1531–7331.

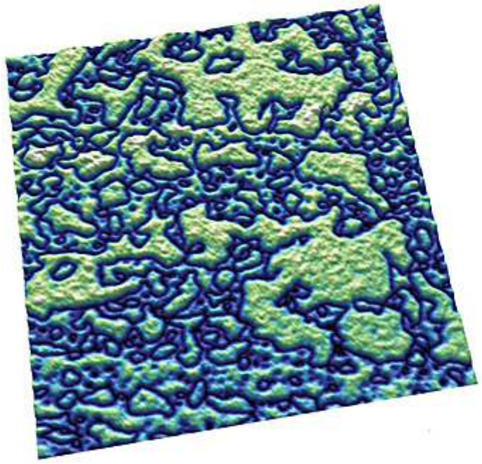

Figure 6.29.: (001) domains in a PZT thin film, 3.8µm scan. (Image courtesy N. Polomoff and B. D. Huey, University of Connecticut Institute of Materials Science. Sample courtesy R. Ramesh, UC Berkeley.)

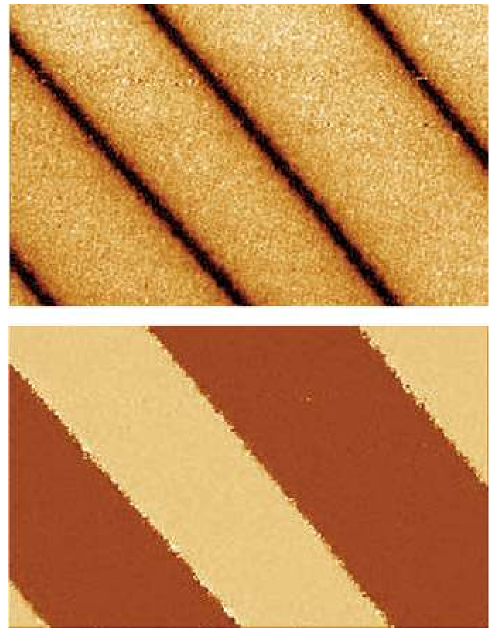

Figure 6.30.: PPLN amplitude (top) and phase image (bottom) acquired with the MFP NanoIndenter, 50µm scan.

The ability to probe forces and directly image the piezoresponse of a sample with the Asylum Research MFP NanoIndenter is an emerging application area[34]. The NanoIndenter consists of a flexure with a calibrated spring constant to which diamond tips are mounted. This flexure is attached to the NanoIndenter AFM head and replaces the standard cantilever holder. The force is computed as the product of the spring constant and the flexure displacement, measurement by an optical signal on the standard MFP-3D photodetector. Because the quantities of indentation, depth, and force are computed based on displacements measured with AFM sensors, the indenter has much better spatial and force resolution than previous systems.

Figure 6.30 on page 77 shows an example image of PPLN acquired with the NanoIndenter. Note that the topographic resolution is not as high as it would be with an AFM cantilever tip, as expected given the larger indenter tip. The amplitude and phase channels show clear, high SNR domain structure, similar to the results one would expect with cantilever-based PFM.

Another example of the experiments that can be performed with the combination of the NanoIndenter and PFM imaging is to study the effects of surface stresses on ferroelectric domain structures with quantitative scratch testing, as shown in Figure 6.31 on page 78. The top image shows the surface topography of PPLN after it has been purposefully scratched with different loading forces using the NanoIndenter tip.

The next image () shows the associated phase signal indicative of the domain structure. The domain boundaries have been distorted by the scratches which implies a lattice change which, in turn, has affected the local polarizability. The final figure in this sequence shows a higher resolution scan where the phase has been overlaid onto the rendered topography, showing a close-up of the

[34] Rar, A et al., Piezoelectric nanoindentation. Journal of Materials Research, 21 MAR 2006, Nr. 3, ISSN 0884–2914.

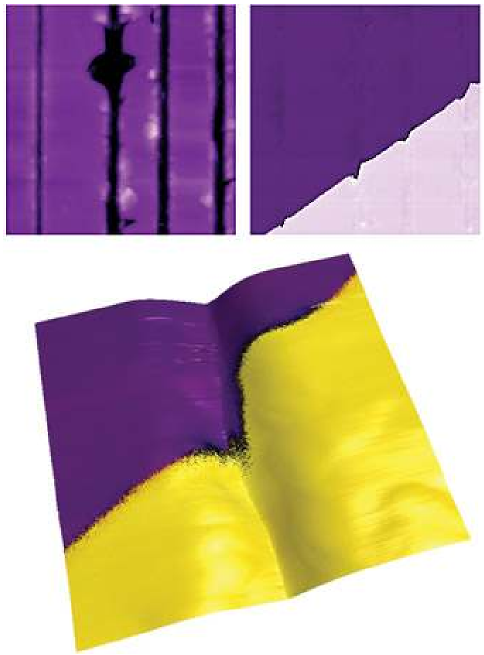

Figure 6.31.: Surface topography of PPLN after it has been purposefully scratched with different loading forces using the NanoIndenter, 10µm scan (top images). 1µm scan (bottom).

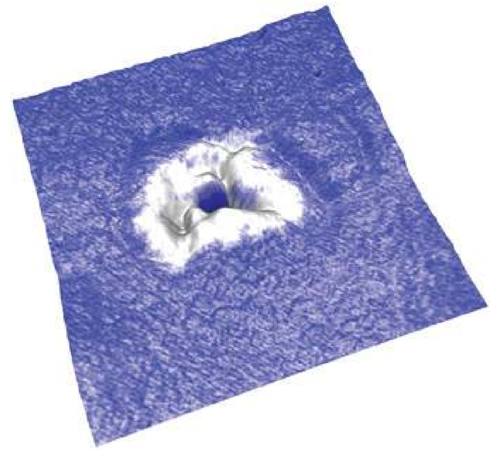

Figure 6.32.: Zoom of the top surface of a red blood cell. The surface shape was rendered to show the topography while the phase channel is overlaid on top to show piezoresponse. A small sub-micron region on top (white) of the cell exhibited a much different piezoresponse than the surrounding cell surface. 2µm scan. (Image courtesy of B. Rodriguez and S. Kalinin, ORNL.)

distortion in the domain structure.

6.6.4. Biological Applications

PFM allows organic and mineral components of biological systems to be differentiated and provides information on materials microstructure and local properties. The use of vector PFM may also enable protein orientation to be determined in real space, for example, the internal structure and orientation of protein microfibrils with a spatial resolution of several nanometers in human tooth enamel. Additional progress will bring understanding of electromechanical coupling at the nanometer level, establish the role of surface defects on polarization switching (Landauer paradox), and probe nanoscale polarization dynamics in phase-ordered materials and unusual polarization states. In biosystems, PFM can also potentially open pathways for studies of electrophysiology at the cellular and molecular levels, for example, signal propagation in neurons. Ultimately, on the molecular level, PFM may allow reactions and energy transformation pathways to be understood and become an enabling component to understanding molecular electromechanical machines. Recently, PFM performed on biomolecules has demonstrated electromechanical behavior in lysozyme

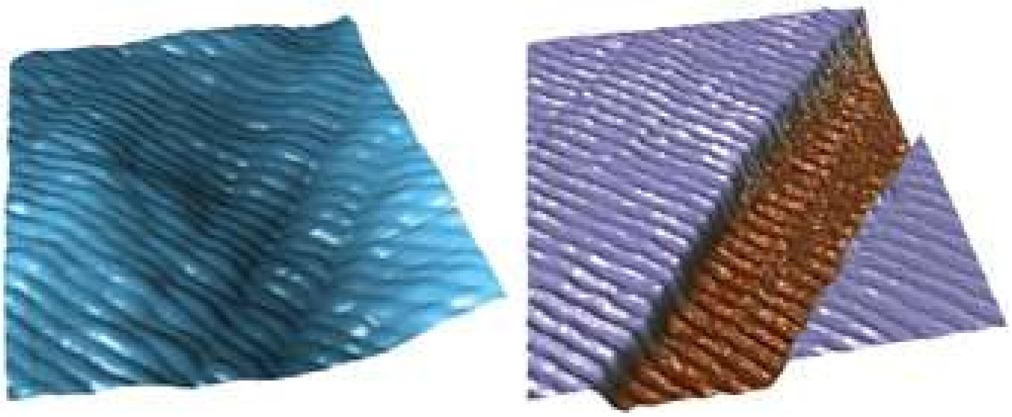

Figure 6.33.: Topographic (top) and PFM phase (bottom) images of collagen fibers, 1.4µm scan. (Image courtesy D. Wu and A. Gruverman, UNL. Sample courtesy G. Fantner.)

polymers, bacteriorhodopsin, and connective tissue[35,36]. 6.33shows an example of vertical PFM height and phase images of collagen fibers. PFM has also recently been performed on biological systems such as cells as shown in Figure 6.32 on page 78[37]. This image shows a zoom of a red blood cell with the PFM phase channel painted on top to show piezoresponse.

6.7. Additional Reading

6.7.1. Scientific Articles of Interest

Although not cited in the application note text, these references may be used for additional reading of the background, theory, and applications of PFM.

- *Theory of indentation of piezoelectric materials*[38]
- *Indentation of a transversely isotropic piezoelectric half-space by a rigid sphere*[39]
- *Point force and point electric charge in infinite and semi-infinite transversely isotropic piezoelectric solids*[40]
- *Nanoelectromechanics of piezoresponse force microscopy*[41]

[35] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007, Nr. 47, ISSN 0957–4484.

[36] Kalinin, Sergei V. et al., Towards local electromechanical probing of cellular and biomolecular systems in a liquid environment. NANOTECHNOLOGY, 18 OCT 24 2007, Nr. 42, ISSN 0957–4484.

[37] Rodriguez, B J et al., Nanoelectromechanics of Inorganic and Biological Systems: From Structural Imaging to Local Functionalities. Microscopy, 16 January 2008, Nr. 1.

[38] Giannakopoulos, AE/Suresh, S, Theory of indentation of piezoelectric materials. Acta Materialia, 47 MAY 28 1999, Nr. 7, ISSN 1359–6454.

[39] Chen, WQ/Ding, HJ, Indentation of a transversely isotropic piezoelectric half-space by a rigid sphere. ACTA MECHANICA SOLIDA SINICA, 12 JUN 1999, Nr. 2, ISSN 0894–9166.

[40] Karapetian, E/Sevostianov, I/Kachanov, M, Point force and point electric charge in infinite and semi-infinite transversely isotropic piezoelectric solids. PHILOSOPHICAL MAGAZINE B-PHYSICS OF CONDENSED MATTER STATISTICAL MECHANICS ELECTRONIC OPTICAL AND MAGNETIC PROPERTIES, 80 MAR 2000, Nr. 3, ISSN 0141–8637.

[41] Kalinin, SV/Karapetian, E/Kachanov, M, Nanoelectromechanics of piezoresponse force microscopy. PHYSICAL REVIEW B 70 NOV 2004, Nr. 18, ISSN 1098–0121.

- *Nanoelectromechanics of piezoelectric indentation and applications to scanning probe microscopies of ferroelectric materials*[42]
- *Modeling and measurement of surface displacements in BaTiO3 bulk material in piezoresponse force microscopy*[43]
- *Nanoscale piezoelectric response across a single antiparallel ferroelectric domain wall*[44]
- *Materials contrast in piezoresponse force microscopy*[45]
- *Electromechanical detection in scanning probe microscopy: Tip models and materials contrast*[46]
- *Local probing of ionic diffusion by electrochemical strain microscopy: Spatial resolution and signal formation mechanisms*[47]
- *Spatial resolution, information limit, and contrast transfer in piezoresponse force microscopy*[48]
- *Nanoscale phenomena in ferroelectric thin films*[49]
- *Encyclopedia of Nanoscience and Nanotechnology*[50]
- *Nanocrystalline multiferroic BiFeO3 ultrafine fibers by sol-gel based electrospinning*[51]
- *Local bias-induced phase transitions*[52]

6.7.2. Comprehensive Material

These references provide key papers and comprehensive reviews on PFM:

- *Scanning probe microscopy : electrical and electromechanical phenomena at the nanoscale*[53]
- *Nanoscale characterisation of ferroelectric materials : scanning probe microscopy approach*[54]

[42] Karapetian, E/Kachanov, M/Kalinin, SV, Nanoelectromechanics of piezoelectric indentation and applications to scanning probe microscopies of ferroelectric materials. Philosophical Magazine, 85 APR 1 2005, Nr. 10, ISSN 1478–6435.

[43] Felten, F et al., Modeling and measurement of surface displacements in BaTiO3 bulk material in piezoresponse force microscopy. Journal of Applied Physics, 96 JUL 1 2004, Nr. 1, ISSN 0021–8979.

[44] Scrymgeour, DA/Gopalan, V, Nanoscale piezoelectric response across a single antiparallel ferroelectric domain wall. PHYSICAL REVIEW B 72 JUL 2005, Nr. 2, ISSN 1098–0121.

[45] Kalinin, Sergei V./Eliseev, Eugene A./Morozovska, Anna N., Materials contrast in piezoresponse force microscopy. Applied Physics Letters 88 JUN 5 2006, Nr. 23, ISSN 0003–6951.

[46] Eliseev, Eugene A. et al., Electromechanical detection in scanning probe microscopy: Tip models and materials contrast. Journal of Applied Physics 102 JUL 1 2007, Nr. 1, ISSN 0021–8979.

[47] Morozovska, A. N. et al., Local probing of ionic diffusion by electrochemical strain microscopy: Spatial resolution and signal formation mechanisms. Journal of Applied Physics, 108 2010.

[48] Kalinin, S. V. et al., Spatial resolution, information limit, and contrast transfer in piezoresponse force microscopy. NANOTECHNOLOGY, 17 JUL 28 2006, Nr. 14, ISSN 0957–4484.

[49] Hong, Seungbum, editor, *Nanoscale phenomena in ferroelectric thin films*. Kluwer Academic Publishers, Boston, 2004.

[50] Gruverman, A; Nalwa, H S, editor, Chap. Ferroelectric Nanodomains In Encyclopedia of Nanoscience and Nanotechnology. Volume 3, American Scientific Publishers, Los Angeles, 2004.

[51] Xie, S. H. et al., Nanocrystalline multiferroic BiFeO3 ultrafine fibers by sol-gel based electrospinning. Applied Physics Letters 93 DEC 1 2008, Nr. 22, ISSN 0003–6951.

[52] Kalinin, Sergei V. et al., Local bias-induced phase transitions. MATERIALS TODAY, 11 NOV 2008, Nr. 11, ISSN 1369–7021.

[53] Kalinin, S/Gruverman, A, editors, *Scanning probe microscopy : electrical and electromechanical phenomena at the nanoscale*. Springer, New York, 2007.

[54] Alexe, M/Gruverman, A, editors, *Nanoscale characterisation of ferroelectric materials : scanning probe microscopy approach*. Springer, N, 2004.

- *Imaging and control of domain structures in ferroelectric thin films via scanning force microscopy*[55]

6.8. Glossary

Band Excitation A scanning technique whereby the cantilever is excited, and the response is recorded over a band of frequencies simultaneously, rather than at a single frequency as in conventional SPM. This allows very rapid data acquisition and enables the direct measurement of energy dissipation through the determination of the Q-factor of the cantilever.

Electromechanical Coupling The mechanical response to an applied electrical stimulus and the electrical response to an applied mechanical stimulus.

Domain Nucleation The event of polarization reversal when an oppositely polarized domain is formed in a ferroelectric material.

Dual AC Resonance Tracking (DART) A scanning technique used in PFM that allows dual excitation of the cantilever to independently measure both the amplitude and resonance frequency of the cantilever, improving spatial resolution and sensitivity. Overcomes limitations of traditional Phase-Locked Loops used in conventional SPM.

Ferroelectric Polarization A spontaneous dipole moment existing due to the distortion of a crystal lattice that can be switched between two or more stable states by the application of electrical or mechanical stress.

Landauer Paradox The electric fields required to induce polarization reversal correspond to unrealistically high values for the activation energy for domain nucleation.

Lateral PFM A PFM technique where the in-plane component of polarization is detected as lateral motion of the cantilever due to bias-induced surface shearing.

Nucleation The onset of a phase transition or chemical reaction in which a nanoscale region of a new phase forms, e.g., a bubble during boiling of a liquid or a crystal from a liquid.

Phase-Locked Loop (PLL) In AFM imaging, the PLL measures the phase lag between excitation and response signals as the error signal for a feedback loop that maintains the cantilever phase at a constant value (typically 90°) at resonance by adjusting the frequency of the excitation signal to maintain precise control of tip-surface interactions.

Piezoresponse Force Microscopy (PFM) Scanning probe technique based on the detection of the electromechanical response of a material to an applied electrical bias.

Piezoelectric Surface A 3D plot depicting the piezoresponse as a function of the angle between the direction of the applied field and the measurement axis.

Q-factor Typically referred to as the “Q-factor of the cantilever,” this is a dimensionless quantity inversely dependent on the cantilever energy dissipation. Typical values of Q range from ten to several hundred.

[55] Gruverman, A/Auciello, O/Tokumoto, H, Imaging and control of domain structures in ferroelectric thin films via scanning force microscopy. Annual Review of Materials Science, 28 1998, ISSN 0084–6600.

Resonant Frequency Typically referred to as the "resonant frequency of the cantilever," it is the natural frequency at which the cantilever is oscillated to achieve maximum amplitude.

Switching Spectroscopy Mapping A quantitative measurement that reveals local switching characteristics for real-space imaging of imprint, coercive bias, remanent and saturation responses, and domain nucleation voltage on the nanoscale.

Vector PFM The real space reconstruction of polarization orientation from three components of piezoresponse, vertical PFM, and at least two orthogonal lateral PFM.

Vertical PFM (VPFM) Out-of-plane polarization is measured by recording the tip-deflection signal at the frequency of modulation.

7. Single Frequency Piezo Force Microscopy (PFM)

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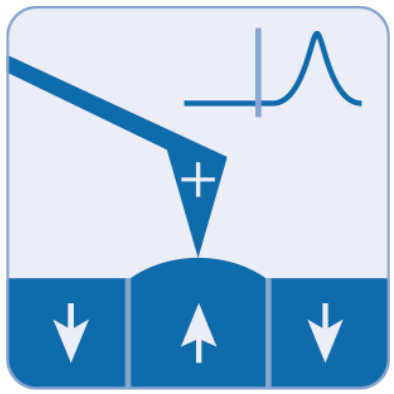


Chapter Contents

Single Frequency Piezo Force Microscopy (PFM) is used to characterize the electromechanical response of piezoelectric materials. Typically, a conductive cantilever is scanned over the sample surface in contact mode. While scanning the surface, an AC bias is applied to the tip. The electric field causes a strain in the surface which in turn causes a periodic deflection of the cantilever.

This chapter describes how to run single frequency PFM, lateral PFM, and lithography on ferroelectric materials. The deflection sensitivity producing these deflections with this technique is usually quite small, sometimes only a few picometers (pm) per volt of excitation. Noise floors of an optical lever are usually somewhere in the neighborhood of tens of pm, so measuring these samples requires either using a large AC voltage or some other amplification technique.

Large voltages can be a convenient way of boosting the small response of piezo samples. However, large voltages come with potentially problematic large electric fields and, with some samples, potentially large, damaging currents.

Here, we describe a method of using the cantilevers's contact resonance to boost small piezo signals. By selecting a Frequency close to the contact resonance, the piezo signal can be amplified. Figure 7.1 on page 84 shows a schematic of the drive Frequency and resulting Amplitude response. By using a lock-in amplifier, the Amplitude and Phase of the response can be measured.

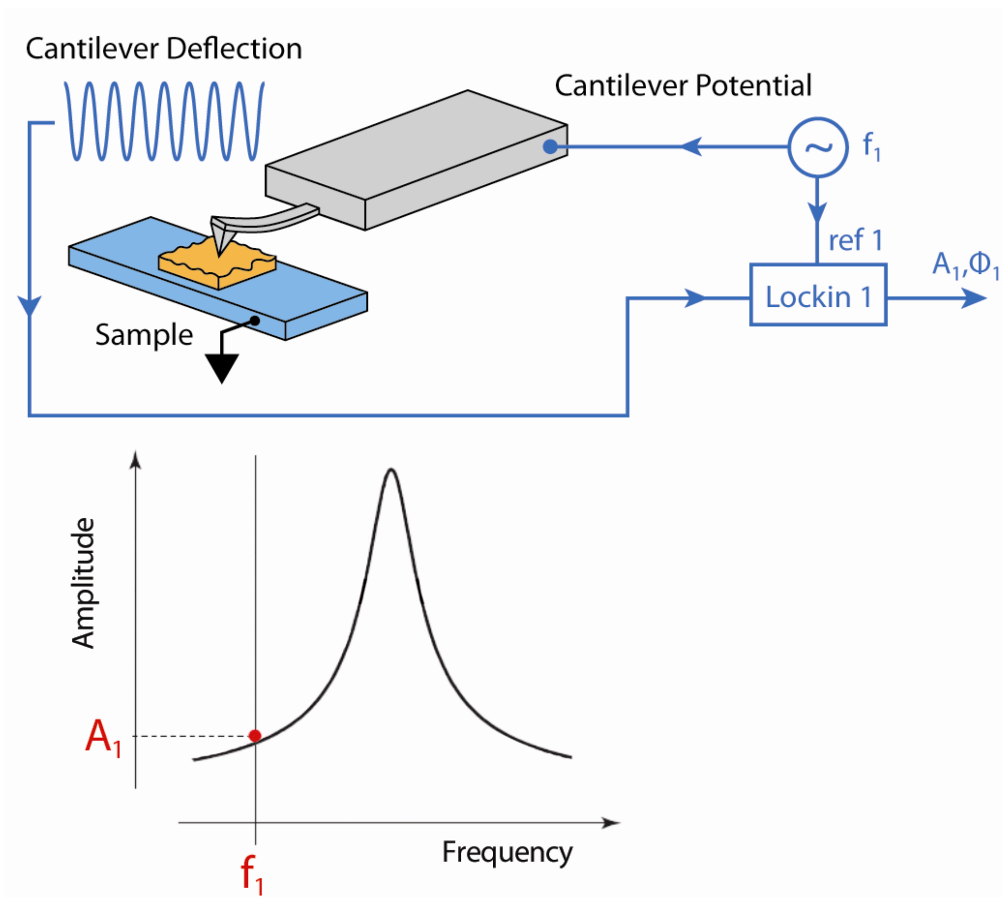


Figure 7.1.: A diagram of Single Frequency PFM

The use of contact resonance to boost small piezo signals is more complex than might be expected at first examination. The contact resonant frequency depends strongly on details of the contact mechanics–the elastic modulus, tip shape, and sample topography, which can contribute to cause the resonant frequency to vary many tens of kilohertz (kHz) while the tip scans over the surface. Because of this resonance variation, and because the phase also varies, fixed-frequency drive techniques are subject to large amounts of topographic crosstalk. For advanced users, we suggest using Dual AC Resonance Tracking (DART) as discussed in chapter 8.

7.1. Choosing a PFM Cantilever

Cantilevers for PFM generally should have a spring constant greater than 1 N/m. The cantilever should have a conductive coating or be sufficiently doped to provide an electrical contact from the spring clip to the tip. If the cantilever is doped Si, it may be necessary to scratch the chip to break through the oxide layer and then use a tiny patch of silver paint to ensure electrical contact between the spring clip and the chip.

Single Frequency PFM uses the cantilever resonance to boost the PFM signal. A good rule of thumb for most diving board shaped Si cantilevers is that the contact resonance is typically 3-5 times the free air resonant frequency. For example, if a probe is nominally 70 kHz resonance, start with a center frequency of ~300 kHz and a sweep width of a few hundred kHz. You may need to experiment, especially if you are working with a new type of cantilever.

Cantilever	Suggested Contact Resonance Range for Tuning
Olympus AC240 Electrilever	200-400 kHz
Olympus AC160	900-1,100 kHz
Nanosensors PPP-NCHR	800-1,200 kHz

Table 7.1.: Typical contact resonance frequency ranges for some common cantilevers

7.2. Single Frequency PFM

For this instruction set, we assume that you are proficient with basic AFM operation. This technique uses the standard cantilever holder for low-voltage PFM or a high voltage option for your microscope.

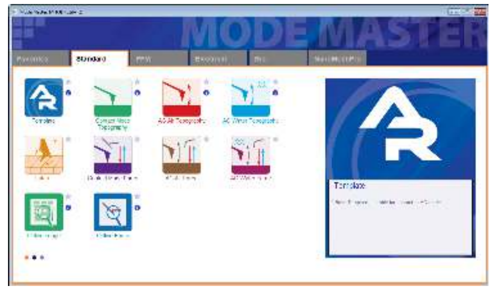
7.2.1. Sample and Cantilever

The sample used in this example is a 3mm x 3mm x 0.5 piece of Periodically Poled Lithium Niobate (PPLN). It is described in Data Sheet 32 `http://www.asylumresearch.com/Products/AR-PPLN/AR-PPLN.shtml` and is available from Asylum Research. The cantilever used here is an Olympus Electrilever, PtIr coated Si cantilever with a nominal 2N/m spring constant and a free air resonance of ~70kHz.

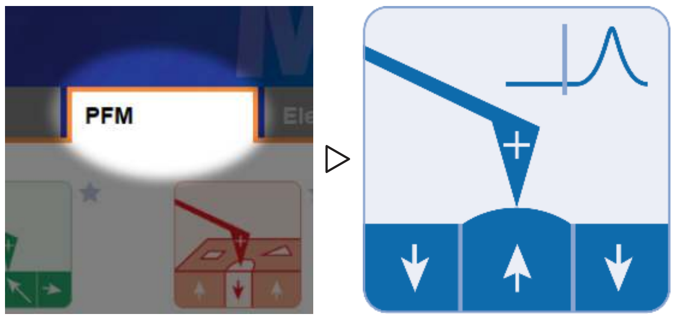
7.2.2. Preparation

1. **The Mode Master window:**

- The software should be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.



Select mode:

- Select *PFM tab > Single Freq PFM*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

3. Load a cantilever into your cantilever holder. For PFM, a good starting point is an Olympus Electrilever. There are several versions of cantilever holders. If you have an MFP3D with the HVA220 Amplifier option for PFM, make sure you use either the HV DC or AC cantilever holder. These have a small red wire attached to the spring clip terminated with a tiny gold magnet. Or, if you have a Cypher with the high voltage PFM option, there is also a specific cantilever holder to use the high voltage.

4. Maximize the sum, and then set deflection near -0.5V. We typically operate at a Set Point 0V to take advantage of the optional 10x gain on the photodetector signal (very useful when measuring the tiny motions in PFM).

5. Load your sample. For most highly insulating piezo and ferroelectric samples, grounding seems to be optional; we do not see significant differences in the response with or without the ground attached.

6.

Position the head above the sample:

- Check that the red wire is seated into the HV connector slot (glowing red on the sample holder. The magnet should suck the end of this wire into the HV connection slot when the head is positioned above the sample.
- MFP3D only: Take care to ensure that the cantilever holder and the sample clip do not interfere mechanically with each other. At the least, this can cause false engages, at worst, it could lead to dangerous and damaging arcing between the high voltage spring clip and the grounded sample clip.

7.

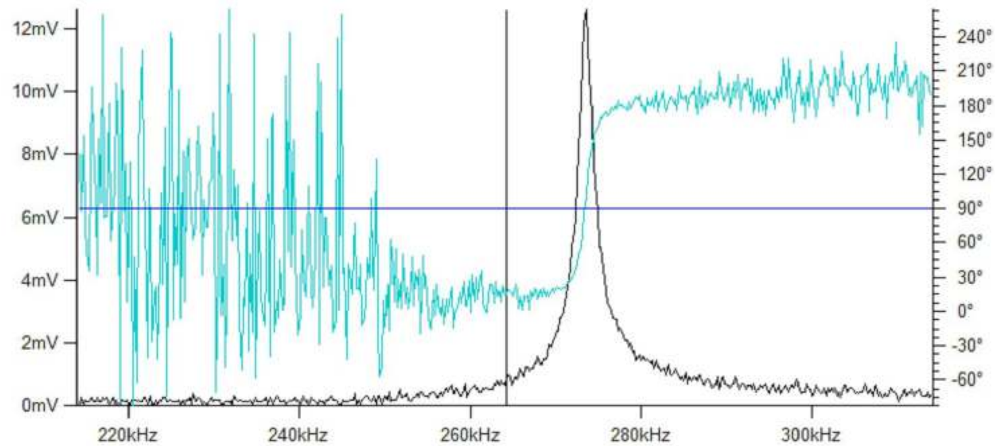
Engage the surface:

- Hit the ‘Engage’ button.
- Make sure that the z-piezo indicator moves down towards the sample.
- Lower the head with the thumbscrews. (You may find it useful to look at the cantilever and sample with the video camera during this process.)
- Once contact has been made, center the z-piezo at ~70 volts, as usual.

7.2.3. Tuning

1. Once you are engaged, it is time to tune. Table 7.1 on page 85 gives ballpark values for contact resonance for a few different cantilevers.
2. Assuming you are starting with an AC240, try a center frequency of 320 kHz and sweep width of 100 kHz. You should see a peak. If not, try widening the sweep width and gently increasing the drive voltage. For an AC240, once the drive goes above ~5 volts, you may start to degrade the tip quality. For PPLN and PZT samples, 200mV is usually sufficient to see the peak with the high voltage amplifier. Your goal is to see a contact resonance peak of ~10-50mV. Going higher than that will typically cause problems with feedback stability and will lead to rapid tip degradation. On these samples, a drive voltage of 1-3V will give a decent peak.
3. Right click close to the peak and click ‘Set Drive Frequency’. Your goal is to select a frequency close to but not on the peak to allow enough amplification and avoid effects by changes in contact resonance.

4.



Center Phase

- Click 'Center Phase Offset' and set the resonance at 90°.

7.2.4. Imaging

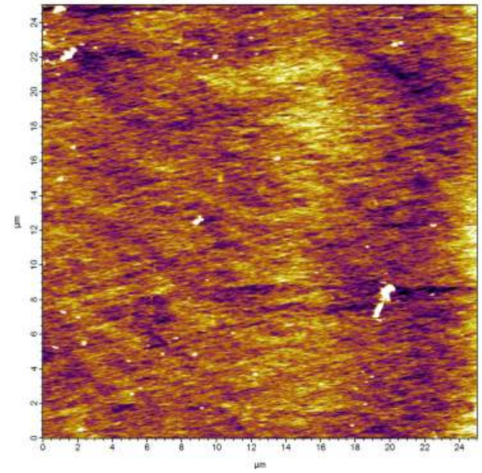
At this point, you are ready to image. Since PPLN domains are typically many microns across, it can be advantageous to scan a larger size. Also note that you will have more reproducible results if you scan at 90°.

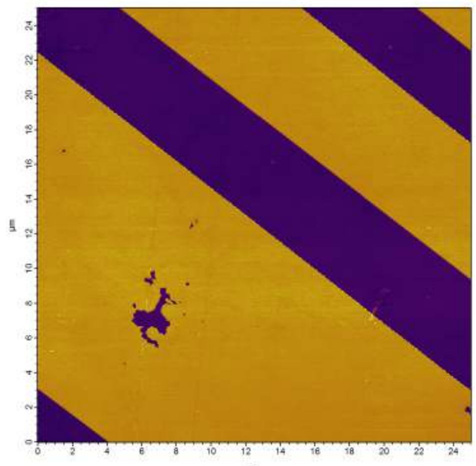
1. Hit 'Do Scan' to start imaging.

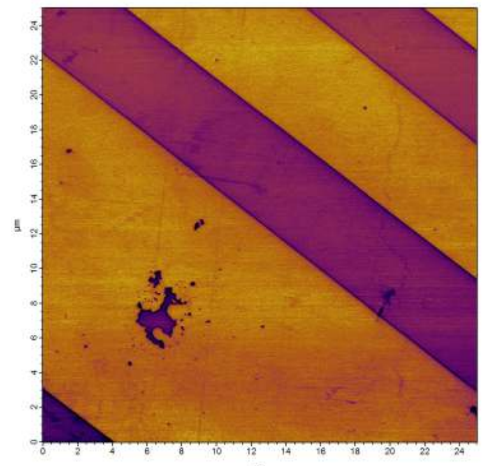
2.

Inspecting data:

- At this point, you should see some 4-10µm periodic signals on the Amplitude, Phase, and Frequency channels corresponding to the poled domains in the PPLN sample. You can also adjust drive voltage to optimize images.
- Clockwise, starting at top right: Topography, Amplitude, and Phase.





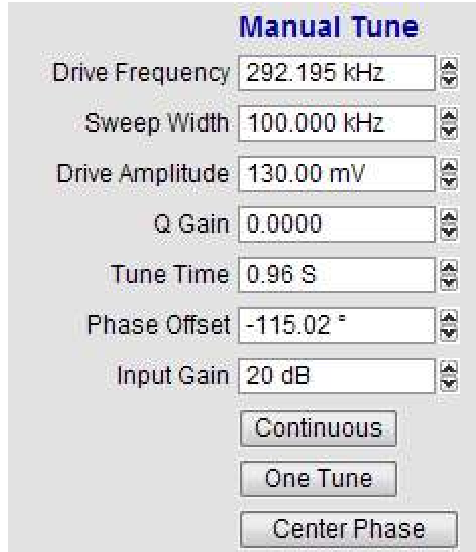


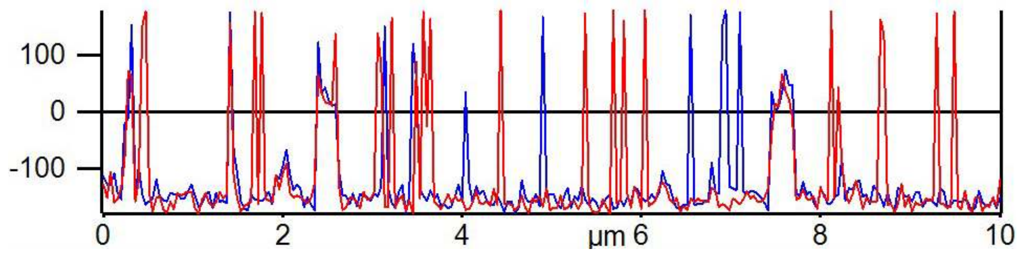
3. A common problem is the loading force being too small. If you have tried adjusting the drive voltage and still do not see good domain contrast, try increasing the loading force. Click 'Stop' and decrease the deflection with the PD knob (or PD motors on Cypher), then retune on the surface. The contact resonance changes depending on the loading force.

4.

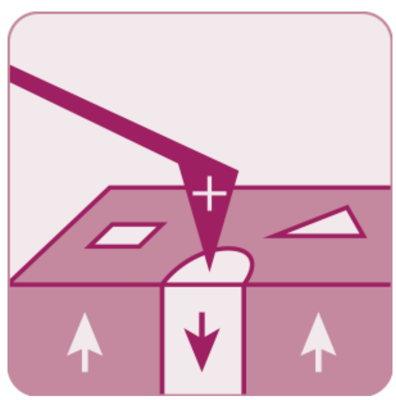
Phase wrapping:

- If phase wrapping occurs (where PFM phase goes below -180º or above 180 º), you will see white spots in the phase image. The scope trace (shown below) shows these spots as peaks.
- Adjust the *Phase Offset* during scan from the Manual Tune panel, shown to the right.
- Live phase flattening should always be off.





7.3. PFM Lithography



PFM can also be used to modify the ferroelectric polarization of the sample through the application of a bias. When the applied field is greater than the coercive electric field, the field can induce ferroelectric polarization reversal. The MFP3D Lithography PFM mode can be used to write complex patterns by importing a grey scale image that is transformed to a bias map.

If the switching voltages are not known, hysteresis loops can be made to determine what they are. Hysteresis loops are discussed in chapter 8.

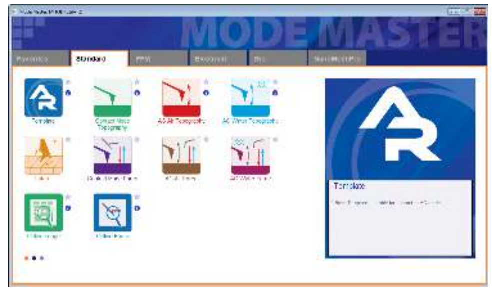
In the following example, lithography is done on a Sol Gel PZT using the High Voltage PFM option. Using the High Voltage holder, a bias of 13.2V was used to guarantee polarization reversal.

To perform PFM Lithography:

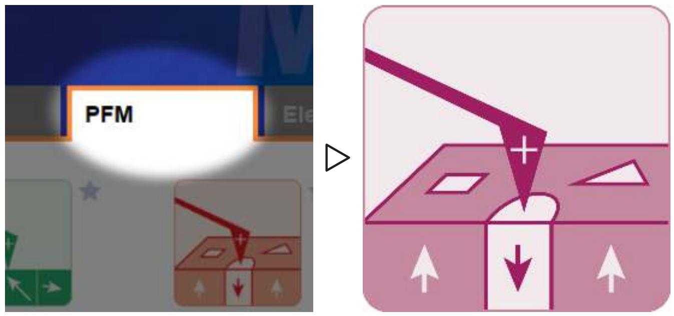
1.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.



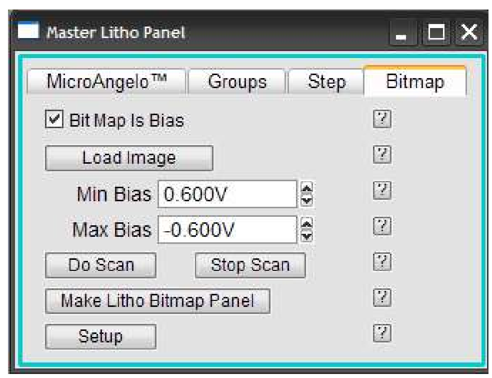
Select Mode:

- Select *PFM tab > Litho PFM*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

3.

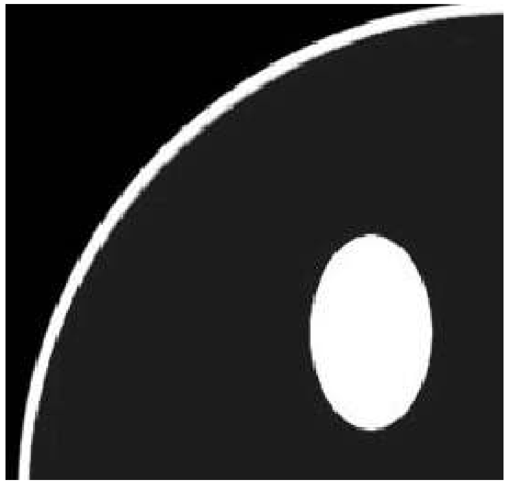
Loading a bitmap image:

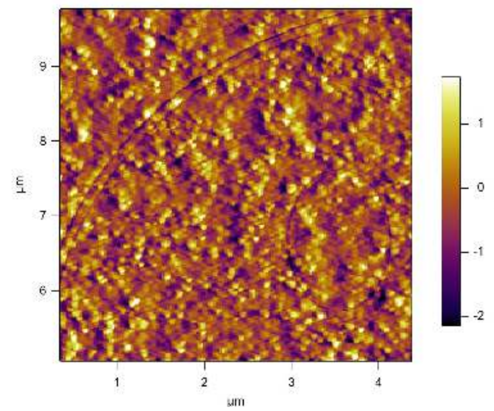
- Click 'Load Image' and select a file.
- The bit-mapping lithography requires a 256 x 256 or 512 x 512 image. A file named *Smile.jpg* is used for this example.
- You will need Quicktime installed to load the graphic into Igor `http://www.apple.com/quicktime/download/`.



4. Click ‘Do Scan’ to start the bitmap bias lithography.

5.

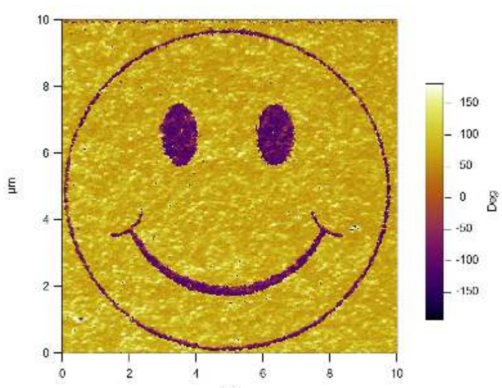


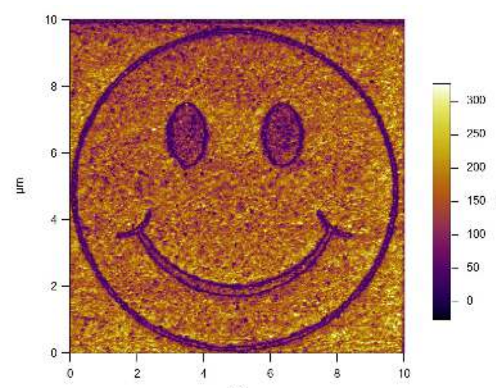


Writing bitmap:

- The minimum and maximum bias voltages are mapped to the gray scale in the imported image.
- During the lithography scan, the voltages are applied to the surface from the bit map image. If the voltage is very large, the electrostatic attractive forces will cause a change in the deflection and topography.
- A zoom of the Deflection channel (above right image) shows the pattern appearing in the deflection during a lithography scan, a good sign that lithography is working

6.



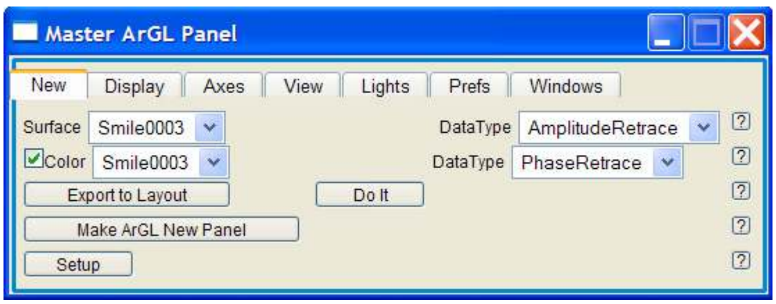


- Switch to the Tune panel and tune for Single Frequency PFM and image as normal. Left image is Amplitude, right Phase.

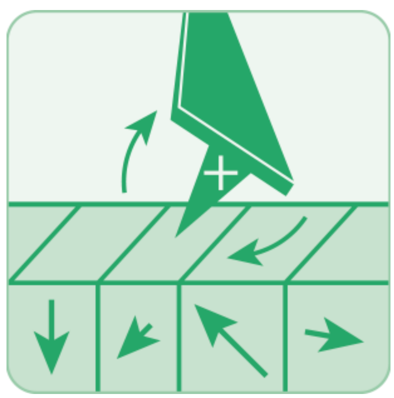
7.

3D Overlay

- A good way to show PFM lithography is to overlay the Phase onto the Amplitude image. This can be done through the Master ArGL Panel found under *AFM Analysis > 3D Surface Plots*.
- At right, the 3D Overlay image, bottom 3D Panel is shown.
- See also AR Software Manual:Argyle 3D:Overlaying Data on a Surface.



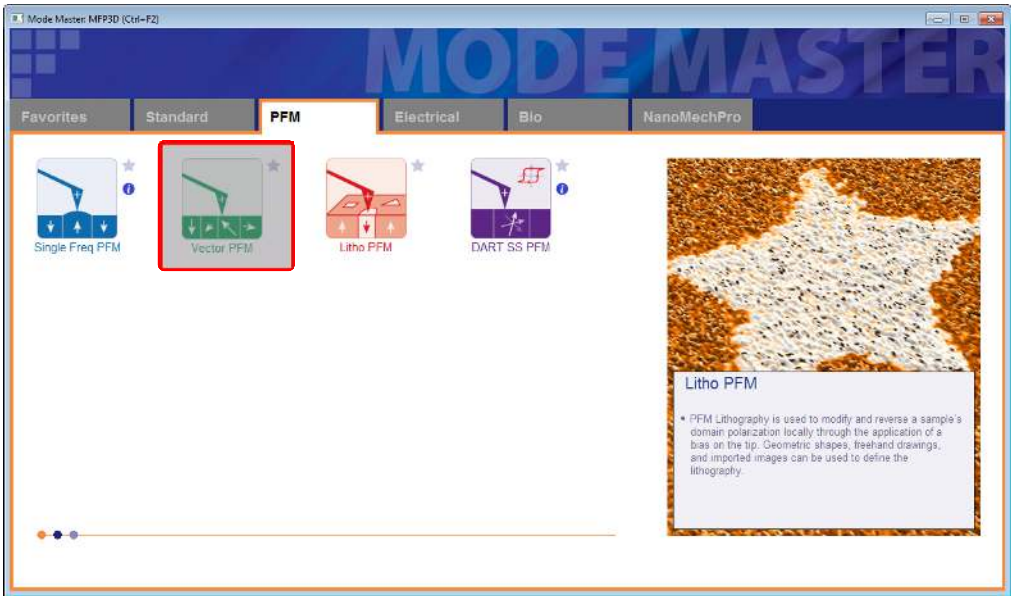
7.4. Vector PFM



Lateral PFM, or Vector PFM, is a two-pass technique (Chapter 10 on page 133) on the MFP-3D Classic/Origin+/Origin AFM and a single-pass technique on the MFP-3D Infinity and Cypher AFM. For the MFP-3D Classic/Origin+/Origin, it first measures the vertical PFM signal in one pass and then the lateral PFM signal in the second pass. The enhanced LFM head option is required to mechanically center the lateral signal to take advantage of higher gains. The sample used in this example is BiFeO3/SrRuO3/SrTiO3.

To perform Vector PFM:

1.



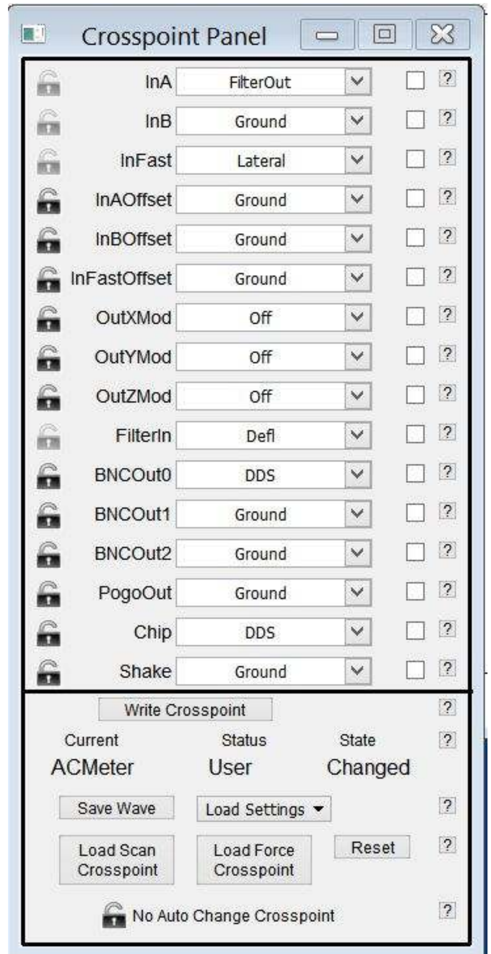
The Mode Master window:

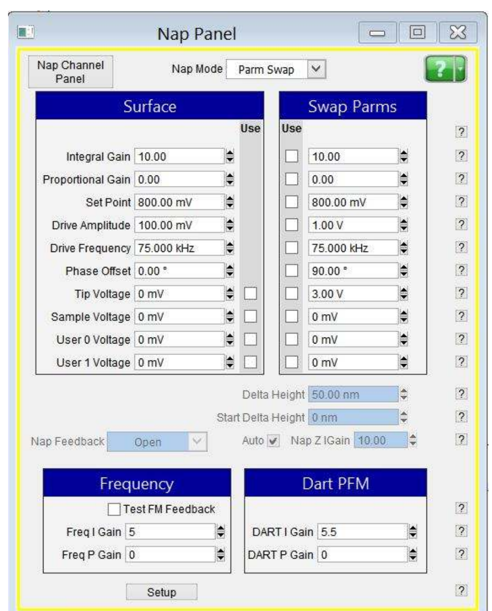
- When the Mode Master window appears, choose the *PFM* tab.
- Select the 'Vector PFM' mode icon.
- All the necessary panels for Vector PFM operation automatically load and arrange on the desktop.

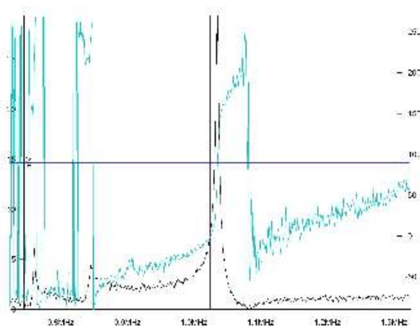
2.

Lateral tuning on MFP-3D:

- To be able to tune for the lateral signal, the Crosspoint switch must be manually changed. To set the parameter to allow this:
  - Select to *InFast > Lateral*.
  - Click 'Write Crosspoint' and
  - Click to select *No Auto Change Crosspoint*.
- Engage and tune as needed. The resonance frequency is usually around 1MHz.
- Manually copy the values for *Drive Frequency*, *Drive Voltage*, and *Phase Offset* from the Surface parameter fields on the Nap Panel.
- Paste them to the corresponding Swap Parms fields. The surface values will be changing in the next step.
- Be sure to unlock the Crosspoint.



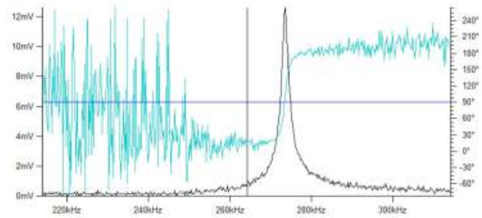




3.

Vertical tuning:

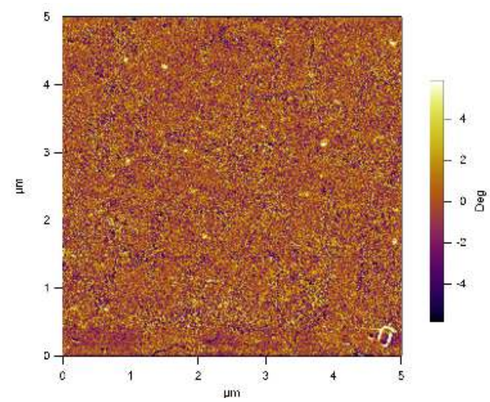
- To tune the vertical deflection signal:
  - Change *Infast* to "ACDefl".
  - Click 'Write Crosspoint'.
  - Make sure *No Auto Change Crosspoint* is NOT selected.
- Enter the *Drive Amplitude* value.
- Click 'Engage', 'One Tune, 'and then right-click *Set Drive Frequency* close to the peak.
- The Surface parameter fields in the Nap Panel for *Drive Amplitude*, *Drive Frequency*, and *Phase Offset* are now filled.

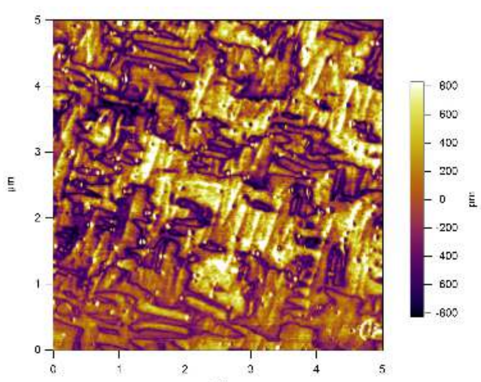


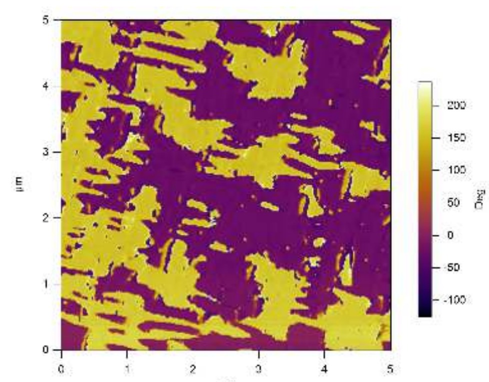
4.

Start scan:

- Click 'Do Scan' to start imaging.
- Clockwise, starting from top right, Vertical Phase, Lateral Phase, and Lateral Amplitude scan images are shown.

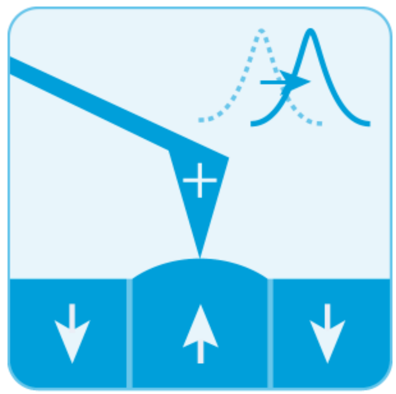






8. Piezo Force Microscopy (PFM) using Dual AC Resonance-Tracking (DART)

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

This chapter describes how to run Dual AC Resonance Tracking Piezo Force Microscopy (DART-PFM), including using the technique to run hysteresis loops on ferroelectric materials.

Piezoresponse Force Microscopy (PFM) is used to characterize the electromechanical response of piezoelectric materials. Typically, a conductive cantilever is scanned over the sample surface in contact mode. While scanning the surface, an AC bias is applied to the tip. The electric field causes a strain 5-10nm below the surface, which in turn causes a periodic deflection of the cantilever.[1]

Recently, a variation on this technique called Electrochemical Strain Microscopy (ESM) has been developed at Oak Ridge National Laboratory[2]. This technique is sensitive to ion transport into and out of the lattice in energy storage (battery) materials such as $LiCoO_2$[3,4,5]. For more information on this powerful new technique, please refer to http://www.asylumresearch.com/Applications/ESM/ESM-DS-HR.pdf

In both PFM and ESM, the electromechanical and electrochemical displacements are usually quite small, sometimes only a few picometers (pm) per volt of excitation. Noise floors of an optical lever are usually somewhere in the neighborhood of tens of pm, so measuring these samples requires either using a large AC voltage or some other amplification technique.

Large voltages can be a convenient way of boosting the small response of piezo samples. However, large voltages come with potentially problematic large electric fields and, with some samples, potentially large damaging currents.

In the following material, we describe a novel method of using the contact resonance of the cantilever to boost small piezo signals. This is more complex than might be expected at first in that the contact resonant frequency depends strongly on details of the contact mechanics– the elastic modulus, tip shape, and sample topography can contribute to cause the resonant frequency to vary many tens of kilohertz (kHz) as the tip scans over the surface. Because of this resonance variation, and because the phase also varies, both fixed frequency drive techniques and conventional phase-locked loops are subject to large amounts of topographic crosstalk[6].

In the following pages, we cover:

8.1. DART concepts

DART is a technique that dramatically reduces the crosstalk due to the shift in resonant frequency by tracking the contact resonant frequency and, using a feedback loop, adjusting the drive fre-

[1] Gruverman, A. et al., Scanning force microscopy as a tool for nanoscale study of ferroelectric domains. Ferroelectrics 184, 184 1996, Nr. 1-4.

[2] Balke, N. et al., Real Space Mapping of Li-Ion Transport in Amorphous Si Anodes with Nanometer Resolution. Nano Letters, 10 2010, Nr. 9.

[3] Balke, N. et al., Nanoscale mapping of ion diffusion in a lithium-ion battery cathode. Nature Nanotechnology, 5 2010.

[4] Kalinin, S. V./Balke, N., Local Electrochemical Functionality in Energy Storage Materials and Devices by Scanning Probe Microscopies: Status and Perspectives. Advanced Materials, 22 September 2010, Nr. 35.

[5] Morozovska, A. N. et al., Local probing of ionic diffusion by electrochemical strain microscopy: Spatial resolution and signal formation mechanisms. Journal of Applied Physics, 108 2010.

[6] Jesse, S et al., Resolution theory, and static and frequency-dependent cross-talk in piexoresponse force microscopy. NANOTECHNOLOGY 21 2010.

quency of the cantilever to match the resonance. Rather than using the phase as the input to the frequency feedback, DART uses the difference between the two amplitudes as the input feedback. Figure 8.1 on page 99 shows a schematic of the two drive frequencies, and the resulting amplitudes (A1' and A2') when the resonant frequency shifts. As the frequency shifts downward, A1 moves up to A1' and A2 moves down to A2'. The change in the A2-A1 signal causes the feedback loop to respond by shifting the drive frequency until the A2-A1 signal is zero again.

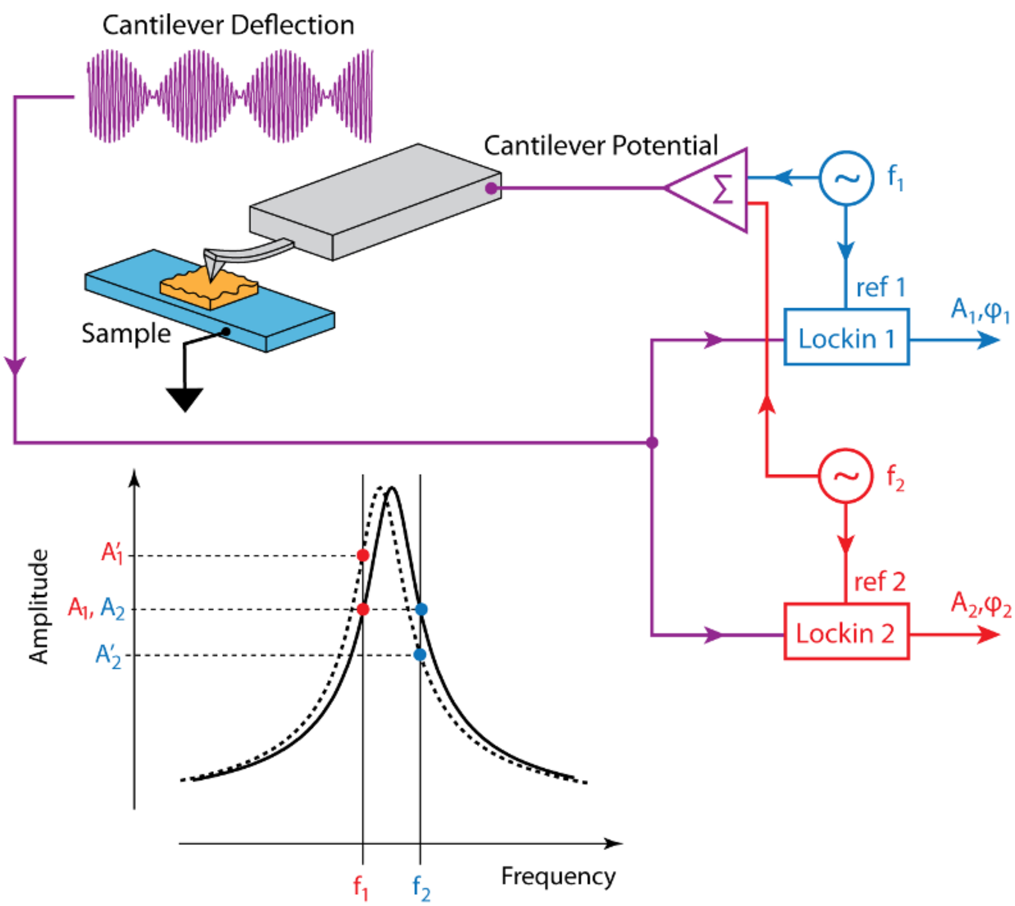


Figure 8.1.: Dart Operation

8.2. Choosing a cantilever and starting a DART template

This section explains how to choose a cantilever and start a DART template using Mode Master. This technique uses the standard cantilever holder for low-voltage PFM or the HV option.

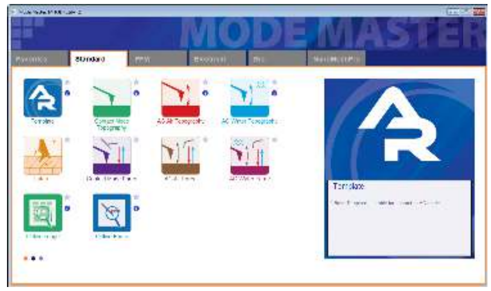
It is assumed in this instruction set that the user is proficient with basic AFM operation.

To choose a cantilever and start a DART template:

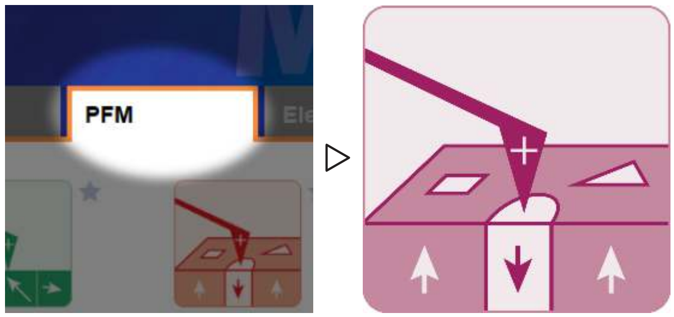
1.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.



Select Mode:

- Select *PFM tab > Litho PFM*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

Note Loading DART SS PFM mode also loads the template for Point Switching Spectroscopy (SS) PFM measurements. You may not need to perform this for PFM with DART imaging, but this technique is covered separately (see Section 8.4 on page 106).

Note	The sample used in this example is a 3mm x 3mm x 0.5 piece of periodically poled lithium niobate (PPLN). It is described in Data Sheet 32`http://www.asylumresearch.com/Products/AR-PPLN/AR-PPLN.shtml`, and is available from Asylum Research. The cantilever used here is an Olympus Electrilever, PtIr coated Si cantilever with a nominal 2N/m spring constant and a free air resonance of ~70kHz.

Cantilevers for DART should have a spring constant greater than 1 N/m. The cantilever should have a conductive coating or be sufficiently doped to provide an electrical contact from the spring clip to the tip. If the cantilever is doped Si, it may be necessary to scratch the chip to break through the oxide layer and then use a tiny patch of silver paint to insure electrical contact between the spring clip and the chip.

DART uses the cantilever resonance to boost the PFM signal. A good rule of thumb for most diving board shaped Si cantilevers is that the contact resonance is typically 3-5 times the free air resonant frequency. For example, if a probe is nominally 70 kHz resonance, start with a center frequency of ~300 kHz and a sweep width of a few hundred kHz. You may need to play around here, especially if you are working with a new type of cantilever.

Cantilever	Suggested Contact Resonance Range for Tuning
Olympus AC240 Electrilever	200-400 kHz
Olympus AC160	900-1,100 kHz
Nanosensors PPP	800-1,200 kHz

Table 8.1.: Typical contact resonance frequency ranges for some common cantilevers

8.3. DART PFM Example

This section describes how to perform an example of DART PFM.

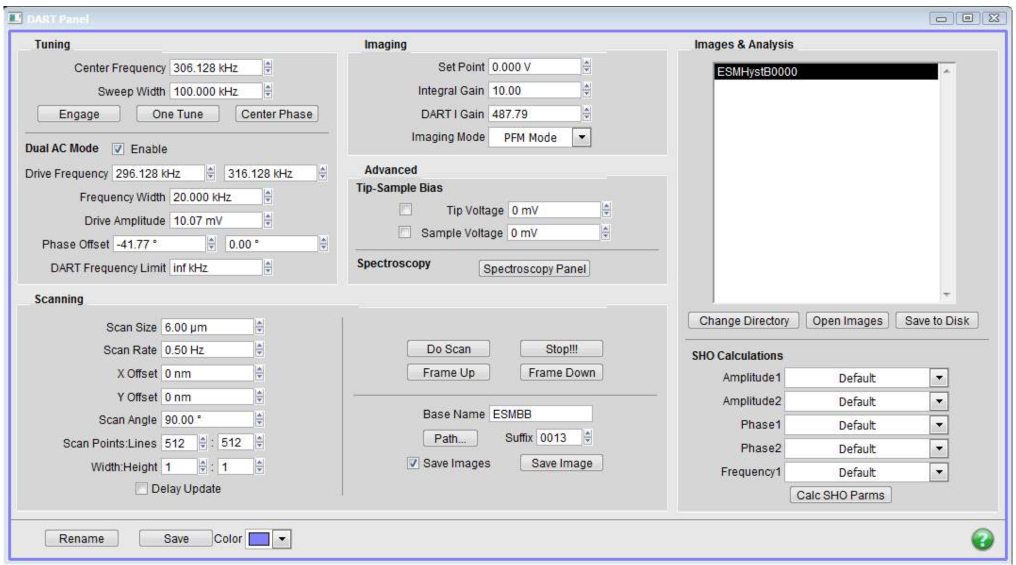


Figure 8.2.: The DART panel

Note	These instructions are for Igor software version 13 or later. (The current version is 18 as of July 2021.) Please update your software accordingly. You can find the latest software at `https://support.asylumresearch.com/forum/content.php?4-Software`.

1.

DART PFM: Getting Started

- Open a new AFM template by launching the AR AFM software or by selecting *File > New AFM template* from Main menu bar.
- From the Mode Master window, select the *PFM* tab.
- In the next window, click 'DART-SS-PFM' (see Section 8.2 on page 99).

Note If the Mode Master window does not appear, press ctrl+F2 or select *User Settings > Mode Master* from the Main menu bar.

2.
The DART Panel:

- The DART panel shown in Figure 8.2 on page 101 should appear. If it does not, in the Main menu bar go to *Programming > User Panels*. You should see a list of available panels, including DART. Select it and the panel below should appear.

3.
PFM initialization:

- In the Tuning section of the DART Panel:
  - Set the *Drive Amplitude* to “10 mV”. This sets the drive amplitude of both frequencies. (If you start with larger values, you may end up unnecessarily degrading your tip.)
- In the Master Channel Panel (Ctrl +7):
  - Record the *Frequency*, *Height*, *Phase1*, *Phase2*, *Amplitude1*, *Amplitude2*. You can use the difference between the phase channels to evaluate the dissipation (Q) of the tip-sample interactions.

4. Load a cantilever into your cantilever holder. For PFM, a good starting point is an Olympus Electrilever. (There are several versions of cantilever holders. If you have the HVA220 Amplifier [MFP3D] option for PFM, make sure you use either the HV DC or AC cantilever holder. These have a small red wire attached to the spring clip terminated with a tiny gold magnet.)

5. Load your sample. For most highly insulating piezo and ferroelectric samples, grounding seems to be optional; we do not see significant differences in the response with or without the ground attached.

6. Position the head above the sample.

7. Maximize the sum, and then set the *Deflection* at approximately “-0.5V”. (We typically operate at a Setpoint of 0V to take advantage of the optional 10x gain on the photodetector signal. This is very useful when measuring the tiny motions in PFM.)

8.
When using the MFP-3D HV sample holder with metal clip:

- Check that the red wire is seated into the HV connector slot glowing red on the sample holder. The magnet should suck the end of this wire into the HV connection slot when the head is positioned above the sample.

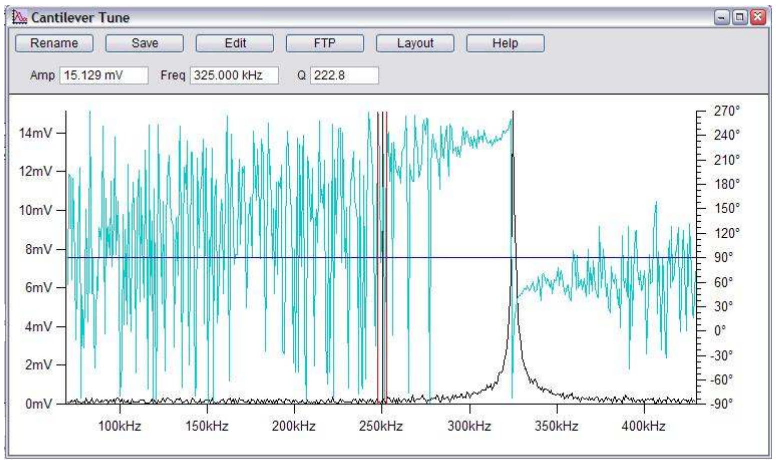
Caution For MFP3D only, take care to ensure that the cantilever holder and the sample clip do not interfere mechanically with each other. At the very least, this can cause false engages; at worst, it could lead to dangerous and damaging arcing between the high voltage spring clip and the grounded sample clip!

9.

Engaging the Surface:

- Click the 'Engage' button.
- Make sure that the z-piezo indicator moves down towards the sample, increasing Z voltage.
- Lower the head with the thumbscrews. You may find it useful to look at the cantilever and sample with the video camera during this process.
- Once contact has been made, center the z-piezo at ~70 volts as usual.

10.



Tuning the cantilever:

- Refer to Table 8.1 for ballpark values of contact resonance for a few different cantilevers. We assume you are starting with an AC240.

  Note To find the contact resonance of an unfamiliar cantilever, do a thermal tune off the sample surface (*Master Panel > Thermal Tune > Start Thermal*). Look for the first and second resonance; they should appear after 10-100 averages of the thermal tunes. The contact resonance will be between these two values
- In the DART panel (Figure 8.2 on page 101), try a *Center Frequency* of 320 kHz and *Sweep Width* of 100kHz.
- The *Frequency Width* refers to the width of the separation between f1 and f2 around the selected drive frequency. Putting these values too close together may cause the two lock-in amplifiers to interfere with each other. *Frequency Width* should generally be set to 5 - 10kHz.
- Click the 'One Tune' button, and you should see a peak.

Note If no clear peak is visible, as in the graph above, try widening the sweep width and gently increasing the drive voltage. For an AC240, once the drive goes above ~5 volts, you may start to degrade the tip quality. For PPLN and lead zirconium titanate (PZT) samples, 200mV is usually sufficient to see the peak though you may need more. Your goal is to see a contact resonance peak of ~10-50mV. Going higher than that will typically cause problems with feedback stability and will lead to rapid tip degradation.

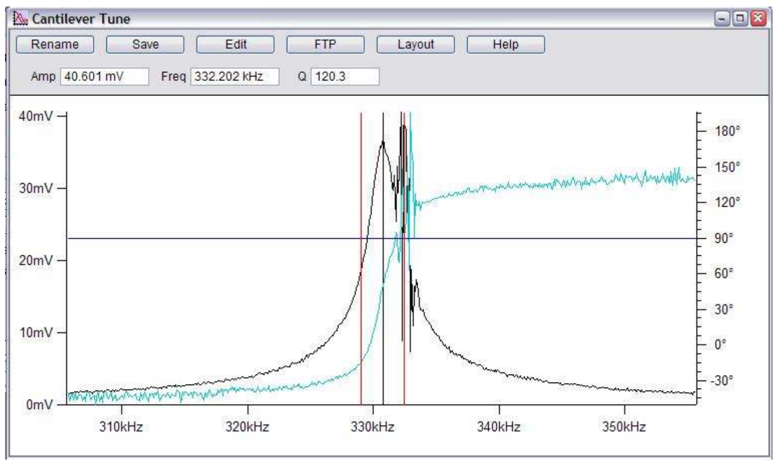
11. Right-click on the tuning peak and select *Center Drive Frequency*. Your goal is to get the

peak to be between the two red bars. Set the *Sweep Width* to 50 kHz and do another tune. The "noise" centered on the high-frequency red bar is normal interference between the two lock-ins and means things are functioning normally.

12. In the Tuning section of the DART panel, click on *Center Phase*, this sets the resonance at 90°. You should see amp1 and amp2 as roughly equal.

Note In contact resonance, it is normal for the resonance to move around many kHz. You may need to make multiple tunes before you have stabilized the peak between the red bars. If you wait between tuning and imaging, you may also have problems; when in doubt, tune again!

13.



Successful tuning result:

- The shape of the curve is nice, and the amplitude at the two drive frequencies are between 10 and 20mV, as shown above. The squiggles in the amplitude and phase around the second frequency are from interference between the first and second lock-in as the first lock-in frequency is swept through the tune range. The ripple in the amplitude and phase channels around the higher drive frequency originate from interference between the first and second lock-ins as the first sweeps through the tune frequencies.

Note It is possible that the position of the resonance peak could shift around during scanning, especially if the surface is rough or the ferroelectric domains are small compared to any thermal drift the system may be experiencing. You may need to "chase" the resonance a bit, especially after you first engage with a fresh cantilever. Finally, you should not wait too long between tuning and starting the DART scanning. If the peak moves from in between the red bars, the feedback loop will actually drive the frequencies away from resonance, rather than tracking it.

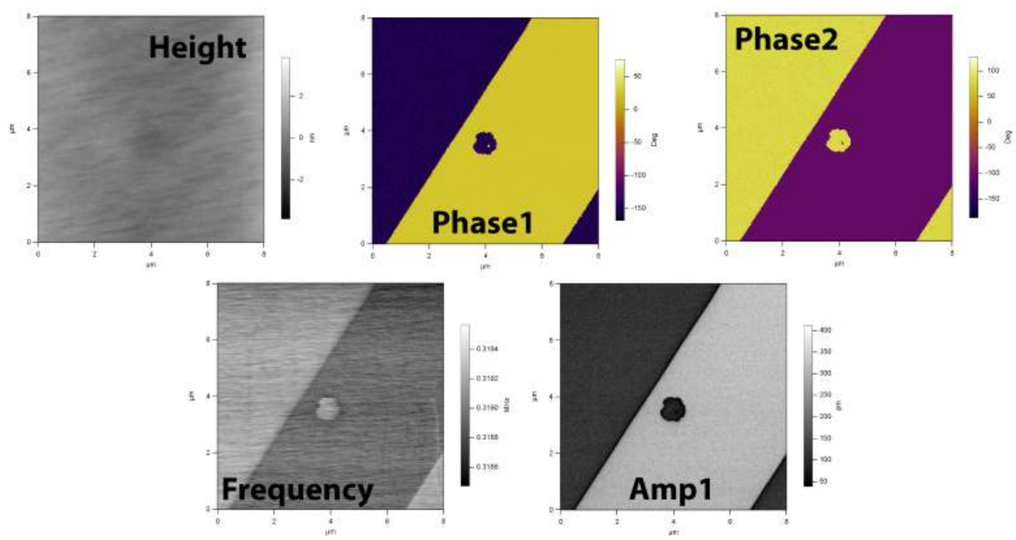
14. **Set Scanning parameters**

- In the Scanning section of the DART panel (8.2), you can start with a reasonably large scan range, such as 20 by 20 µm.
- Set the *Scan Angle* to 90°. (This generates more reproducible results.)

15. **Set Imaging parameters and start imaging:**

- Click 'Frame Down' or 'Frame Up' to start imaging.
- Adjust the *Integral Gain* for good image tracking.

16.



Typical images, PPLN sample:

- If all is well at this point, you should see some 4-10µm periodic signals on the amplitude, phase and frequency channels corresponding to the poled domains in the PPLN sample.
- The images above show a typical 6 µm scan over PPLN with nice contrast over different domains. A small, reversed domain is visible in the center of the image where a ~100V positive voltage was applied.

17.

Optimizing images:

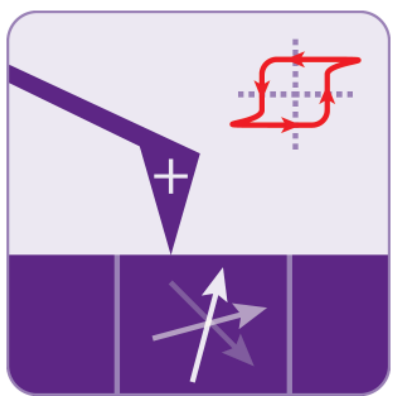
- Right-click on one of the images and select *Fix All Scales*.
- Depending on the balance of electrostatic and piezo forces, you may see more or fewer signals split between the amplitude and the phase channels.
- If frequency tracking is smearing, or trace and retrace look very different, the *DART I Gain* should be increased. Don't be afraid to play here, as there are a wide range of values that may be appropriate; ranges from 5 to 300 are common for normal imaging.
- The frequency tracking may have also "lost lock". In this case, you may need to stop imaging and find the contact resonance again. If this is happening systematically, it may mean that your sample is very rough, and you may need to decrease the scan speed.

18.

Adjusting imaging parameters:

- A common problem is the loading force being too small. If you have tried adjusting the drive voltage and still do not see good domain contrast, try increasing the loading force. Do not change the setpoint; rather click St"op, adjust the PD thumbwheel to a more negative value, and re-engage. Note that you will need to go through the surface tune process again.

8.4. Switching Spectroscopy PFM (SS-PFM)



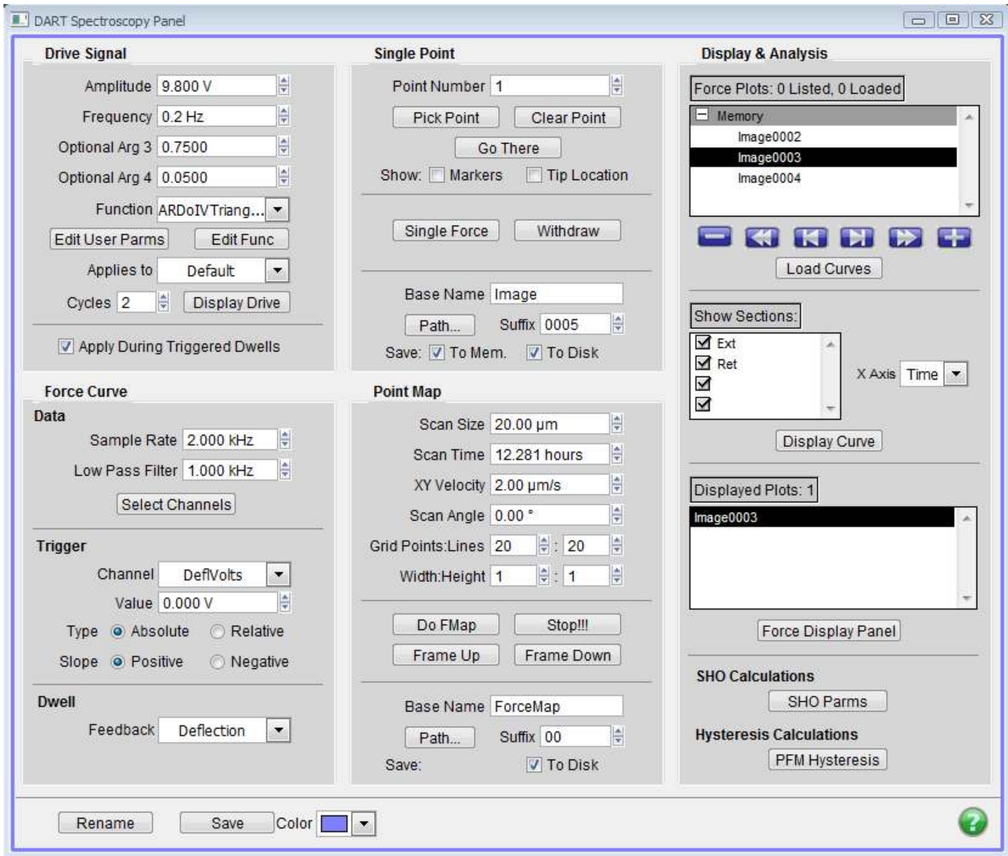


Figure 8.3.: The DART Spectroscopy Panel

In this section, we will measure point hysteresis loops. We start with loops at an arbitrary point and finish with an array of loops using the panel above. We will go through the process of measuring SS-PFM hysteresis loops as described by[7]. First, we'll review some of the terminology related to the triangle step functions that will excite the tip voltage.

There are four parameters for the triangle step function. The effects of varying them are illustrated in Figure 8.4 on page 108 below.

Amplitude The largest square waves will peak at this value and is symmetric around zero. Care should be taken that the sum of this amplitude plus the drive (oscillating) amplitudes never exceeds 10V for the standard holder, 220V for the MFP3D high voltage option, and 150 for the Cypher high voltage option. Also, remember that since DART applies two sinusoidal drives, you must use two times the oscillator drive amplitude when calculating this. It is usually better to start small and increase the number gradually, say ~1V.

Frequency The frequency one cycle of the hysteresis loop. For example, 200 mHz implies a single cycle is acquired in 5 seconds.

Optional Argument 3 This controls the phase of the overall hysteresis measurement. This value ranges from 0 to 1; a value of 0.5 means the voltage starts at 0 and ramps in the positive direction first. For examples, see Figure 8.5 on page 108.

[7] Jesse, S et al., Resolution theory, and static and frequency-dependent cross-talk in piexoresponse force microscopy. NANOTECHNOLOGY 21 2010.

Optional Argument 4 This controls the period of the individual pulses in units of seconds. For example, a value of 0.25 here means that a single on-off measurement will last for 250 ms total. Of that, 125 ms will be at the applied voltage, and 125 ms will be at 0 volts.

Note: The switching voltage for PPLN is >10V, so you will need a high voltage option to see hysteresis loops. With the standard configuration, you will not see the loops.

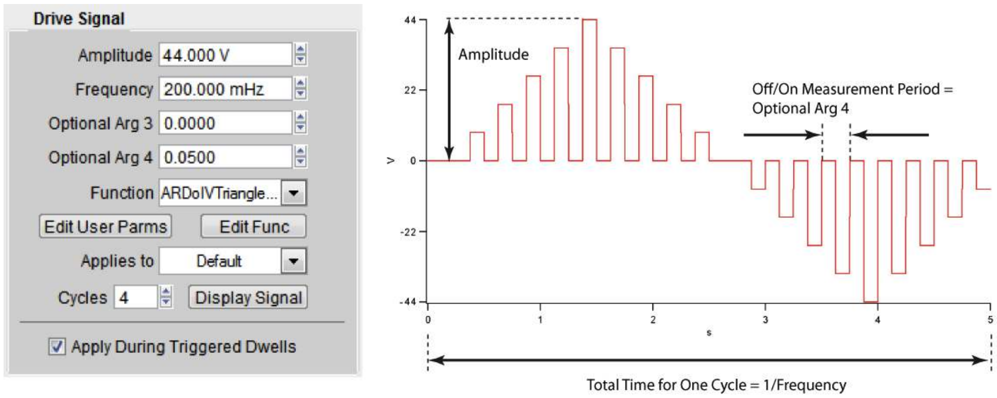


Figure 8.4.: PFM drive waveform parameters

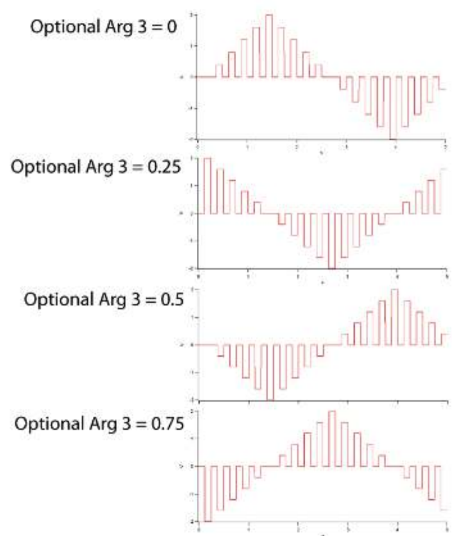


Figure 8.5.: Examples of the effect of Optional Argument 3

8.4.1. Dart PFM Spectroscopy Example

1. The *DART I Gain* for spectroscopy should be smaller than values used for imaging. A suitable number to start with is 10.
2. On the Spectroscopy Panel, (Figure 8.3 on page 107), go to the Drive Signal section.
3. Click the *Display Drive* button. This brings up a voltage versus time display for the loop measurements. This is useful for ensuring that you are applying the proper waveform.

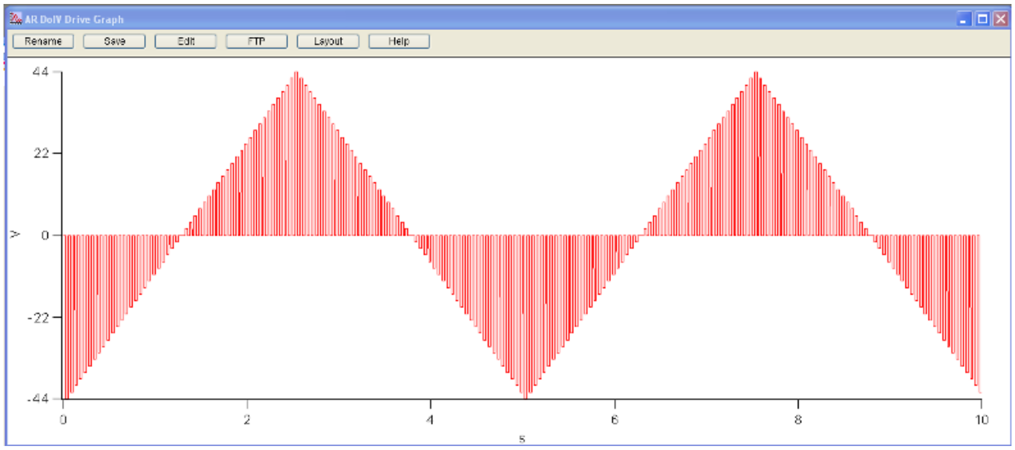


Figure 8.6.: A typical scan waveform for measuring in-field and remnant hysteresis loops for SS-PFM.

4. Select the "ARDoIVTriangleSquare" waveform in the *Function* pulldown menu. This waveform will generate applied steps with a ramping maximum value. It generates two hysteresis loops: one with the field on (the "applied loop"), and the other with it off (the "remnant loop").
5. Next go to the Force Curve section. For measuring hysteresis loops, we use some of the machinery used in measuring force curves and force volumes, including:
   a) For Data, by default, *Amplitude1*, *Phase1*, and *Frequency* are selected versus Time . You can check this by clicking 'Select Channels'.
   b) For Trigger, the *Channel* is set to "DeflVolts" and should have an absolute value set to the setpoint used for imaging, usually zero volts.
   c) For Dwell, the *Feedback* is set to "Deflection". This enables the feedback loop keeping the cantilever deflection constant during the hysteresis loops.
   d) Now you select a point or points for the hysteresis loops. Select a reference image by clicking on it to bring it to the top, as follows:
      i. Click the 'Pick Point' button.
      ii. On the reference image, position the round cursor on the location for your hysteresis loop.
      iii. Click 'That's It' to finalize your choice.
      iv. Repeat for as many locations as you want. You can also select additional points this way after you start measuring hysteresis loops.
      v. Select 'Show Markers' to bring up the locations and indices of the various locations you've selected.
      vi. Select 'Tip Location' to place a red ball on the image at the current tip XY position. Using the 'Go There' button and the position index, you can put the tip on any of the locations you have selected.
   e) Go to one location and do a surface tune. As before, center the peak between the red bars.

f) Click 'Single Force'. The data shown in the Force Review Graph 8.7 is the entire time record of all the saved channels. It will take one more step to turn them into the hysteresis loops.

6. To process the time waves to extract the applied and remnant hysteresis loops, do the following:

a) Go to the Display and Analysis section.

b) Select the data wave that you are interested in. This should bring up the force review wave.

c) Click the *PFM Hysteresis* button. This will process the given time data and return the applied ('On') and remnant ('Off') hysteresis loops. See Figure 8.9 below for a nice example of data extracted from the time waves shown in Figures 8.7 and 8.8.

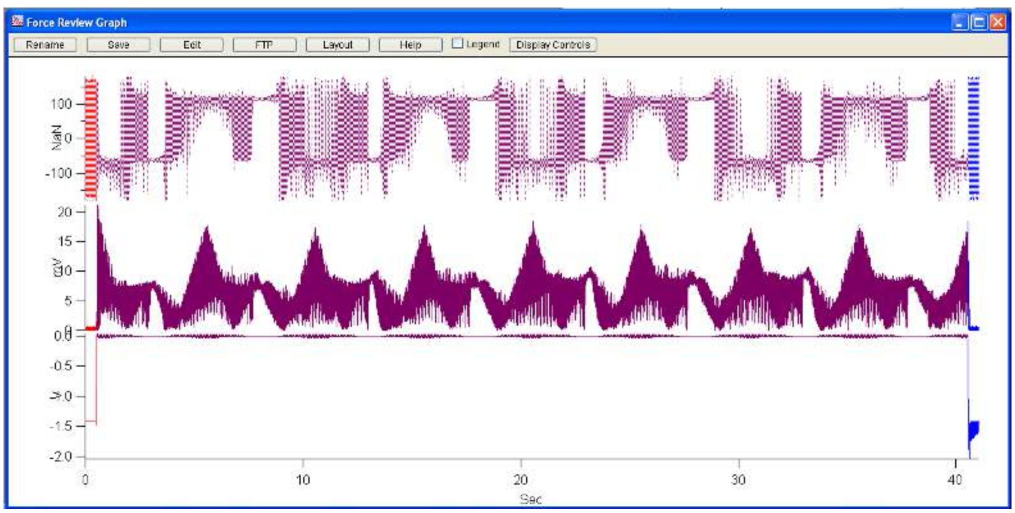


Figure 8.7.: The SS-PFM raw data set. The phase1, amp1, and deflection traces for an 8-cycle, 40 second hysteresis loop. The details of the switching are difficult to discern from the time data and need to be processed to produce hysteresis loops.

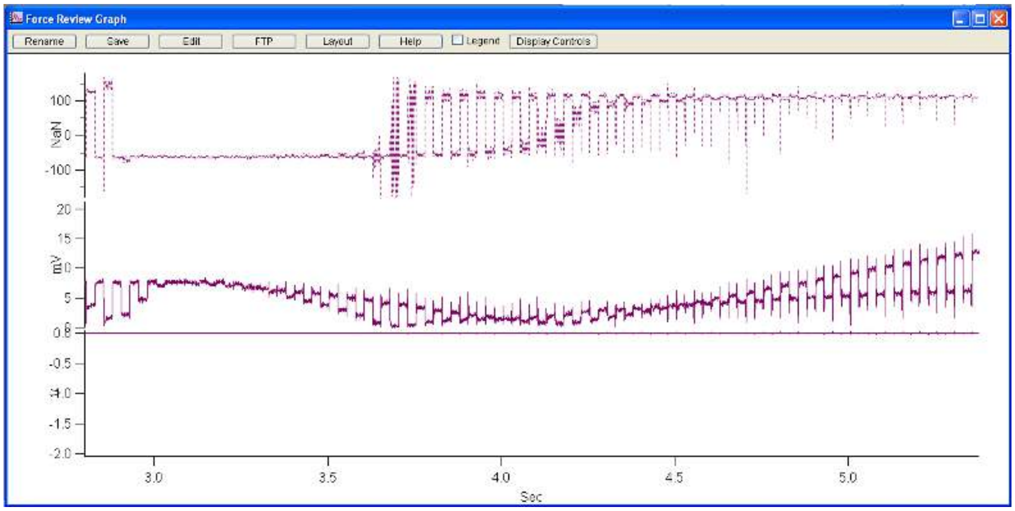


Figure 8.8.: Zoomed in region from Figure RawData . Here, the individual steps from the stepped SS-PFM method of[8] are evident. Repeat the above steps as many times as you want, adjusting the drive parameters accordingly. If you notice noise during the higher voltage portions of the curves, you may have your *Freq Igain* set too large. Reduce it until the effect disappears.

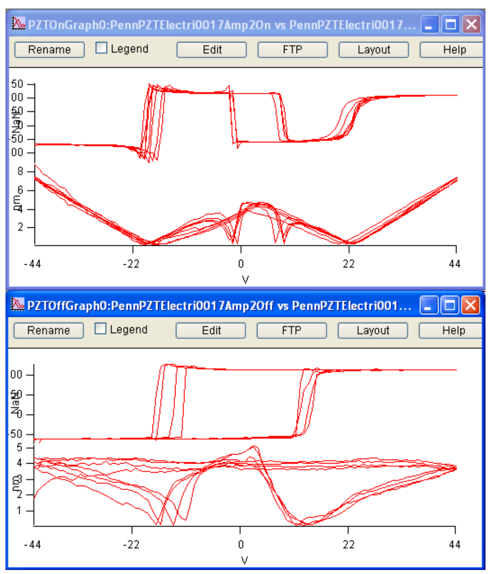


Figure 8.9.: The applied ("On", top graph) and remnant ("Off", bottom graph) hysteresis loops obtained from the processed time waves of figures 8.7 and 8.8. The phase loops are on the top of each window, and the amplitude "butterfly" loops are on the bottom.

8.4.2. SS-PFM Array Measurements

1. Start with single point measurements described earlier (allowing you to adjust parameters and optimize the conditions for your sample and tip), as follows:

   a) The array of points uses the current image size and location as a reference. Using the usual tools, select your scan size and location.

b) In the Point Map region of the DART Spectroscopy Panel, select the number of *Grid Points* and *Grid Lines*. Each is required to be an even number.

c) As in the previous examples, do a surface tune.

d) Click 'Do Scan'. The red spot should start moving on the image, and you should see data start to appear in the Force Review Graph window. (Force maps can take some time; you should budget a few seconds for moving from one point to another in addition to the time it takes to measure the hysteresis loop itself.)

e) If there is a problem, click the 'Stop!!' button, then adjust the parameters and start again.

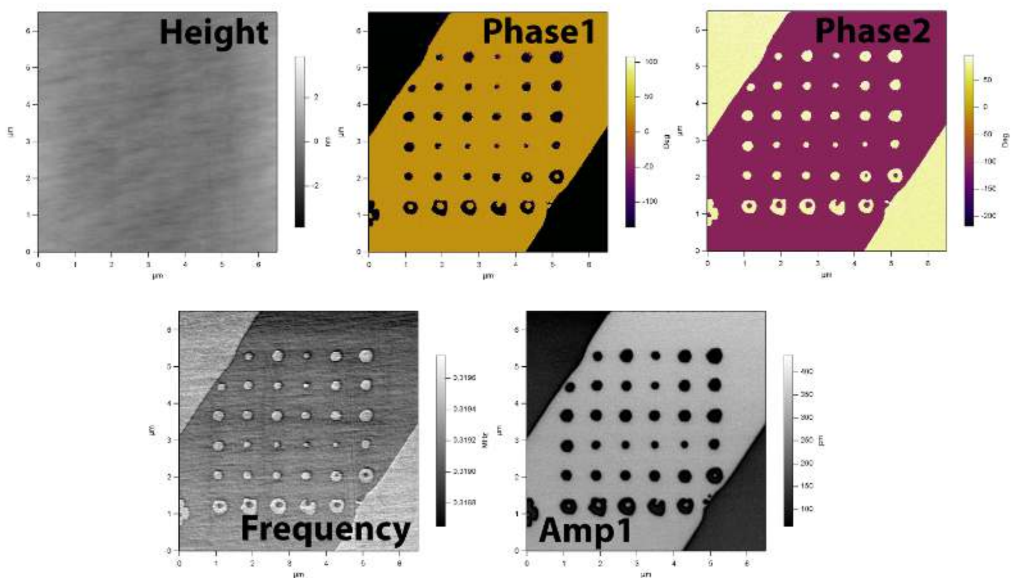


Figure 8.10.: DART PFM image of PPLN after a 6 by 6 array of hysteresis loops have been measured

8.5. DART Imaging: Guidelines & Troubleshooting

8.5.1. Amplitudes of Drive and Response

Start out with a drive voltage of 250 mV. If you need to drive the tip at >5.5 volts, the tip will most likely experience some degradation. These drive amplitudes should result in response peak amplitudes between 5-50 mV. Generally, amplitudes less than that may not result in stable feedback, and amplitudes larger than ~50 mV can lead to instabilities in the frequency feedback loop.

8.5.2. Image Size

Set the image size to a small value (such as 1 μm) to begin with. This is usually a convenient range for optimizing the feedback and scanning parameters. Typically, it is a good idea to scan at 90º to minimize tip shape effects that can potentially have a large effect on the contact resonant frequency.

8.5.3. Feedback Gains

One significant difference between Contact Mode imaging and DART is that there are two gains : one for the topographic feedback (marked *Integral Gain*) and another for the frequency feedback loop (*DART I Gain*). First, you should optimize the topographic tracking in the Height trace and retrace curves with the *Integral Gain* and then do the same thing for the Frequency trace and retrace curves using the *DART I Gain* parameter. This gain can vary over a wider range than you may expect from your experience with topographic gain. Ranges from 2 to > 300 are common. As you would expect, scanning faster requires higher *DART I Gain* settings.

For typical operation, the trace and retrace on the frequency channel should match and also have distinct features correlating to topography or material properties. The amplitude should show signal within different domains and drop to zero at domain boundaries, indicated by regions where phase shifts 180°.

8.5.4. “Pre-imaging” and Image Region

It may be a good idea to take an AC mode image of the surface either with the tip that will be used for the experiment, or a standard AC mode probe (such as an Olympus AC240) to be sure there are no surprises on the surface. This is often true for Contact Mode electrical techniques, since any debris on the surface can have a dramatic effect on electrical characterization, which is often not easy to see in Contact Mode.

8.5.5. Loss of Frequency Lock

If the sample has a weak piezoelectric response, or the tip is damaged, or the *DART I Gain* setting is too small, it is possible for the resonance peak to move outside of the two drive frequencies. When this occurs, lock is lost and is unlikely to occur again without user intervention.

8.5.6. Tip Damage and Contamination

PFM is a technique that is extremely sensitive to the condition of the tip and sample. Contaminants and tip wear can have very large effects on image quality. The downside of this behavior is that you may see degradation in image quality as you scan. PFM can be treated in the same manner. It is often worthwhile to tweak the image parameters a little and wait for a scan or two, as the contaminant will often detach and give you beautiful contrast again.

8.5.7. MFP-3D HVA220 Setup

For optimal PFM imaging and spectroscopy, the High Voltage option on the MFP-3D HVA220 is highly recommended. There are numerous examples where the standard 10V range is simply not quite sufficient to get good contrast or to get a closed hysteresis loop. In typical PFM examples, often a small improvement in range, say to 15-30V, will make all the difference in contrast.

The HVA220 allows a high voltage to be applied to the tip in a very safe and secure manner. The tip itself is not energized until the head is securely in place over the sample by means of a simple magnetic interlock.

You can check to see if the SmartStart system has detected the High Voltage module by clicking on the gear icon at the bottom of the screen. A list of detected devices should appear, including the HVA220, as shown in Figure 8.11 on page 114.

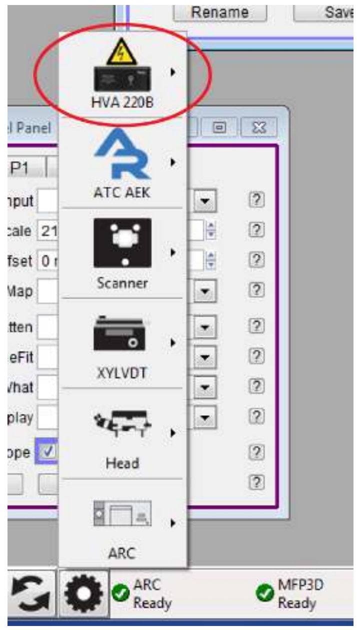
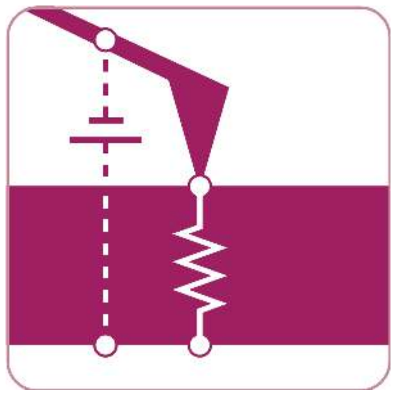


Figure 8.11.: Testing communication with the HVA using the SmartStart Device interface. The HVA icon indicates that the HVA is present and active.

If the HVA icon does not appear, rescan the SmartStart bus by clicking on the black circle icon. If this still does not show the HVA220, check the expansion cable connections and the HVA220 power cord, and make sure that the HVA220 power switch on the back of the unit is on. If you still have problems, contact Asylum Research Support for assistance.

9. Conductive AFM (ORCA)

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

9.1. Introduction

Conductive AFM or CAFM (called ORCA on an Asylum Research instrument) can be considered to be anything that looks at electrical current passing through the tip and the sample. This can include several techniques with many acronyms. For the most part, with minor deviations, these techniques all do the same thing in principle; they measure one of the three variables in Ohm's Law V=IR, where *V* is the voltage applied to the sample, *I* is the current passing through the sample and the tip, and *R* is the resistance of the entire circuit.

A schematic of CAFM is shown in 9.1.

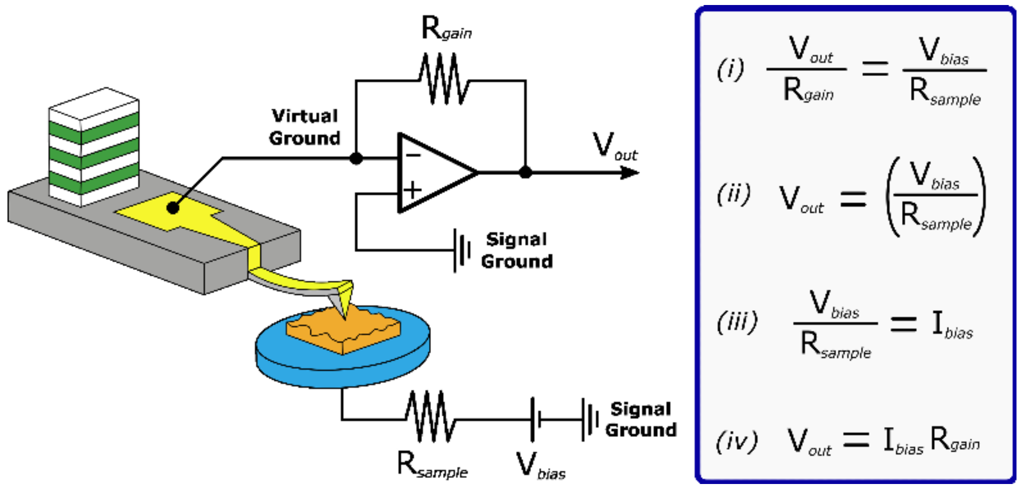


Figure 9.1.: Schematic of CAFM with relevant equations. Current is passed through the tip and into a transimpedance amplifier, converting the current to a voltage.

9.2. Video Tutorial

Consider watching this introductory video tutorial: Conductive AFM (ORCA), Imaging & IV curves (internet connection is required).

9.2.1. Terminology

Terms used in CAFM:

CAFM Conductive AFM, defined here as anything that looks at current passing through a sample and a tip in atomic force microscopy.

Subcategories: TUNA Tunneling AFM. This refers to the specific application of CAFM where the current tunneling through an oxide or insulating layer rather than the ohmic current passing through a sample. In practice, the circuit is generally the same as CAFM, but a higher gain resistor is used.

SSRM Scanning spreading resistance microscopy: Here, a tip with a tougher coating is used with a higher force so that the tip is digging through the contamination/oxide layer on the semiconductor under study. Typically, a logarithmic current detection circuit is used to increase the range of current detectable, but a multi-stage gaining circuit can accomplish the same thing.

SGM Scanning gate microscopy: Here, current is passed through a transistor instead of the tip-sample. The tip is used to bias the gate, and the same current detection circuit is used to measure the current.

Orca Optimized Resistance Conductance Amplifier. Asylum's name for CAFM.

9.2.1.1. Spectroscopy vs. Imaging

With CAFM, you can either collect a current map of a surface at a given bias, essentially giving you a single slice of the conductivity of your sample, or you can take current-voltage spectra at points either predefined by the user individually (i.e., 'Pick a Point') or on a grid of n×n points. Spectroscopy normally entails taking AC mode images of a surface, then changing over to contact mode and collecting the spectra where desired.

9.3. The Hardware

The hardware is specific to your mode of AFM. Please consult the appropriate manual:

- *Cypher User Guide, Chapter: Conductive AFM*
- *MFP-3D User Guide, Chapter: Conductive AFM (ORCA) Hardware*

The following sections explain which cantilever holder to use, how to calibrate it, how to mount the sample, and how to connect the bias wire.

9.4. Probes

There are several probes available for the CAFM technique. Ideal spring constant range is between 0.5 and 5 N/m. Cantilevers that have lower spring constants have difficulty to overcome the contact resistance between the tip and the sample. Cantilevers that have too high of a spring constant will wear down too quickly.

Useful probes include the following:

- **Metal-coated Si probes.** Pt, PtIr, Au, CoCr, Tungsten Carbide, and Ir-coated probes, such as the AC240TM-R3 (see the Probe Store for other Conductive Probes as well). These probes perform similar to each other. The Tungsten Carbide and Ir probes last a little longer. For Polymer samples or soft materials, these probes are fine. Radii are typically 50nm.
- **Conductive diamond-coated probes.** These probes use a diamond-like carbon film coated onto a Si probe. Available from Nanosensors. They last quite a lot longer than metal-coated probes but have a larger radius (~100nm). ADT makes a nano crystalline coating that wears at least as well as the Doped DLC, but typically has a smaller radius (<50nm).
- **Solid W probes from Multiprobe.** Very consistent probes. More expensive than most but last a long time. Form an oxide over time that has to be worn away with a bit of imaging.
- **Solid Pt probes from Rocky Mountain Nanotechnology.** Best overall probes for CAFM. Small radius (5-20nm). Good wear characteristics. Even if the probe wears to larger sizes, it does not lose its ability to conduct.

9.5. Repeatability in CAFM

The follow list includes common examples of repeatability in CAFM:

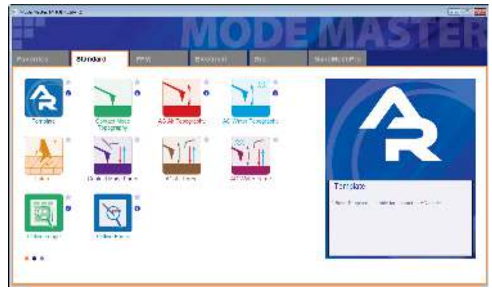
- **Tip change.** This is the most common. Metal coated probes are infamous for chipping, breaking, and otherwise acting in an obstinate manner. This normally shows up as a systematic asymmetry in the current to topography comparison. In other words, the current will appear on only one side of all the grains or features on the surface.
- **Debris on the surface.** Probably the easiest factor to diagnose. Normally, this is some soft material that does not show up in the contact mode image because it is being swept away during the scanning. To diagnose this, simply take an AC mode image of the surface before you begin your contact mode image. If there are any soft, organic particles on the surface that may foul your measurement, they should show up. It is a good habit to always take an AC image first when working with CAFM.
- **Contact resistance variation.** The most difficult to diagnose. Here, you may be using just enough force to get a current image of your surface, but not enough force to maintain a constant contact resistance over all the varying features on the surface. This one can be particularly difficult on soft surfaces, where if the user applies too much force, the surface will be modified, but if the user applies not enough force, the contact resistance will vary significantly over the sample surface. This is one of the more difficult aspects of imaging conductive polymers.
- **Surface change.** Normally easy to diagnose, but often not suspected by the user. Common ways this can happen is surface oxidation, surface destruction, or surface modification. Surface oxidation is common with Si surfaces and other semiconductors and is normally worse in high humidity. Surface destruction is common with polymers and carbon nanotube (CNT) surfaces. Some materials can be modified by biases to become either conducting or insulating with a current or a bias.
- **Sample preparation.** Other problems that can lead to poor reproducibility involve the circuit, rather than the tip or sample. This is more common when comparing results from sample to sample but can also occasionally occur in multiple experiments on the same sample. This includes things like poor silver paste used to mount the sample, or poor contact being made with one of the connections in the circuit. This could be where a clip contacts a pad on the sample or a break in the silver paint once it has dried. It is a good idea to double-check this, even if you think the circuit is fine.
- **Silicon Oxide.** This one really belongs in the previous item, but it is so common that it should be stressed. On Si samples, you need to scratch away the SiO_2 from the surface, then as quickly as possible apply conductive paint to the surface (Ted Pella (`http://www.tedpella.com/`) "Leitsilber" Conductive Silver Cement is very good) to ensure you don't have a metal-oxide-semiconductor (MOS) contact in your circuit.

9.6. Current Imaging

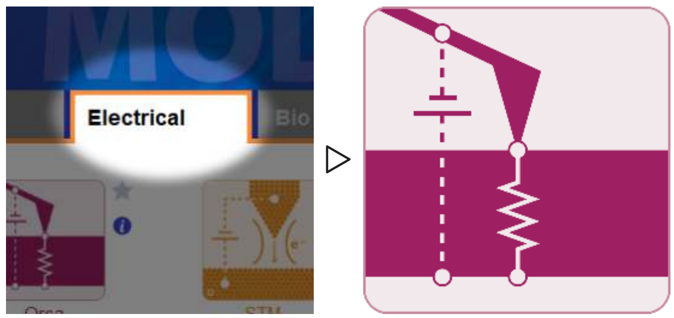
9.6.1. How to perform CAFM imaging:

1. **The Mode Master window:**

- The software should be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2.



Select ORCA mode:

- Select *Electrical tab > ORCA*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

Note The ORCA cantilever holder must first be attached to the AFM to load the ORCA template.

3. Load a conductive cantilever and sample as standard for Contact Mode imaging. This includes any necessary bias wires and/or resistors. (See the appropriate manual for setup instructions.)

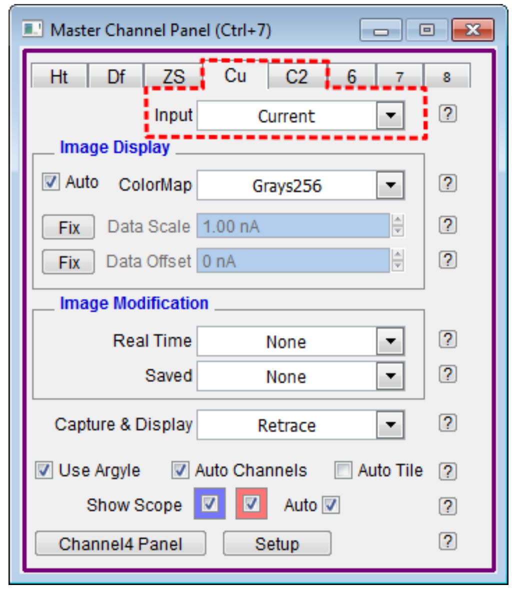
4. Set the AFM to operate in Contact Mode.

5. Set *Scan Angle* to 90º.

6.

Open the Channels:

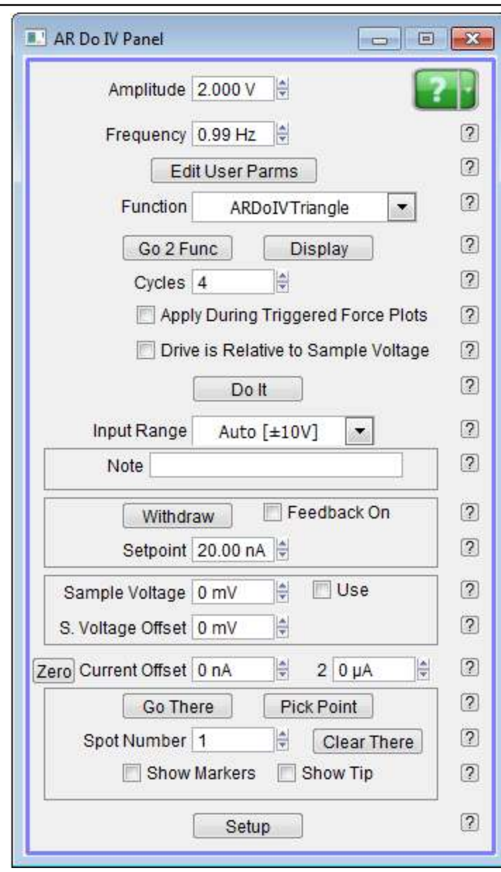
- Select *AFM Controls > Master Channel Panel* (Ctrl+7)).
- Select “Current” as the channel *Input*.
- For the Dual-gain ORCA holder, you will have two choices: “Current 1” and “Current 2”.



7.

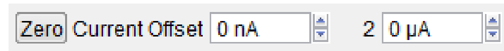
Open the Do IV Panel:

- Select *AFM Controls > Do IV Panel.*
- The **AR Do IV Panel** is where you will control the majority of the electrical-based paraments in your experiment.



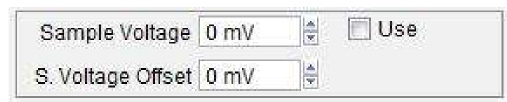
8. **Set the current offset to zero:**

- There are often small, persistent current offsets associated with the AFM electronics that must be subtracted.
- Zero the *Current Offset* by clicking the 'Zero' button in the **AR Do IV Panel**.



9. **Applying voltage:**

- Like the current, there are also often small, persistent voltage offsets between the AFM and the sample. These need to be zeroed as well. However, this is done manually with a voltmeter.
- It is best to measure the voltage between the "PogoOut" bias wire coming from the cantilever holder sample puck and the cantilever holder clip.
- Enter the opposite value measured into the *S. Voltage Offset* parameter. A typical offset is between 10-100mV.
- Sample bias for ORCA experiments can be set with the *Sample Voltage* parameter on the **AR Do IV panel**.



10. When you first engage on the sample, you should start with a light contact force or low *Setpoint*, but you may need to slowly increase the force. This reduces the tip-sample contact resistance until the dominant resistance is the spreading resistance from the tip, rather than the contact resistance. Typical deflections for conductive AC240-based cantilevers, or other similar cantilevers with spring constants around 2.5 N/m, are 0.2-0.3 V. For cantilevers with spring constants of 20-40 N/m, deflections of less than 0.1-0.15 V are typical. For Contact Mode based probes with less than 0.1 N/m, deflections should be 1 V or above.

11. Once you begin the image in Contact Mode, begin raising the *Sample Voltage* until you see a current response from your sample. You may need to, once again, readjust the Deflection at this point.

9.7. Current-Voltage (I-V) Spectroscopy

An I-V curve is a measurement of the current as a function of voltage applied to a single point on the sample surface.

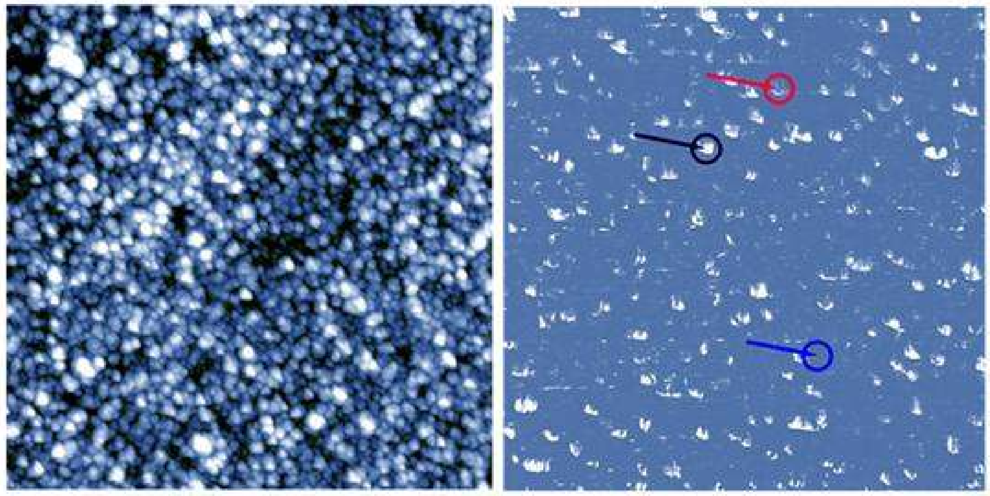


Figure 9.2.: Fairly typical thin film CAFM image. Note that tip shape artifacts are visible on the current image (left), but not on the top graphic image (right). As the metal coating on the probe tip wears, or if the tip is contaminated, these artifacts will be more prominent. Rocky Mountain Nanotech now makes a solid Pt probe that does not suffer from this problem.

I-V curves are done using the **AR Do IV Panel**, which can be opened from the menu *AFM controls > Do IV Panel*. A description of all of the individual listings on the **AR Do IV Panel** can be found in the software's embedded Help file by clicking on any question mark box to the right of the control in question.

The **AR Do IV Panel** contains all the controls that you would need to perform the following:

- Position the tip over specific features on the sample
- Engage the tip on the surface
- Apply a specific voltage signal or function
- Carry out any custom user functions that you may want to write during the routine.

9.7.1. How to Guide

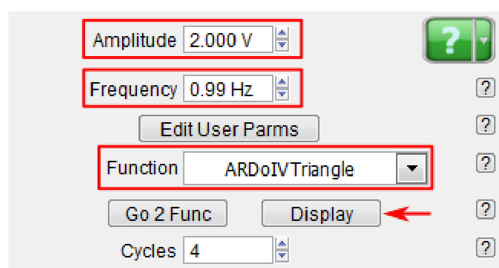
1.

Note	• Before loading your sample, you may want to first measure the resistance of the sample with an Ohmmeter. This may help to get reasonable starting values, as the resistance will normally be about an order of magnitude (10x) higher for the CAFM measurement than the macro measurement. This occurs because the number of conductive paths is much more numerous for the larger radius probe than for a ~20nm radius probe. Of course, this is not always the case, particularly for hydrophilic or metallic samples. • Once the resistance of the current loop is known, you can then estimate the bias you will need to apply by using Ohm's law, Voltage = Current × Resistance.

2. Before attempting to do I-V curves on a sample, we recommended that you first run I-V curves on the included test resistor to confirm everything is working properly (see[1]).
3. Engage the tip on the sample and acquire an image of your sample surface. For delicate samples that would be damaged or modified by Contact Mode, AC mode is generally advised to take this image.

9.7.1.1. Setting up the I-V Curve

1. Set up the bias function you want to execute on your sample.
2. **I-V curve functions:**
   - I-V curves are usually treated like periodic functions. The prewritten functions are listed near the top of the **AR Do IV Panel** in the dropdown menu labeled *Function*.
   - These functions include a triangle wave, square wave, triangle-square wave, and sinusoidal wave. You can also select "Function Editor" from the dropdown menu to create a custom form.

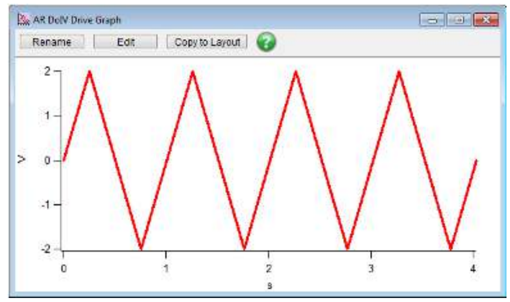


[1] *MFP-3D User Guide, Chapter: Conductive AFM (ORCA) Hardware..*

3.

Function controls:

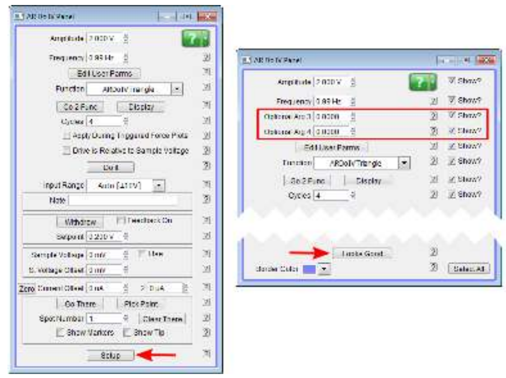
- The most convenient way to design your I-V curve is to display that function on a graph before you execute it. To do this:
  - Click the ‘Display’ button below the *Function* dropdown menu. This brings up a graph of the voltage that will be applied to the sample as a function of time.
- You can adjust the *Amplitude*, *Frequency*, and *Cycles* parameters of your function on the **AR Do IV Panel**. These allow you to control the high and low voltage and how fast that voltage will change.
- *Amplitude* gives you the maximum voltage above and below 0. For example, 1 Volt will produce a ramp that will start at 0, rise to 1 Volt, then drop to negative 1 Volt, and then return to 0.
- *Frequency* shows you how long one function cycle will last. For example, a 1 Hertz frequency will complete one full cycle in one second.
- *Cycles* determines how many full cycles of your function are repeated per execution.



4.

Additional Function controls:

- If you need to adjust other parameters, such as including a DC offset in the curve, shifting the phase so that the bias is ramped from positive to negative, or so that the ramp is only half of a curve, you can use the *Optional Arg 3* and *Optional Arg 4* parameters:
  - *Optional Arg 3*, generally speaking, controls the function phase. Phase is a ratio, where 1 is a 360° phase shift, 0.5 is a 180°, etc.
  - *Optional Arg 4*, generally speaking, controls the offset. The offset is in units of Volts. Typing in “1” will produce an offset of 1 Volt, etc.
- These parameters are hidden by default but can be revealed by clicking the ‘Setup’ button at the bottom of the **Do IV Panel**. Once set, click the ‘Looks Good’ button to save.



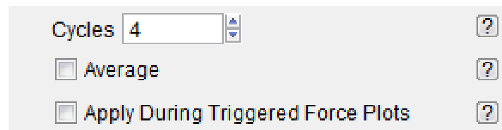
Note The “Arg” stands for “argument” and will augment different things for the different prewritten functions.

5.

Function Averaging

- Checking the *Average* box will average each cycle of the periodic function to produce a single I-V curve.

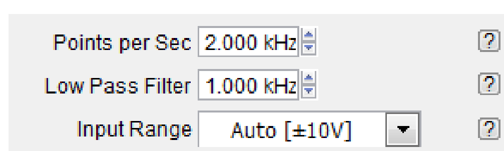
  Note Use caution with this function, as the current will often change drastically over multiple I-V cycles, and the changes will be lost in the averaging. On stable curves, averaging is a good way to improve signal to noise.
- You can also append and apply an I-V function to the end of a force curve, or a force volume map, by checking the *Applied During Triggered Force Plots* box. If this is box is checked, whenever the system performs a force curve, it will automatically execute an I-V curve during a dwell on the surface immediately after the trigger. This ultimately allows force mapping to be applied in order to produce a current spectroscopy map.



6.

Function frequencies:

- For I-V functions with frequencies faster than 20 Hz, using a triangular, square, or triangle-square drive wave is not recommended. The construction of these waves requires DACs with much higher frequencies than 50kHz. To perform I-V curves above 20Hz, instead use a sinusoidal wave as the function.
- For higher frequencies, you will typically need to increase the number of points present in the function. You can do this by increasing the *Points per Sec* parameters on the **Do IV Panel**. Increase this parameter until the function wave looks smooth on the displayed graph.

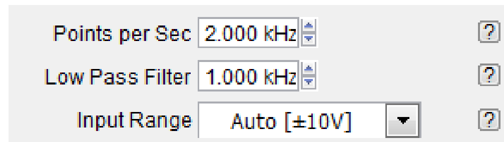


7.

Function filtering:

- The filter placed on the Analog-to-Digital Converter (ADC) that collects the current signal from the ORCA cantilever holder can also be controlled. To do this:
  - Change the value in the parameter labeled *Low Pass Filter* in the **Do IV Panel**. Values from 50 Hz to 25kHz are possible.
- For I-V curves, the system uses the 5MHz ADC.
- For normal ORCA imaging, one of the 50kHz ADCs is used.

Note You can set the digital filter to as low as needed; however, below 50Hz, artifacts may appear. Ensure that your filter value is set to at least twice the frequency of your function wave. More complex wave forms (non-sinusoidal) require much higher bandwidths to drive and acquire.



8. You can also write custom functions with the **Do IV Panel**. Custom waves use the Waves included in the **User Parm Table** that can be brought up by clicking on the 'Edit User Parms' button.

9.7.1.2. Picking points for the I-V curve

Now that you have taken your image and set up your I-V function, you can set points on that image to perform your I-V curves.

1.

Setting points:

- Select the 'Show Markers' and 'Show Tip' boxes.
- Drag the red marker to the position you want to use.
- For each location, click 'Pick Point' for the first time; for subsequent uses, click 'That's It'.

2. Once you have set all the points where you want to take I-V curves:

   a) Select the *Spot Number* you want for your first I-V curve.

b) Click the 'Go There' button to move the scanner so that point is under the tip. If you would like to see that the tip is there, check the box that reads Show Tip Location. This will put a red dot at the present location of the tip.

3. When you are satisfied that you are in a good spot:

a) Enter a *Setpoint* (on either the ARDoIV panel or on the Main panel) for the deflection.

b) Check the box labeled 'Feedback On.' This will put the tip onto the surface and turn feedback on.

4. Now you are ready to get an IV curve. If you have selected the 'Apply During Triggered Dwells' box, go ahead and take a force curve. Otherwise, click the 'Do It' button.

9.7.2. Dithering

It is optional to dither the tip before executing an IV curve. This could be useful if there is excess debris on the surface, or if you are attempting to drive the tip through oxide or contamination on the surface to get a good measure of the bulk material.

To do this, click the 'Setup' button and enable the dither controls at the bottom of the AR DoIV panel.

Figure 9.3.: The Dither function creates a spiral pattern the probe traces out. This is intended to remove debris or to score the surface in order to get through oxides, thin films, or contamination on the sample surface.

The dither itself is a spiral that starts at the point of the IV curve, moves outward to a diameter of the amplitude, and then moves back to the point where the IV curve is taken.

Dither amplitude is the size of the spiral traced with the tip.

Dither Cycles is the number of times the tip will turn within the circle to be dithered.

Dither fraction is the ratio of how many turns the tip will take on the way out before spiraling back in to do the IV curve.

9.7.3. Custom drive waves using the Function Generator

It is also possible to drive the bias with a custom wave form created in a stepwise manner using the built in function generator. This allows you to insert each step of the wave, then assign a ramp, oscillation, amplitude, and many other sets of parameters to each part of the drive wave.

1. To begin, select the "Function Editor" as the *Function* on the **Do IV Panel**. This brings up the Function Editor window with a graph and a few controls at the top (see 9.4). A default segment is shown that can be modified using the controls at the top of the graph. The time for the segment, the start and end amplitude, and modulation can all be added. The static controls drive the overall shape of the wave, and the dynamic can be used to add an AC dither to the segment.

2. Whatever segment is red on the graph is the segment that is being modified by the Segment Parms at any given time. Click on a segment to select it.

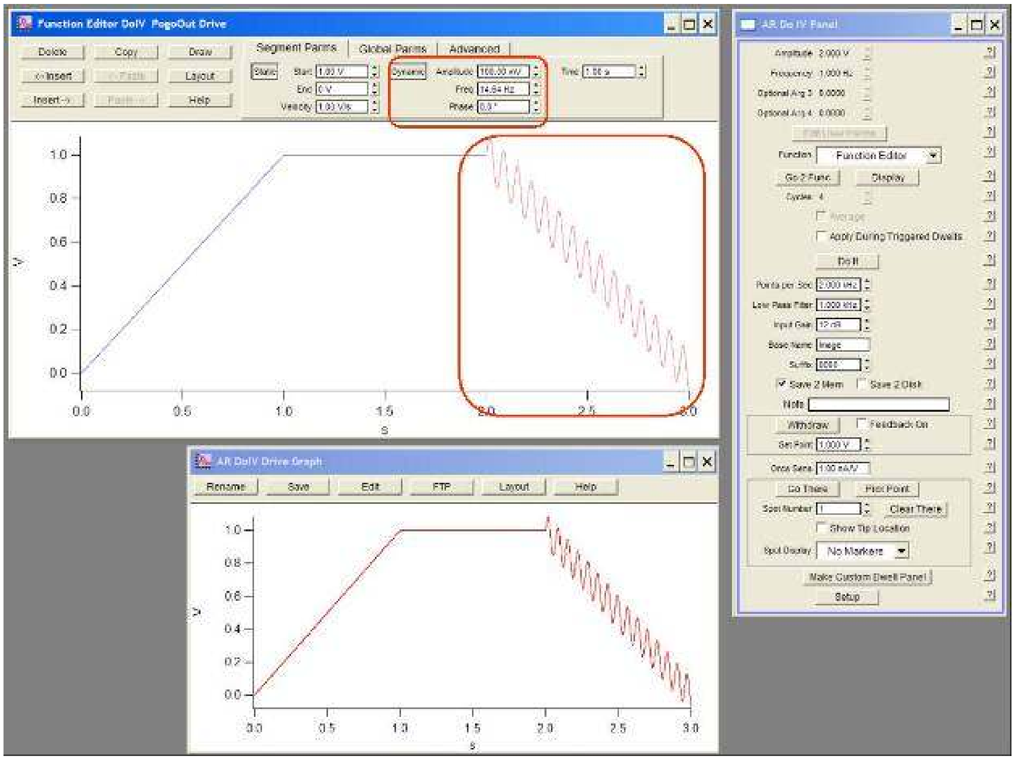


Figure 9.4.: The Function Editor can be used to make a variety of drive waves for current measurements. You can insert new segments using the Insert button, select different segments by clicking them., and modify each independently. The Segment Parms (parameters) tab is shown here.

3. On the Global Parms tab (see 9.5), the entire drive wave can be modified with a multiplier, and offset, or a sine wave dither. By combining local dithering with global dithering, it is even possible to drive the wave with two sine waves.
4. The buttons on the upper left of the Function Editor can be used to insert, delete, copy, paste segments, and you can even draw segments by hand using the 'Draw' button.
5. On the Advanced tab, you can click the 'Save' button to save the drive wave for later use.

9.7.4. Viewing I-V curves

I-V curves are treated like force curves for viewing. The actual data is treated as a dwell on a force curve. See Figure 9.6 on page 131.

It is often useful to display the current and bias data as a function of time, especially for multiple curves taken consecutively, and those taken at frequencies higher than 20 Hz. To do this, under

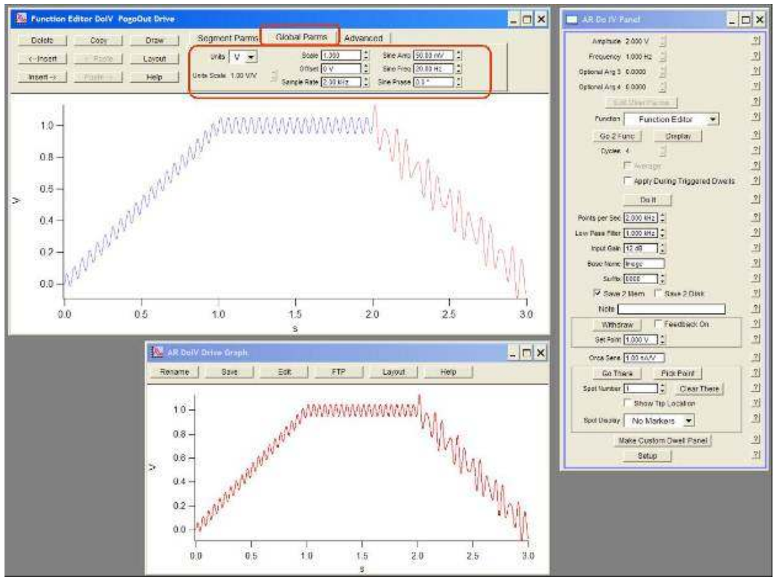


Figure 9.5.: Use the Global tab to modify all the individual segments at one time. Of particular use in this section is the *Scale* field. This function allows you to scale up or down the entire wave function you have created. For example, if you had a custom wave from 0-1V with a dwell, scaling it by 5 would make it go from 0 to 5V.

Axis 1 choose 'current' as the data type and under Axis 2 choose 'bias' (see Axes Data Types). Then choose time as the X axis see Force X axis).

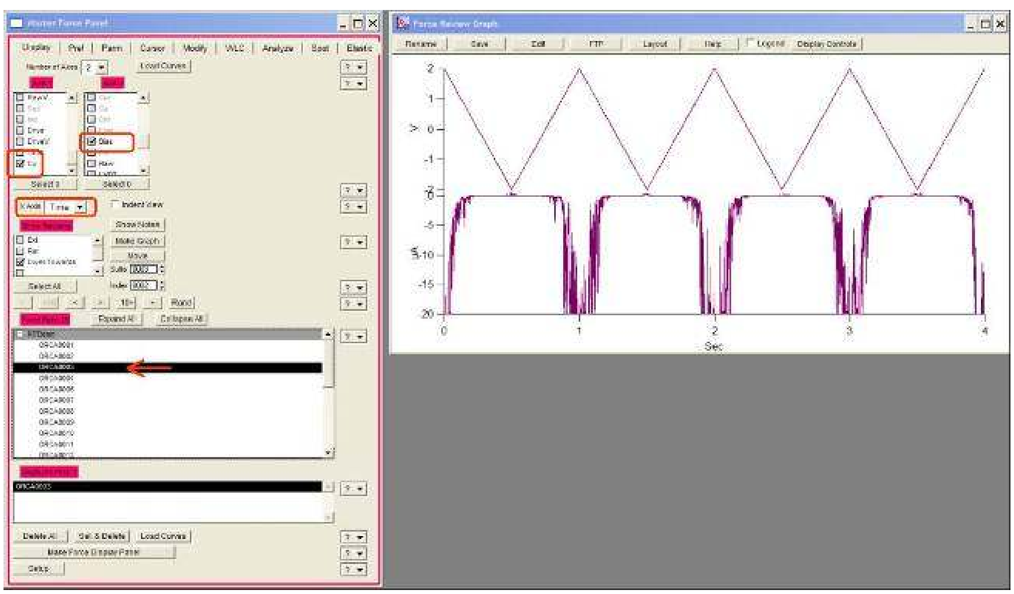


Figure 9.7.: Current vs time. It is often useful to display the current vs time, particularly for samples that degrade or improve in conductivity with time. Si will oxidize as more current is passed through it if there is moist air near the surface.

If you select the last IV curve in the list of loaded curves, then the force panel will automatically move to the next curve as it is taken so that you can view your I-V curves in real time. Simply

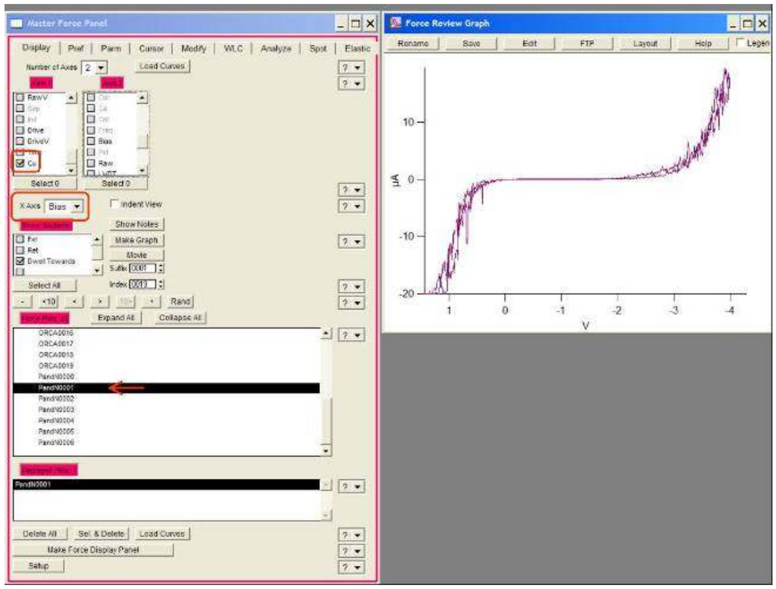


Figure 9.6.: Current vs. Bias. To display IV curves, open the force review panel, view current (and current2, if you have a Dual Gain ORCA), set the *X Axis* to "Bias", and then select the dwell towards under the sections window. If the graph does not appear, click 'Make Graph'.

select the last curve, then on the **AR Do IV Panel**, click 'Do It', and the force display panel will jump to the newest curve.

9.7.5. Correcting Offsets on I-V curves

The DACs on our system do not have built-in analog adjustments to remove small voltage offsets in their output. To correct for this while running CAFM measurements, we have two simple tools: One is a sample voltage offset, and the other are current offsets for every ORCA channel that is being captured. For Single Gain ORCA modules, this will be a single channel with a single sensitivity setting. For the Dual Gain ORCA, there are two separate channels with different sensitivities. Each channel has a different offset adjustment to compensate for offsets in both the voltage drive and the current measurement.

The best way to adjust these is to first measure the output from the PogoOut wire that hangs down from the ORCA holder to bias the sample. Use a multimeter to measure this and adjust the S. Voltage offset until it reads zero.

Then click on the 'Zero' button, which adjusts the current offset to get zero current when there is no bias on the tip.

Figure 9.8.: Bias and current offsets from the DoIV panel. These can be used to correct for small variations in the outputs of our DACs and the transimpedance current to voltage converter that we use to measure current passing through the sample.

10. Nap Mode

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

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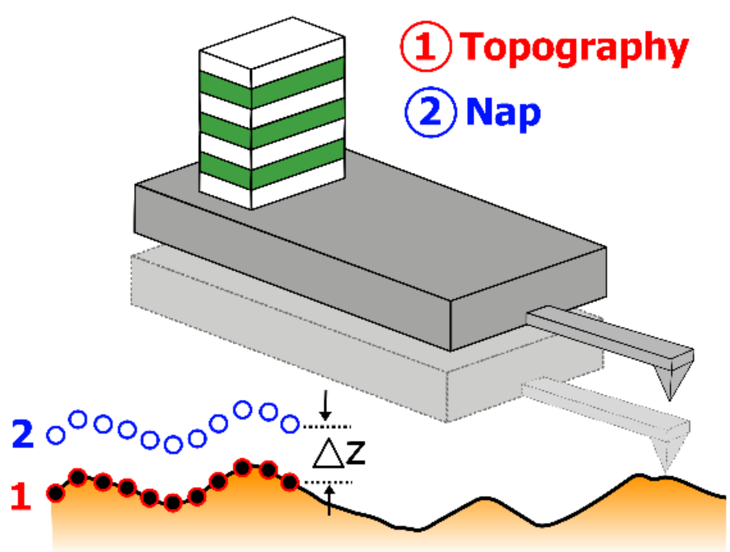


Figure 10.1.: During Nap mode, the probe is first scanned across the sample surface, then raised to a delta height defined by the user. This allows for detection of long-range forces between the tip and sample.

Nap mode uses two panels in addition to the normal controls for standard AC mode imaging.

- **Nap Panel**: Contains all the controls needed to run conventional EFM, MFM, SKPM, and other two pass techniques. See Chapter 12 on page 147, Chapter 13 on page 155, and Chapter 11 on page 136 for more these techniques. The Nap Panel can be accessed by selecting *AFM Controls > Nap Panel* (Ctrl + 9) in the menu.

- **Nap Channel Panel**: Allows the user to set display settings for the Nap images; much in a similar fashion to the main Channel Panel. The Nap Channel Panel can be opened directly from the Nap Panel or from the menu *AFM Controls > Nap Channel Panel*.

Nap mode is a fairly simple technique in principle, as it is fundamentally a two-pass technique. First, a topographical trace and retrace pass are made on the sample surface (the surface pass). This is a normal topographic scan that can be done in any imaging mode, though it is most typically done in AC mode. The second trace and retrace pass is the Nap pass. and the method and specifics of this pass are set by the parameters in the Nap Panel.

10.1. Nap Modes

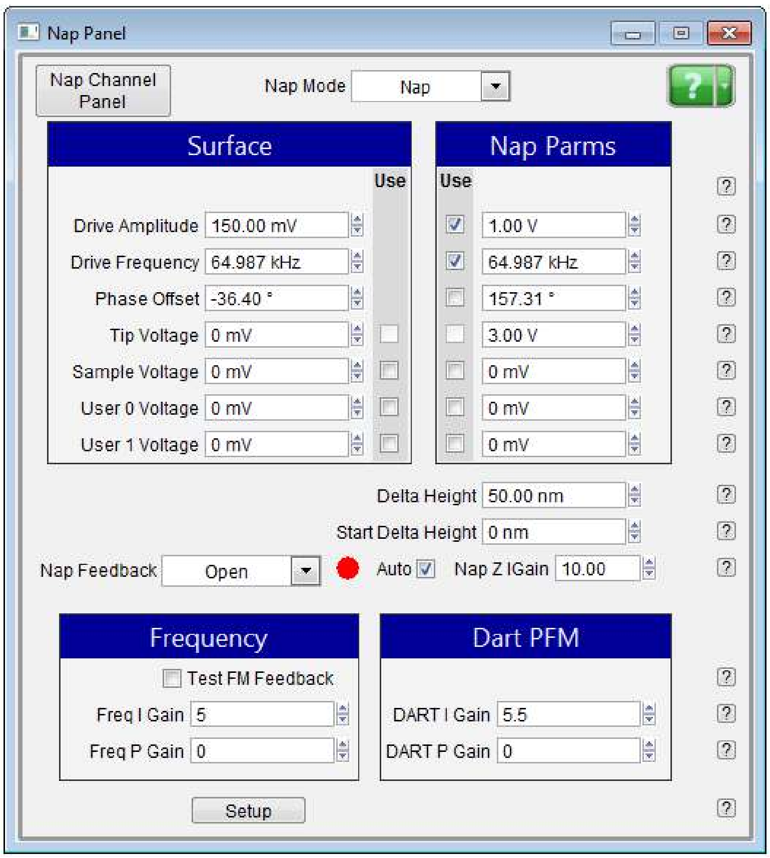


Figure 10.2.: Nap Panel, opened from the menu: *AFM Controls > Nap Panel*(Ctrl + 9)

The Nap mode is set at the top of the Nap Panel and can be set to any of the following:

- **Off**: Nap mode off disables Nap mode, and performs a normal ordinary scan, with a single pass for trace and retrace.
- **Nap**: Standard Nap mode. The system scans twice for every scan line as explained above see 10. The Nap passes are above the surface, as specified by the *Delta Height*. The standard Z feedback loop is off during the Nap pass. Most two-pass techniques use this Nap mode (see Chapter 12 on page 147, Chapter 13 on page 155 and Chapter 11 on page 136).

- **Parm Swap**: This is an interleaved scan but leaves the standard imaging mode Z feedback loop running for both passes. It may change the parameters of that loop (*Setpoint*, *Drive Amplitude*, etc.), but there is no delta height used in this mode.
- **Snap**: This refers to linear, or "simple" Nap mode. This is the same as Nap mode, but instead of following a set height above the sample, it follows the slope of the sample. The surface pass data is fit to a line, an offset is put into that slope, and that is what is followed during the Nap pass. This is generally used when testing theories of long-range interactions, by comparing Nap and Snap data.

10.2. Parameters

The following parameters can be switched or toggled between two different states: one value for the surface pass, and a different value for the Nap pass.

- Integral Gain (for Parm Swap only)
- Proportional Gain (for Parm Swap only)
- Setpoint (for Parm Swap only)
- Drive Amplitude
- Drive Amplitude1 (for Dual AC only)
- Drive Frequency
- Drive Frequency 1 (for Dual AC only)
- Phase offset
- Tip Voltage
- Sample Voltage
- User 0 Voltage
- User 1 Voltage

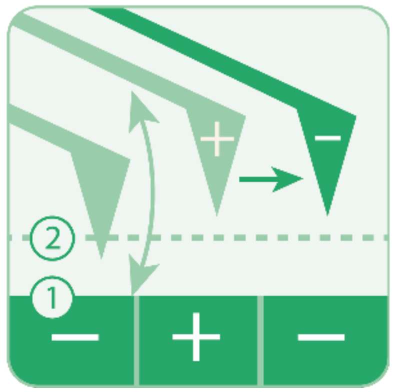
To select one of these, check the box in the column marked *Use* to the left of each of the items in the Parm Swap column.

As an example, consider that you want to compare attractive and repulsive imaging (see Section 5.3 on page 48) with a single Nap scan. You could use Parm Swap to image repulsively in one pass and attractively in the next.

1. Set the *Nap* mode to "Parm Swap".
2. Swap the *Drive Frequency*.
3. Set the Surface pass to be on the low side of the resonance.
4. Set the Nap pass to be on the high side of the resonance.
5. Swap the *Setpoint*.
6. Set the Surface pass to a setpoint of 75% of the free air amplitude.
7. Set the Nap pass to a setpoint of 85 - 90% of the free air amplitude.
8. You may also want to swap the *Drive Amplitude* and drive less on the Nap pass, see Section 5.3.1 on page 49. This will require you to adjust the setpoints accordingly.

11. Scanning Kelvin Probe Microscopy (SKPM)

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

11.1. Introduction

Scanning Kelvin Probe Microscopy (SKPM) is a technique that detects the potential difference between the probe tip and the sample. A few synonyms for this technique include:

SKPM Scanning Kelvin Probe Microscopy, which is used in this chapter, as it seems to be a widely used and accepted descriptor for this technique.

KPFM Kelvin Probe Force Microscopy, another widely used and accepted descriptor.

SSPM Scanning Surface Potential Microscopy

SKFM Scanning Kelvin Force Microscopy

SPM Surface Potential Microscopy

SP-AFM Surface Potential Atomic Force Microscopy

The term “Kelvin force” refers to similarities between this microscopic technique and the macroscopic “Kelvin probe” method. Although the methodology of the microscopic technique is somewhat different, the measured value is equivalent. For clarity, this note will refer only to the microscopic technique hereon as SKPM.

11.2. Principles

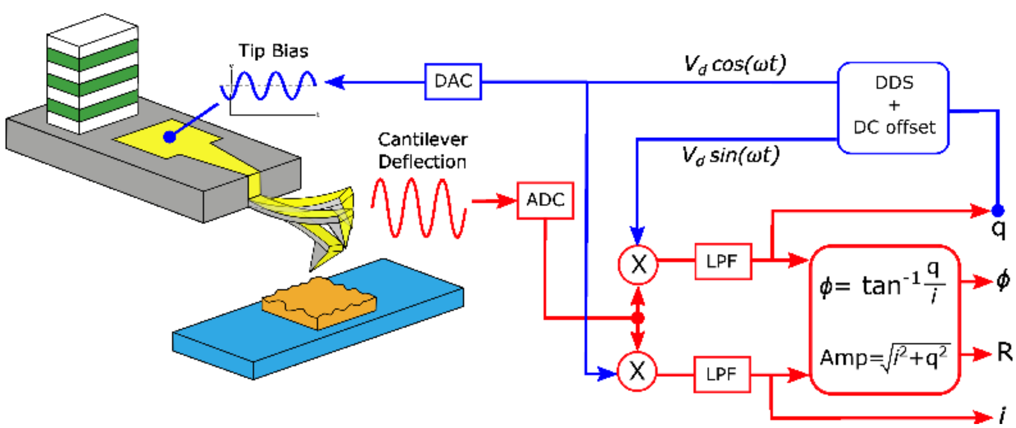


Figure 11.1.: Schematic showing how SKPM is performed on Asylum Research AFMs. The probe tip is driven electrically with an AC bias. The potential difference between the tip and the sample causes the probe to mechanically oscillate. These mechanical oscillations are then negated by a feedback loop on the bias. The voltage required to negate the oscillations (i.e., match the probe to the sample) is captured and recorded as the surface potential channel in the AFM software.

This technique relies on an AC bias applied to the tip to produce an electric force on the cantilever that is proportional to the potential difference between the tip and the sample. During the Nap pass, there is no mechanically induced drive, such as with a drive piezo in standard AC mode imaging or in Electric Force Microscopy. The only oscillations that the probe will have will be induced by an applied AC bias. Figure 11.1 on page 137 shows a schematic diagram of the setup for the technique.

An AC bias applied between the tip and the sample produces an electrostatic force between the two. If they are modeled as a parallel plate capacitor, then the force between the two plates is proportional to the square of the applied voltage:

$$F = \frac{1}{2}\frac{\partial C}{\partial z}V^2 \tag{11.1}$$

The total potential difference between the probe and the sample is the sum of the applied AC bias (V_{ac}), the potential difference we are trying to measure (V_{sp}), and any DC voltage we wish to apply (V_{DC}).

$$V = (V_{DC} - V_{sp}) + V_{ac}\sin(\omega t) \tag{11.2}$$

We need to square this to go into Equation 11.1 on page 138.

$$V^2 = (V_{DC} - V_{sp})^2 + 2(V_{DC} - V_{sp})V_{ac}\sin(\omega t) + V_{ac}^2\sin^2(\omega t) \tag{11.3}$$

and from

$$\sin^2\theta = \frac{1}{2}(1 - \cos(2\theta)) \tag{11.4}$$

We have

$$V^2 = (V_{DC} - V_{sp})^2 + 2(V_{DC} - V_{sp})V_{ac}\sin(\omega t) + \frac{1}{2}V_{ac}^2(1 - \cos(2\omega t)) \tag{11.5}$$

which shows the important point, that there are DC, $1\omega t$ and $2\omega t$ components to the signal. Putting this into 11.1 and a little rearranging gives us:

$$F = \frac{1}{2}\frac{\partial C}{\partial z}\left(\left[(V_{DC} - V_{sp})^2 + \frac{1}{2}V_{ac}^2\right] + 2\left[(V_{DC} - V_{sp})V_{ac}\sin(\omega t)\right] - \left[\frac{1}{2}V_{ac}^2\cos(2\omega t)\right]\right) \tag{11.6}$$

Note the 2F component may have a significant force, but it is typically not enhanced by the resonance of the cantilever, meaning that the cantilever does not respond to that frequency.

The main point to SKPM is the middle part of this equation, which shows you will minimize the force (amplitude) if $V_{DC} - V_{sp} = 0$ or $V_{DC} = V_{sp}$.

Which is exactly what SKPM is, using a feedback loop to adjust the DC bias on the lever to minimizing amplitude.

In practice there are multiple contributors to the DC potential difference:

- Difference in work function between the tip and surface
- Trapped charge
- Any permanent or applied voltage between the tip and the sample

For these reasons, the technique is generally considered a pseudo-quantitative technique, in that it gives an accurate measurement of the potential difference, but that number likely has multiple contributors.

11.3. Methodology

11.3.1. Probes

The probes needed for SKPM must be electrically conductive, as a bias must be applied to the tip - insulating probes will not work. There are many probes available, however a good probe to first try would be the AC240TM-R3, see the Probe Store for other Conductive Probes as well.

11.3.2. Two-Pass Method

SKPM is a technique that uses two separate passes per scan line. This two-pass method on an Asylum Research instrument is called **Nap Mode**. The first pass is used to determine the topography of the surface, which is done exactly like a standard **AC Mode** scan line. The second pass, in this technique, has the tip raised at a fixed distance above the determined topography and is used to measure the surface potential. See Chapter 10 on page 133 for more details on this method.

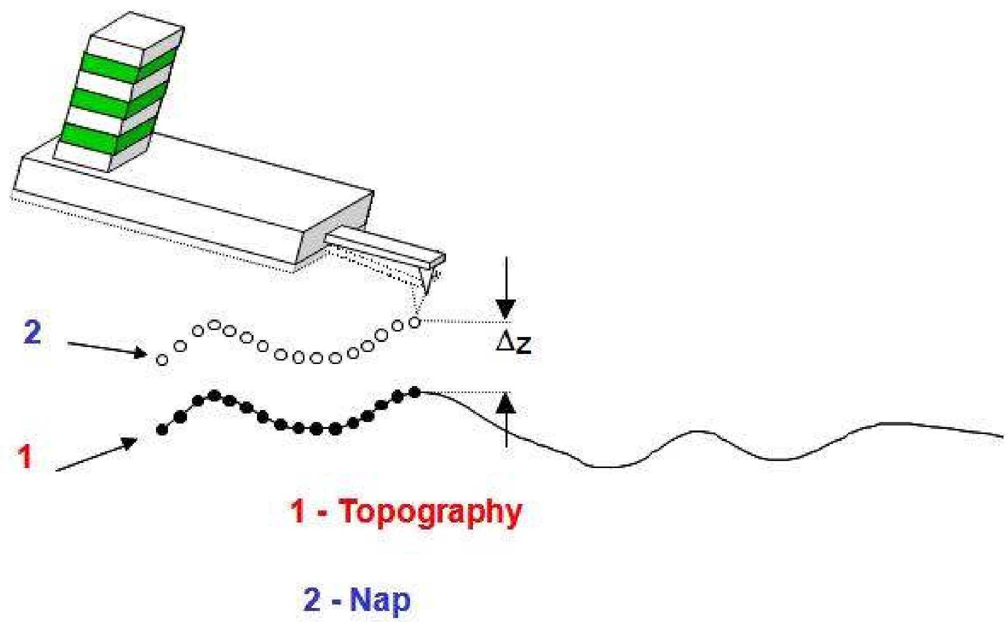


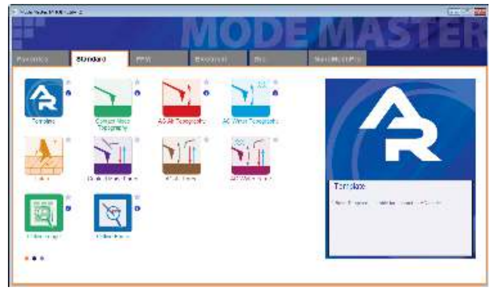
Figure 11.2.: Schematic of Nap Mode. For each scan line, the system captures the topography. The system then retraces that topography on the same line to keep a constant distance from the sample surface.

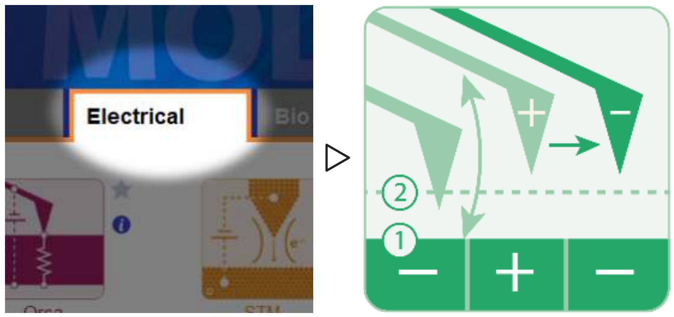
11.4. How to Guide

Note that this instruction set assumes the user is already familiar with standard AC Mode imaging, see Chapter 4 on page 34.

1. **The Mode Master window:**

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



2. 

Select mode:

- Select *Electrical > SKPM*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

3. Mount and load the sample. Insulating and semiconducting samples need not be grounded; however, metal samples should be grounded to signal ground. This can be either the pogo out wire that drops down from the probe holder or one of the grounds on the BNCs of the ARC2 controller. A lack of ground can cause arbitrary offsets in the potential that may mask the surface potential/work function between the tip and the sample.

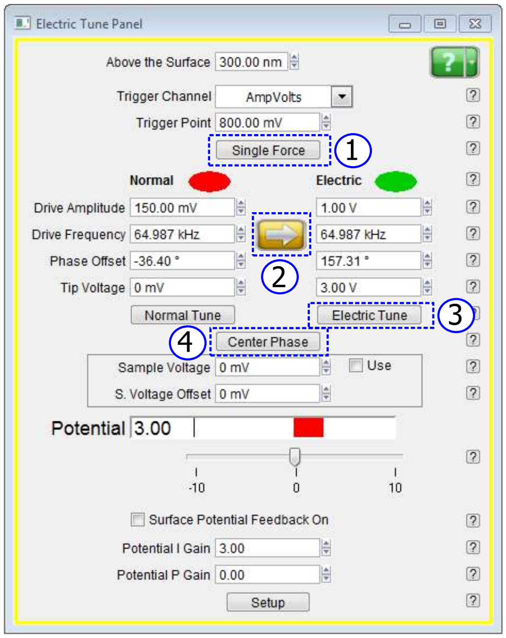
4. Load an electrically conductive AC Mode probe into the cantilever holder and attach to the AFM.

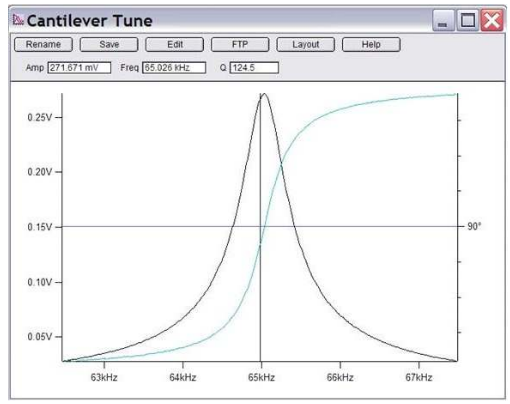
5. Tune the probe as described in the AC Mode tutorial.

6. Open the Electrical Tune Panel (*AFM Controls > Other > Electric Tune Panel*). This panel allows the user to find the resonance frequency and phase of the probe when it is driven electrically close to the sample surface. This electrical drive phase will often be considerably different than that of the same probe driven mechanically with the Shake piezo and is very important for the correct determination of surface potential. Note that this panel is only enabled while in AC imaging mode.

7. 'Engage' the tip on the sample surface while in AC mode as if doing standard AC mode AFM.

8.





Electric Tune Panel with the 'Single Force' (1), 'Drive Copy' (2), 'Electric Tune' (3) and 'Center Phase' (4) buttons highlighted. The graph on the right is a properly electrically tuned cantilever.

9. At the top of the Electric Tune Panel set the *Above the Surface* parameter to "300nm". Set the *Trigger Channel* to "AmpVolts", and the *Trigger Point* to "800mV". Click the button marked 'Single Force'. When the force curve is finished, the tip will be positioned 300nm just above the surface.

10. Click on the yellow arrow button (➡) in the center of the Electric Tune Panel to copy the *Drive Frequency* from the Normal (mechanical) tune column over to the Electric tune column *Drive Frequency* field.

11. Click the 'Electric Tune' button. This will sweep an AC bias at the tip of the probe and show the Amplitude and Phase response in the tune graph. The resonance frequency should be very close to that of the normal/mechanical tune. Ensure that the *Tip Voltage* parameter is set to 3.0V under the Electric column and that you have at least 500mV in the *Drive Amplitude* under the Electric column. Now click the 'Center Phase' button. The software sets the phase properly so that the feedback loop can function properly. These buttons are shown in Step 8 on page 141.

Note	The AFM software assumes that the tip is at a positive potential relative to the sample. If this assumption is false, then the *Phase* from the Electric tune will be 180° off. For samples with very high potential offsets, it may be necessary to set the *Tip Voltage* higher than 3.0V. The easiest way to check if the sample has too high of a voltage applied is to tune everything as described, then change the sign of the probe tip to -3V. The phase should flip 180°. If the surface potential of the sample is higher than the 3V offset on the probe, the phase will not flip. Repeat as nedded.

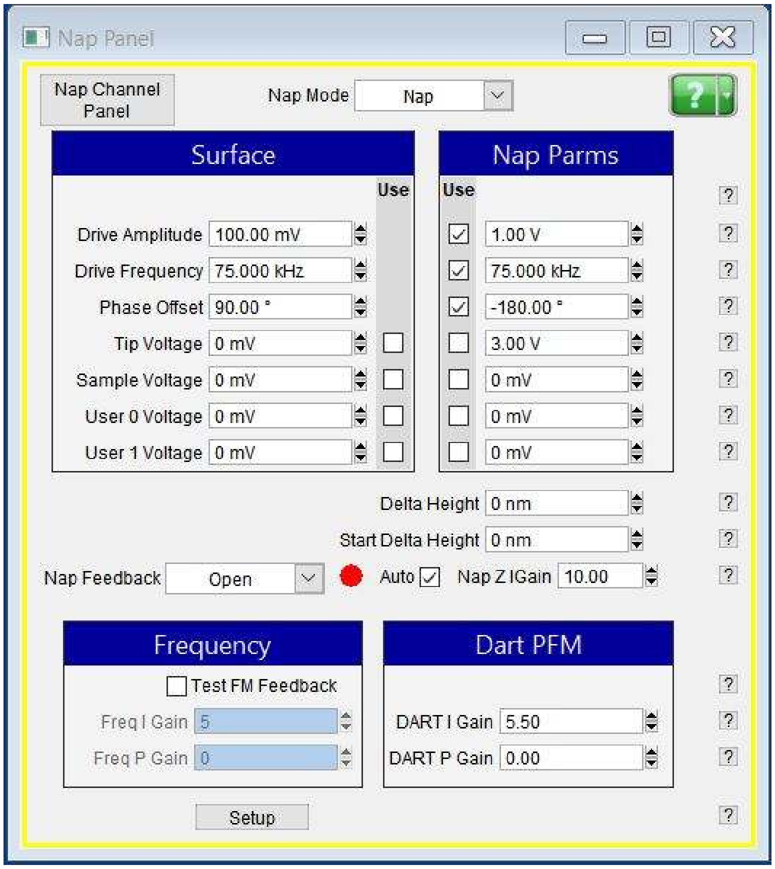


Figure 11.3.: Nap Panel configured for SKPM scan. The values in the parameters may vary depending on your tip and sample.

12. Open the Nap Panel (*AFM Controls > Nap Panel* (Ctrl+9)).

13. Select *Drive Amplitude*, *Drive Frequency*, and *Phase Offset* under the Swap column. See Figure 11.3 on page 142

14. Set the *Nap Mode* to “Nap”.

15. Set the *Delta Height* parameter to “0 nm”. This will be lowered later, if you want to try to get better lateral resolution during your image.

Note	The reference height is based on the center point of the cantilever oscillations, and the potential feedback loop will maintain the cantilever amplitude at zero during the potential scan; because of this, it is actually possible to enter a value lower than 0nm as a *Delta Height*. However, if a value that is too low is entered, the tip will strike the surface, and you will have a poor image.

16. Open the Nap Channel Panel (*AFM Controls > Nap Chanel Panel*) or click the ‘Nap Channel Panel’ button at the top-left of the Nap Panel. See Figure 11.3 on page 142

17. Make sure “Potential” is enabled as a channel in the Master Channel Panel (Ctrl+7). Enabling this will automatically turn on the Potential feedback loop when scanning. Be sure that the

Nap Potential channel (set on Nap Channel Panel) have *Real Time* and *Save*set to **“**None” under Image Modification.

18. On the Image tab of the Master Panel (Ctrl + 5), set your *Scan Speed* and *Scan Size* to appropriate values for the sample you are imaging. Slower scan speeds of 0.25-0.75Hz typically yield the best results in SKPM.

19. Begin the scan.

20. Look at the Potential channel data. If the trace and retrace do not match, but seem to be tracking roughly, raise the *Potential I Gain* and *Potential P Gain* on the Electrical Tune Panel. If the Potential data appears unstable or chaotic, try lowering these gains quite a bit. A good starting value is around “3” for I Gain (integral) as well as “3” for P Gain (proportional).

21. To slightly improve lateral resolution of the image, try lowering the *Delta Height*. Generally, negative delta heights produce the highest lateral resolution.

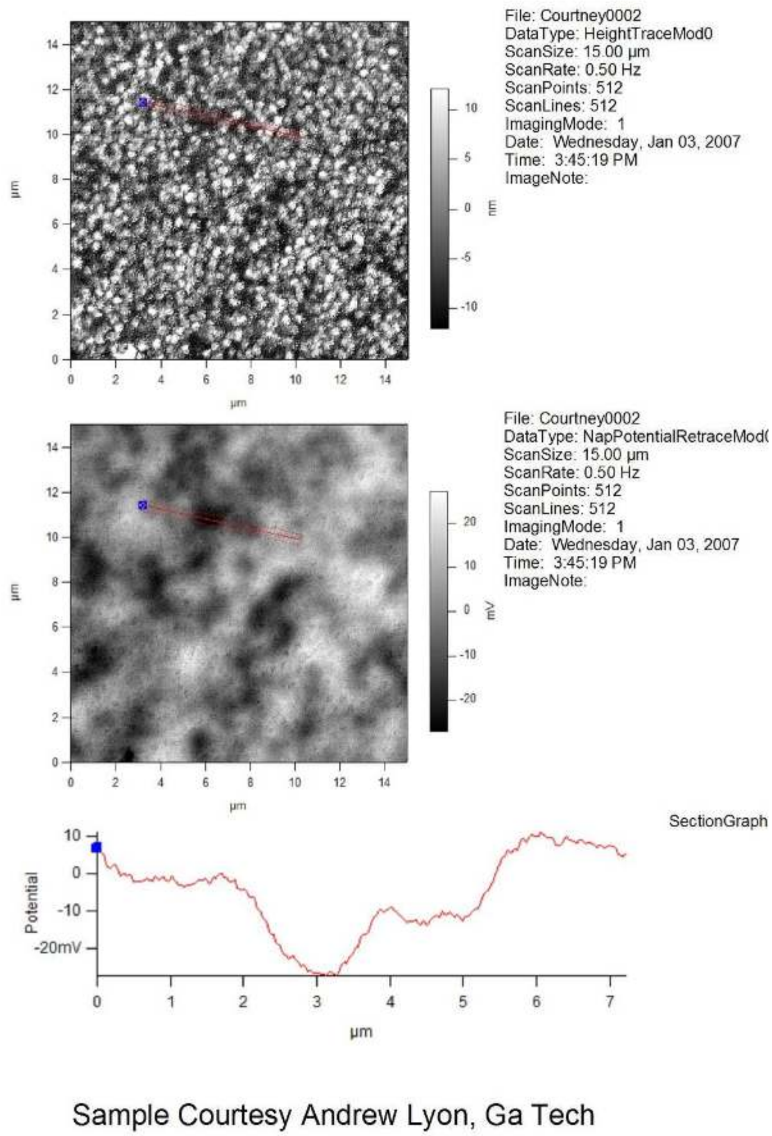


Figure 11.4.: Height (top) and Surface Potential (middle) image of a three-layer microgel sample. The section (bottom) shows three discreet potential levels on the sample, which is mostly likely attributable to incomplete coverage of the three different microgel layers. (Sample and images courtesy of C. Sorrell and A. Lyon, Georgia Institute of Technology.)

11.5. Single Pass Method

Single-pass SKPM is possible on the Cypher, Infinity, and Jupiter systems. In general, the probe is tuned as for standard AC mode, and a second AC voltage is applied directly to the probe. Any mismatch in the surface contact potential difference (CPD) causes the probe to oscillate at the frequecency of that second drive signal. The software then detects this in the form of an amplitude and phase and cancels that oscillation with a DC potential. This feedback loop operates much like the potential feedback in the Two-pass method. However, the potential feedback is applied either below the resonance or on the second resonance. Either are possible, and the choice falls on the user.

In general, off-resonance SKPM offers slightly lower CPD resolution, and on-resonance or 2nd-resonance SKPM produces higher CPD resolution but can add artifact caused by shifts in the resonance frequency. This frequency shift is caused by variations in the tip-sample interactions as the probe tracks the surface.

11.5.1. How to Guide

Note	The single-pass option is only possible on the Cypher, Jupiter, and the MFP-3D Infinity

1. Load the *Single Pass SKPM* template under the electrical tab of the Mode Master template.
2. Load an electrically conductive AC mode probe into the cantilever holder and attach to the AFM.
3. Tune the cantilever as described in the AC Mode tutorial.
4. Engage on the surface as described in the AC Mode tutorial. In general, the SKPM feedback loop is easier to set up in attractive mode, but imaging single-pass SKPM is possible in both repulsive (phase below 90°) and attractive mode (phase above 90°).
5. Click the button labeled 'Tip Voltage Offset Tune'.
6. Look at the graphs. They should look like the image in Figure 11.5 on page 146. The tune is meant to put all of the probe response to the AC drive voltage into the blue trace, which is the input q or the y output from the lock in. This is needed so that the system knows whether to apply a positive or negative DC voltage to the probe to match the potential of the surface.

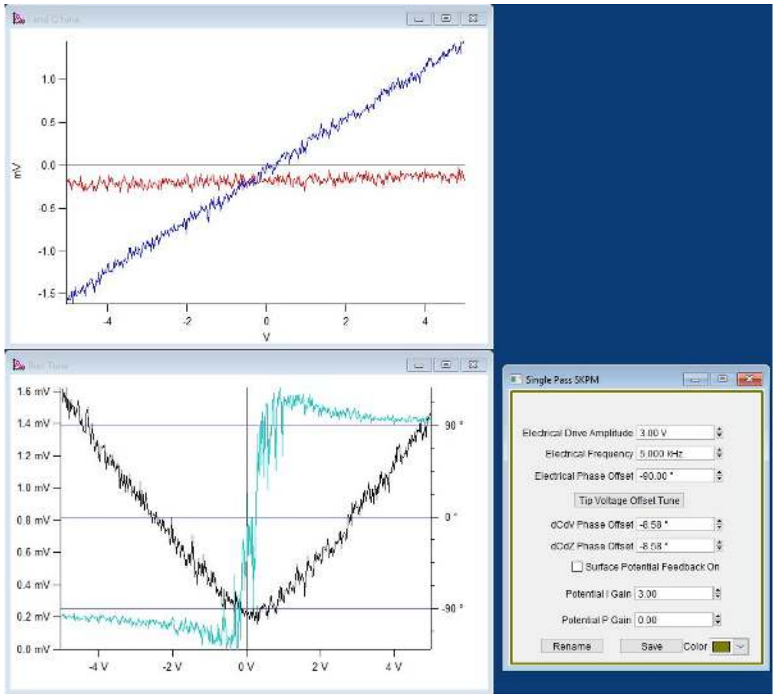
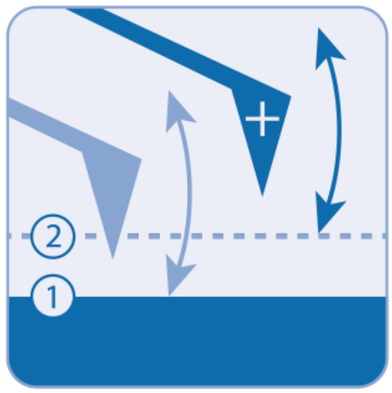


Figure 11.5.: The surface tune panel and graphs. Note that the blue trace (input q), has a slope that increases with increasing voltage, and the red trace (input i) is flat. This shows that the probe response has been pushed into the input q channel. The bottom graph shows the amplitude and phase response of the probe with a ramped voltage. The potential feedback loop uses the phase to set the sign of the DC potential voltage to the tip, and the amplitude is used to set the magnitude of that DC potential.

7. If the red curve (input i or x output from the lock in) has any slope to it, adjust the *Electrical Phase Offset* until the red curve is flat and the blue curve has maximum slope.
8. Note that on the minimum of the amplitude trace (black trace), you will see an offset on some samples. This is the surface contact potential of the sample relative to that of the probe tip.
9. Begin imaging your sample. Slowly raise the *Potential I Gain* and *Potential P Gain* until trace and retrace of the potential channel match.

12. Electrostatic Force Microscopy (EFM)

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

12.1. Introduction

Electric Force Microscopy (EFM) is relatively simple in concept and operation, but often requires some considerable thought in interpretation. The technique oscillates the tip at its resonance frequency above the surface while a direct current (DC) bias is applied between the tip and the sample. The electric field between the tip and the sample creates a force gradient between the two that causes a shift in the resonance frequency of the cantilever. This shift in resonance in turn causes a shift in the phase lag between the drive and response of the cantilever. By monitoring the phase shift, you can find areas under the tip that perturb the electric field between the tip and the sample.

EFM is the technique most commonly used on samples that are a mix of conductive and non-conductive areas. EFM tends to show contrast where the conductivity changes dramatically.

Conceptually, you can consider that the electric field lines between the tip and the sample take the shortest possible path to the ground plane.

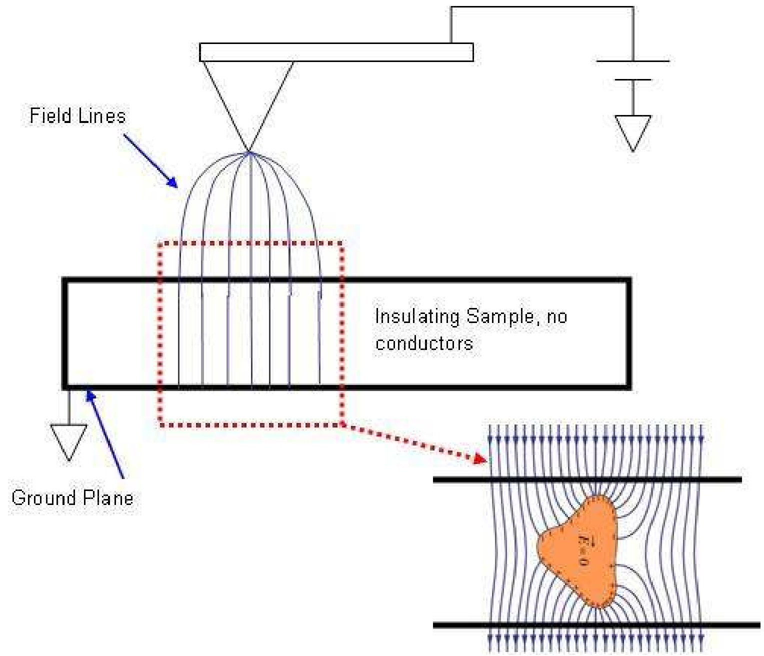


Figure 12.1.: This schematic shows how the introduction of a conductor into the electric field between the tip and sample can affect the field lines. The shift in the gradient due to the presence of the conductor causes a shift in the resonance frequency of the cantilever, and therefore a shift in the phase and amplitude, which are then collected as the EFM data. The phase indicates whether the shift is attractive or repulsive. Attractive forces show a shift in the phase to higher than free air value. Repulsive forces shift the phase below the free air value.

When a conductor is introduced into the electric field (see Figure 12.1 on page 148), the lines move perpendicular to the surface of the conductor in order to make the electric field inside the Gaussian surface zero. Any change in the field lines causes a change in the field gradient, which in turn changes the force on the lever, which shifts the resonance frequency. This is observed as a shift in the phase, as the drive frequency is now driving off resonance. This is particularly apparent when dealing with conductors in an insulating matrix. For the opposite of that, insulators in a conducting matrix, the only contrast can be achieved when the insulators are on the surface, and that phase shift is minimal and often difficult to detect. If the insulating particles are below the conductive surface, then they cannot be detected by EFM (see Figure 12.2 on page 149).

12.2. Theory

EFM relies on electrostatic forces between the tip and the sample to cause a shift in the phase lag of the cantilever that is driven mechanically. The tip-sample interaction is most often treated as parallel plate capacitors. Through the fundamental equations on capacitance, the force acting on the capacitor plates is given by:

The electric field is decoupled from topography by performing two scans over each scan line.

$$F = \frac{1}{2}\frac{dC}{dZ}V^2 \tag{12.1}$$

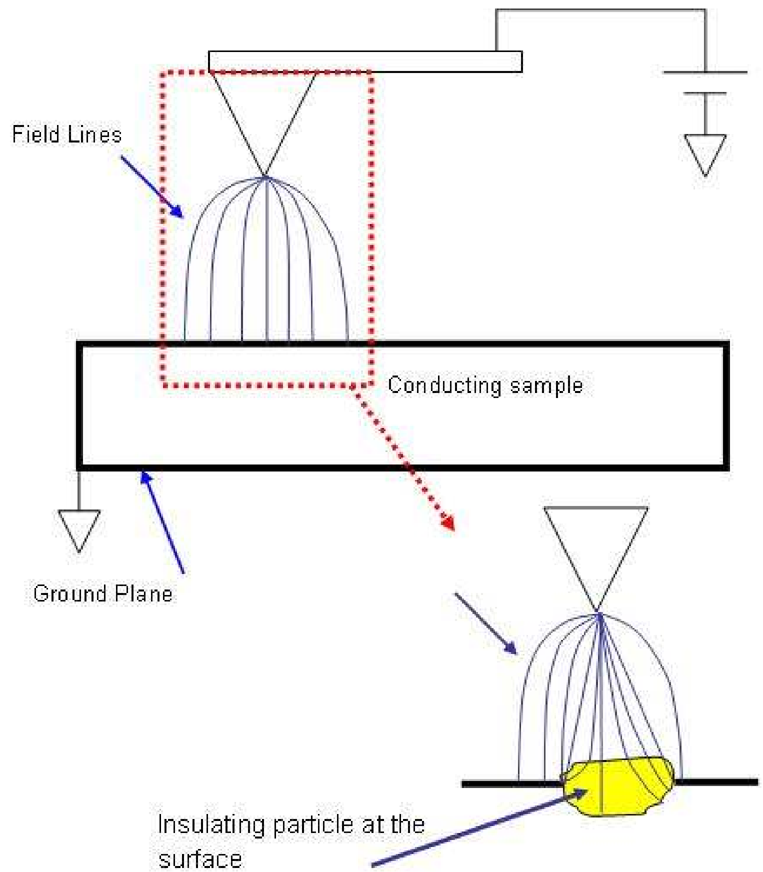


Figure 12.2.: Effects of introducing an insulator into a metal sample. EFM is generally not suitable for this sample configuration, as the field lines show little shift. Any shift would be difficult to discern from the changes in topography.

The force depends on the capacitance and the applied voltage. The capacitance depends on the geometry of the capacitor(s), meaning the location, size, conductivity, dielectric constant, etc. of the area between the tip and the ground plane. For example, in Figure 12.1 on page 148, before the conductor is introduced into the insulating sample, the capacitor can be considered to be the tip to the ground plane, with two dielectrics between. One is the sample, and the other is the air between the tip and the sample (of course this is much more complicated if you consider the contamination layer). When the conductor is introduced, you now have essentially two capacitors: the tip to the conductor and the conductor to the ground plane. Areas where there is no conductor will have a different dC/dV than areas where conductors exist.

It is also possible to get contrast due to the presence of charge, but this is normally very faint compared to the contrast due to the capacitor geometry under the tip. For samples that have varying charge, a much better technique to use is scanning Kelvin probe microscopy (SKPM), described in detail in Chapter 11 on page 136.

EFM is a two-pass technique. In the first pass, the normal topography is taken. On the second pass, the tip is raised above the surface and a bias is applied between the tip and the sample. Phase data is collected to show the electrostatic induced shift in resonance frequency. A general illustration of nap mode is shown in Figure 10.1 on page 133. This is done to maintain the separation between

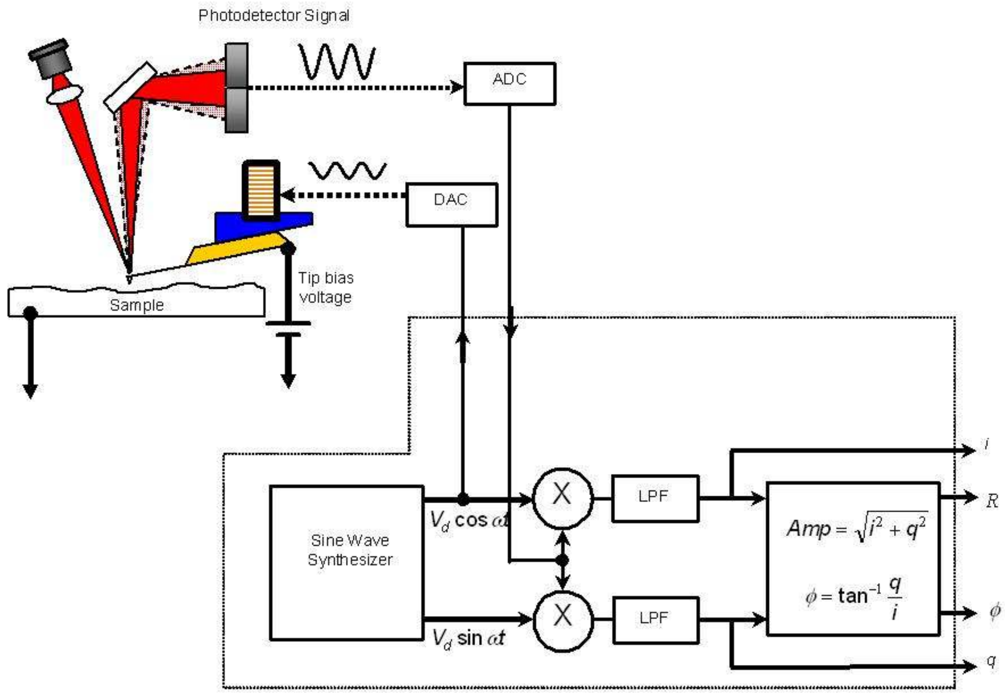


Figure 12.3.: Schematic of Electric Force Microscopy. The Probe is oscillated both during the main scan and during the nap scan. During the nap scan, the probe is lifted off the surface, and a bias applied between the tip and sample to show the location of areas that are conductive or strongly charged.

the tip and the sample constant while collecting the EFM data. You note from equation 12.1 that the force is dependent on changes in Z.

12.3. Nap Mode

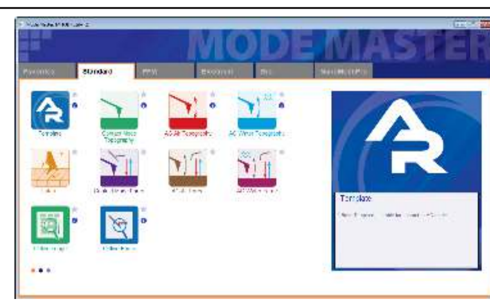
Nap Mode uses two panels in addition to the normal controls for standard AC mode imaging. For more details on Nap mode, refer to 10.

12.4. How to Guide

1.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



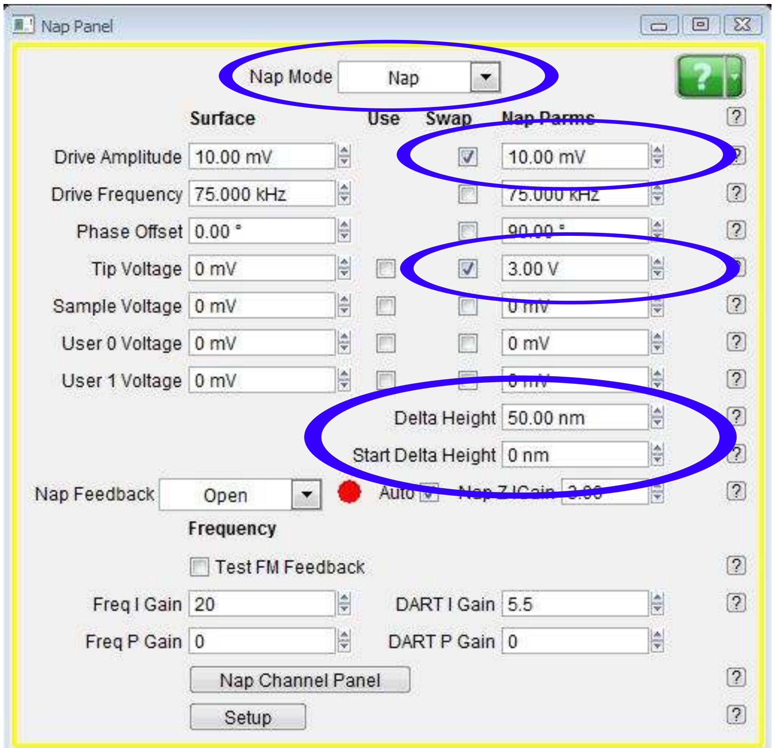


Figure 12.4.: Here the Nap Panel is set up for EFM operation. The relevant fields have been circled. The "Use" column in the middle left has check boxes that, when checked, cause that parameter to be used during the main scan. The "Swap" column has check boxes that, when checked, allow those fields to be used during the parm swap/ nap scan.

2.

Select Mode:

- Select *Electrical tab > EFM*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

3. Load the sample as usual, but if possible ground the sample; or if it is an insulator, mount the sample to a good ground plane, such as a gold coated slide or a metal sample puck. Though EFM can be done on an ungrounded sample, it is usually preferable to have the sample grounded. This is because the field lines emanating from the tip have a higher density if the ground plane is flat and closer to the tip. It is the perturbation of the field lines that cause the phase shift in the EFM image.

4. Load a probe that is conductive and align the light as for normal AC mode. The standard cantilever used for EFM and a good first lever to try would be the electri lever, see also AFM Probe Selection Guide; however, any conductive probe is fine, provided that the cantilever can be driven at its resonance frequency. The key factor in choosing a probe is that you do not want the probe tip to be much larger or smaller than your surface features. This is because your phase shift due to the shift in the force gradient is maximized when the tip and the sample features are on the same order. However, if your features are larger than 100nm, this is not important. Any probe less than that amount should do fine, as the signal should be quite good.
5. Open the Nap Panel (Ctrl + 9) and then open the Nap Channel Panel.
6. Tune the cantilever as usual for an AC mode image. Once this is done, it is wise to approach and engage the surface in AC mode and then take a quick standard AC mode image to be sure the tip has landed on a good area of the sample. Once this is done, the settings specific to EFM can be adjusted.
7. To set the system for EFM, first set the Nap mode to ‘Nap’ or ‘Snap’. For most applications, Nap is preferred, as it reduces the effects of topography on the EFM image. Generally Snap mode is used if the researcher has plans to model the data later to extract quantitative results. For the purposes of this description, Nap mode is used. Note that the two are identical in operation other than the initial selection of Nap or Snap. Once the system is set to Nap mode, choose the parameters that will be used in EFM. These should be *Drive Amplitude* and *Tip Voltage*. To select these, check the boxes by the corresponding parameter boxes under the Parm Swap column on the Nap Panel.
8. Next, set the *Delta Height* to the value you want to use. A reasonable start point for this value is 40 or 50nm if the free air amplitude is 100 nm (1V with 100 nm / V AmpInvols). This value should be linearly higher if the free amplitude is higher. Figure 12.4 on page 151 shows the nap panel with typical settings for EFM.
9. To begin the scan, the *Drive Amplitude* is set to half the drive used for free air scans, and *Tip Voltage* can be set to zero.
10. Start scanning. The first priority is to be sure that the topographic scan is good, as the nap scans rely on this for the nap pass. These adjustments are not different from normal imaging, so refer to the standard AC Mode Imaging chapter for more detailed information.
11. Once the topography looks good, the *Drive Amplitude* and *Tip Voltage* can be adjusted under the Parm Swap parameter column on the Nap Panel. Slowly raise these two numbers until data begins to appear in the Nap Phase Channel.
12. The amplitude during the main and nap scans can be viewed in real time on the Sum and Deflection Meter Panel. By watching the amplitude in real time, you can ascertain fairly quickly if the tip is striking the surface during the nap scan. To do this, watch the amplitude during the nap scan. If the nap amplitude drops to zero, the tip is striking the surface during the nap pass. Try to improve the surface tracking, scanning slower, and/or decrease the nap pass drive amplitude. The drive amplitude during the nap scan should generally be lower than the drive amplitude during the main, or surface, scan.
13. At this point, the operation section of performing EFM is finished. From here, you must adjust parameters to improve contrast to a point where the images are usable. To be sure

that images are related to the electrical properties of the sample and not the topography, the tip bias can be slowly changed during scanning. If the signal is related to electrical properties, then the phase contrast between the different features should increase with increasing voltages, rather than a general offset of the phase for the entire surface.

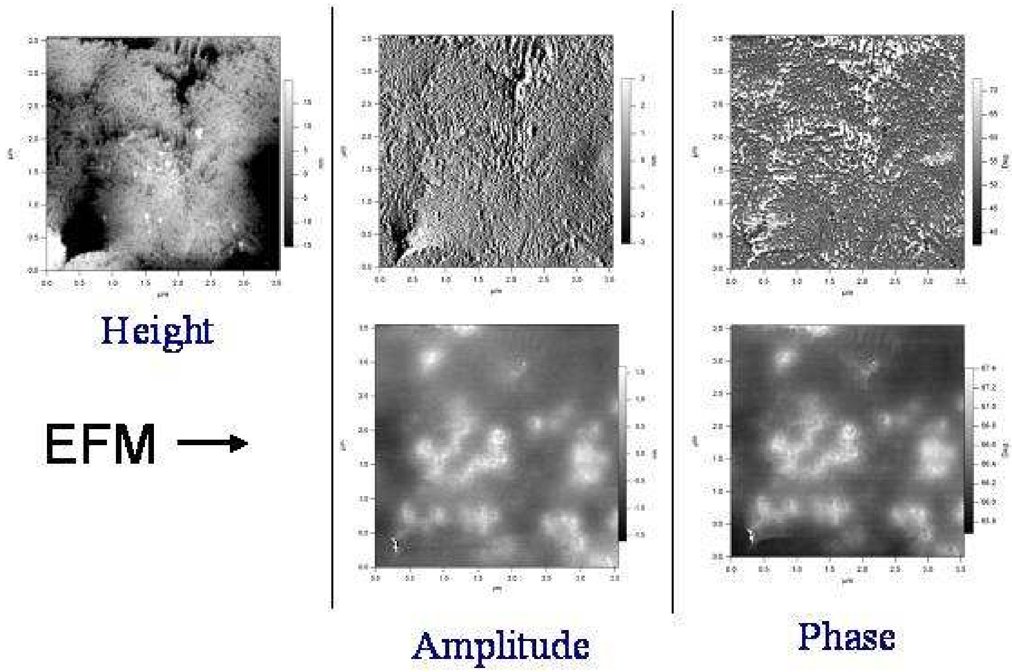


Figure 12.5.: EFM image of polypropylene with carbon black particles (CB) imbedded into it. The areas of contrast in amplitude and phase are due to the presence of CB particles either at or below the surface of the polypropylene.

Note	Note also that the EFM contrast is a result of the interactions between the sample and the cantilever, tip cone, and tip apex. The highest resolution is when the tip apex is the dominant contributor. This happens when the tip is very close to the surface, on the order of the tip radius. As minor adjustments are made, the tip should always be kept as close to the surface as possible. Furthermore, using a larger tip radius helps; just be sure that it is still small enough to see the features you are looking for.

12.5. Frequency Modulated EFM (FM-EFM)

EFM can also be operated using a Phase Lock Loop (PLL) to track the frequency during the nap scan. When a PLL is turned on, the system adjusts the drive frequency of the probe to keep the phase at 90° or on resonance. This has two primary effects. The first is that the phase and amplitude changes are separated, meaning that a shift in resonance will no longer result in both a drop in amplitude and a shift in phase. Instead, the system will stay on resonance, and any shift in phase before the application of the PLL will result in a shift in the frequency. Additionally, any changes in amplitude represent a change in the Q factor of the resonance peak. This leads to the second effect of the application of a PLL, which is to allow modeling of the system for quantitative field shifts. The detail of this modeling is beyond the scope of this document.

How to implement FM-EFM

1. Read the procedures above for standard EFM. Follow steps 1-9, then move to step 2 in this section.
2. In the Master Channel Panel, select "Frequency" as a channel. This opens the Frequency Channel in the Nap Channel Panel. Check that the *Capture & Display* setting should be "None" for the Master Channel Panel and set to "Retrace" in the Nap Channel Panel.
3. When the probe is in z piezo range of the surface, withdraw the probe, then click the Tune tab on the Master Panel.
4. Set the *Sweep Width* to 5kHz, then click 'One Tune'.
5. If you do not see the 'Center Phase' button, under User Settings, check *Normal Panels*.
6. On the *Center Phase* drop-down arrow, click "Set phase to 90".
7. On the Nap Panel, check the *Test FM Feedback* box.
8. Observe the frequency and phase readouts on the Sum and Deflection Meter Panel, then raise the *Freq I Gain*. Raise the value until the field is unstable, then lower it back to stability.
9. Observe the frequency and phase readouts on the Sum and Deflection Meter Panel, then raise the *Freq P Gain*. Raise the value until the field is unstable, then lower it back to stability.
10. Begin imaging. The information previously in the phase will now be transferred into the frequency. The amplitude will now rise and fall with the varying Q factor of the cantilever.

13. Magnetic Force Microscopy (MFM)

CHAPTER REV. 950, DATED 07/10/2012, 15:54. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

13.1. Introduction

Magnetic Force Microscopy (MFM) and EFM imaging using Nap mode is based around the pioneering work of Hosaka et al. This method is now widely used for a variety of imaging modes where separating long and short ranged (usually topographic) forces is accomplished by moving the probe a prescribed distance above the measured topography.

Before you dive into MFM imaging, please spend some time reading **??** on page **??** to familiarize yourself with the theory behind this imaging technique. Note that those who understand the theory tend to have more success with imaging challenging samples.

This is a somewhat advanced imaging technique, and it is assumed you:

- have followed the AC Mode Imaging Tutorial associated with your model of AFM.

It is also helpful if you:

- Are familiar with the more advanced aspects of AC Mode Imaging. The theory of this can be found in Chapter 5 on page 46 and a practical guideline in Chapter 4 on page 34.
- Again, Are familiar with MFM Theory, covered in **??** on page **??**.

13.2. Required Materials

Materials required in MFM imaging include:

- MFM cantilevers (part number ASYMFM)
- Rare earth magnet, either the sample holder magnets or another comparable rare earth magnet
- MFM test sample
- Standard AC Mode capable cantilever holder

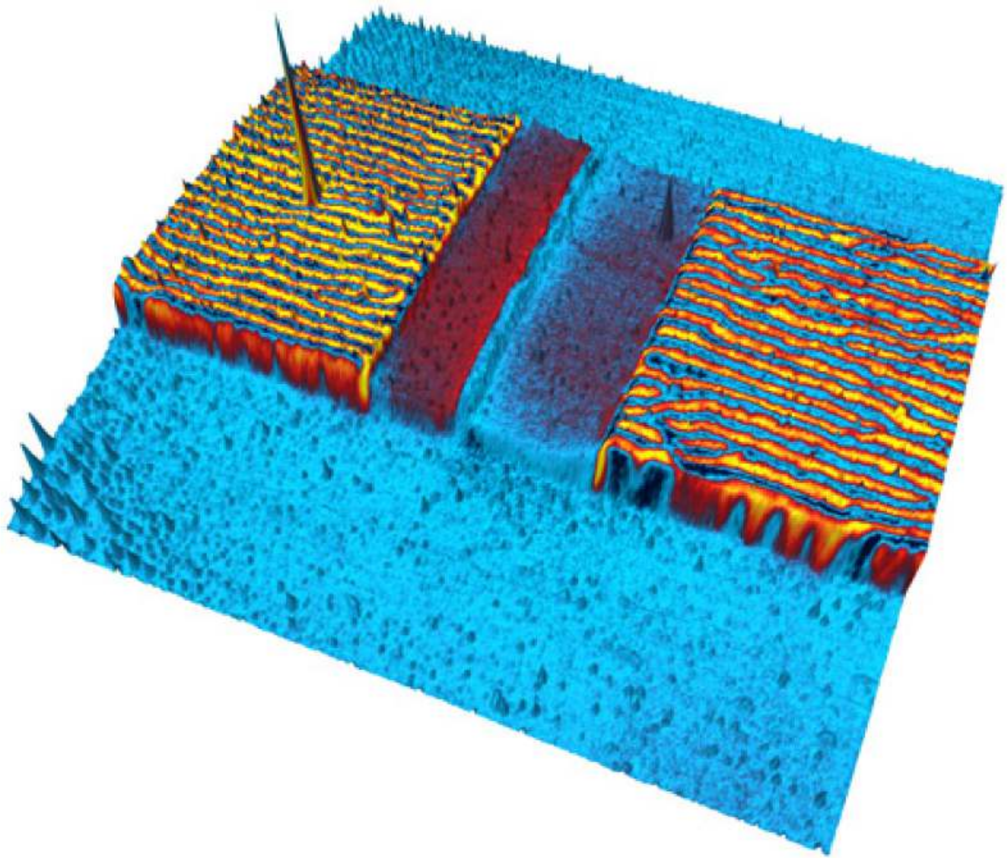


Figure 13.1.: The MFM signal showing the magnetic fields above an Iomega Zip™ 1 GB drive write head. The MFM phase signal was overlaid on top of the topography, 20 µm scan.

13.3. How to Guide

1. The direction of the tip's magnetic moment will affect the image. Imagine a cantilever with a magnetic dipole tip $\overrightarrow{m} = m_x\hat{x} + m_y\hat{y} + m_z\hat{z}$, where $\vec{m}$ is the magnetic dipole moment, m_x, m_y and m_z are the vector components of the magnetic dipole moment, and $\hat{x}$, $\hat{y}$ and $\hat{z}$ are unit vectors. If this dipole is oscillating along the z-axis with a small amplitude, the force gradient F_z' acting on the tip is given by:

$$F_z' = m_x \frac{\partial^2 H_x}{\partial z^2} + m_y \frac{\partial^2 H_y}{\partial z^2} + m_z \frac{\partial^2 H_z}{\partial z^2}. \tag{13.1}$$

2.

Magnetize the cantilever:

- To magnetize the tip along the z-axis of the cantilever:
  - Take one of the magnetic sample holders from the MFP-3D (or a comparable rare earth magnet):
  - Load an MFM probe into the probe holder.
  - Orient the magnet so that the side with the circles is facing the tip.
  - Hold the magnet in one hand and the cantilever holder in the other about 6 inches apart.
  - Move the magnet toward the cantilever tip until one of the circles on the facing side is very close (~1 mm) to the tip.
  - Move the magnet away from the tip to a distance of >6 inches.
  - Make sure that when you are moving the magnet toward and away from the cantilever, doing so in a straight line along the normal tip axis.

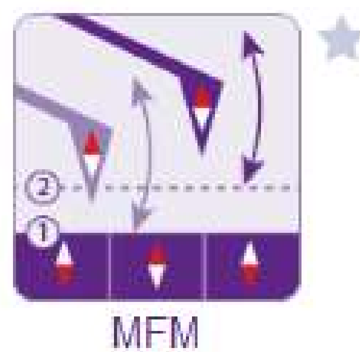
Note By changing the orientation of the magnet, it is possible to select other field components such as m_x or m_y.

3. Load the cantilever into the cantilever holder as you would for normal AC mode imaging.

4.

Launch the MFM Profile:

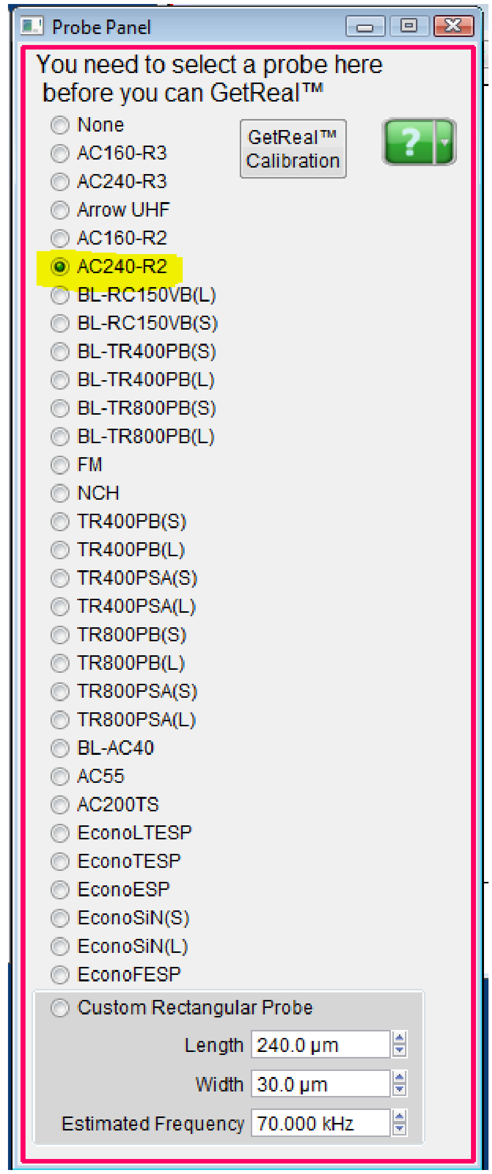
- If Mode Master is not already shown, from the main menu bar select *AFM Controls > Mode Master*.
- Tab over to the Electrical tab on the Mode Master Panel and click the 'MFM Mode Master' button.



5.

Calibrate the spring constant (k) of the cantilever:

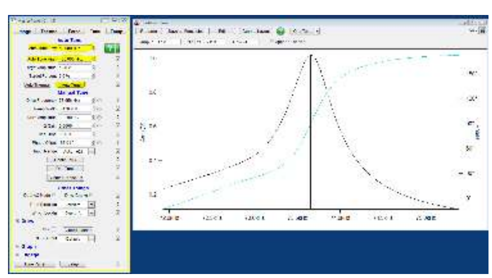
- If you are using Asylum Research branded MFM probes, it is possible to characterize the probe using GetReal(TM).
- Tab over to the Thermal tab on the Master Panel.
- For Asylum MFM probes labeled without a revision appended (e.g., ASYMFM, ASYMFMHC, etc.), select 'AC-240-R2'.
- For other probes, select 'Custom Rectangular Probe', then enter the dimensions and estimated resonance frequency of the probe.
- Click the 'GetReal™ Calibration' button.



6.

Tune the cantilever:

- Tab over to the Tune tab on the Master Panel.
- Set the *Auto Tune Low* and *Auto Tune High* so that the range includes the resonance frequency range of the probe manufacturer. (For ASYMFM probes. use 40kHz - 110kHz.)
- Click 'Auto Tune'.



7.

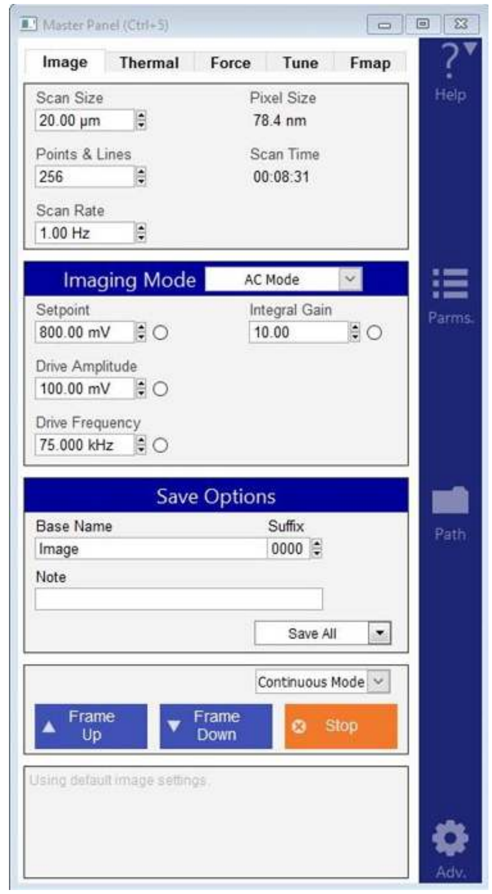
Approach the surface:

- The method of approach depends on the instrument you are operating.
  - For Cypher-based systems, consult the Cypher User Guide.
  - For the MFM-3D series of AFMs, consult the MFP-3D User Guide.

8.

Set the Master Panel imaging parameters:

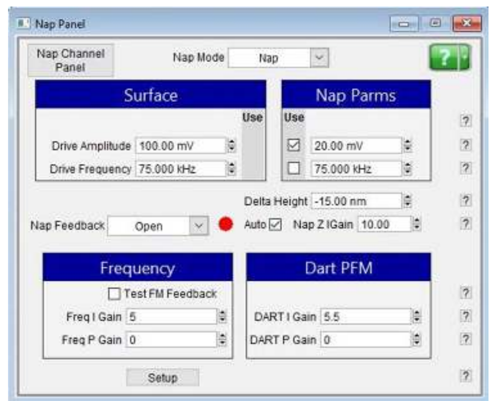
- Typically, the amplitude *Setpoint* needs to be larger than the largest vertical features expected to on the sample surface. If this information is not known, this parameter can be adjusted once the scan has begun. To calculate this value, look on the thermal tab of the Master Panel to find the Amp InvOLS. This value, multiplied by the amplitude setpoint, is the physical amplitude of the probe–typically 50-100nm.
- Mechanical bandwidths on MFM probes are typically around 500-700 pixels per second, so the *Scan Rate* should typically be 0.5Hz to 0.75Hz.
- The *Drive Amplitude* during the nap scan should be 1/2 to 1/5 of the main drive amplitude. Higher drive amplitude will give better signal to noise, while smaller drive will give better lateral resolution.



9.

Set the Nap Panel imaging parameters:

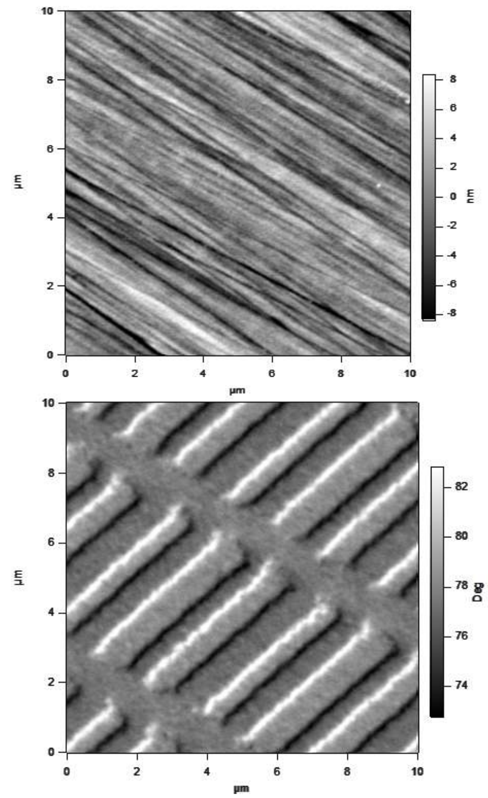
- The *Delta Height* on the Nap Panel should range from zero to a negative value, depending on the main *Drive Amplitude* and the nap *Drive Amplitude*. Generally, the delta height should be as low as possible without hitting the surface. If the probe is striking the surface during the nap scan, any MFM data will be obscured by the tip-sample interaction, and the nap phase or frequency will resemble the main scan phase channel.
- When adjusting the *Delta Height* and *Drive Amplitude* important principles to consider are:
  - A smaller drive amplitude will allow the probe to be passed closer to the sample surface during the nap scan, improving the lateral resolution.
  - At some point, however, this improvement in lateral resolution will eventually be reversed by a reduction in the phase detection as the noise in the measurement surpasses the signal.
  - Likewise, the absolute phase that can be detected can be increased by increasing the drive during the nap scan, resulting in a reduction in the lateral resolution. This is generally true for all nap-based imaging.



10.

MFM images:

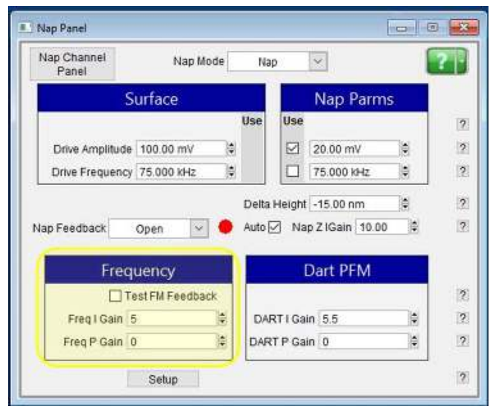
- The MFM image should clearly show the magnetic domains independent of the topography and phase. In plane, domains will show bright and dark contrast regions, as shown in the image at right. Out of plane will show simpler contrast.
- If the phase or topography is clearly apparent in the nap phase or nap frequency image, it is likely that the probe is striking the surface during the nap scan.



11.

Set frequency feedback using PLL - Part 1:

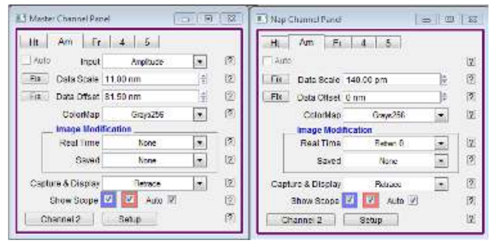
- It is possible to set up a feedback loop during the nap scan to track the resonance frequency using a Phase Lock Loop (PLL). This primarily allows two goals to be achieved. The first is to separate the phase and amplitude responses of the signal. (Changes in amplitude are primarily due to a change in the quality factor (Q) of the resonating probe, and changes in phase are due to frequency shifts in the resonance of the probe due to a change in the magnetic field gradient under the probe tip.)
- To turn on a PLL, it is necessary to first find the gains needed for the PLL feedback.
  - Once the probe has been calibrated and tuned, check the box on the Nap Panel labeled *Test FM feedback*. "FM" stands for frequency modulation and is another way to describe the PLL.
  - Once the box is checked, raise the integral and proportional gains (*Freq I Gain* and *Freq P Gain*, respectively) until the *Phase* and *Amplitude* i\on the Sum and Deflection Meter Panel become unstable. The instability will be apparent because the amplitude and phase will begin to oscillate uncontrollably.)



12.

Frequency feedback using a PLL - Part 2:

- Next the frequency channel must be opened during the nap scan.
- On the Master Channel Panel:
  - Select “Frequency” as a channel.
  - Set *Capture and Display* to “None”.
- On the Nap Channel Panel, the frequency *Capture and Display* setting should be set to “Retrace”.
- When imaging, the phase data should now be an error channel in the frequency feedback loop, or PLL. Changes in the amplitude will now reflect variations in the Q factor of the cantilever, and the frequency data will represent the adjustment to the drive frequency needed to keep the phase channel at 90°, or on resonance.



14. Bimodal Dual AC™ Mode

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

14.1. System Requirements for Bimodal Dual AC™ Mode

Bimodal Dual AC Mode is possible on all MFP-3D AFMs and Cypher AFMs.

System requirements for Dual AC Mode include:

- A **High Frequency Cantilever Holder** is highly recommended but not necessary for lower frequency cantilevers.
- If Cypher AFM is equipped with **blueDrive**, this can be used instead with any standard cantilever holder.

Depending on your model of AFM, please refer to:

- *MFP-3D User Guide, Chapter: AM-FM Viscoelastic Mapping Hardware*.
- *Cypher User Guide, Chapter: Cantilever Holder Guide*.

14.2. Introduction

Dual AC™ mode imaging is a relatively new AC imaging mode offered both on the MFP-3D series and Cypher series of AFMs. The concept is to simultaneously drive the cantilever at two of its flexural resonances, giving an AC waveform similar to the one depicted in Figure 14.1 on page 166.

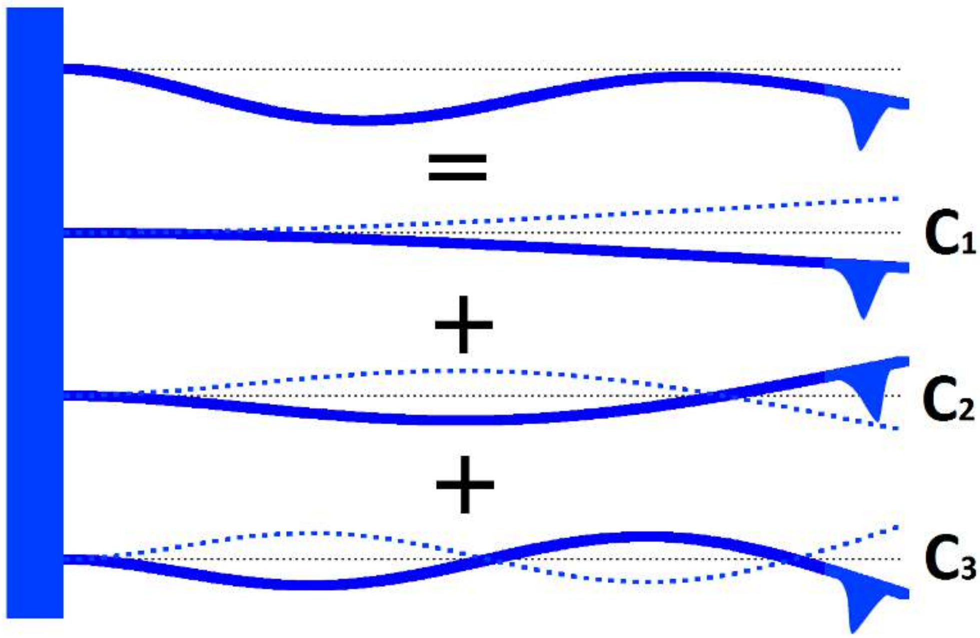


Figure 14.1.: Depiction of bimodal resonance operation.

The Dual AC™ mode imaging requires two independent lock-in amplifiers to accomplish this technique, which the ARC2 Controller is capable of performing, as seen in 14.2. The fundamental cantilever resonance frequency (i.e., first Eigenmode) is typically locked in for an amplitude feedback loop, which ultimately constrains the movement of the cantilever at this mode. What's interesting is that the second Eigenmode is not constrained by the feedback loop, quite often producing increased image contrasts in the second amplitude and/ or second phase channels. Since this is based on the flexural resonant frequencies of a cantilever, these higher eigenmodes are not integers (as with harmonics), but rather follow the relationship: C1 = 1; C2 = 6.1; C3 = 17.5

Thus, it is possible for the cantilever selection for this technique to be limited to the bandwidth of the detector system (i.e., the second or third Eigenmode of stiffer cantilevers is often at too high a frequency to be driven by the MFP-3D which has a maximum bandwidth of ~2.5MHz and ~ 7Mhz for Cypher).

Keep in mind that not all samples will show good contrast in the second mode. Likewise, some samples show good contrast between the first and second amplitudes while showing little difference between the first and second phase, or even vice versa. Heterogeneous samples seem to show good contrast with this technique.

The same attractive/repulsive imaging mode rules seem to apply to the second mode as does the first. The author suggests using the protocols described in Section 4.6.3 on page 40to avoid mode hoping in the second mode.

For the total beginner, we have found that just a small amount of water-based latex paint on a glass microscope slide (allowed to dry) gives excellent contrast in the Amplitude and Phase images and

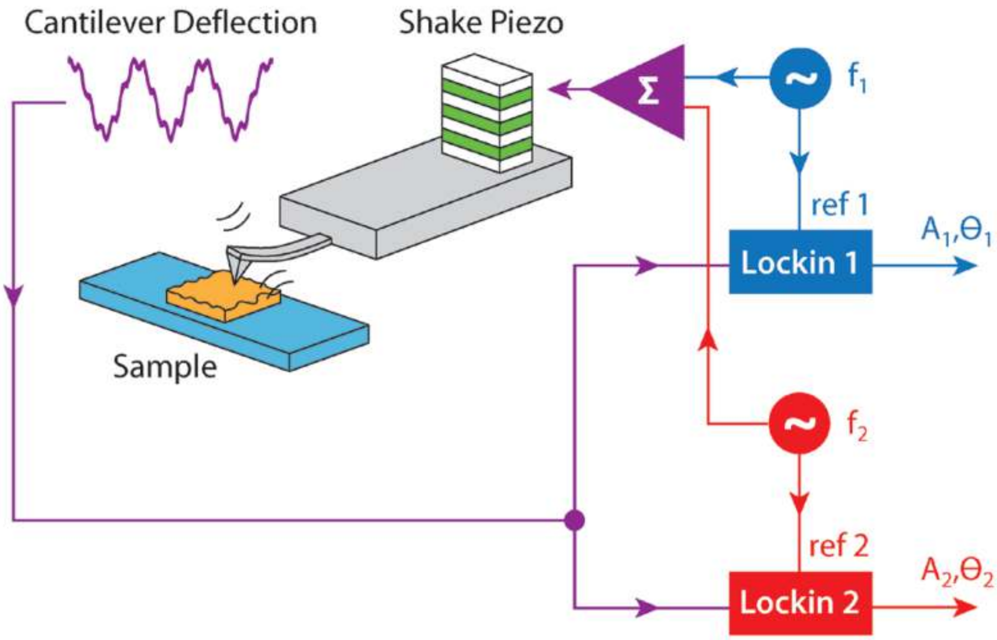


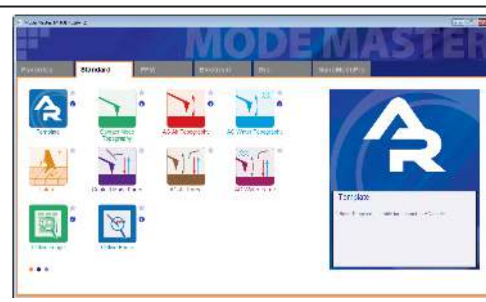
Figure 14.2.: In Bimodal Dual AC, the cantilever is both driven and measured at two (or more) frequencies. The sinusoidal "shake" voltage is a sum of voltages at frequencies f1 and f2. The cantilever deflection then contains information at both of those frequencies, as shown in the red curve. The amplitude and phase at the two frequencies are then separated again by the two lock-ins and passed on to the controller. The controller can use one or both of the resonant frequencies to operate a feedback loop.

can be easily accessed by most users wanting to learn and practice how to do this technique. Water-based paints are heterogeneous because of the small emulsion polymer particles and other fillers used as the ingredients that give the paint its opaque character.

14.3. DualAC™ Imaging in Air

1. **The Mode Master window:**

   - The software should now be showing the Mode Master window.
   - If not, click the 'Mode Master' button at the bottom of the screen: .



2.

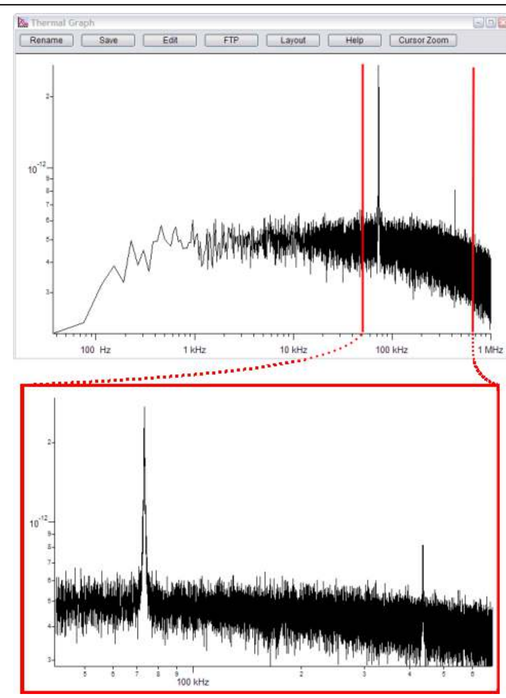
Select mode:

- Select *NanoMechPro > Bimodal Dual AC*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

3.

Thermal tuning:

- First, determine the fundamental resonant frequency of the cantilever by performing a thermal tune (seeChapter 21 on page 293).
- To determine the second Eigenmode, roughly multiply the first Eigenmode frequency by 6.1. This is important for entering high and low tune values for the DualAC™ Auto Tune later.
- An example of a thermal tune for an Olympus AC240-R3 Silicon cantilever (f~70kHz; k~2N/m) can be seen in the images at right.

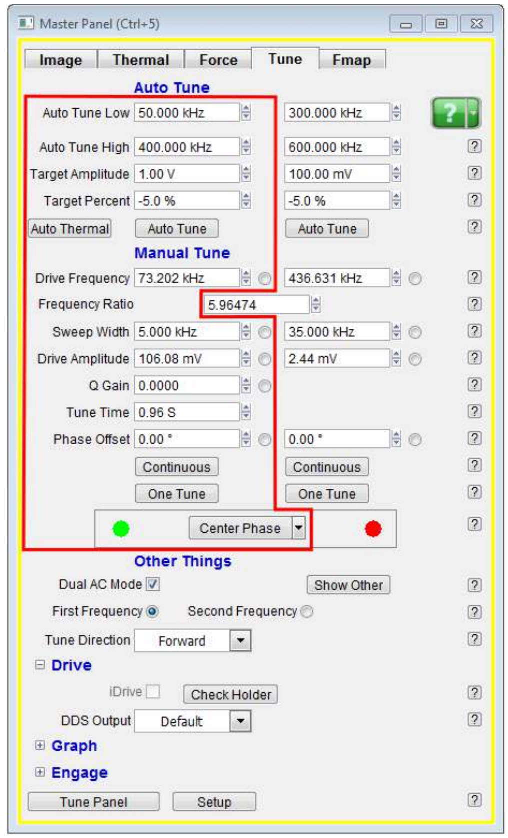


4. Go to the Tune tab of the Master Panel (Ctrl+5).

5.

Auto Tuning: First Mode:

- Confirm that the *Auto Tune Low* and *Auto Tune High* field values cover a range of frequencies to capture the cantilever resonances earlier determined. (The left column includes the first Eigenmode tune parameters, and the right column includes the higher Eigenmode tune parameters.)
- Choose a *Target Amplitude* and *Target Percent* for the first Eigenmode resonant peak. Typically, 1-2 V and -5%, respectively, works well to be in Repulsive mode (see5.3.1).
- Click the ‘Auto Tune’ button in the left column. This will tune the cantilever as is it typically does in standard AC imaging.

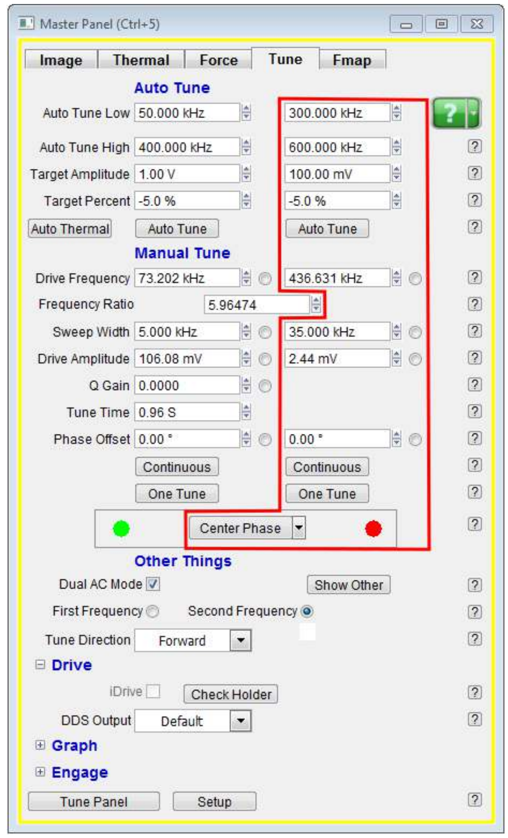


6.

Auto Tuning: Higher Mode:

- Confirm that the *Auto Tune Low* and *Auto Tune High* field values cover a range of frequencies that will capture the cantilever resonances determined earlier.
- Choose a *Target Amplitude* and *Target Percent* for the higher Eigenmode resonant peak. Typically, 100-200 mV and -5%, respectively, works well to be in Repulsive mode
- Click on *Auto Tune* button in the right column. The software chooses a frequency and updates the *Frequency Ratio* value in the center column.

Note: If the *Auto Tune* for the higher mode is not successful, it is possible the mechanical coupling of the probe chip to the cantilever holder may be too good. This will cause the *Drive Amplitude* to be too small of a signal for the controller to achieve and maintain the defined *Target Amplitude*. If this happens, increase the *Target Amplitude*, and then step it down later once imaging commences.

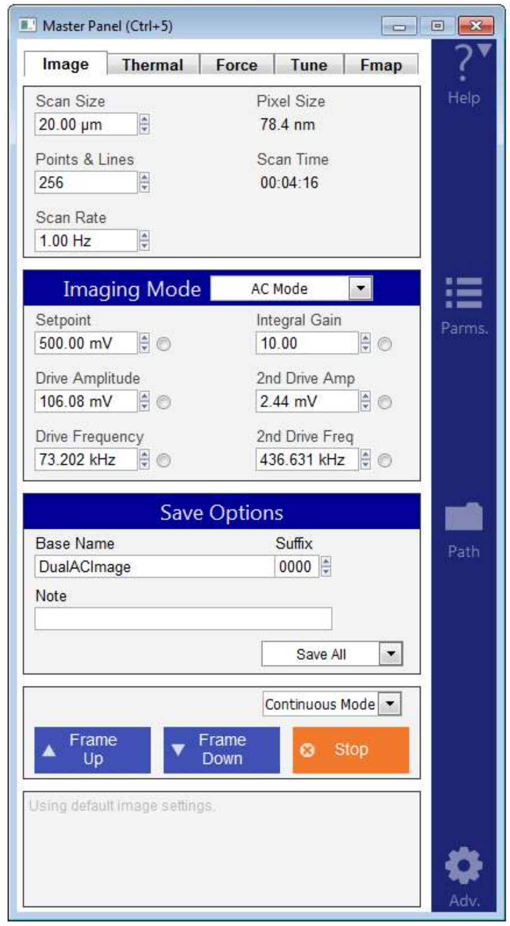


7. Perform a “soft engage” as described in video tutorial for the Soft Engage Method.

8.

Start the scan:

- Once the tip is firmly engaged on the surface, adjust the *Drive Amplitude* appropriately to keep the Phase signal of Amplitude 1 within the imaging mode desired (e.g., repulsive, or attractive).
- Notice that the main tab of the Master Panel now has an expanded column offering *2nd Drive Amp* and *2nd Drive Freq* parameters with radio buttons for image tuning as well.
- Click the 'Start' button to start the scan.



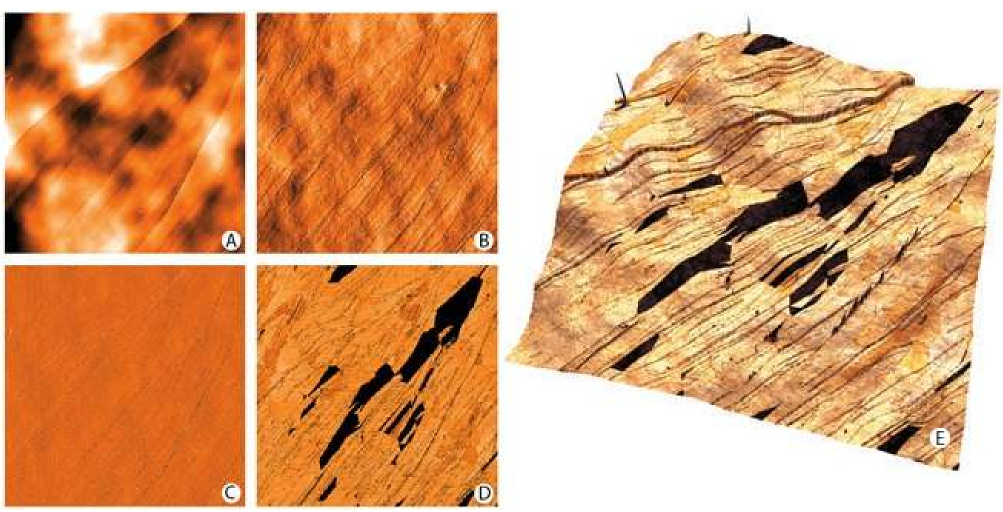


Figure 14.3.: 30µm image of a HOPG surface acquired with an Olympus AC240-R3 Silicon cantilever. (A) Height channel, (B) Amplitude 1 channel, (C) Phase 1 channel, (D) Amplitude 2 channel, (E) Phase 2 channel overlaid on 3D rendered Height channel.

14.4. DualAC™ Imaging in Liquid

DualAC™ in liquid while driving the cantilever acoustically (e.g., shake piezo) is generally not successful. The main reason is likely because, in liquid, the entire cantilever holder is ultimately driving the liquid between the tip and sample; this creates an unstable resonant cavity.

Asylum Research has found that DualAC™ has had success through direct cantilever actuation using either photothermal actuation: blueDrive or magnetic actuation: iDrive™. Through direct actuation, just the cantilever is actuated, eliminating all the other crazy waves that normally occur in solution when driving via acoustic mode.

References:

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15. AM-FM: Viscoelastic Mapping

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

15.1. System Requirements for AM-FM

AM-FM Mode imaging is possible on all MFP-3D AFMs and Cypher AFMs.

System requirements for AM-FM include:

- A **High Frequency Cantilever Holder** is highly recommended but not necessary for lower frequency cantilevers.
- If Cypher AFM is equipped with **blueDrive**, this can be used instead with any standard cantilever holder.

Depending on your model of AFM, please refer to:

- *MFP-3D User Guide, Chapter: AM-FM Viscoelastic Mapping Hardware.*
- *Cypher User Guide, Chapter: Cantilever Holder Guide.*

15.2. Introduction

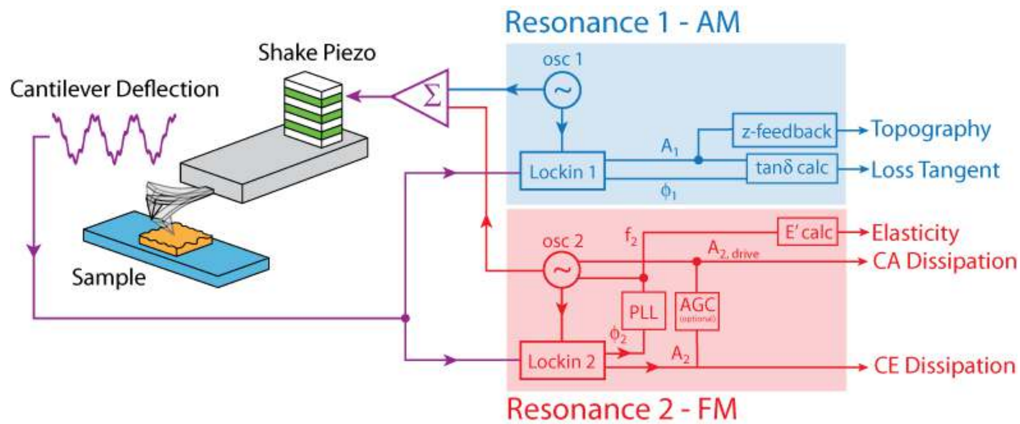


Figure 15.1.: AM-FM Mode. The first mode amplitude is controlled to create a topographic image of the sample (blue). The second mode is used to calculate elasticity and tip-sample stiffness (red).

AM-FM Mode imaging combines the features and benefits of normal AC mode/ Tapping mode (also called AM, or Amplitude Modulation) with the high sensitivity of Frequency Modulation (FM) mode. In this mode, the cantilever is simultaneously driven at two different mechanical resonances, as shown in Figure 15.1 on page 174. When operating in standard AM-FM mode, there are two, or an optional three, feedback loops running simultaneously.

- The topographic feedback on the cantilever oscillation amplitude operates as in typical AC mode/ Tapping mode, adjusting the Z-piezo position, or tip-sample separation, to keep the cantilever amplitude on the first resonant mode constant.
- The second feedback loop adjusts the drive frequency to the second resonant mode such that it is always oscillating at 90° or, in other words, at resonance. This resonant frequency is a very sensitive measure of the tip-sample interaction forces. Simply put, a stiffer sample shifts the second resonance to a higher value while a softer sample shifts it to a lower value. This can be converted into a quantitative modulus measurement through a variety of mechanical models.
- The third feedback is optional, but when enabled, maintains a constant second resonance mode cantilever amplitude by either increasing or decreasing the drive amplitude. This is used to calculate the tip-sample stiffness.

As with conventional Frequency Modulation (FM) mode, AM-FM is a quantitative technique in which the conservative and dissipative tip-sample force interactions can be deconvolved. However, unlike conventional Frequency Modulation (FM) mode, the feedback loop tracking topography (AM) is completely decoupled from the FM loop tracking the interactions, which both greatly simplifies and stabilizes the operation of the technique.

The second mode spring constant can be calculated from this equation:

$$K_2 = \left(\frac{\omega_2}{\omega_1}\right)^2 K_1 \tag{15.1}$$

15.3. Video Tutorial

Consider watching this introductory video tutorial: AM-FM Viscoelastic Mapping Mode *(internet connection required)*.

15.4. How to Guide

15.4.1. Setup

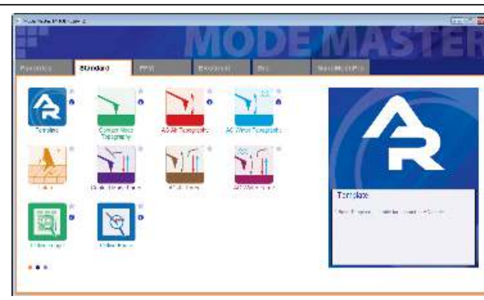
1. For the most recent features use the latest AFM software version 15 or later.

2. **Load your sample in to the AFM:**

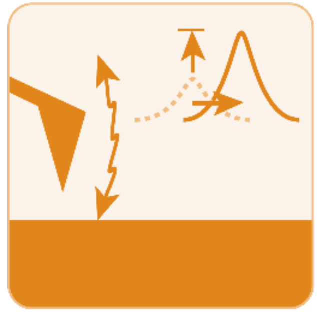
   Note The AM-FM technique is very sensitive to sample cleanliness since the indentation depths during imaging are in the picometer to nanometer range. Especially in the case of stiffer samples, they should be freshly prepared or cleaned shortly before imaging.

3. Load your probe into the High Frequency Cantilever Holder and attach it to the AFM. Recommended probes for AM-FM in air include:
   - AC240TS-R3
   - AC160TS-R3
   - FS-1500AuD(Cypher only)

4. **The Mode Master window:**
   - The software should now be showing the Mode Master window.
   - If not, click the 'Mode Master' button at the bottom of the screen: .



5.



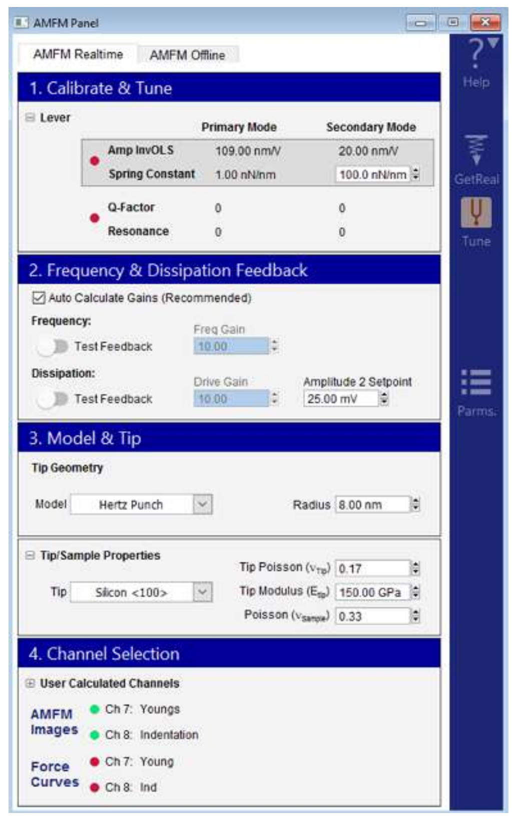
Select mode:

- Select *NanoMechPro > AMFM*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

6.

The AMFM Panel

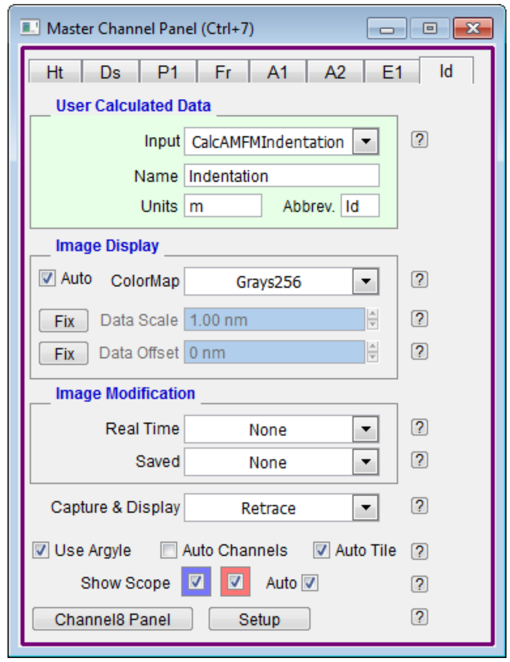
- The **AMFM Panel** includes both *Realtime* and *Offline* tabs.
- The *Realtime* tab includes **calibration** parameters, **feedback controls** for the FM and AM feedback loop of the higher resonance mode, and real-time **tip modeling** parameters.
- The *Offline* tab includes parameters to calculate additional **stiffness/elasticity** images with various **tip models** for AM-FM images after they have been collected.



7.

The Master Channel Panel:

- The eight default enabled channels are: Height (Ht), Dissipation (Ds), Phase1 (P1), Frequency (Fr), Amplitude 1, Amplitude2 (A2), either Stiffness (Ks) or Elasticity (E1), and either Indentation (Id) or Loss Tangent (L1).



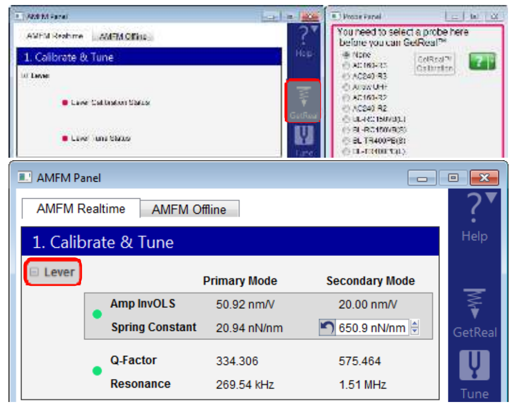
15.4.2. Calibration

1. If you are measuring the tip-sample stiffness (Ks) or Young's modulus (E1), you will first need to calibrate your cantilever using GetReal™: Section 20.2 on page 282 or the standard Thermal Method: Section 20.3 on page 282. These instructions outline GetReal™ cantilever calibration for AM-FM as it is easier, faster, more accurate, and preserves the probe sharpness.

2.

Probe calibration:

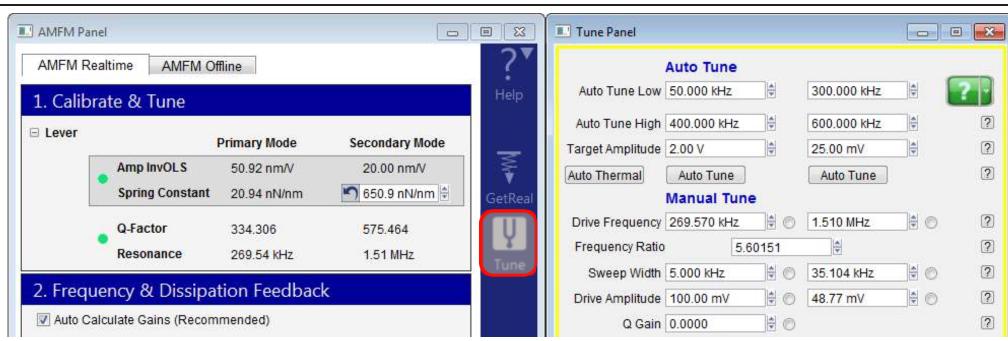
- Align your laser on the cantilever and zero your Deflection signal.
- Click the 'GetReal™' icon, located in the right column of the AMFM Panel and choose your probe from the list.
- Click the 'GetReal™ Calibration' button in the Probe Panel and wait for the system to calibrate your cantilever sensitivity (Amp InvOLS) and spring constant (k).
- The Primary Mode and Secondary Mode calibrations are now automatically calculated. Their values can be viewed by selecting the expand button for 'Lever' in the AMFM Panel.



Note The software automatically calculates the Secondary Mode spring constant using an equation 15.1.

15.4.3. Tuning

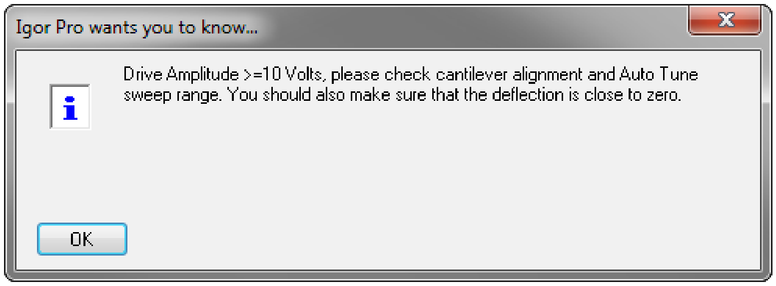
1.



Tuning the cantilever:

- Click the 'Tune' icon on the right column of the AMFM Panel.
- Autotune the Primary Mode using the left column of the Tune Panel. A target or free amplitude of 2V is typically a good preliminary or general value to start with.
- Autotune the Secondary Mode using the right column of the Tune Panel. A target or free amplitude of 25mV is typically a good preliminary or general value to start with.

Note It is very important that your Secondary Mode free amplitude is significantly lower than your Primary Mode free amplitude.



2. **Tune error message:** After autotuning, if you receive an error message, as shown above, you may need to:

- If you are using an MFP-3D, flip the attenuation switch on your cantilever holder. Remove the cantilever holder, find the switch on the backside of the holder near the quartz window, and move it to the other side. Reattach it to the AFM and retry the tune.
- Reposition the cantilever in the holder. Typically securing most of the probe chip under the clip will improve drive coupling.

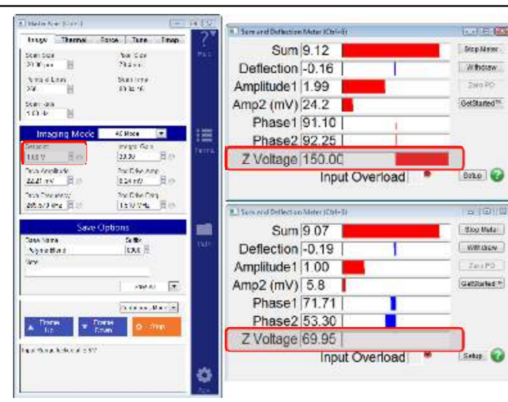
3. **A note on spot position:**

- You want the sensitivity of the optical beam deflection to be as high as possible for the Secondary Mode. To accomplish this, after you have successfully tuned, you should move the spot as far out to the end of the lever as possible. Observe the Amplitude 2 signal while you do this and pick a laser spot location where you've maximized the Amplitude 2 signal. If you lose a bit of signal in Amplitude 1 to maximize Amplitude 2, this is just fine. Once you have found the optimal laser location, retune the Primary and Secondary Modes.

15.4.4. Engaging

1. **Engaging on the surface:**

- Select your imaging *Setpoint* in the Master Panel. ~40% of the free amplitude is a good preliminary value. This would be ~800mV if you have chosen a free amplitude of 2V.
- Engage on the surface and set *Z Voltage* to ~70V.
- Withdraw from the surface.

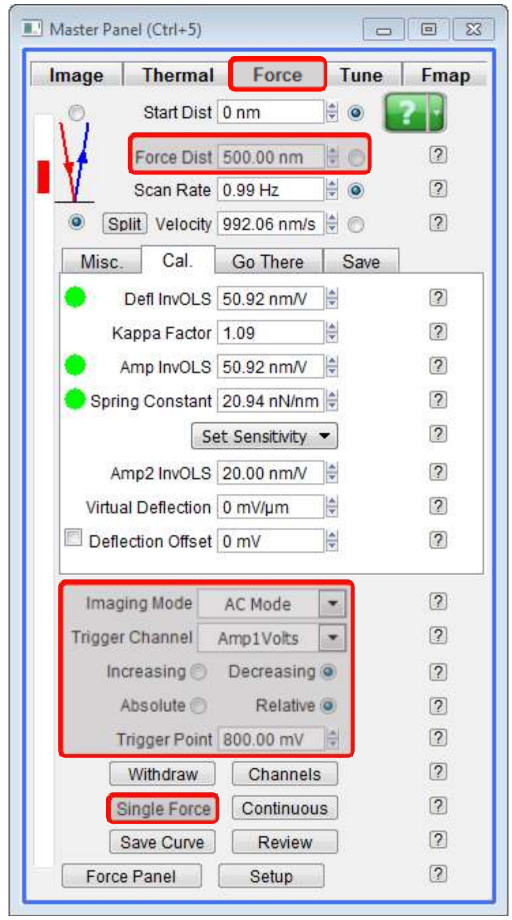


2.

AC force curve:

- Do an ~800mV Amplitude 1 Volts triggered AC force curve with a force distance of 500nm.
- Retune the Primary and Secondary Modes of the cantilever. Make sure you are right on the resonance peaks and that Phase 1 and Phase 2 are both set to 90°.
- Re-engage on the surface.

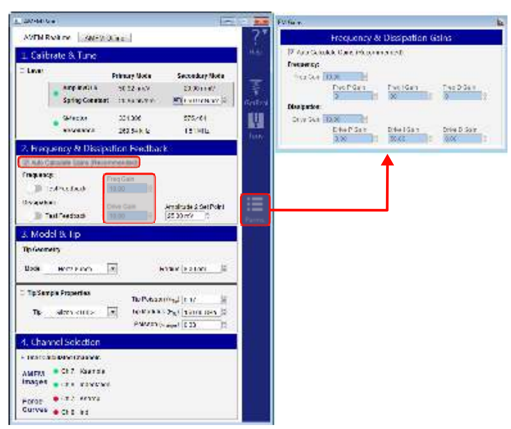
Note This step helps to ensure you are exactly on resonance right at the sample surface, as this typically will shift a bit from the initial tune as you near the surface.



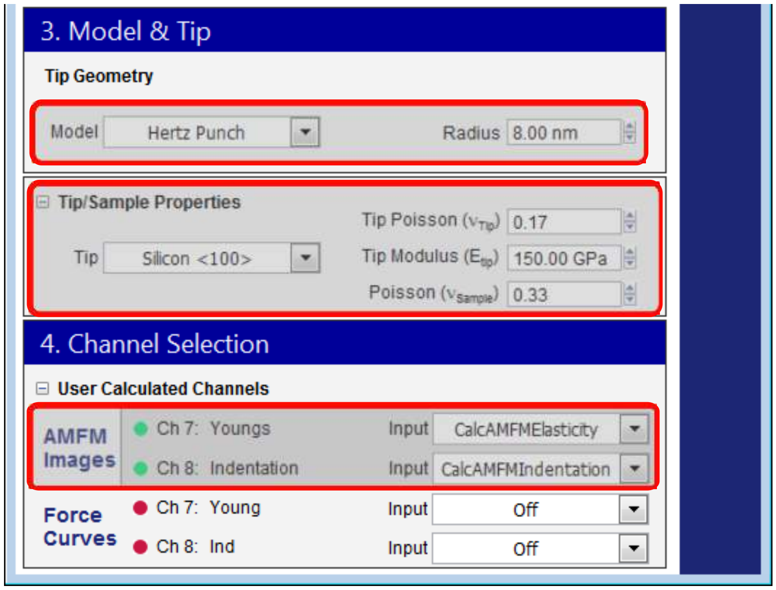
3.

Frequency & Dissipation feedback:

- While engaged, on the AMFM Panel, click the *Test Feedback* switches for both Frequency and Dissipation.
- On the Sum and Deflection Meter Panel, ensure that both Phase 2 and Amp2 appear stable with *Test Feedback* on.
  - If they are not, you may need to manually adjust the feedback gains. Deselect the *Auto Calculate Gains* check box, click the 'Parms' icon, and adjust the P, I, D gains (on the FM Gains panel), accordingly until stable.



4.



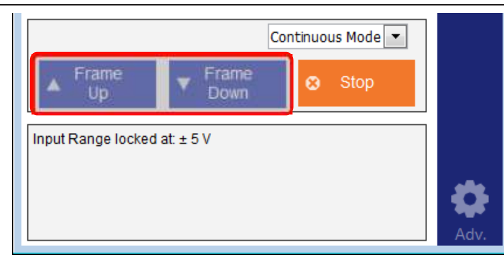
Model & Tip: The last steps to do before scanning is to:

- Choose the tip model.
- Adjust the tip/sample properties.
- Decide which calculated channels you want to display during the scan.

5.

Begin the scan:

- Click 'Frame Up' or 'Frame Down' in the Master Panel.

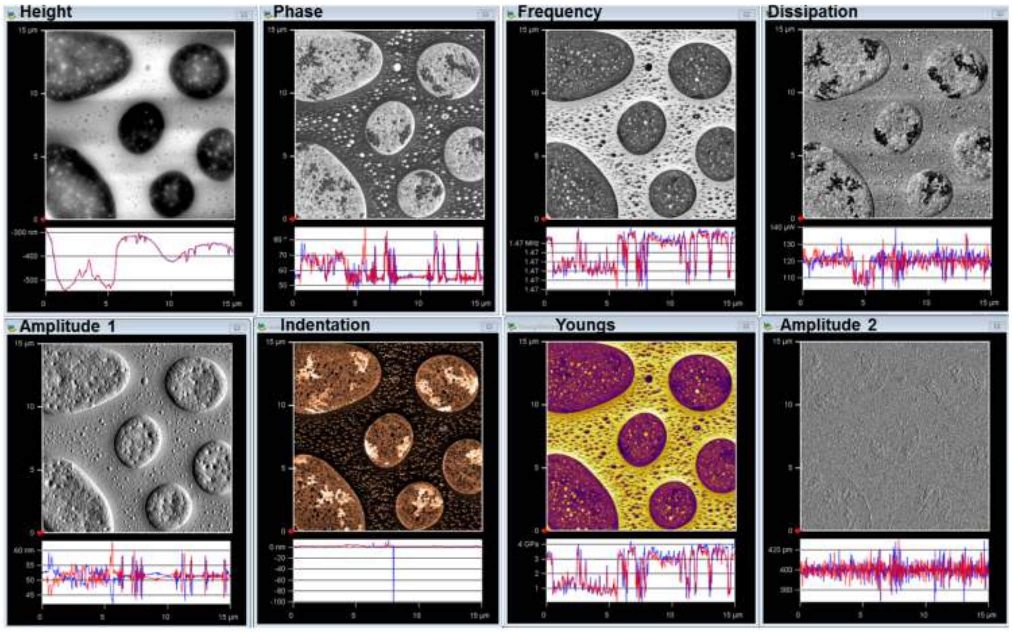


15.4.5. Optimizing the scan

1.

Repulsive mode:

- The cantilever must be firmly in Repulsive mode (Phase 1 <90°) for AM-FM imaging. (If the Phase 1 is consistently hopping between Attractive (>90°) and Repulsive (<90°), the AFM mode will struggle to work well.)
- Initially, the amplitude *Setpoint* is ~40% of the free amplitude, but you may need to adjust this to keep the cantilever firmly in repulsive mode. If Phase 1 is ≥90°, gradually increase the *Drive Amplitude* or decrease the Amplitude 1 *Setpoint* in the Master Panel to maintain a Phase 1 in range ~40°-80°.
- If you have increased the *Drive Amplitude* and decreased the *Setpoint*, and imaging in Repulsive mode is still not achievable, the cantilever tip may have become too blunt or contaminated and should be changed.



2\. **Optimizing the scan:** Adjust the feedback parameters to optimize Topography, Frequency, and, if enabled, Dissipation. If *Auto Calculate Gains* is selected and the Frequency and Amplitude 2 channels are not ringing, you likely do not need to manually adjust the Frequency & Dissipation feedback at all. However, if you do, for best results adjust the feedback gains for these 3 channels in the following order:

a) **Topography** (Ht): **Adjust Integral Gain in the Master Panel**. To improve tracking, you may need to adjust the Integral Gain; typically, increase the gain until you see a little ringing in Amplitude 1 channel and then decrease it just until the noise just disappears.

b) **Frequency** (Fr): **Adjust the Freq I Gain in the AMFM Panel**. Increase until the Trace and Retrace lines are overlapping in the scope plot of the Frequency channel and there is contrast in the image.

c) **Dissipation** (Ds): **Adjust the Drive I Gain in the AMFM Panel**. Increase until the Trace and Retrace lines are overlapping in the scope plot of the Frequency channel and there is contrast in the image.

Note The Frequency and Drive gains can have enormous dynamic range (~10 to 100,000). If, in the off chance, you are manually adjusting these, don't be afraid to turn these up. If you start to see ringing in either the Amplitude 2 or Frequency channels, back down the gains a little. This is causing instabilities only in the very small amplitude of the Secondary Mode drive frequency and should have almost no effect on the Primary mode that is tracking topography. It will not damage the tip.

15.5. Quantitative Analysis

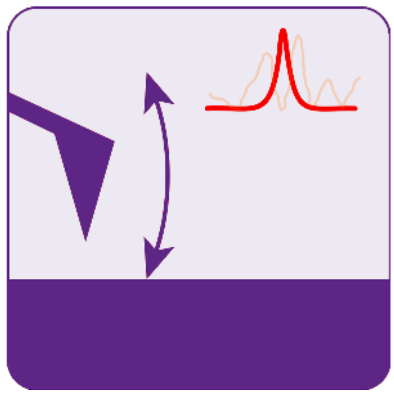
15.5.1. Reference Sample

The AM-FM technique requires a reference sample to determine accurate quantitative nanomechanical properties of unknown materials. This is mainly due to the fact that it is very difficult to characterize the *exact* tip geometry and/or tip radius of a specific cantilever. The most accurate method for AM-FM experiments that you want to measure modulus information for is to first image a reference sample of know modulus and adjust the tip model parameters to match this value, then swap out the reference sample with the unknown sample and use these same tip model parameters while imaging.

Note: For best results, it is most optimal to use a reference sample with a modulus value somewhat close to that of the estimated value of the unknown sample modulus.

16. iDrive Imaging

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



Chapter Contents

NOTE The iDrive option is obsolete and has limited support.

16.1. Theory

16.1.1. Lorentz Force

iDrive is a patented technique which uses Lorentz Force to magnetically actuate a cantilever with an oscillating current that flows through the legs. The Lorentz force acting on a current flowing through a wire is shown in Figure 16.1 on page 185. The force vector $\vec{F}$ is a cross product of the current $\vec{i}$, flowing through a wire with length l, and the magnetic field $\vec{B}$ vectors and is, therefore, orthogonal to both $\vec{i}$ and $\vec{B}$:

$$\vec{F} = \vec{i} \times \vec{B} l \tag{16.1}$$

$$F = ilB \sin\theta \tag{16.2}$$

where θ is the angle between the magnetic field and the current vectors.

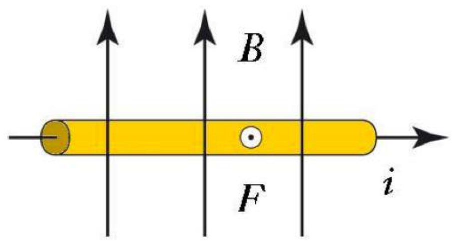


Figure 16.1.: The Lorentz Force F is orthogonal to both current i and the magnetic field B and is pointing out of the page.

Since the force is proportional to the current, if the current is oscillated, the force acting on the wire also oscillates. In 2001, Silberzan and colleagues realized this would make an ideal cantilever actuation mechanism that directly drives the cantilever[1]. Their patented invention avoids the well-known complications of driving the cantilever acoustically and allows for magnetic actuation without the need for coating the cantilever with a ferromagnetic film.

Advantages of iDrive

- **Avoids magnetic materials in the solution**. Magnetic materials typically include rare earth ions that easily oxidize (rust) and perhaps worse, can be toxic. The iDrive cantilevers are coated with inert gold, avoiding this complication.
- **Avoids the use of an oscillating magnetic field**. Experimental studies have shown that an oscillating magnetic field can inadvertently lead to acoustic driving of the cantilever[2]. This unwanted acoustic energy often manifests itself as bumps and dips in the cantilever tune[3]. Because the current loop is made by the cantilever itself, the drive is completely localized. This means that the tunes are smooth and that other parts of the microscope mechanics are not being excited.
- **The phase signal of the cantilever can be used in a manner similar to the phase signal in air**. This is because the drive is well-defined and is a smooth function of frequency, even in liquid. This can lead to some very interesting contrast (Figure 16.2 on page 186, Insulin Fibrils) and even allows advanced techniques such as Q-control.

[1] Buguin, A./Roure, O. Du/Silberzan, P., Active atomic force microscopy cantilevers for imaging in liquids. Applied Physics Letters, 78 2001, Nr. 19 ⟨URL: `http://link.aip.org/link/?APL/78/2982/1`⟩.

[2] Revenko, I./Proksch, R., Magnetic and acoustic tapping mode microscopy of liquid phase phospholipid bilayers and DNA molecules. Journal of Applied Physics, 87 2000, Nr. 1.

[3] Han, Wenhai/Lindsay, S. M./Jing, Tianwei, A magnetically driven oscillating probe microscope for operation in liquids. Applied Physics Letters, 69 1996, Nr. 26 ⟨URL: `http://link.aip.org/link/?APL/69/4111/1`⟩.

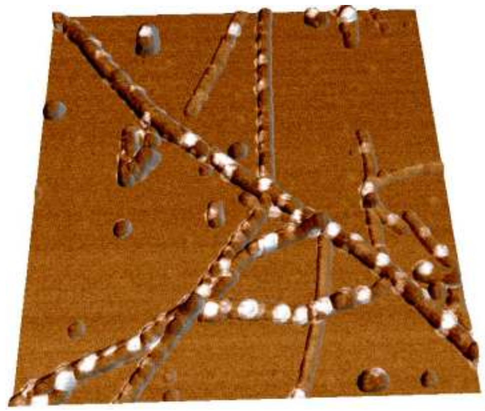


Figure 16.2.: Insulin Fibrils imaged in water. Phase signal overlaid onto topography. The "white" regions in the image are from the phase channel and correspond to higher energy dissipation. 1µm scan.

16.2. iDrive Requirements

Attention	iDrive imaging requires a specialized iDrive cantilever holder and specialized cantilevers for operation.

16.2.1. Cantilevers

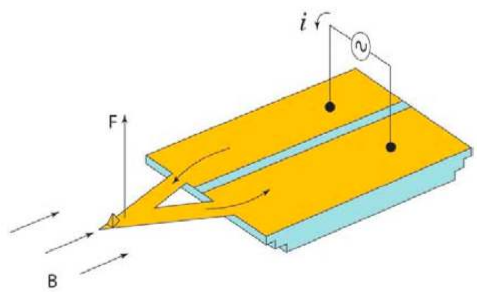
Note	The TR400/800 probes are permanently discontinued.

A generalized schematic of a iDrive cantilever is shown in 16.3a. This is how the bias is applied to one side of the chip and will flow through the legs of the lever, inducing the Lorentz forces on the lever.

A list of available probes can be found on the Asylum Research Probe Store. We currently have two iDrive compatible probes, model numbers BL-TR400PB and BL-TR800PB. The BL-TR400PB is the standard cantilevers included with the iDrive accessory. The resonant frequency of these cantilevers in liquid is approximately 6-10 kHz in liquid, and a spring constant of approximately 0.09N/m making them ideal for imaging soft samples. A stiffer cantilever is also available (Model Number BL-TR800PB) if a higher spring constant of 0.61N/m is desired. A close-up view of the probe (Figure 16.4 on page 187) clearly shows each electrical contact along the length of the cantilever chip, connected to one leg of each of the two short 100µm long cantilevers. Only these two 100µm long cantilevers, not the 200µm long cantilever, can be used for iDrive AC mode. This selection is consistent with liquid bio imaging using standard piezo-driven AC mode, where one does not typically use the 200µm long lever. The iDrive cantilever still retains its full functionality as a standard non-iDrive cantilever and can be operated in both contact mode (air and liquid) and piezo driven AC mode in liquid. All of the iDrive cantilevers are coated in a thin layer of biologically safe gold. Other metals such as Platinum are available upon request, just fill out a request on our probe store.

16.2.2. Cantilever holder

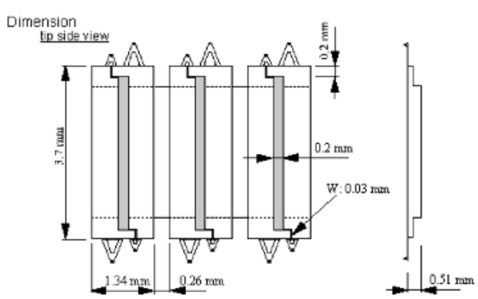
The specialized cantilevers require a specialized cantilever holder to apply the bias to one side of the chip. The specialized cantilever holder is a modified standard holder with an included NbFeB magnet in the glass insert to provide that magnetic field (Figure 16.3b on page 187). The fully enclosed magnet is completely sealed from the sample region. The cantilever chip clamping design has two electrically isolated and conductive spring clips. The iDrive cantilever holder still retains its piezo actuation capability so you can switch between piezo-driven (acoustic) AC mode and iDrive AC mode via the software.



(a) Schematic diagram of Lorentz Forces exerted on a cantilever with integrated circuit loop.

(b) iDrive cantilever holder for the MFP-3D AFM.

Figure 16.3.



(a) Schematic diagram with dimensions.

(b) SEM micrograph.

Figure 16.4.: iDrive cantilevers (Model AR-iDrive-N01)

16.3. Testing Your Cantilevers

It is good practice to test the resistance of the iDrive levers before you use them to make sure they are in good working order. The resistance across the cantilever chip should be between 8-20 ohms, depending on whether the two 100µm long cantilevers are intact or not. The value will be higher if there is only one cantilever attached to the chip. Although it is not necessary to check the resistance every time a new cantilever has been placed in the holder, you may want to make this measurement to troubleshoot the iDrive holder (e.g., no peak appears in the cantilever tune).

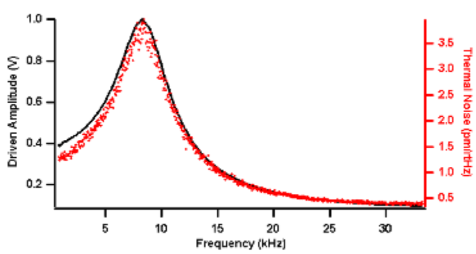
The instructions to measure the resistance across the cantilever chip are listed below (you will need a multimeter):

1. Select “resistance” on your multimeter and, if necessary, the low resistance range.
2. Place the probes on each side of the cantilever chip (one to the left and the other to the right of the terminals).
3. Measure the resistance and confirm a value of 8-20 ohms. If the resistance measured is 0, the cantilever chip has shorted. If the resistance is higher than 30 ohms, the cantilever is likely damaged.
4. Replace the cantilever and repeat the measurement.

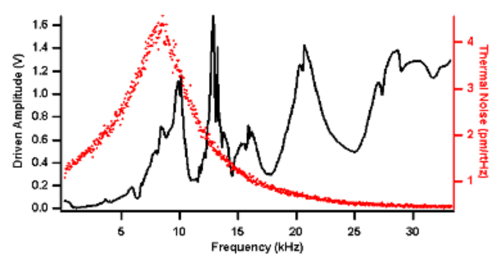
Note	It is not necessary to place the cantilever chip into the iDrive cantilever holder to measure its resistance. You can measure the resistance with your multimeter probe while the chips are still in their storage box.

16.4. iDrive Works in Water

iDrive was specifically designed for imaging soft biological and polymer samples in a liquid environment. iDrive simplifies liquid imaging by eliminating the multitude of resonance peaks due to the mechanical coupling of the shake-piezo to both the cantilever and liquid and, therefore, allows the user to auto tune the cantilever in liquid. A comparison between cantilever tunes acquired with iDrive AC mode and piezo-driven (acoustic) AC mode can be seen in Figure 16.5 on page 188, where both methods are compared to the Brownian “thermal” tune.



(a) iDrive

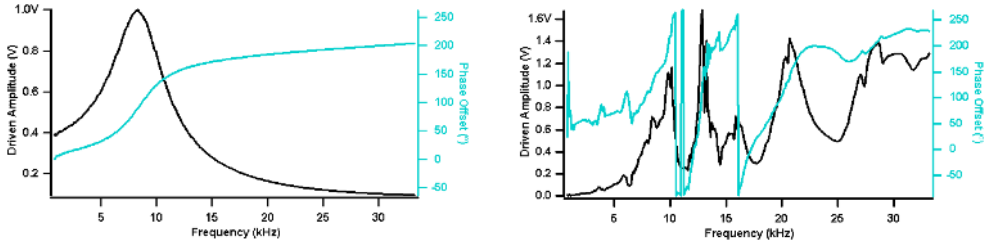


(b) Acoustic

Figure 16.5.: Cantilever tunes acquired with iDrive AC mode (left) and piezo-driven (acoustic) AC mode (right) showing both the mechanical response (black curves) and the thermal noise power spectrum (red curves).

16.4.1. Phase imaging

When using iDrive AC Mode, the phase response in liquid is very similar to that seen in air. This is quite different compared to piezo-driven acoustic AC mode. A comparison of the phase response can be seen in Figure 16.6 on page 189.

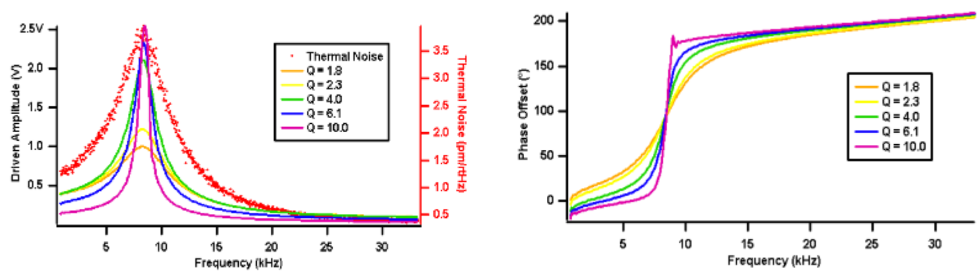


(a) iDrive **(b)** Acoustic

Figure 16.6.: Cantilever tunes acquired with iDrive AC mode (left) and piezo-driven AC mode (right) showing both the mechanical response (black curves) and the phase response (teal curves).

16.4.2. Q-Control

With a “clean” iDrive cantilever tune, it is feasible to use Q-control (see Section 4.6.2.3 on page 39) in liquid; whereas before, there were several issues with using piezo-driven AC mode. It was difficult to decide which peak to select, and some peaks were more responsive than others; and even when the Q gain was increased, the Q for that given peak did not always increase. Similar to air, the phase contrast with iDrive in liquid increases with greater Q (Figure 16.7 on page 189).



(a) Q Control amplitude **(b)** Q Control phase

Figure 16.7.: Cantilever tunes (left) and phase offset (right) acquired using iDrive AC mode with increasing Q.

16.4.3. Cantilever damping

When using iDrive, there is both a significant damping of the cantilever’s oscillation and a shift in the resonance frequency with a decrease in the tip-sample separation. Both the damping and the resonance shifts can be clearly seen in Figure 16.8 on page 190, where the cantilever tune is measured at fixed distances above the sample surface. This decrease in the cantilever’s oscillation amplitude can be explained by the Simple Harmonic Oscillator Model. The equation is as follows:

$$\omega_{\max} = \omega_0(1 - {}^1/_{4Q^2}) \tag{16.3}$$

where $\omega_{\max}$ is the maximum resonance, ω_0 is the free cantilever resonance, and Q is the quality factor.

These results are consistent with theoretical and experimental data that explains the observed decrease in cantilever resonance and shift to a lower value[4]. The hydrodynamic effect on the cantilever is not seen with piezo-driven acoustic mode since the multitude of peaks that are observed in the cantilever tune correspond to non-cantilever resonances, typically due to the resonances of various hardware components.

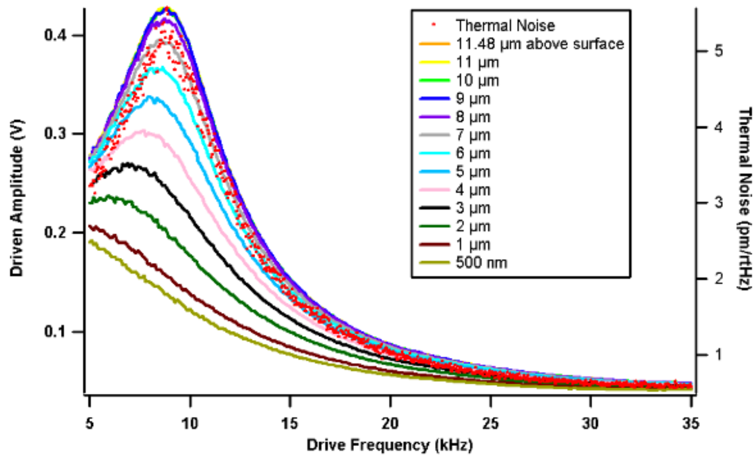
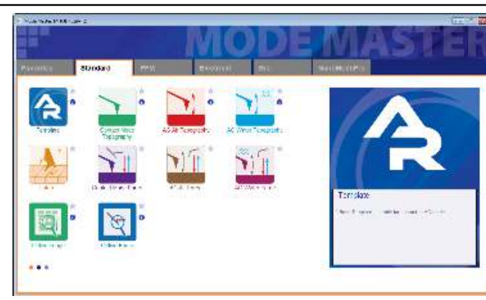


Figure 16.8.: Cantilever amplitudes at different Z positions using iDrive AC mode

16.5. iDrive Operation

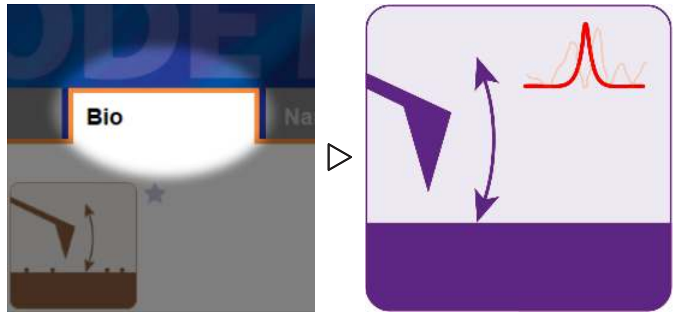
1. **The Mode Master window:**

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



[4] Rankl, Christian et al., Hydrodynamic damping of a magnetically oscillated cantilever close to a surface. Ultramicroscopy, 100 Aug 2004, Nr. 3-4 ⟨URL: http://dx.doi.org/10.1016/j.ultramic.2003.12.014⟩.

2.



Select Mode:

- Select *Bio > iDrive*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

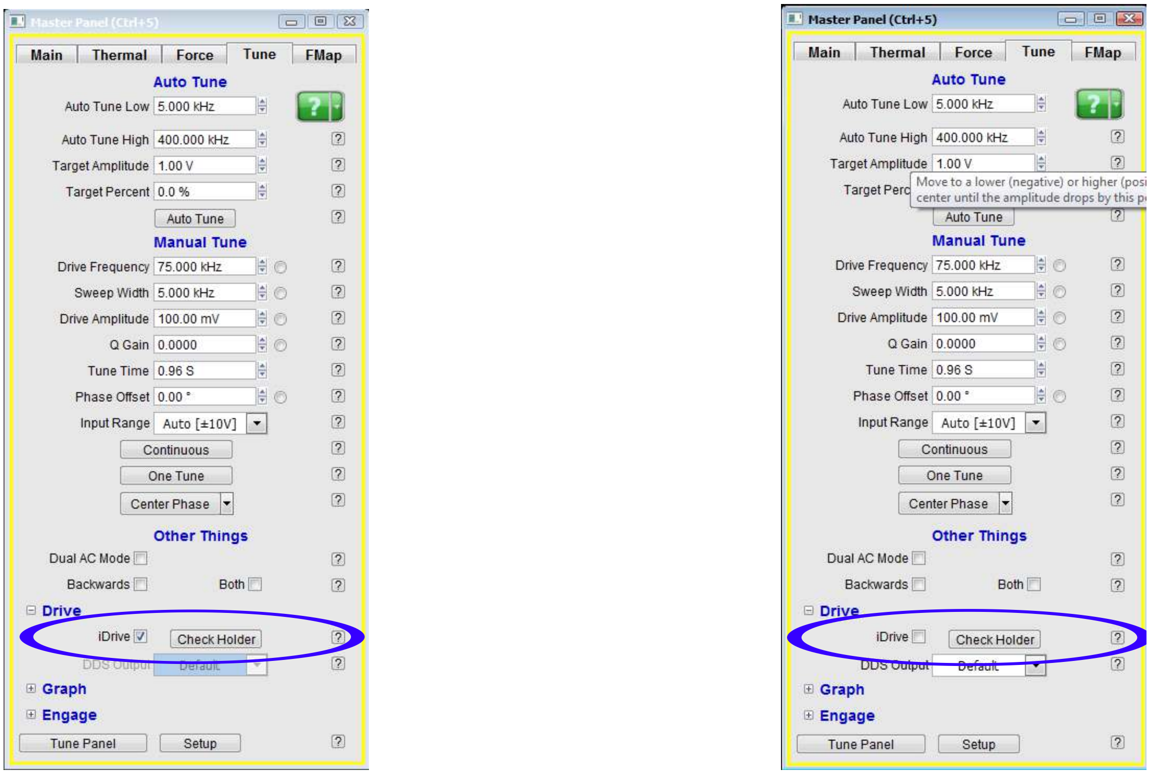
Note The iDrive cantilever holder must be first attached to the AFM to load the iDrive template.

There is no special sample preparation required for iDrive. Standard glass slides with mica, gold, or any other substrate can be used.

Install an iDrive compatible cantilever (Model AR-iDrive-N01) into the cantilever holder pocket, as usual and tighten the PEEK screw until the cantilever chip is secure (do not over tighten).

Similar to operating in piezo-driven AC mode, you need to:

1. Tune the cantilever, much as described in Chapter 5 on page 46. The exception is that you need to make sure that the iDrive checkbox is checked as shown in 16.9a.
2. Select the appropriate amplitude setpoint value and approach towards the surface.



(a) standard cantilever holders

(b) iDrive cantilever holders but iDrive not selected

Figure 16.9.: Tune Panel configurations.

16.5.1. Tuning the cantilever

To tune the cantilever:

- If the software is opened before the iDrive cantilever holder is attached to the head, or when a standard cantilever holder is already attached to the head, the iDrive box will be shaded (Figure 16.9a on page 192). To enable the iDrive box, attach the iDrive cantilever holder to the MFP-3D head and click the 'Check Holder' button (Figure 16.9b on page 192).
- When iDrive is selected, the *Auto Tune Low* parameter decreases to 5 kHz so that the resonance of the AR-iDrive-N01 cantilevers, which is approximately 6-10 kHz, will be within the range for the Auto Tune function.
- Once the iDrive box has been selected, verify that the *Target Amplitude* is set to 1V, and then click the 'Auto Tune' button.
- Confirm that the *Drive Frequency* is between 6-10 kHz and that the *Free Amplitude* in the Sum and Deflection Meter Panel is "1V".

16.5.2. Engage on the surface

The iDrive cantilevers require a greater decrease in the setpoint before the tip is engaged onto the surface. To simplify the approach process:

- Enter an amplitude setpoint value of 500mV.
- Engage onto the surface as usual.
- When the Free Amplitude in the Sum and Deflection Meter Panel equals the Amplitude Setpoint and the computer beeps (if you have your speakers on), continue to lower the head until the Z Voltage position is approximately 35V (i.e., Z is 1/2 way in the BLUE).
- Decrease the amplitude setpoint, one click at a time, until the Z Voltage position stops increasing. With this amplitude setpoint value, the tip should be tracking the surface, and only slight modifications to the amplitude setpoint may be necessary.

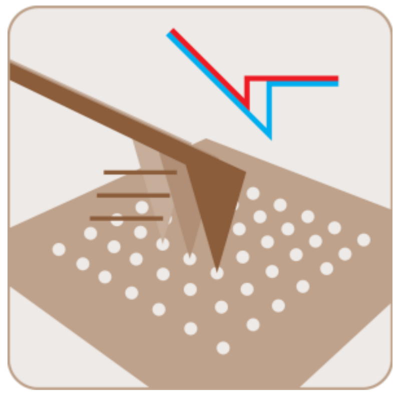
Due to the damping of the cantilever and shift in resonance, see Section 16.4.3 on page 189, it is recommended that you re-tune the cantilever once the tip is on the surface. To do this:

- Click the 'Stop' button, and then go to the Tune Panel and click the 'Auto Tune' button.
- Alternatively, you can choose to enter a *Target Percent* of negative (-) 5-10%, which automatically selects a resonant frequency 5-10% off and to the left of the resonant peak. This offset compensates for the shift in resonant frequency. This step is recommended since the jump into contact is quicker due to the attractive forces, and therefore, less force is exerted onto the sample.

For these particular cantilevers, the optimal amplitude setpoint is 200mV or less. Decrease the drive amplitude and setpoint to achieve this final setpoint value. For example, if the tip is tracking the surface well with an amplitude setpoint of 325mV then decrease first the drive amplitude then the amplitude setpoint, one click at a time, until the final amplitude setpoint is approximately 200mV. You can use even lower amplitude setpoint values if lower oscillation amplitudes are desired.

17. Fast Force Mapping

CHAPTER REV. 2007, DATED 03/27/2018, 20:30. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.



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17.1. System Requirements for Fast Force Mapping

Attention	• Must be using a Cypher AFM or MFP-3D Infinity AFM (FFM is not available for MFP-3D Origin or Origin+) • Fast Force Mapping is an optional AFM technique and requires an additional software license to be activated. • AFM software version 16 or higher is required in order to run Fast Force Mapping mode.

17.2. Introduction

Fast Force Mapping (FFM) is a deflection (sub-cantilever resonance frequency) feedback technique that allows you to acquire data, such as topography, adhesion, mechanical, electrical properties, etc., on a very wide range of materials. In contrast to Contact Mode or AC Mode, the principle behind FFM is based on force/deflection feedback force-distance curves performed at rates much higher than standard Force Maps. FFM force curves are acquired at frequencies ranging from 10 Hz up to 300 Hz for MFP-3D Infinity AFMs only and up to 1000 Hz for all Cypher AFMs while a feedback loop monitors the maximum force detected for each individual force curve. Each force curve is recorded and analyzed to generate both topography information and mechanical properties of the sample. Additional signals, e.g., electrical conductivity, can also be measured to obtain supplementary information about the sample through use of the ORCA cantilever holder. The main and obvious advantage of the FFM technique is much faster data acquisition, compared to standard Force Mapping (Section 18.3 on page 248), while still capturing all the information provided by force curves. With FFM, you can collect force maps with a much higher pixel density and aquistion times much lower than standard force maps. The FFM technique also provides a novel method for imaging a class of samples that are both extremely adhesive and delicate, which might otherwise be challenging to image with dynamic mode imaging techniques (e.g. AC mode).

A sinusoidal driving voltage applied to the Z-actuator is used to oscillate the cantilever position at frequencies ranging from 10 Hz to 300 Hz (Infinity) and up to 1000 Hz (Cypher); far below cantilever resonance. For each force curve, the maximum deflection signal, read on the photodetector, is used to calculate the Z height measured by the Z-sensor. Maximum force, “Max Force”, is calculated in real time and is used as an error signal for the Z feedback loop. While the probe position is driven (sinusoidal wave) in the Z-axis, the sample is moving underneath it in a raster pattern in the X-Y plane. The resolution of the FFM image is determined by the number of points in each scan line, as each pixel is an individual force curve.

The software continuously digitizes Deflection, Z-Sensor, Z-Voltage and Current (or Current2) signals at 2MHz (Cypher) or 500kHz (Infinity).

Each force curve is then analyzed to provide sample topography and its Young’s modulus (based on the chosen model and fit parameters). If an electrical bias is applied to the sample, and a conductive tip is used, ORCA measurements (see 9) can be performed at the same time.

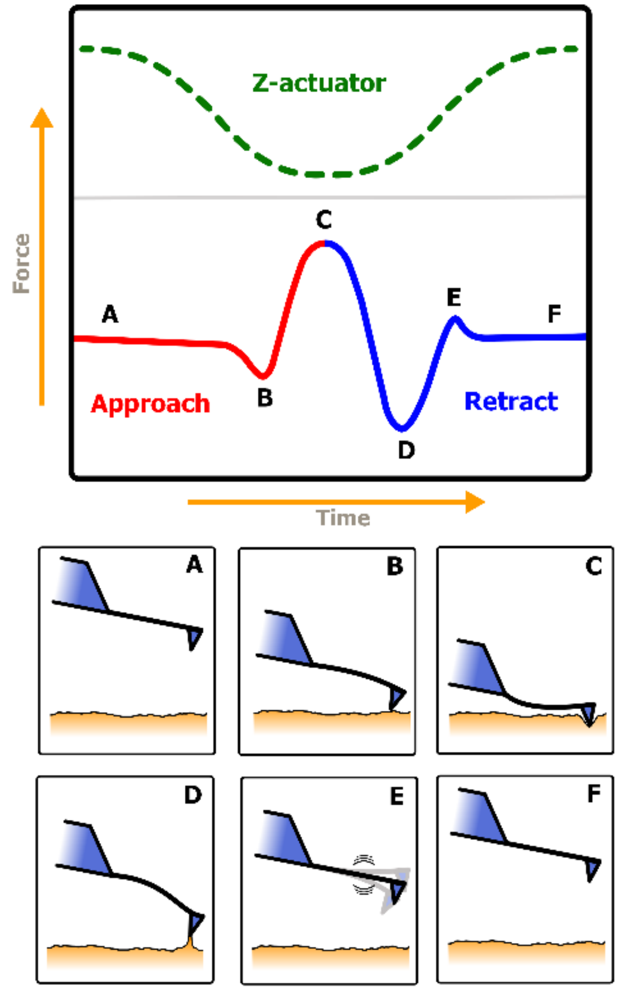


Figure 17.1.: Diagram of general Fast Force Map technique. As the Z-actuator changes position sinusoidally versus time, the cantilever force does as well. A, B, C, D, E, and F show discrete points of interest along the curve during the cantilever approach and retraction.

17.3. Fast Force Mapping in Contact Mode

FFM is natively a contact mode-based technique; therefore, you need to choose a probe with a spring constant matching the stiffness of the sample.

For this demonstration and example, we use the following items:

- A polystyrene/polypropylene thin film sample
- AC200TS Olympus probe with ~150 kHz resonance and ~10 N/m spring constant

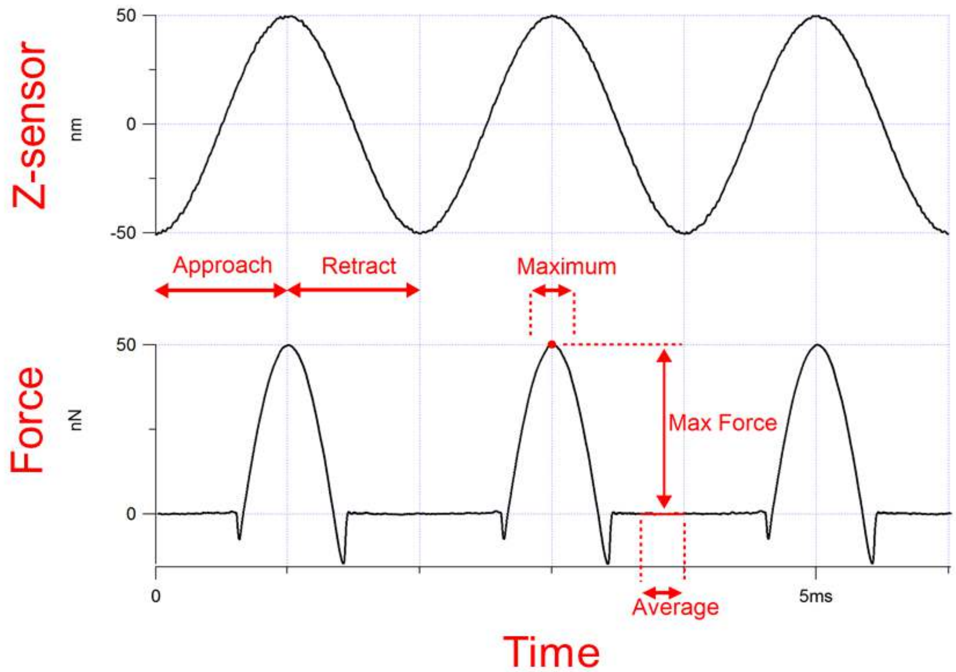


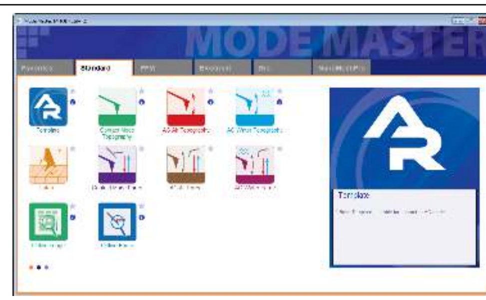
Figure 17.2.: Plot of cantilever oscillation measured by the Z-Sensor (excited by driving voltage) (top) and the resulting force (bottom) when the tip is pushing on a hard surface. The “Max Force” arrow indicates how the setpoint force is calculated for each force curve.

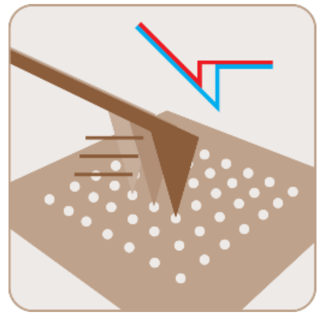
17.3.1. FFM Automated Setup: GetStarted™

1.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the ‘Mode Master’ button at the bottom of the screen: .





2.

Select Mode:

- Select *NanoMechPro tab > Fast Force Map*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

Note Loading FFM from the Mode Master automatically starts in the GetStarted™ configuration.

3.

Automatic Imaging Setup - GetStarted™:

- Follow the instructions to set up your experiment.

Specific Parameters for FFM - GetStarted™:

Note Some of these parameters are adjusted automatically depending on user input

- *Scan Points & Lines*
  - These two parameters determine the number of scan points (pixels) in each scan line and the number of lines in the scanned area. In contrast to other techniques, during FFM the number of force curves corresponds exactly to the number of pixels in the image. For comparison, during AC mode, the tip oscillates at a much higher frequency than the number of pixels. Therefore, for each pixel, it is the average value of the amplitude of oscillations that is measured by the feedback loop. To improve the resolution of images, these two parameters can be increased. However, the acquisition time will increase at the same time.
    - Acquisition time = (lines x points per line x 2.5)/ Z rate
- *Sample Roughness*
  - This parameter is used to determine the force distance. It should be chosen to roughly represent the height difference between the lowest and highest features on the sample.
- *Z-Rate*
  - This is the ramp rate at which the force curves will be performed and how fast the Z actuator is moving up and down above the surface. You can increase the Z-rate until you see instability in the force curve; the instability arises when the deflection is changing rapidly and the setpoint cannot be met on all curves. On Infinity, the maximum Z-rate is 300 Hz. On Cypher S or ES, the maximum Z-rate is 2000 Hz.

4.

- *Setpoint*
  - This is the setpoint value used for feedback. Maximal force is calculated in real-time and used as error signal for the Z-feedback loop. When the setpoint value is reached, the lever is pulled away from the surface.
  - The setpoint value is displayed in units of Newton and Volt (they are related by the cantilever calibration values InvOLS and k). The Newton value provides information about the amount of normal force applied on the sample, and the Volt value can be compared with the deflection value displayed on the Sum and Deflection Meter Panel. For example, before engaging on the surface, the *Deflection* should read “0 Volt”, and the *Setpoint* should be set to a value (Volt or Newton) greater than 0.
  - The setpoint value is also used to obtain an image of the surface topography. For each pixel, the software measures the Z height at which the setpoint value was reached. The Z height for each pixel is then used to map the topography of the surface.
- *Force Distance*
  - Force distance is the distance the piezo travels during the force curve (extend or retract portion). This distance should match or exceed the topography variations of the sample.
- *Sample Stiffness*

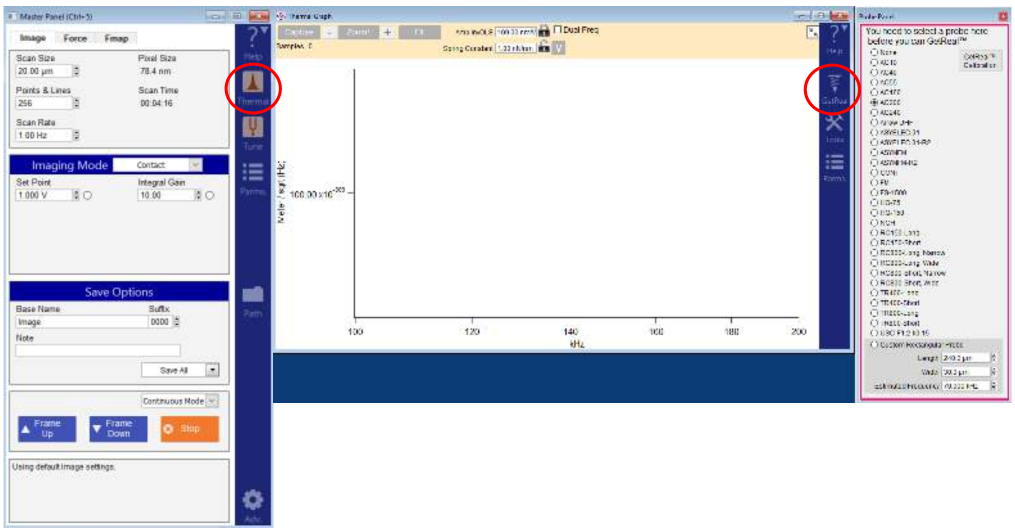
17.3.2. Experimental setup

1.

Align laser:

- Open the Video Panel.
- Use the arrows in the Video Panel to move the laser spot onto the cantilever and maximize the SUM signal.

2.



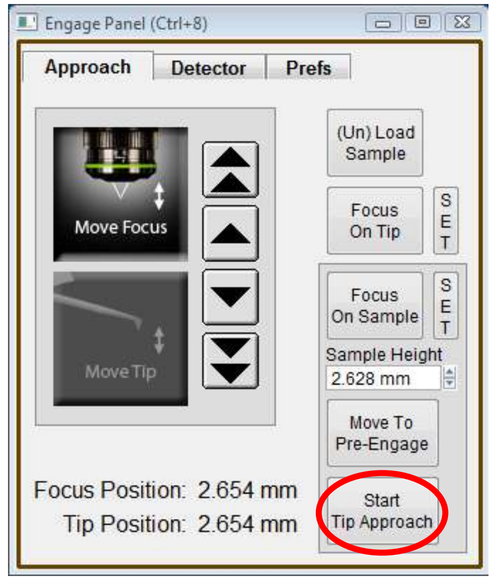
Calibrate probe:

- Click the ‘Thermal’ icon in the Master Panel. The Thermal Graph will appear.
- Click the ‘GetReal’ icon (right side of panel). The Probe Panel opens.
  - Select the probe you are using for the experiment.
- Click the ‘GetReal Calibration’ button.
- When the calibration has completed, the Amp InvOLS and Spring Constant values at the top part of the thermal graph will be updated.

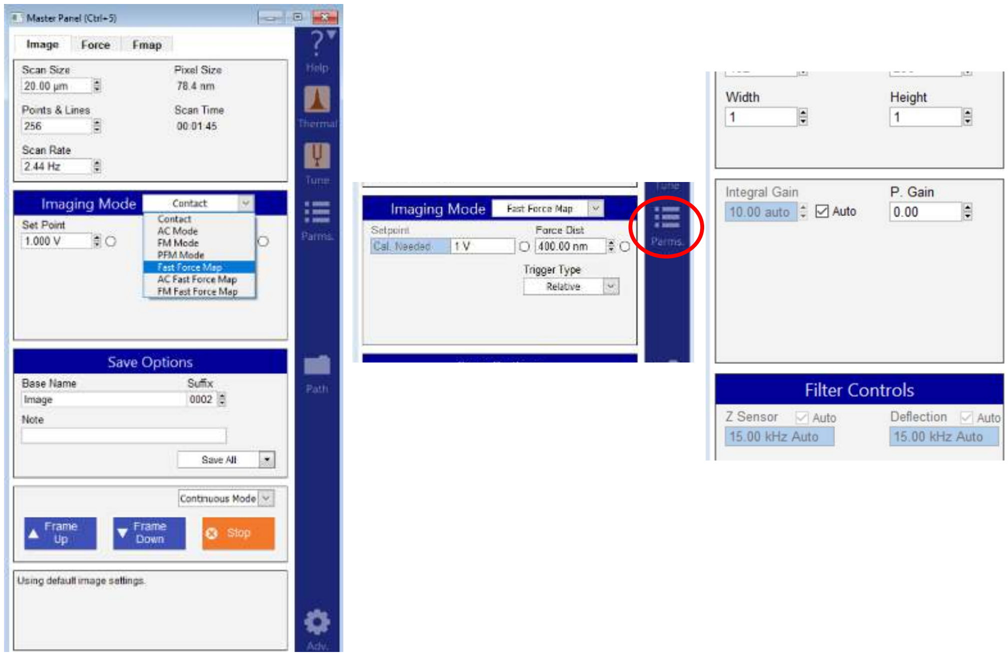
3.

Approach the sample surface:

- In Master Panel, set the following parameters:
  - *Imaging Mode*: Contact
  - *Set Point*: 0.2 V (for this particular probe and sample)
- In the Engage Panel, set the following parameters:
  - Set the tip position while focused on the tip.
  - Set the sample position while focused on the sample.
  - Click the 'Start Tip Approach' button.



4.



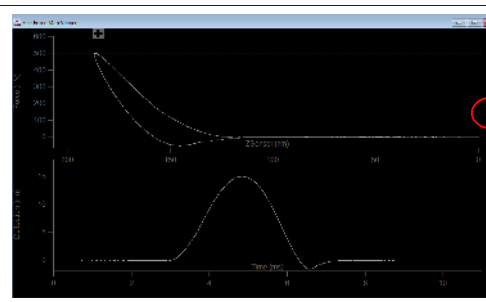
Switch to FFM mode and start imaging:

- In Master Panel, switch the *Imaging Mode* from “Contact” to “Fast Force Map”.
- Parameters related to FFM are now shown. Set as follows:
  - *Setpoint* (now displayed in units of Newtons and in Volts): 10 nN
  - *Force Distance*, set to 200 nm
  - *Trigger Type*: Relative
- Set the other parameters in the Master Panel:
  - *Scan Size*: 5 µm
  - *Points & Lines*: 256
  - *Z-rate*: 500 Hz
- The Gains are located in the Parms panel. To open, click the *Parms* icon in the right column of the Master Panel.
- Click the ‘Engage’ button in the Sum and Deflection Meter Panel.

5.

Engaging:

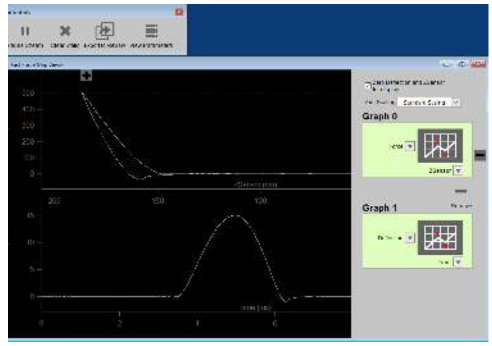
- As the tip engages on the surface, Fast Force Map Viewer will appear.
- Display choices are available by clicking the “plus” sign.



6.

Real time Force curves:

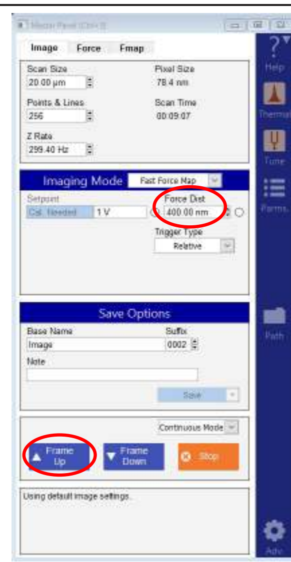
- The Fast Force Map Viewer can show up to two channels.
- Each graph has several options for each axis. In this example:
  - Graph 0 is Force vs. ZSensor
  - Graph 1 is Deflection vs. Time
- The green line on the top graph indicates the setpoint.
- Top button controls allow for superposition of several already acquired curves over the real-time curve.

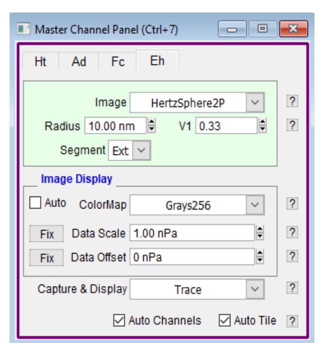


7.

Imaging parameters:

- Four channels can be collected simultaneously during FFM imaging. On the Master Channel Panel, they are:
  - Height (Ht)
  - Adhesion (Ad)
  - Max Force (Fc)
  - Calculated Modulus (Eh). Model, Hertz in this example, and tip radius must be adjusted prior to imaging.
- Adjust the force distance (*Force Dist*) and the *Setpoint* as needed.
- When the imaging parameters are optimized and the force curves look good in the FFM viewer, click 'Frame up' to start imaging.





17.4. FFM in AC Mode

The FFM technique can also be operated while simultaneously oscillating the cantilever at resonance, instead of in Contact mode.

The following demonstration is for use with a Cypher AFM equipped with blueDrive and is intended for imaging a flat sample in liquid.

17.4.1. Preparation and calibration

1.

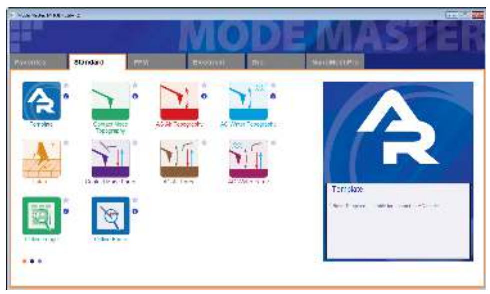
Prepare:

- Fresh calcite sample
- Clean cantilever holder to be used for imaging, either liquid holder or perfusion holder.
- Load a FS-1500AuD probe (f = ~1.5 MHz, k = ~6 N/m) into the cantilever holder.
- Launch AFM software version 16 or higher and enable FFM mode.
- Use blueDrive for the cantilever drive and with the 0.1x ND filter cube.

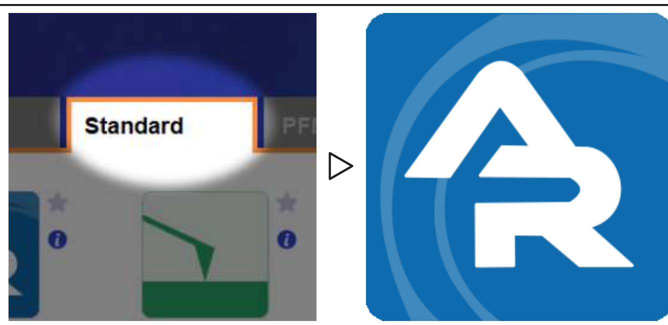
2.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the ‘Mode Master’ button at the bottom of the screen: .



3.



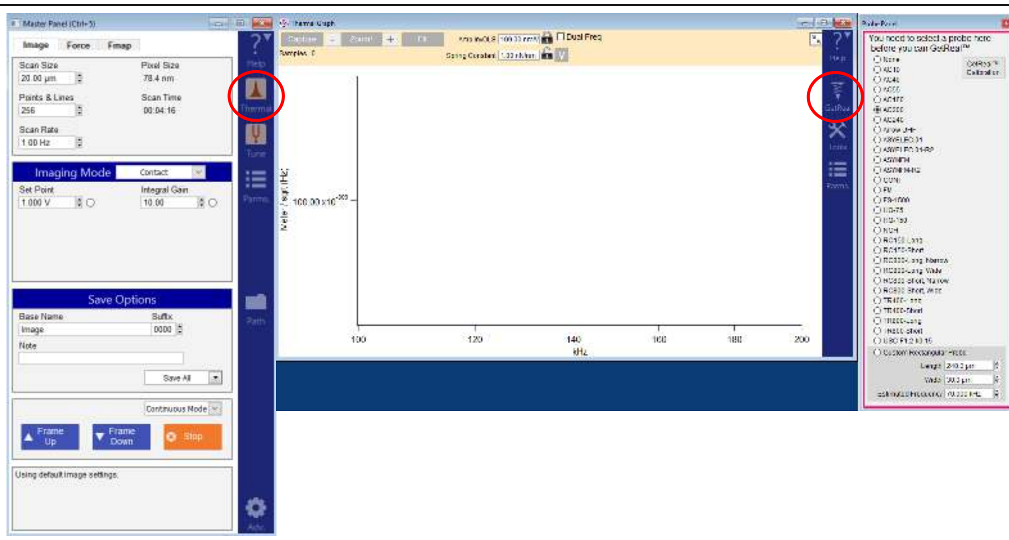
Select mode:

- Select *Standard > Template*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

Note Loading Template from the Mode Master will automatically start in a nonspecific set up of Contact mode.

4. **Align laser:**

- Open the Video Panel.
- Use the arrows in the Video Panel to move the red laser spot onto the cantilever. Try to maximize the SUM signal.

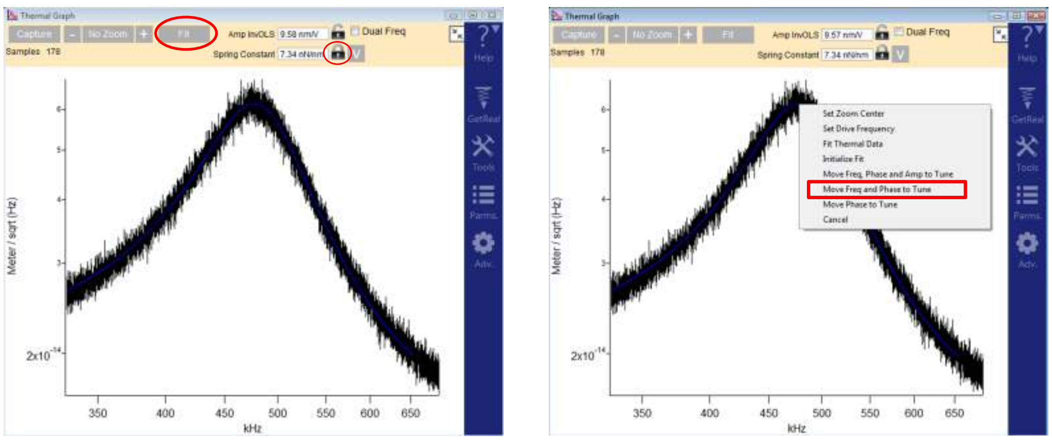
5. 

Calibrate probe (in air):

- Click the 'Thermal' icon in the Master Panel. The Thermal Graph appears.
- Click the 'GetReal' icon to open the Probe Panel.
- Select the probe to use for the experiment.
- Click 'GetReal Calibration'.
- When the calibration has completed, the Amp InvOLS and Spring Constant values at the top part of the Thermal Graph will be updated

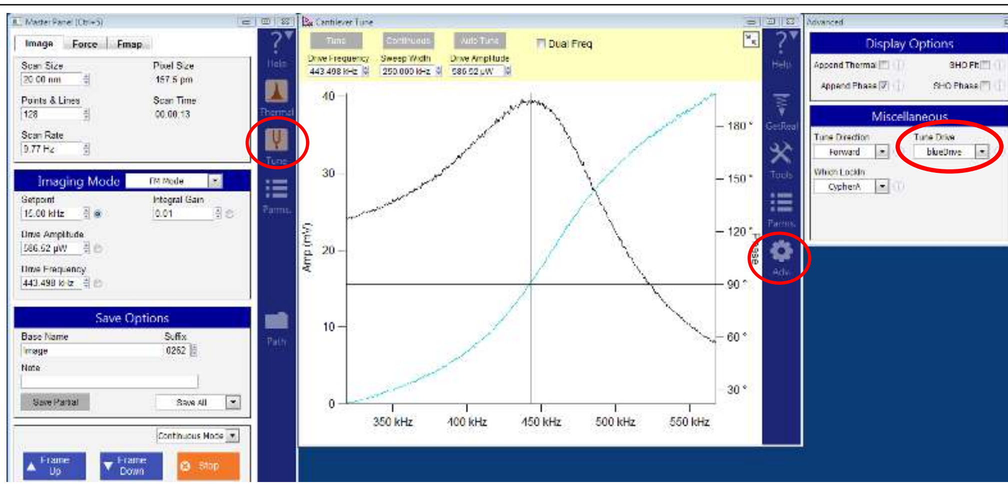
6. Remove the cantilever holder and place the freshly cleaved calcite sample on the scanner.

7. Add a drop of water to the sample

8. Put a drop of water on the probe and place the cantilever holder back on the scanner
9. While looking at the distance between the probe and the sample, approach the sample to the tip so that both water drops join and the sample and lever are both in water environment.
10. Refocus and realign the laser on the cantilever, now in liquid. The Sum should be ~ 6V.
11. 

Capture a thermal of the cantilever in liquid:

- Make sure that the padlock besides Spring Constant is locked.
- Refit the thermal data to obtain the updated (water) InvOLS value.
- Transfer the frequency of thermal peak to the Tune Panel.
  - Right-click on the peak of the Thermal Graph and choose *Move Freq and Phase to Tune.*

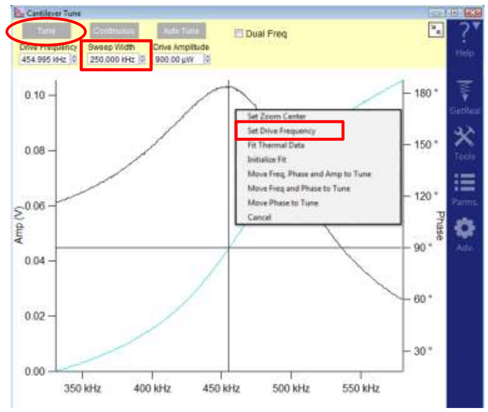
12. 

Turn on blueDrive photothermal excitation:

- In the Cantilever Tune graph, click the 'Adv.' icon (located in the right column, as shown in the above screenshot).
- In the Advanced panel, select "blueDrive" from the *Tune Drive* dropdown menu.

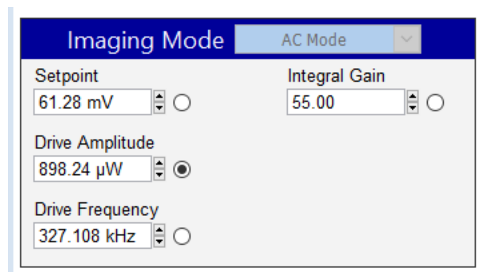
13. **Cantilever Tune panel settings:**

- Set the *Sweep Width* to “250 kHz”.
- Click one ‘Tune’.
- When tune is done, right-click on the peak and select *Set Drive Frequency*.
- Move the blue laser spot around the base of the cantilever to maximize the amplitude.
- Adjust the *Drive Amplitude* in the Master Panel until the amplitude reaches ~ 80 mV. This will be visible on the graph and in the Sum and Deflection Meter Panel.



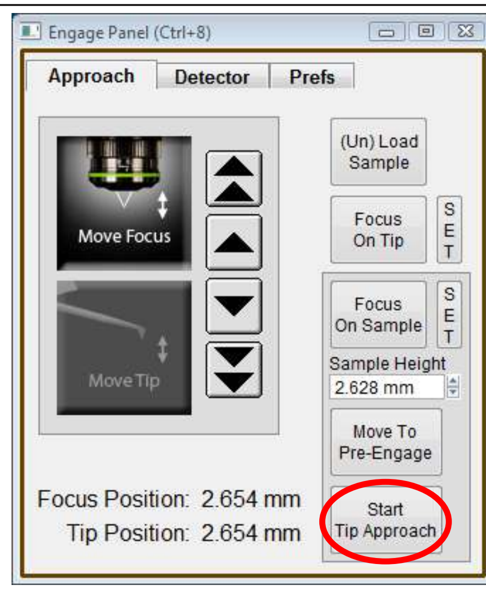
14. **Master Panel settings:**

- Set the *Setpoint* to ~ 60 mV.
- Set the *Integral Gain* to “55”.



15. **Engage Panel settings:**

- Set the tip position while focused on the tip.
- Set the sample position while focused on the sample.
- Click the ‘Move to Pre-Engage’ button.
- Click the ‘Start Tip Approach’ button.



16. Once the tip is in piezo range of the sample, capture another Thermal Tune with z-piezo at 0V (sample is 2-3 um from surface).

17. Lock padlock beside Spring Constant on Thermal Graph to recalculate Amplitude InvOLS.

18. Right-click on the peak in Thermal Graph and select *Move Freq and Phase to Tune*.

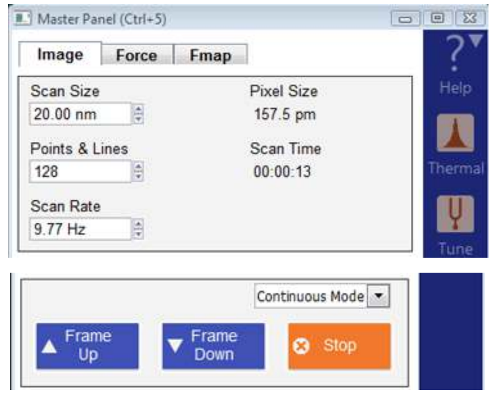
19. When the tune is complete, right-click on the peak and center phase.

17.4.2. Imaging in AC Mode

1.

Master panel imaging parameters:

- Set the *Scan Size* to “20 nm”.
- Set the *Scan Rate* to “10 Hz”.
- Start imaging by clicking ‘Frame Down’.



2. Start imaging:

a) Decrease the free amplitude by decreasing *Drive Amplitude* in the Master Panel.

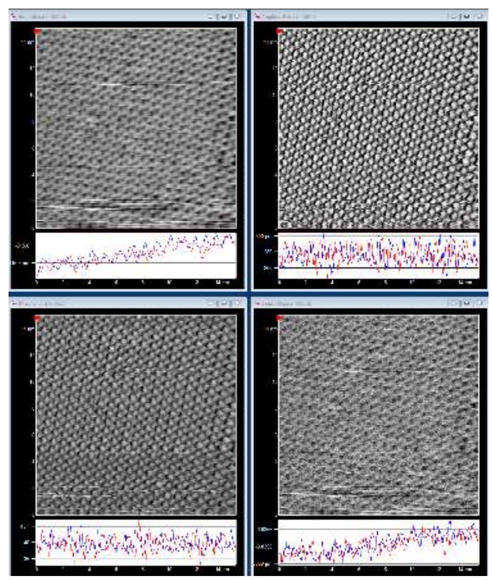
b) Decrease the *Setpoint* until trace and retrace overlap each other.

c) Repeat several times to image with the lowest amplitude possible.

3.

Imaging:

- Acquire a 20 nm image and resize the scan to 10 nm.
- Channels that are acquired include:
  - Height
  - Amplitude
  - Phase
  - ZSensor
- Once the imaging looks optimized, stop the scan and switch to AC-FFM mode.

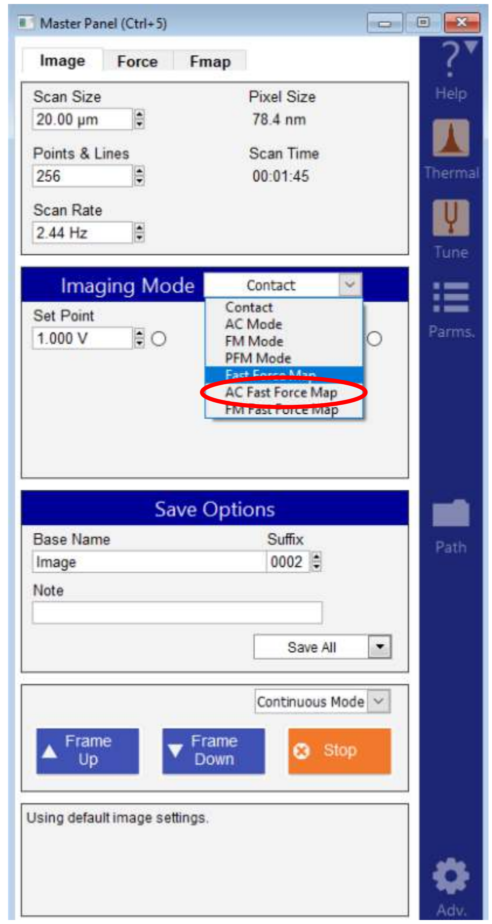


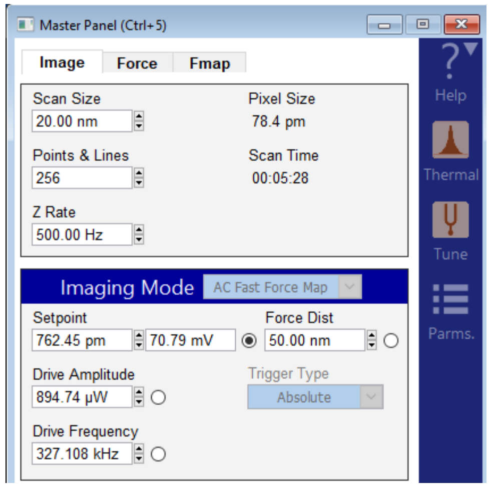
17.4.3. Imaging with AC-FFM

1.

Switch to AC-FFM:

- Switch to “AC Fast Force Map” from the *Imaging Mode* dropdown menu in the Master Panel.



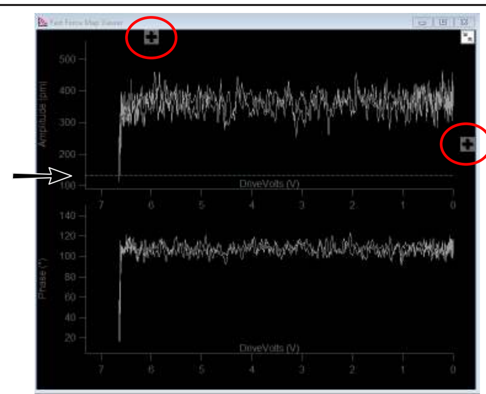


2. **Set imaging parameters:** The Master Panel should now display parameters related to AC-FFM mode

- Set the Imaging Mode parameters:
  - Setpoint (now displayed in units of meters and in Volts): 70 mV
  - Force Distance: 50 nm
  - Trigger Type - Absolute
- Set the other parameters in the Master Panel:
  - *Scan Size*: 20 nm
  - *Points & Lines*: 256
  - *Scan Rate* (Z-rate): 500 Hz
- The Gains are located in the Parms panel. Set to "10".
- Click the 'Engage' button in the Sum and Deflection Meter Panel.

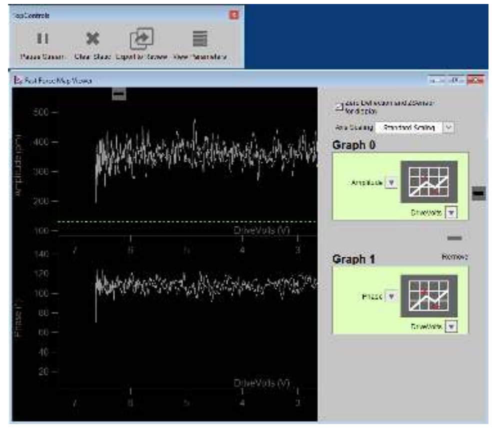
3. **Engage:**

- As the tip engages on the surface, the Fast Force Map Viewer appears.
- Display choices are available by clicking on the "plus" sign.
- The arrow in the top graph indicates the setpoint value.



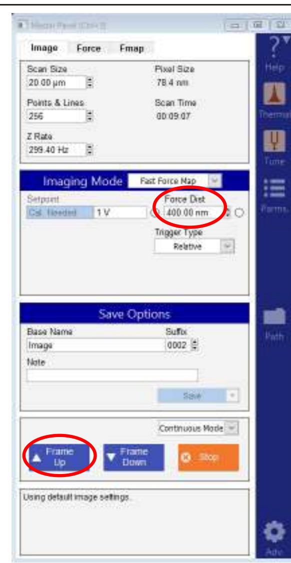
4. **Real time force curves:**

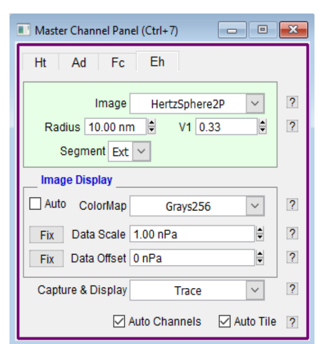
- Fast Force Map Viewer can display up to 2 channels (graphs)
- Each graph has several options for each axis, choose the appropriate axes for the imaging mode. For AC FFM, choose:
  - Graph 0 (top): Amplitude (pm) vs DriveVolts (V)
  - Graph 1 (bottom): Phase (°) vs DriveVolts (V)
- Controls at the top allow for the superposition of several already acquired curves over the realtime curve.



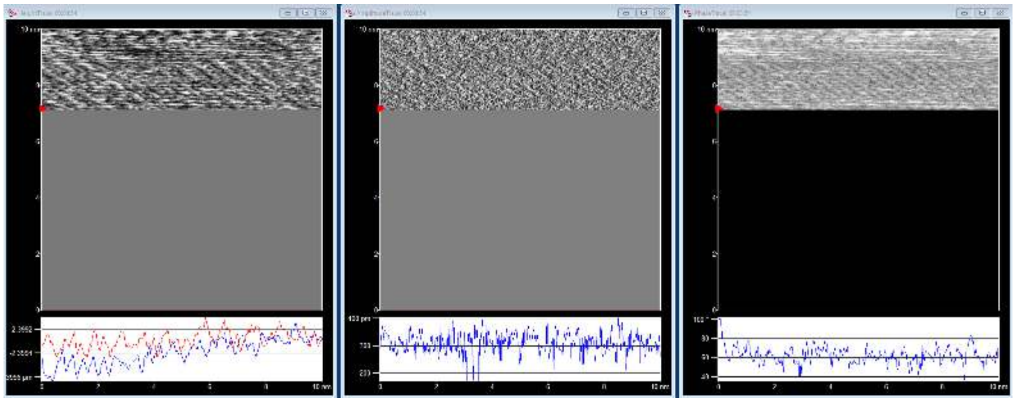
5. **Imaging parameters:**

- Adjust the force distance and setpoint until the curves in the viewer look good.
- When the imaging parameters are optimized, click 'Frame up' to start imaging.





6.



Imaging

- Data is collected in 3 channels during AC-FFM:
  - Height
  - Amplitude
  - Phase

17.5. Fast Force Mapping in FM mode (FM-FFM)

The FFM technique can also be operated while simultaneously oscillating the cantilever at resonance, instead of in Contact mode, by using a Phase-Lock Loop (PPL) to maintain the cantilever drive is on resonance.

The following demonstration is for use with a Cypher AFM equipped with blueDrive and is intended for imaging a flat sample in liquid.

You are guided through the following steps:

1) Calibration of a probe

2) Acquisition of an AC mode image. This step allows you to determine if the probe is sharp and if the area of interest is flat/clean.

3) Acquisition of AC force curves - this step will allow the user to approach the sample to a defined distance

4) Acquisition of FM curves - this step will allow to check FM gains

5) Acquisition of FM-FFM images and force curves

17.5.1. Preparation and Calibration

1.

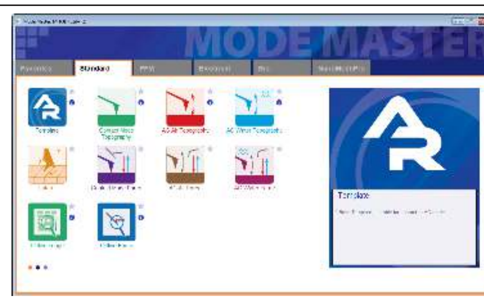
Preparation:

- Fresh calcite sample
- Clean cantilever holder to be used for imaging, either liquid holder or perfusion holder.
- Load a FS-1500AuD probe (f = ~1.5 MHz, k = ~6 N/m) into the cantilever holder.
- Launch AFM software version 16 or higher with the FFM mode enabled.
- Use blueDrive for cantilever drive and with the 0.1x ND filter cube.

2.

The Mode Master window:

- The software should now be showing the Mode Master window.
- If not, click the 'Mode Master' button at the bottom of the screen: .



3.

Select mode:

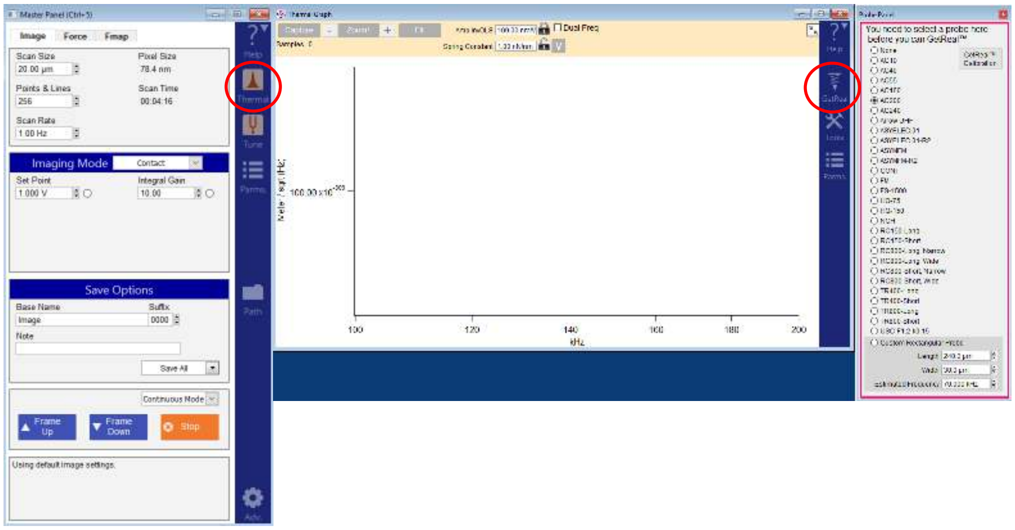
- Select *Standard > Template*
- The screen rearranges to present all the controls necessary for this type of AFM imaging.

Note Loading Template from Mode Master automatically starts in a nonspecific setup of Contact mode.

4.

Align laser

- Open the Video window.
- Use the arrows in the Video Panel to move the red laser spot onto the cantilever. Try to maximize the SUM signal but keep the laser spot at the end of the lever (see image at right).

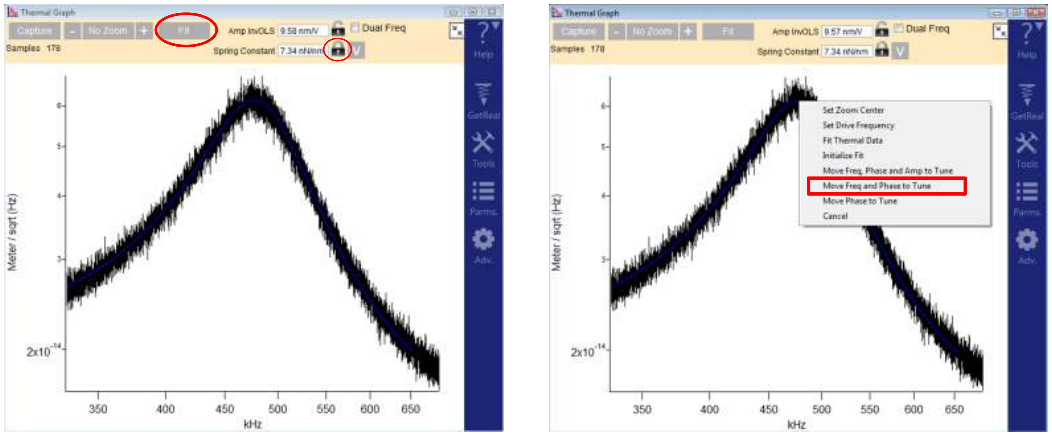
5. 

Calibrate probe (in air):

- Click the 'Thermal' icon in the Master Panel. The Thermal Graph appears.
- Click the 'GetReal' icon to open the Probe Panel.
- Choose the probe to be used for the experiment, for this experiment choose "Arrow UHF".
- Click the 'GetReal Calibration' button.
- When the calibration has completed, the Amp InvOLS and Spring Constant values at the top part of the thermal graph will be updated.

6. Remove the cantilever holder and place a freshly cleaved calcite sample on the scanner.
7. Add a drop of water onto the sample.
8. Add a drop of water onto the probe and place the cantilever holder back on the scanner.
9. While looking at the distance between the probe and the sample, approach the sample to the tip so that both water drops join.
10. Refocus and realign the laser on the cantilever, now in liquid. The Sum should be ~ 6V.

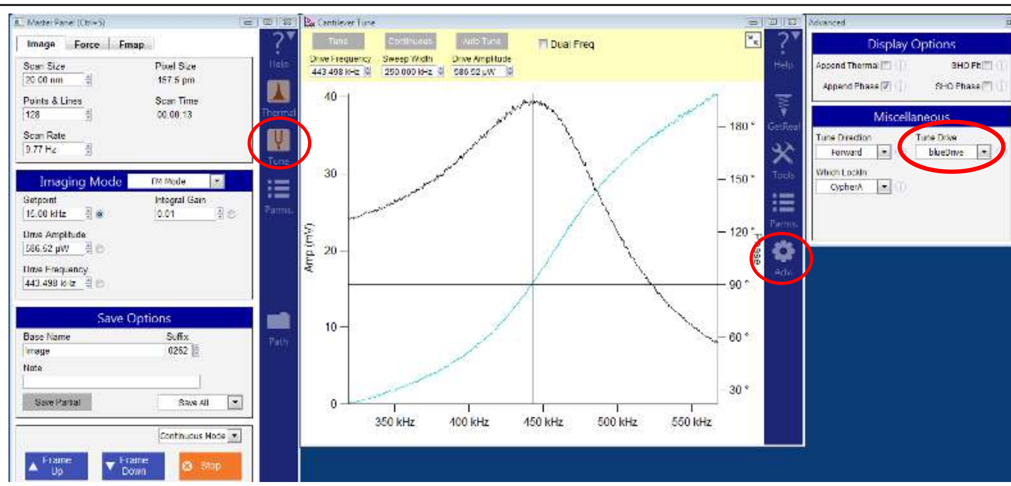
11.



Capture a thermal of the cantilever in liquid:

- Make sure that the padlock besides Spring Constant is locked.
- Refit the thermal data to obtain the updated (water) InvOLS value.
- Transfer the frequency of the thermal peak to the Cantilever Tune PANEL.
  - Right-click on the peak of the Thermal Graph and choose *Move Freq and Phase to Tune.*

12.



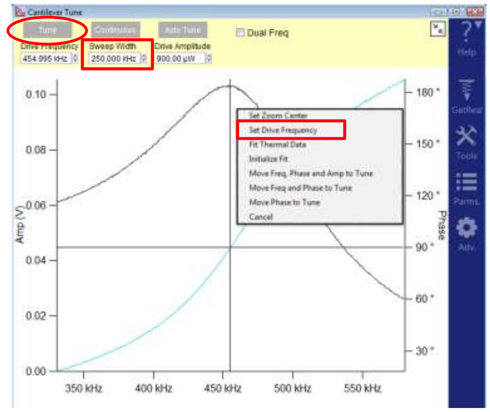
Turn on blueDrive photothermal excitation:

- In the Cantilever Tune graph, click the 'Adv.' icon.
- In the Advanced panel, choose "blueDrive" from the *Tune Drive* dropdown menu.
- Use the blue arrows in the Video panel (top right side) to move the blue laser spot to the base of the cantilever.

13.

Cantilever Tune panel:

- Set the *Sweep Width* to “250 kHz”.
- Click the ‘Tune’ button.
- When tune is done, right-click on the peak and select *Set Drive Frequency*.
- Move the blue laser spot around the base of the cantilever to maximize the amplitude (look at the amplitude value in the Sum and Deflection panel).
- Adjust the drive amplitude until the tune amplitude reaches ~ 80 mV (visible on the graph and in the Sum and Deflection Meter Panel)



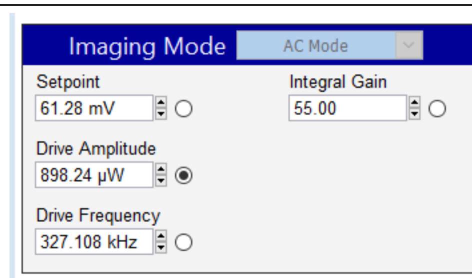
17.5.2. Imaging in AC Mode

Acquire an AC image of the calcite sample to first check probe tip sharpness and to define the area of interest.

1.

Master Panel settings:

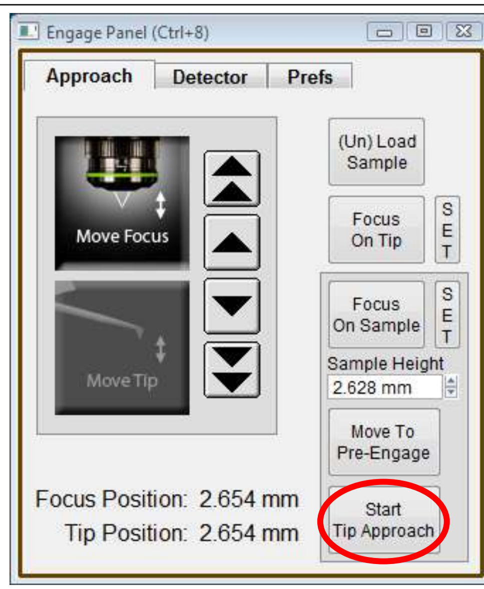
- Set the Setpoint to ~ 60 mV.
- Set the Integral Gain to “55”.



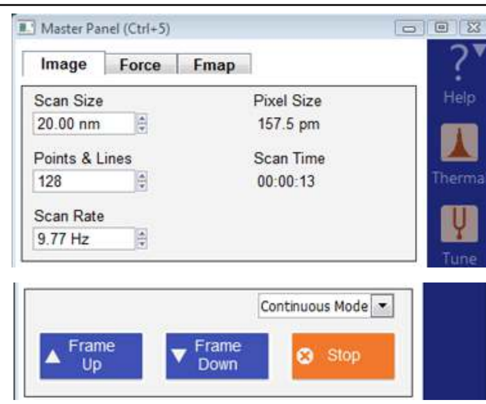
2.

Video and Engage panel settings:

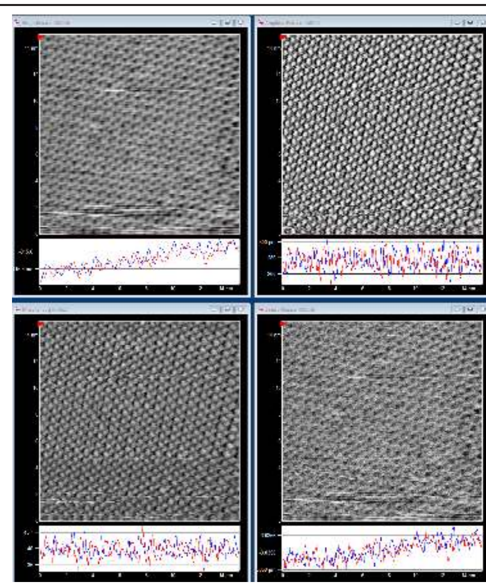
- While focused on the tip, click the ‘SET’ button beside Focus on Tip.
- While focused on the sample, click the ‘SET’ button beside Focus on Sample.
- Click the ‘Move to Pre-Engage’ button. Tip is now 50 µm from surface.
- Click the ‘Start Tip Approach’ button.



3. Once the tip is in piezo range of the sample, capture another Thermal Tune with z-piezo at 0V (tip is 2-3 μm from surface).
4. In the Thermal Graph panel, keep the Spring Constant padlock locked and recalculate Amplitude InvOLS.
5. Right-click on the peak in Thermal Graph and select *Move Freq and Phase to Tune*.
6. When the tune is complete, right-click on the peak and center phase.
7. **Master Panel Image parameters:**
   - Set the Scan Size to "20 nm".
   - Set the Scan Rate to "10 Hz".
   - Start imaging by clicking 'Frame Down'.



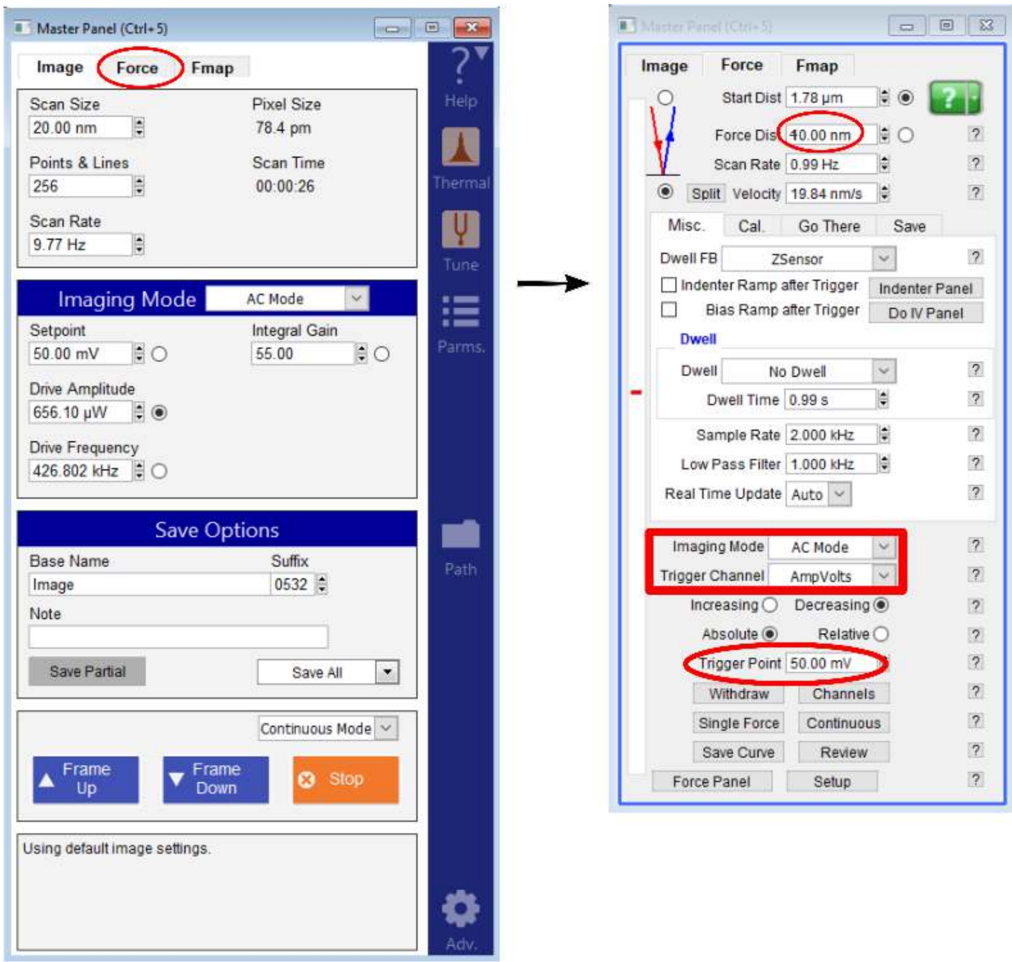
8. Start imaging and optimize imaging parameters:
   a) Decrease the free amplitude by decreasing *Drive Amplitude* in the Master Panel.
   b) Decrease the *Setpoint* until trace and retrace overlap each other.
   c) Repeat several times to image with the lowest amplitude possible.
9. **Imaging in AC mode:**
   - Acquire a 20 nm image and resize the scan to 10 nm.
   - Channels that are acquired include:
     – Height
     – Amplitude
     – Phase
     – ZSensor
   - Once the imaging looks optimized, stop the scan and switch to the Force tab of the Master Panel.



17.5.3. AC Force-Distance Curves

You will acquire several AC force curves to bring the tip within 50 nm of the surface and retune the cantilever.

1.



Force Tab

- In Master Panel, switch from AC Mode imaging to AC mode force curves.
- The Force tab now shows parameters related to AC mode force curves. Set as follows:
  - Set the *Setpoint* to about 60% of the amplitude (e.g., if amplitude is 80 mV, set the setpoint to 50 mV).
  - Begin with *Force Dist* (force distance) set to ~ 500 nm. Force distance is the distance between the tip and sample after the curve has been acquired.
  - Set the *Trigger Channel* to “AmpVolts”, select *Decreasing* and *Absolute*.

2.	**AC Force Curves:** • Acquire a single force curve. • Click 'Continuous' and, as the curves are being acquired, gradually decrease the force distance down to 50 nm. • Acquire ~20 curves with 50 nm force distance and click 'Stop Curve'. • The Z-Voltage should remain between 70 and 150 Volts.
3.	**Re-tune 50 nm away from the surface:** • Click the 'Tune' button on the Tune Graph. • Right-click on the peak of the graph and select *Set Drive Frequency*. • Right-click on the graph again and select *Center Phase*. The Z voltage should still be at the same value as when the force curve was acquired, i.e., the tip is ~ 50 nm away from the sample.

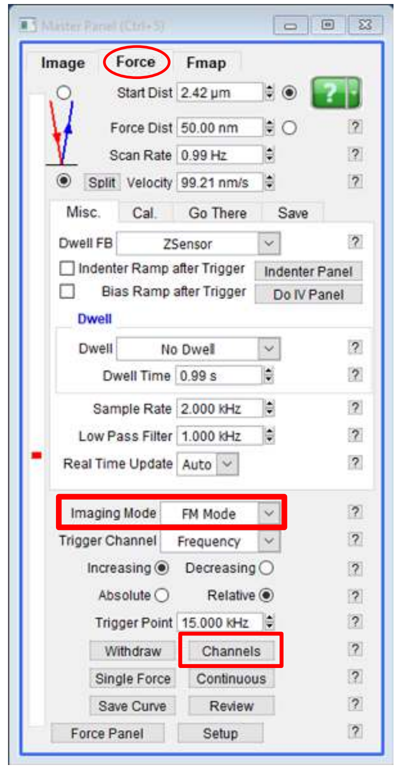
17.5.4. FM Force-Distance Curves

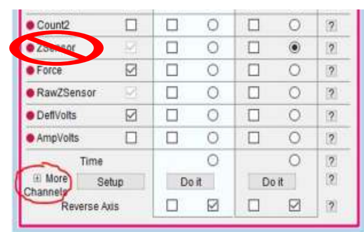
This step allows you to calculate FM gains and check if the setpoint is appropriate.

1.

Master Panel Force settings:

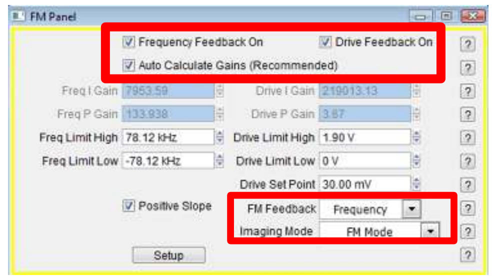
- Select “FM Mode” for the *Imaging Mode*.
- Click the ‘Channels’ button on the Force tab and select:
  - Frequency
  - Dissipation
  - Amplitude
  - Phase
  - Make sure to PLOT versus DRIVE (not ZSensor) for short curve (tens of nm) as Drive is a quieter signal than ZSensor, which has 50 pm of noise in a1 kHz bandwidth.
- Set *Trigger Channel* to “Frequency” and select:
  - Increasing
  - Relative
- *Trigger Point*: 5 kHz is a good start

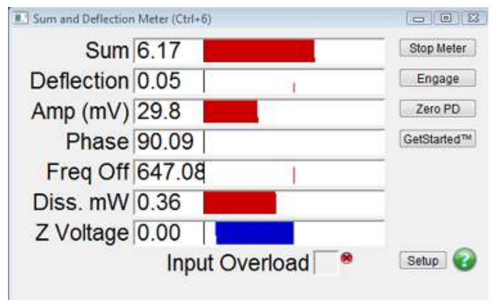


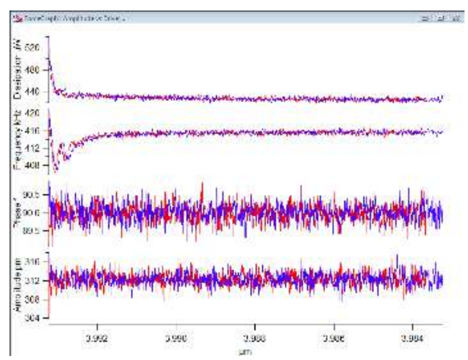


2.

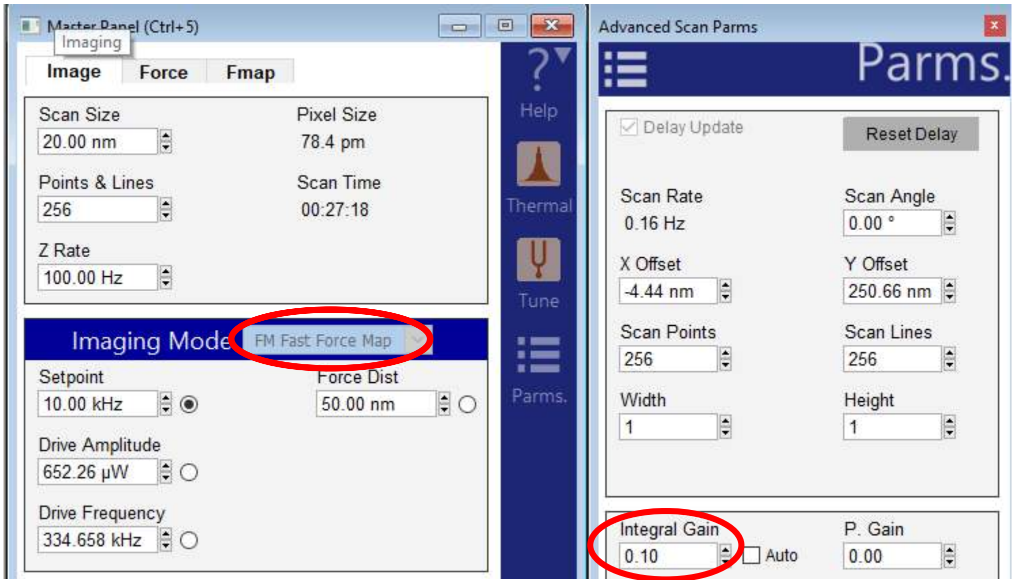
- Open the FM Panel (*AFM Controls > Other > FM Panel*).
- Uncheck and check the *Auto Calculate Gains* checkbox. The values should update.
- Set the *Drive Set Point* to the required amplitude:
  - Start with 3 Angstroms: 3A = 0.3nm.
  - For example, 0.3nm / 10.25nm/V InvOLS = 0.0292V = 29.3mV.
- Activate Feedback Loops by checking the boxes beside:
  - *Frequency Feedback On*
  - *Drive Feedback On*
- When the frequency and drive feedbacks are ON, the following values appear in the Sum and Deflection Meter:
  - *Amp (mV)* = Drive SetPoint Value
  - *Phase* = 90
  - *Freq Off* = frequency offset compared to the value of drive frequency from tune, here 647 Hz
  - *Diss mW* = power (in mW) needed to keep the Drive Set Point Value at 30 mV
- On the Force tab, click 'Single Force' to acquire a FM force curve. If the FM gains are correct, the force curve should appear on the graph.







17.5.5. Imaging with FM-FFM



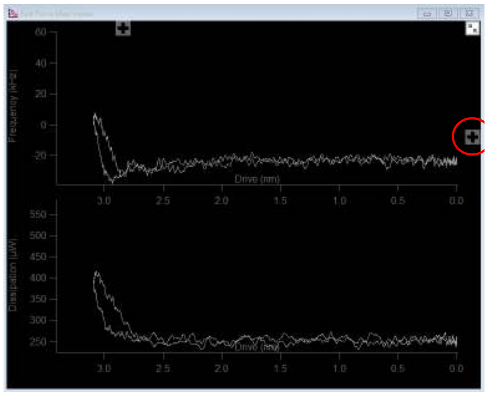
1.

Imaging parameters

- In Master Panel, switch the *Imaging Mode* from “FM Mode” to “FM Fast Force Map”.
- The Master Panel will now display parameters related to FM FFM mode:
  - *Setpoint* is now displayed in units of Hertz (if Frequency Feedback is ON in the FM Panel) and watts (if Dissipation Feedback is ON in the FM Panel).
  - Set *Force Dist* (force distance) to “50 nm”.
- Set the other parameters in the Master Panel:
  - *Scan Size*: 20 nm
  - *Points & Lines*: 256
  - *Z Rate*: 100 Hz
  - Z-voltage should still be between 70 and 150 V
- Click ‘Engage’ in the Sum and Deflection Meter panel. The FM FFM settings should now be reflected in the Sum and Deflection Meter panel .
- Set the *IGain*, located in the Parms panel, to “0.1”.

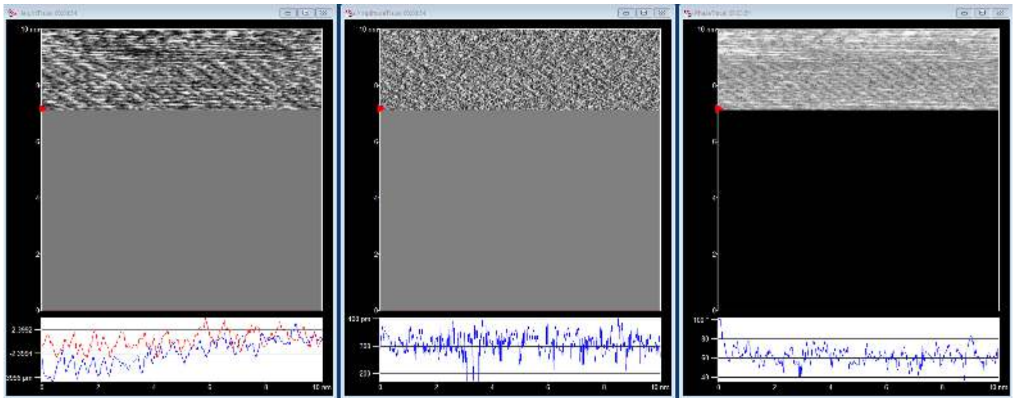
2.

Real time Force curves



- The Fast Force Map Viewer can display up to two channels. (To add a graph, click the plus sign on the right side of the viewer.)
- Each graph has several options for each axis; you must define the appropriate axes for the Imaging Mode. For FM FFM, choose:
  - Graph 0 (top): Frequency (Hz) vs. DriveVolts (V)
  - Graph 1 (bottom): Dissipation (μW) vs. DriveVolts (V)

3.



Imaging data:

- Data is collected in three channels during FM FFM imaging, including:
  - Height
  - Frequency
  - Dissipation

Part II

SPM Non-Imaging Techniques

Part II: Who is it for? Succinct step by step instructions for various non-imaging techniques. Light on theory and gets to the point.

Part Contents

18. Force Spectroscopy

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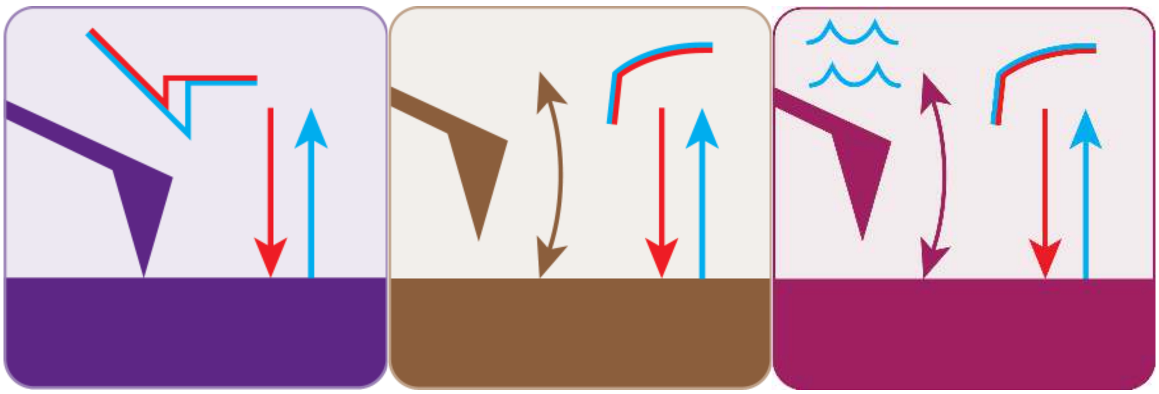


Chapter Contents

18.1. Introduction

Force spectroscopy includes several AFM techniques, including Force Curves and Force Maps, that are used to study the interaction between the AFM tip and sample to measure intra- and intermolecular forces and sample mechanics. A basic force curve displays the deflection of the cantilever (with known spring constant) in the Z direction as the cantilever moves towards then away from the sample surface.

As seen in Figure 18.1 on page 229, the process usually begins with the tip approaching the surface with some 'free air' (non-contact) deflection value (1), makes contact with the surface (2), pushes into the sample for some period/load until it reaches a user-defined cantilever deflection or force, i.e., the trigger point (3), and then reverses direction as the cantilever retracts from the surface. The tip may remain attached to the surface until enough force/distance pulls it off the surface, which appears as adhesion (4), before returning to its rested "free air" (non-contact) deflection (5). Although a typical force curve only moves in the Z direction, you can also acquire a Force Map that is an array of force curves over a user-defined XY scan range.

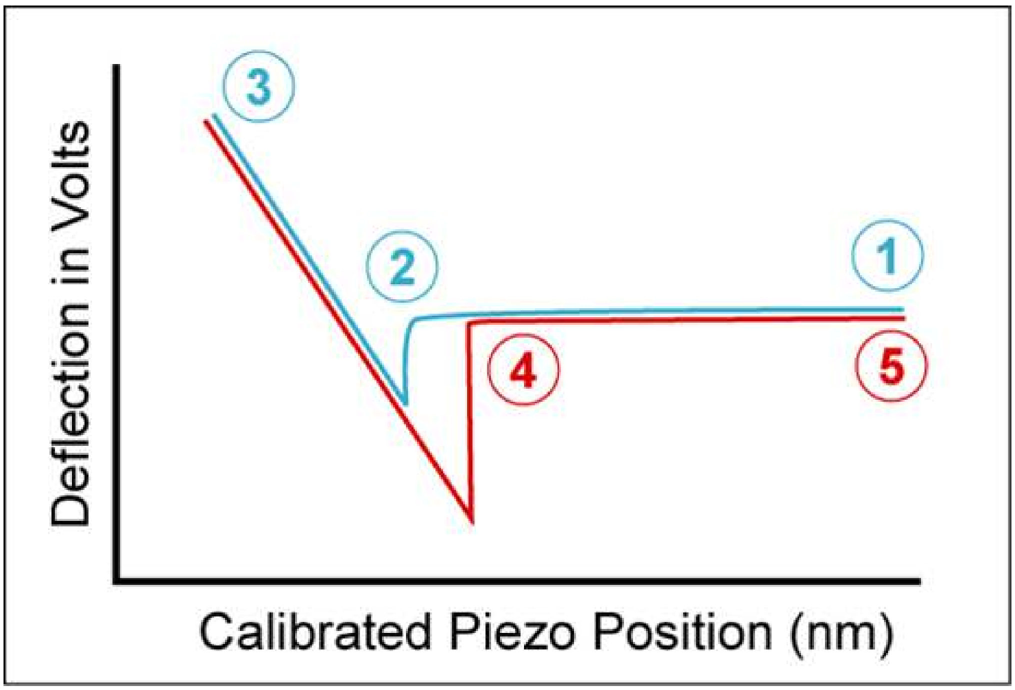


Figure 18.1.: Components of a Force Curve as the cantilever approaches and retracts from the sample surface

There are two major classifications that most force spectroscopy experiments measure:

- A pulling event where the tip interacts with the surface and some adhesion dissociation event between the tip and surface is measured on the retract cycle (Figure 18.2 on page 230).
- A modulus or compliance measurement where the tip pushes into the sample surface. (Figure 18.3 on page 230).

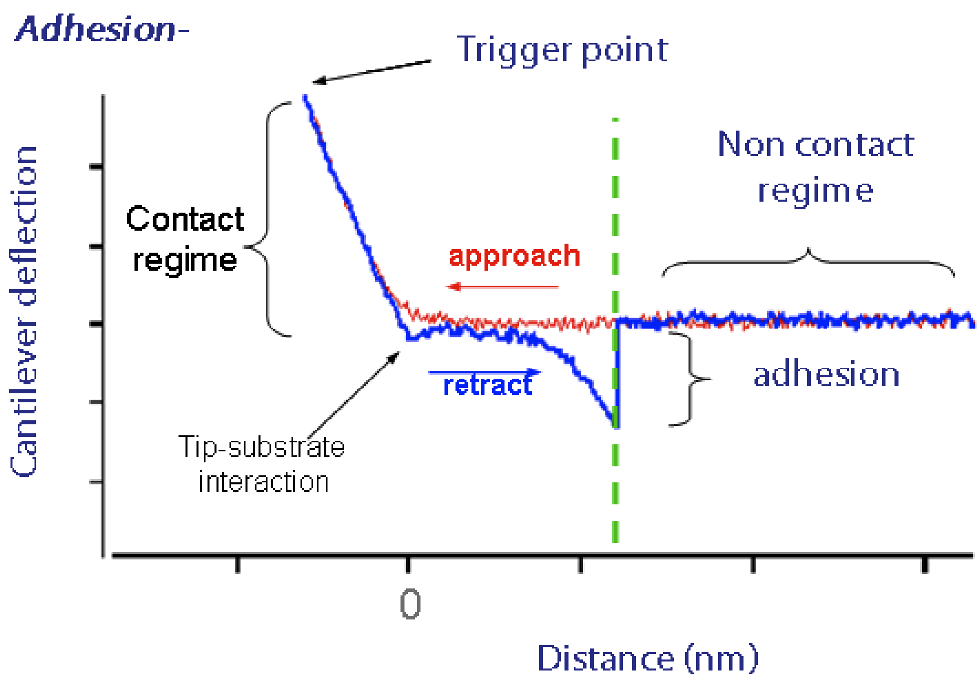


Figure 18.2.: Adhesion force spectroscopy classification. Pulling can yield adhesion forces.

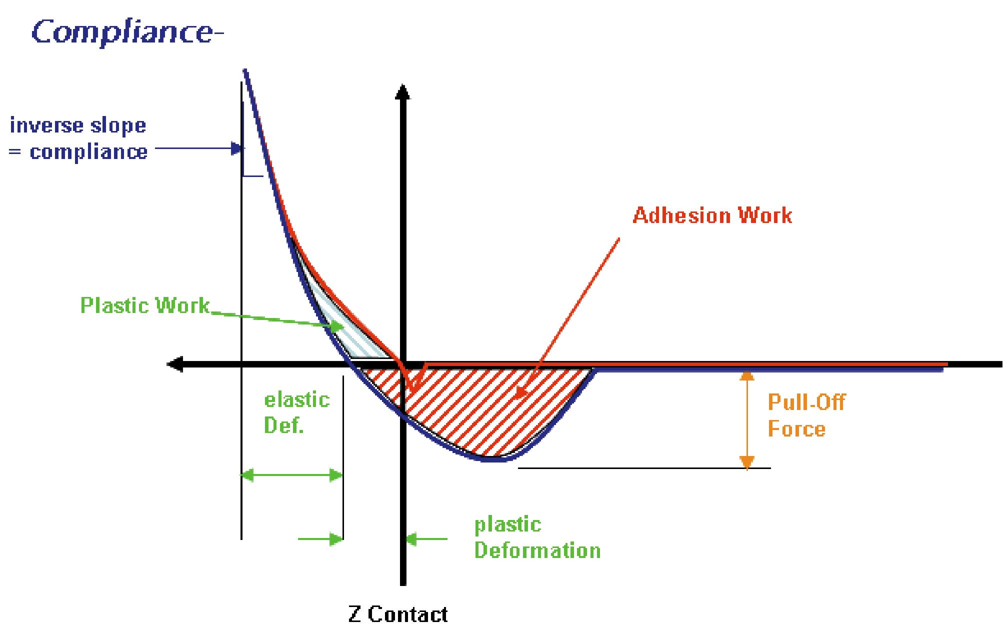


Figure 18.3.: Compliance force spectroscopy classification. Pushing can measure compliance forces.

This chapter is designed to help describe, and in some cases reveal, the versatility of the force spectroscopy capabilities with the Asylum Research AFMs and also includes some empirical considerations.

Cantilever selection is very important in force spectroscopy: If a lever is too stiff, small forces may not be recognized; if a lever is too floppy, perhaps not enough force can be applied during compliance measurements. In other words, for a lever that has too low a spring constant, the sample will appear to be infinitely stiff; and for a probe that is too stiff, a soft sample will appear to be infinitely soft or as no different from the noise in the measurement. Other considerations include the cantilever material, regarding how well it will work in air/ fluid and the ability to do chemical cross-linking to it.

Important The most important thing to remember to do before taking force curves and force maps is to calibrate the cantilever. It's not recommended to use Force Triggers based on the raw DeflVolts, since cantilever sensitivity and spring constant varies with cantilever; and as a result, the applied force will also vary from cantilever to cantilever. Using Deflection (nm) and Force (nN) as your Force Trigger is preferred and ideal for most force experiments. There are a couple of options with our software: The traditional Thermal Method or the built-in GetReal™ Routine. For a complete description and instructions on how to calculate InvOLS and spring constant Chapter 20 on page 281.

To briefly summarize, the Thermal Method requires two steps:

1. A **force curve** is acquired to calculate the cantilever sensitivity (InvOLS).
2. A **thermal tune** is captured and used to calculate the cantilever spring constant (k), using the calculated InvOLS.

GetReal™ is an automated routine that relies on both the Sader Method and a thermal tune to calculate InvOLS and k. It also uses two steps:

1. Cantilever **spring constant** (k) is calculated based on the dimensions and frequency of the cantilever.
2. A **thermal tune** is captured and used to calculate the InvOLS. This is the opposite of what is done in the Thermal Method. GetReal™ "locks" the k and calculates the InvOLS.

Excellent force spectroscopy review papers in the literature include:

- Butt, H.J., Cappella, B. and Kappl, M., 2005. Force measurements with the atomic force microscope: Technique, interpretation and applications. Surface science reports, 59(1-6), pp.1-152.
- Cappella, B. and Dietler, G., 1999. Force-distance curves by atomic force microscopy. Surface science reports, 34(1-3), pp.1-104.
- Dupres, V., Verbelen, C. and Dufrêne, Y.F., 2007. Probing molecular recognition sites on biosurfaces using AFM. Biomaterials, 28(15), pp.2393-2402.
- Gavara, N., 2017. A Beginner's guide to atomic force microscopy probing for cell mechanics. Microscopy research and technique, 80(1), pp.75-84.
- Heinz, W.F. and Hoh, J.H., 2005. Getting physical with your chemistry: Mechanically investigating local structure and properties of surfaces with the atomic force microscope. Symposium: Chemistry at the nanometer scale, Journal of chemical education, 82(5), pp. 695-703.
- Heinz, W.F. and Hoh, J.H., 1999. Spatially resolved force spectroscopy of biological surfaces using the atomic force microscope. Trends in biotechnology, 17(4), pp.143-150.
- Hughes, M.L. and Dougan, L., 2016. The physics of pulling polyproteins: a review of single molecule force spectroscopy using the AFM to study protein unfolding. Reports on Progress in Physics, 79(7), p.076601.

- Janshoff, A., Neitzert, M., Oberdörfer, Y. and Fuchs, H., 2000. Force spectroscopy of molecular systems—single molecule spectroscopy of polymers and biomolecules. Angewandte Chemie International Edition, 39(18), pp.3212-3237.
- Liu, B., Chen, W. and Zhu, C., 2015. Molecular force spectroscopy on cells. Annual review of physical chemistry, 66, pp.427-451.
- Müller, D.J. and Dufrene, Y.F., 2010. Atomic force microscopy as a multifunctional molecular toolbox in nanobiotechnology. In Nanoscience And Technology: A Collection of Reviews from Nature Journals (pp. 269-277).
- Rief, M. and Grubmüller, H., 2002. Force spectroscopy of single biomolecules. ChemPhysChem, 3(3), pp.255-261.

18.2. Force Curve Acquisition

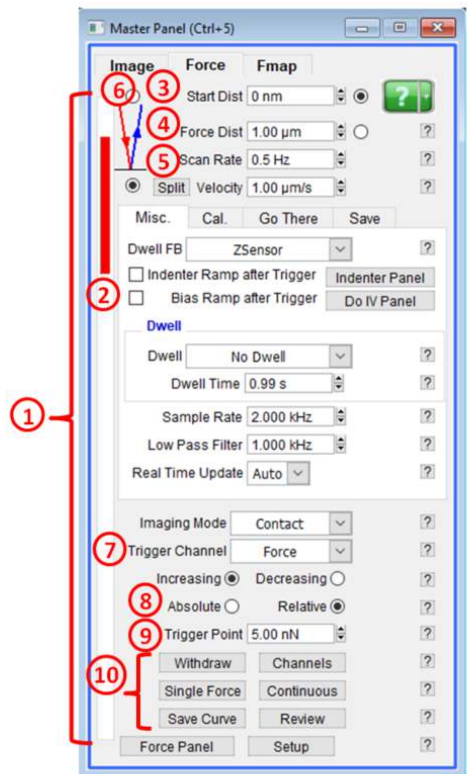


Figure 18.4.: Force tab of the Master Panel.

18.2.1. The Force tab

The Force tab of the Master Panel is where most the basic force spectroscopy software control occurs. First, we'll describe some of the general controls, and then cover controls hidden under the additional tabs on this panel, also known as "sub-tabs", shown in Figure 18.4 on page 232.

① **White Bar** Located along the left side of the panel, representing the entire travel distance of the Z piezo. The top of the white bar represents the piezo as fully retracted; the bottom represents the piezo fully extended.

② **Red Bar** Located within the white bar, representing the "Force Distance" of the force distance cycle relative to the entire travel of the Z piezo . Note that given values in microns are in reference to the piezo sensitivity. The captured and displayed force curve will show slightly different values as corrected by the Z-sensor.

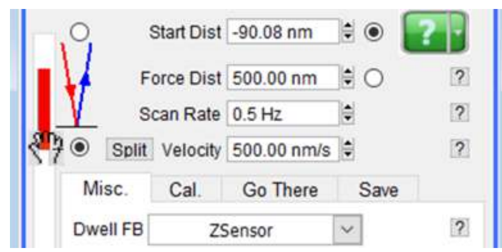
③ **Start Distance** The point at which the piezo begins its Z/ force distance cycle (i.e., the starting voltage applied to the piezot). The red bar's upper position in the white slider bar qualitatively depicts this position. In most cases you don't need to adjust the start distance. Typically, the first force curve acquired begins with the Z piezo fully withdrawn. Then, after the first force curve has been acquired, the software automatically set the parameter to the Z position based on the pull off distance (i.e., Force Distance).

The red bar can be moved with the mouse cursor two different ways:

1.

Method 1

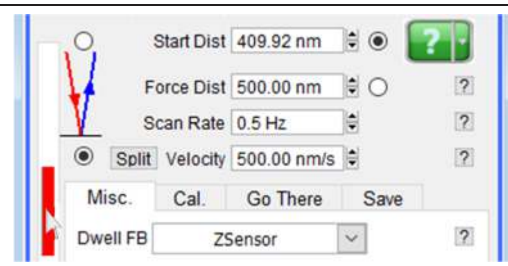
- Place the cursor anywhere in red region.
- Left-click until the hand icon appears (see image at right).
- Drag the red bar throughout the Z piezo range (white slider bar)



2.

Method 2

- Left-click mouse in the white area to roughly scroll the red cursor up or down.



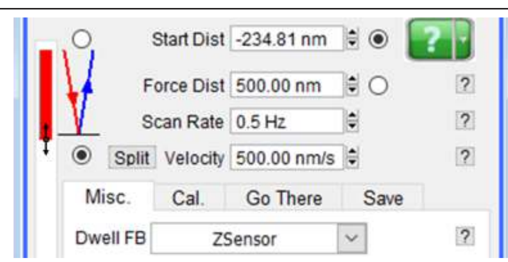
Note The Hamster wheel (detached or on the front of MFP controller) can also execute this movement in real time when the *Start Dist* (start distance) radio button is selected.

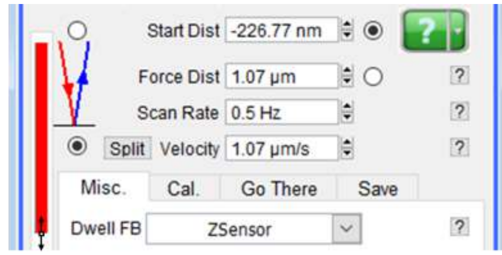
④ **Force Distance** This parameter is how long the force-distance cycle will be, provided a trigger point isn't arrived at prior to that. Notice that as this setvar value is changed, the length of the red bar changes (i.e., the length of the red bar qualitatively depicts the force distance relative to the entire length of the Z piezo's distance (white slider bar)). The length and position of the red bar can be manipulated with the mouse as follows:

1.

Step 1

- Place cursor at the edge of red bar.
- Hold left mouse button down until a double-arrow appears (see image at right).



2.	**Step 2** • Drag cursor to desired force distance value. • Notice the change in the *Force Dist* value in the box to the right.	

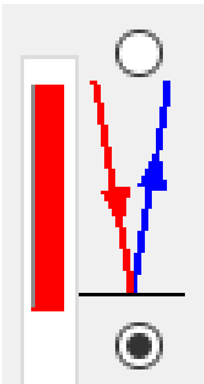
⑤ **Scan Rate/Velocity** This is the estimated piezo velocity (*not* the tip velocity). Changing the velocity, or scan rate, affects the other.

⑤* **Sync** Split/ Synch Approach/ Retract velocities can be made different in “Split” mode. For experiments that require constant velocities, turn on the closed loop in the Z axis (see Section 18.2.7 on page 245). To select velocities that are not subject to hydrodynamic effects, see Section 18.2.8 on page 246.

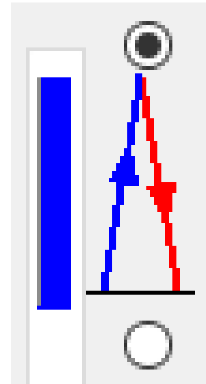
⑥ **Forward/Reverse pulls** These radio labels control whether the tip starts on or above the surface (Red mode/ Blue modes):

Forward pull (**Red Mode**): Tip starts cycle in free air (non-contact) and approaches surface; then retracts; this is the default mode upon MFP3D software start up. This is the most commonly used mode for force spectroscopy experiments. See Figure 18.5a on page 235.

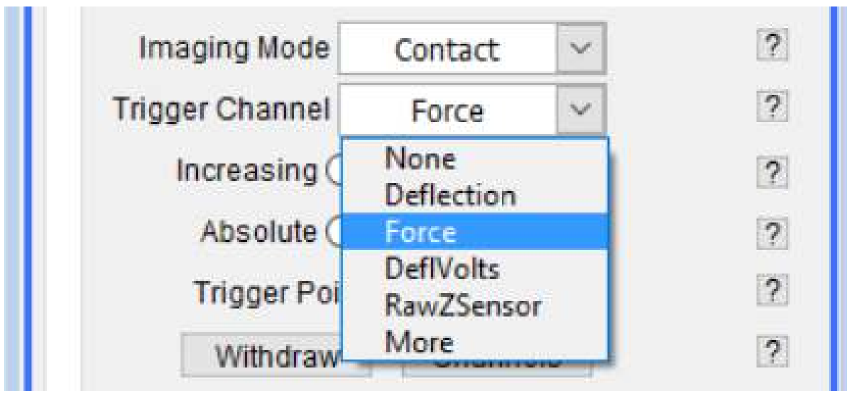
Reverse pull (**Blue mode**) Tip starts cycle at surface and travels (retracts) to free air (non-contact), then returns to surface. This mode is good for ‘Fishing’, possibly Force Clamp. . See Figure 18.5b on page 235.



(a) Forward pull



(b) Reverse Pull



(c) Selecting a Trigger Channel

Figure 18.5.: Examples of different Trigger types.

⑦ **Trigger Channel** Dropdown menu that allows you to define which units to measure for "triggering", essentially a user-defined force that the tip applies before switching direction. See Figure 18.5c on page 235.

- **None** No trigger applied. The piezo will go through its force distance cycle regardless of whether the tip makes contact with surface. However, with an untriggered force curve, you have a higher risk of grinding the tip into the surface.
- **Deflection** Monitors the deflection (nm) on the photodetector. The piezo will only move the cantilever if the InvOLS (and ideally, the spring constant) has been calibrated. See Chapter 20 on page 281.
- **Force** Monitors the force applied to the sample. Accurate only if both the InvOLS and the spring constant have been calculated. See Chapter 20 on page 281.
- **DeflVolts** Monitors the amount of raw volts on the PSD. Since the amount of deflection (in nms) and force varies depending on the InvOLS and spring constant, you typically use this only to calibrate the cantilever; but once the cantilever has calibrated, you would want to switch to either Deflection (nm) or Force (nN).
- **RawZSensor** Monitors the distance on the Z-axis closed-loop sensor.
- **More** Expands the list to show less-used triggers, including:
  - **Lateral ,LateralVolts**

- **Bias**
- **UserIn0 ,UserIn0Volts**
- **UserIn1 ,UserIn1Volts**
- **UserIn2 ,UserIn2Volts**
- **Frequency**
- **Count**
- **Drive, DriveVolts**

⑧ **Relative/Absolute Trigger** Check relative to trigger at the value relative to the value at the start of the approach. For example, if the deflection at the start of the curve is 0.3V, and the trigger is 0.5V, the net deflection would be 0.5V, and the curve would trigger at 0.8V. If *Absolute* was checked, then the net deflection would be 0.2V, and the curve would trigger at 0.5V, ignoring the start value. Most deflection-based force experiments use *Relative*, and AC mode force experiments use *Absolute* trigger. Relative triggers are important if there is any cantilever deflection drift, as it keeps the applied force constant.

Slope A positive slope will trigger on a rising *Trigger Channel* value. A negative slope will trigger on a falling *Trigger Channel* value. Deflection-based force experiments typically use a positive slope, and AC mode force experiments typically use negative slopes. See Section 18.3.4 on page 258 for more information on Dynamic (AC) Force Spectroscopy.

⑨ **Trigger Point** This setvar defines trigger point/ load values. The *Trigger Point* is a user-defined value detected on the photodetector that tells the piezo to switch directions, even if the force (deflection) curve has not achieved the completed force distance (deflection) value.

⑩ **Buttons:**

Withdraw Withdraws the tip; and puts Z piezo voltage at 0V (i.e., almost fully retracted).

Channels Brings up the Force Channel Panel. See Section 18.2.6 on page 244 for more information.

Single Force Activates a single force cycle. (Crtl+3 also does this.)

Continuous Initiates force cycles to be continuously executed until told to stop. The number of continuous curves can be limited by using the *Limit Cont. to* parameter as follows:

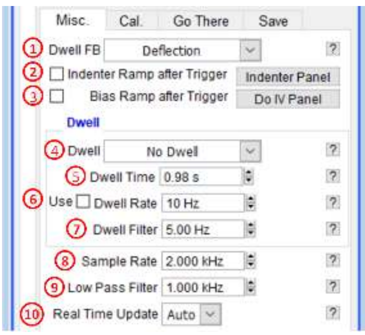
1. *Limit Cont. to* is an advanced parameter that is hidden with the default display. To show it:
   a) Click the 'Setup' button.
   b) Select the checkbox.
   c) Click the 'Looks Good' button. This parameter should now appear.
2. Enter the number of curves needed to collect continuously in the *Limit Cont. to* setvar.
3. Click the 'Continuous' button to acquire your force curves.

Save Curve Use to save a curve if you do not have all curves automatically being saved in the Save sub-tab. By default, all force curves are automatically saved to Memory and Disk.

Review Loads force curves for review/analysis by bringing up the Master Force Panel.

18.2.2. The Misc. sub-tab

The **Miscellaneous (Misc.)** sub-tab in the Master Panel of the Force tab controls dwell periods/ sampling and sampling/bandwidth options. The MFP-3D software allows you to define a period of 'Dwell', where the tip stops moving (or maintains some load) in the middle or end of a scan while continuing to collect data at some rate. This feature is great for data acquisition where the tip (functionalized or not) may need time to interact with the sample (i.e., to accommodate some receptor-ligand event, or to assess some creep/compression event while applying/ maintaining a load to the sample with the 'tip') before the tip gets pulled away from the surface.



(a) Misc. tab overview

(b) Dwell dropdown menu

Figure 18.6.: The Misc. tab allows you to define temporal and low-pass filter settings for dwell and force curve collection.

18.2.2.1. Dwell Periods and Sampling

① **Dwell FB** This dropdown menu allows you to set the *Dwell FB* (Dwell Feed Back) loop input. Options include:

- **ZSensor** This is the default setting. Monitors the cantilever displacement / Z position.
- **Deflection** Monitors the Load / Force.
- **Other Indentation** Monitors indentation by subtracting Z Sensor and Deflection signals in real time. This mode is somewhat experimental and requires a well-calibrated InvOLS.

② **Indenter Ramp After Trigger** When selected, this calls whatever function has been developed in the Indenter Panel. The 'Indentor Panel' button displays the Indenter Panel. Section 18.3.2 on page 251

③ **Bias Ramp After Trigger** When selected, executes an I-V curve using whatever settings are applied to the Do IV Panel. The 'Do IV button' displays the Do IV Panel. See 9.7.

④ **Dwell dropdown menu** Activates the *Dwell* function, as shown in Figure 18.6b on page 237.

None No Dwell period is applied.

Toward Surface Dwell period applied when the trigger point is reached. Bandwidth options during this segment can be chosen.

Away from Surface Occurs for reverse pull. A dwell is applied after the tip pulls away from the surface and before it approaches it again.

Both Applies a dwell when the tip retracts from surface and again as the tip approaches the surface.

⑤ **Dwell Time** Defines the length of dwell. Maximum dwell time is 10 seconds. An example of a force distance curve (plotted as deflection vs. time) with a multi second dwell setting is seen in Figure 18.7 on page 238.

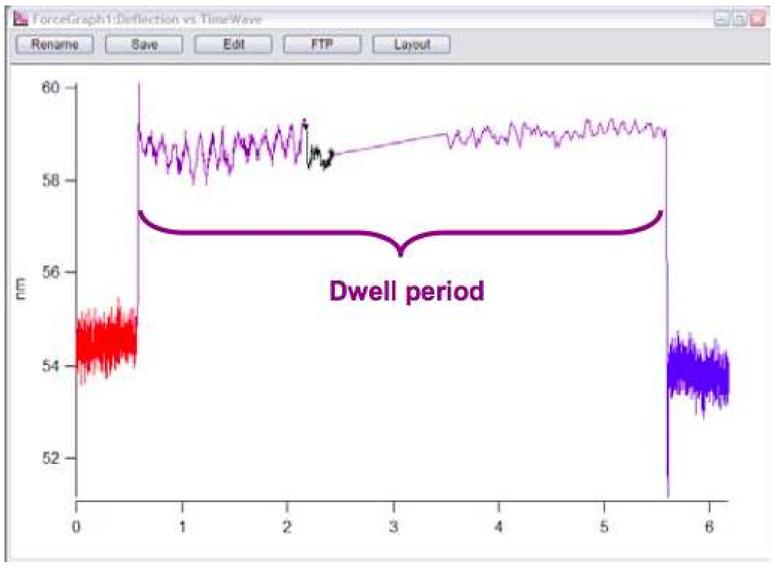


Figure 18.7.: Example of a temporal force distance curve (plotted as cantilever deflection vs. time, with a multi-second dwell towards surface.

⑥ **Dwell Rate** Rate at which data is collected during a dwell (e.g., maybe the experiment calls for more or less points to be collected during this time). It is an advanced parameter that is hidden with the default display. To show it, click the 'Setup' button, then click the checkbox to the right, and finally, click the 'Looks Good' button. The parameter should now appear. Click the 'Use' checkbox (to the left of the setvar) to activate so that any changes made will be executed.

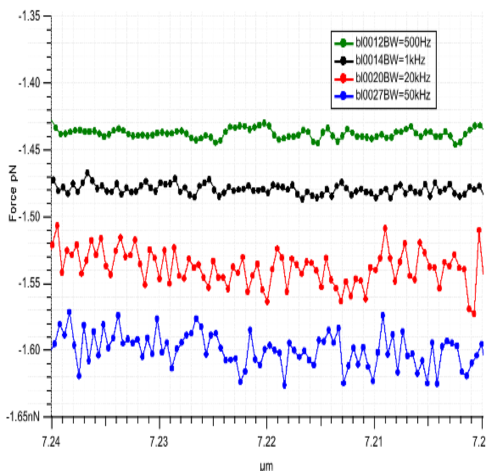
⑦ **Dwell Filter** A low-pass filter that can clip from 1 Hz to 1kHz. Often not a default parameter. Activate via the 'Setup', similar to Dwell Rate.

18.2.2.2. Sampling and Bandwidth Options

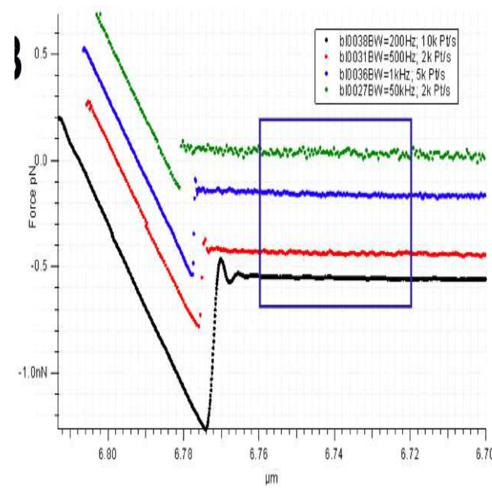
The band width (BW) options for the curve collection. To view the BW options (in most version of Asylum Research software), they must be activated for display using the 'Setup' button. (The Setup menu can also be opened by right-clicking anywhere on the panel.)

Note Annotations are continued below for the Misc. sub-tab from the previous section (see Figure 18.6a on page 237).

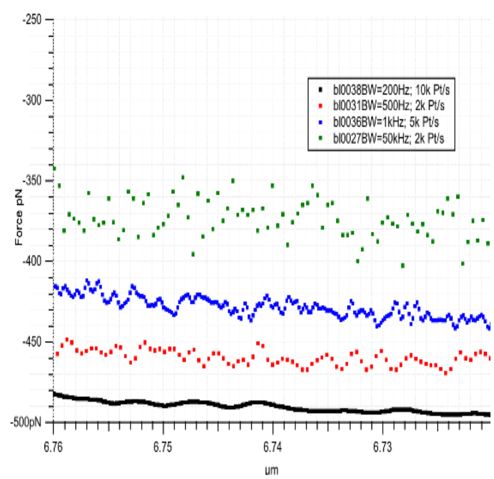
⑧ **Sample Rate** This is essentially how many points per second collected during the force curve acquisition (see Figure 18.8a on page 239 for an example).



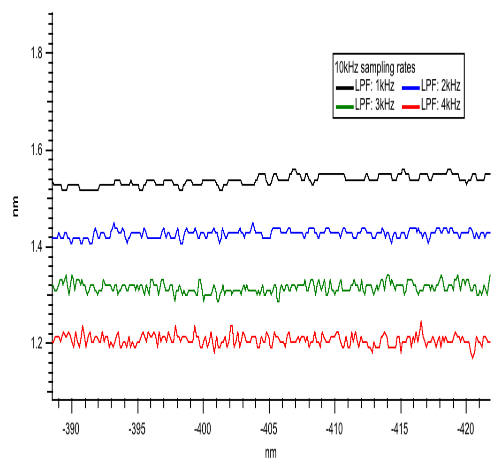
(a) Various data streams from a free air portion of a force curve collected at 2kHz (i.e., 2,000 points/sec) with different low pass filter settings. Notice higher filter settings are noisier.



(b) Examples of force curves collected at various sample Rates and low pass filter settings.



(c) Zoom of boxed area in Sub-Figure B to illustrate sampling point density



(d) Four force curves collected at 10kHz sampling rate with different low pass filter (LPF) band width settings. Notice at low LPF BW, the data is clipped sufficiently such that data fitting might not occur at desired point values (in Y).

Figure 18.8.: Examples of filter settings for force curve acquisition

⑨ **Low Pass Filter** Sets the cut-off value of the low-pass filter applied to the incoming data. Lower values cut off more of the high frequency data. If measuring a high-speed event, care must be taken to have this value high enough to see the event. For example, for an event that takes 2 milliseconds, you need a bandwidth of at least 500Hz (Figure 18.8a on page 239).

- Examples of varying Sample Rate and Low Pass Filter values can be seen in Figure 18.8b on page 239: this was acquired with an Olympus Biolever in water on a mica surface, but is shown to demonstrate using the Sample Rate and Low Pass Filter setvar settings to get the effect needed.
- An example of using high sample rates and lower LPF settings can be seen in Figure 18.8d

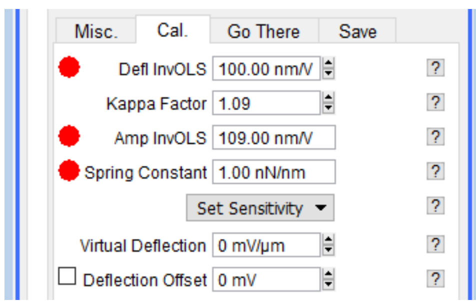
on page 239, where all curves were collected at a sampling rate of 10kHz, while the various (lower) LPF settings were employed. Notice that at lower LPF settings, the data may be clipped too much for certain applications where deflection resolution with distance is important.

⑩ **Real Time Update** Dropdown menu that allows you to choose how the Force graph gets updated. Options include:

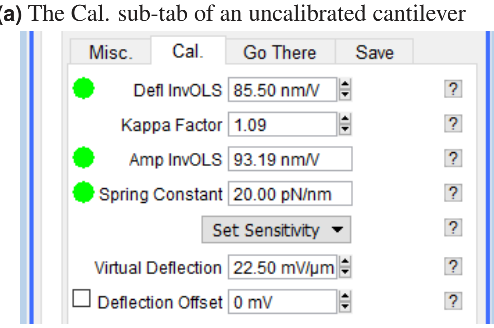
- **Never** The Force plots updates *after* the acquisition is complete.
- **Auto** Updates with some time constraints within the acquisition parameters.
- **Always** The Force plot is updated as it is being collected; this is evident by a black line that appears within the data stream in the force curve. (See Figure 18.7 on page 238 for an example of this.)

18.2.3. The Cal. sub-tab

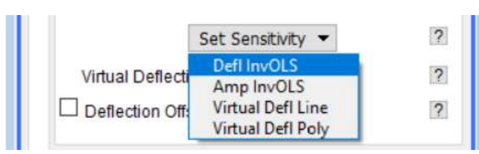
The **Calibration (Cal.)** sub-tab is where the calibrations are executed and displayed for spring constant determination. (See Chapter 20 on page 281 for a complete description of that protocol.)



(a) The Cal. sub-tab of an uncalibrated cantilever



(b) The Cal. sub-tab of a calibrated cantilever



(c) *Set Sensitivity* Pull-down Options.

Figure 18.9.: Examples of the options in Calibration sub-tab.

Defl InvOLS Deflection Inverse Optical Lever Sensitivity is calculated by doing a force curve in Contact mode on an infinitely hard surface and fitting the slope of the contact region to 1. (GetReal

(R) can also be used to calculate the InvOLS.) The units are nm/V (i.e., the amount of detected distance of cantilever deflection per voltage on the photodetector). The red circle indicates the system is still using the default value; and therefore, the cantilever is not calibrated (Figure 18.9a on page 240). Once the value has been updated the red circle changes to a green circle (Figure 18.9b on page 240).

Kappa Factor Coefficient between Defl InvOLS and AmpInvOLS; value is usually 1.09. This value attempts to compensate for the difference in the lever bending freely vs. a lever bending under a load.

Amp InvOLS Amplitude Inverse Optical Lever Sensitivity is calculated by doing a force curve in AC mode on an infinitely hard surface and fitting the slope of the contact region to 1. The units are also nm/V. It is slightly different because it is an AC signal on the photodetector. The red circle indicates the system is still using the default value; and therefore, the cantilever is not calibrated. More commonly, AmpInvOLS is calculated by multiplying the Defl InvOLS by the Kappa Factor. Once the value has been updated, the red circle will change to a green circle (Figure 18.9b on page 240).

Spring Constant The calibrated spring constant is displayed here. (See Chapter 20 on page 281 for protocol for determining spring constant (k) using the Thermal or Sader methods.) The red circle indicates the system is still using the default value, and therefore the system is not calibrated (Figure 18.9a on page 240). Once the value has been updated the red circle will change to a green circle (Figure 18.9b on page 240).

Set Sensitivity The Set Sensitivity dropdown menu under Spring Constant allows the user to select what will get calibrated (see Figure 18.9c on page 240).

- **InvOLS** Refers to Deflection InvOLS.
- **AmpinvOLS** See description above.
- **Virtual Defl Line** Fits the virtual deflection to a linear line.
- **Virtual Deflection Poly** Fits the virtual deflection to a 2nd order polynomial. Commonly used for Extended Z heads, which are identifiable by the horizontal blue strip around the head.
- **InvOLS (LVDT)** Used data from LVDT closed loop sensor channel as the y-axis data
- **AmpInvOLS (LVDT)** Used data from LVDT closed loop sensor channel as the y-axis data
- **Amp2 InvOLS** Amp InvOLS for second frequency in DualAC™.
- **Virtual Deflection** This is how to calibrate the slight aberrant deflection that occurs in the optical detection system using a very long force distance cycle. The free air often has several nm worth of deflection over the entire 15 µms of Z travel. It must be calibrated to ‘level’ the free air part of the curve to allow for more accurate analysis/ determination of contact point; it is updated upon calibration.
- **Deflection Offset Checkbox** This will electronically set the Deflection voltage in the S&D meter to 0V; The offset is the same magnitude made to whatever the free air deflection is, this will electronically zero the deflection, and then an absolute trigger works like a relative trigger (see Figure 18.2.1 on page 236 for more on Absolute and Relative triggers).

  For example, if there was a -0.88V free air deflection in the S&D meter, typing 0.88V into the deflection offset setvar and clicking the checkbox to activate it would electronically set the free air deflection to 0.0V.

- **Virtual Defl. 2nd Term** This is for extended Z range heads that need a polynomial to fit the Virtual Deflection (i.e., using the Virtual Defl Poly fitting function described above); value is updated upon calibration.

For a more complete protocol description of spring constant calibration, see Chapter 20 on page 281.

18.2.4. The GoThere sub-tab

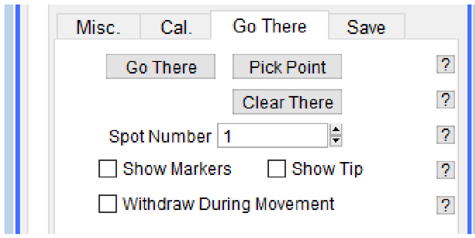


Figure 18.10.: The Go There Sub Tab for Point and Click force curve acquisition.

The Go There sub-tab of Force tab in the Master panel allows you to designate points on an acquired image for subsequent force curves. This is very useful when you have imaged the surface and want to immediately acquire force curves at specific points/features on your image. The MFP-3D's closed-loop sensor accuracy makes this possible.

While acquiring the image or immediately following, you can "pick points" on the image to acquire either force curves or IV curves at those "points".

Procedure:

1. Acquire image in AC or Contact mode.
2. If the image was acquired in AC mode, but the desired force curve acquisition is to be in Contact mode (i.e., not dynamic force spectroscopy), withdraw the tip and change the imaging mode to "Contact" from the *Imaging Mode* dropdown menu, located right above the *Trigger Channel*. If you are relying on either the amplitude or phase channels to locate a feature or area of interest, you may want to unselect *Auto Channels* in the Master Channel Panel before switching to Contact mode, so that the software doesn't reconfigure the displayed channels when switching from AC Mode to Contact Mode.
3. Click the *Show Markers* check box. By default, the *Show Tip* check box will also be selected. The location of the tip, as marked by a red dot, will appear within the XY scan area. A red "X" marked with a "0" will also appear in the center of the image. This is Position 0. A ⊕ cursor will also appear at Position 0. You can grab and drag the ⊕ cursor to new location to mark additional points for either force or IV curves.

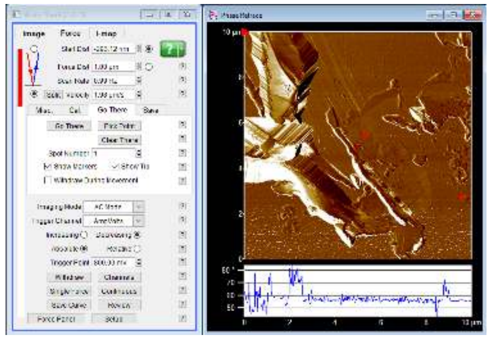
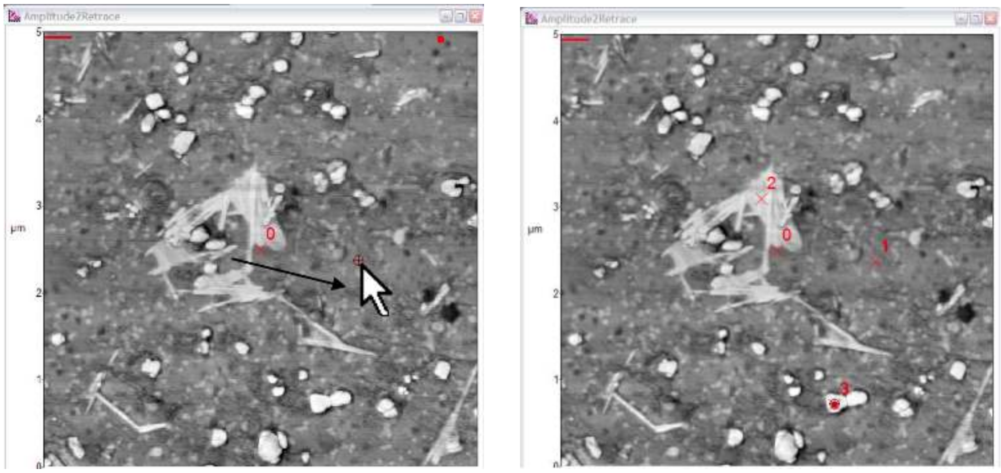


Figure 18.11.: The Go There sub-tab for “Point and Click” force curve acquisition

4. Click the ‘Pick Point’ button.
5. Using the left mouse button, grab and drag the ⊕ cursor to a feature of interest.
6. Click on the ‘That’s It!’ button. The new position will be marked by a “1” for Position 1; this locks the point at that spot. Meanwhile:
   a) The Spot number index is advanced by one in anticipation of the next point location being chosen.
   b) The Spot location you defined is marked on the image with an ‘X’ and also labeled with the spot number (Figure 18.12b on page 244).
   c) The Spot Number base suffix will increase by one, expecting another point to be picked.
7. Continue to grab and drag the ⊕ cursor until you have selected all of your points (Figure 18.12a on page 244).
8. When ready to acquire the force curves, enter the Spot Number for the position on the surface you want to probe.
9. Click the ‘Go There’ button. Notice the red dot designating the tip position will move to that point (Figure 18.12b on page 244), generally at a rate the *Scan Rate* is set to (in the Main tab of the Master Panel).
10. Click the ‘Single Force’ button, ‘Continuous’ button, Crtl+3, or downward function selector toggle on the Controller to acquire force curve(s).

Sub-figures a and b in Figure 18.12 on page 244 show an example of designating some user-defined points on a DualAC™ second amplitude image of some water-based paint (see Chapter 14 on page 165). In Figure 18.12a on page 244, the ⊕ cursor was moved from image center to designate first-defined location (as seen in Figure 18.12b on page 244). In Figure 18.12b on page 244, three different points are selected at different contrasted image locations. Notice that the red dot is at point “3”, marking the current position of the tip.

To acquire multiple curves at a given point, click the ‘Continuous’ button, and designate the *Limit Cont. to* setvar to a desired value (see Section 18.2.1 on page 232). Remember that the *Limit Cont.* to variable isn’t shown in the default MFP-3D software, so it must be activated by clicking the ‘Setup’ button. If the value is set to “inf” it means it will collect curves continually (infinitely) until told to stop.



(a) Drag the cursor from image center to desired location

(b) Many locations selected; tip located at Position 3, indicated by red dot

Figure 18.12.: Force curve location points

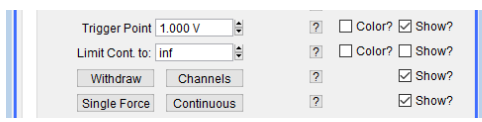


Figure 18.13.: Continuous force limit count of the Force panel.

Note The Go There functions also work for the electrical characterizations techniques.

18.2.5. The Save sub-tab

The **Save** sub-tab is where force curves are saved (Figure 18.14 on page 244). The default filename is “Image”, but it is highly recommended that you change the default to a more meaningful description of the experiment. Use the same 17-character filename as with saving images; the filename can’t start with a number or Igor lets you know its displeasure with your desire to attempt this.

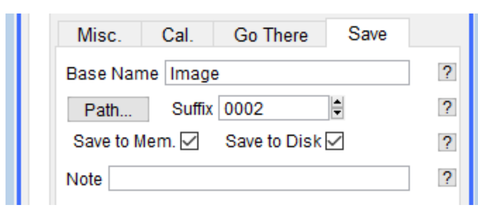


Figure 18.14.: The Save Sub Tab.

As curves are acquired, the base suffix will increase by 1.

18.2.6. Force Channel Panel

The Force Channel Panel allows you to assign the data channels displayed and saved in Force Plots.

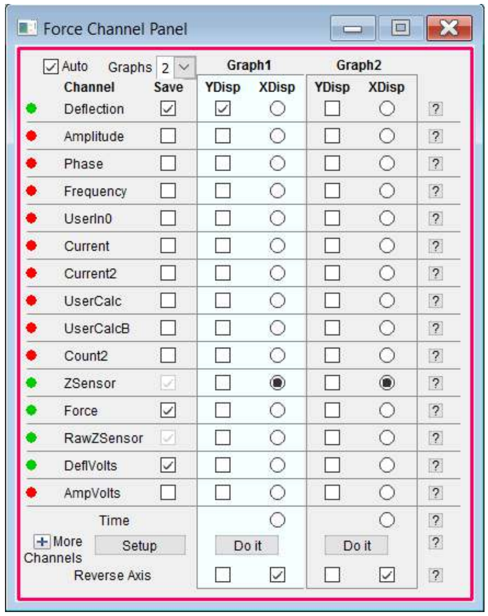


Figure 18.15.: Force Channel Panel

The green, blue, and red circular dots to the left of the channels describe the settings of those channels as follows:

- **Red** The channel is not on (no activation checkmarks).
- **Blue** The channel is ONLY displayed (e.g., *YDisp* checked for a given Channel, but *Save* not checked)
- **Green** The channel is saved, regardless if displayed or not.
- The Channel column gives all the possible data channels. Note that, depending on the experiment, some channels will give no real signal.
- The Save column is what data is always saved - the channels checked here are what can be called up in the Force Display Panel in the Analysis section of this software (i.e., there is a pull-down menu that you can select the Y axis for).
- The *Graphs* dropdown menu allows up to five channels to be plotted simultaneously. For the number of graphs selected, that many X&Y columns will be displayed.
- Each Graph column (1,2, . . .5) allows for selection of two dimensions to plot the desired curve/signal in. On each plot, multiple Y-axis dimensions can be plotted vs. a single X-axis.

To display time as the X-axis, activate the 'Time' radio button towards the bottom of the Force Channel Panel. Figure 18.16 on page 246 shows an example of a Deflection (Y) vs. Time (X) plot assigned via the Force Display Panel. The inset shows the X-axis radio label selected to assign time as the X axis; 'Deflection' would be checked in the Column tab.

18.2.7. Closed Loop (Z) Activation

Closed Loop force measurements are necessary for constant velocity or consistent loading. Note that it will have more noise associated with it than in "Open Loop".

1. To turn on, go to the Setup menu to activate it.

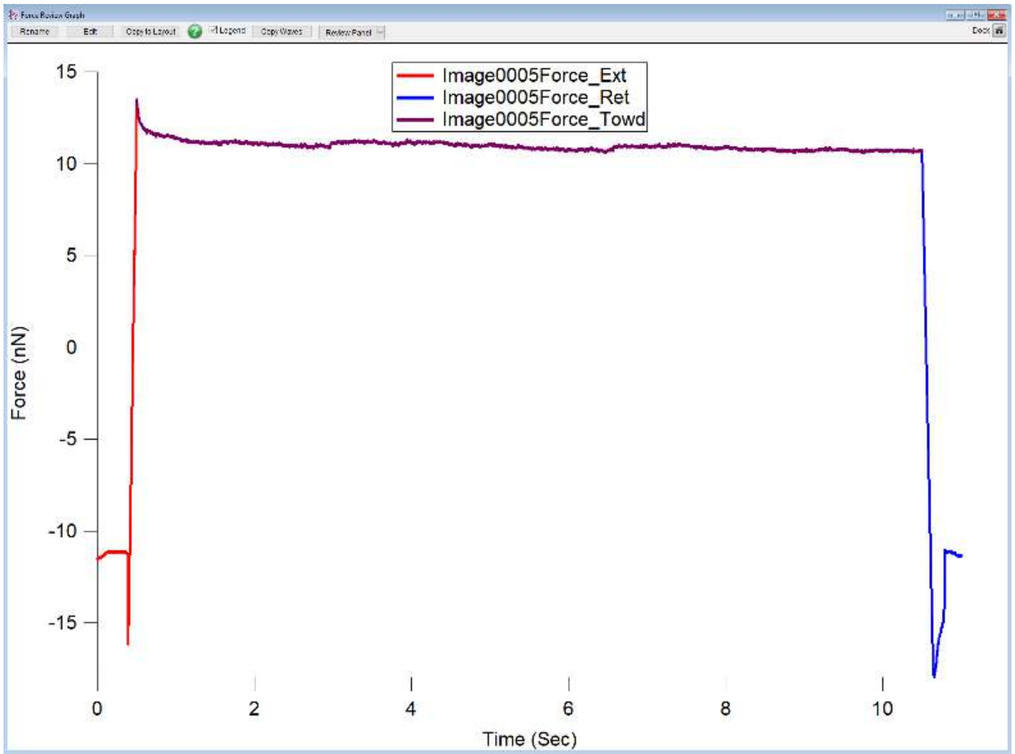
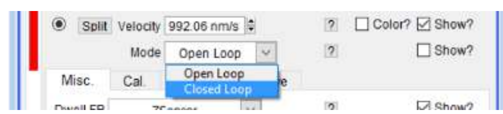


Figure 18.16.: Deflection vs. Time Plot

2. **Select Closed Loop:**

- From the dropdown menu, select "Closed Loop".



3. Closed Loop can also be activated when activating the 'Indenter' checkbox in the Indenter Panel.

18.2.8. Empirical considerations

18.2.8.1. Software parameters:

1. **Force Distance:**

- Choose a force distance that is large enough to have some of the Free Air (non-contact) portion in the curve, such that the contact point can be properly determined during the analysis.
- Using force distances that are too long (i.e., mostly all free air in the curves) reduces the portion of the curve dedicated to the displacement by the surface and therefore reduce the resolution of the curve.

2. **InvOLS / k Changes:**

- Sometimes you can get different InvOLS values numbers before and after an experiment. There are a few possible causes of this. Contamination of the probe can change the SUM, which can change the InvOLS. Thermal drift can change the superluminescent diode (SLD) position over long periods of time (hours to days). Degradation of the probe coating can also cause this. This can also manifest itself to some degree in a wider than expected force distribution results.
- Floppy cantilevers can be electrostatically bent by the surface even when the tip is retracted several microns. This in turn compromises the thermal peak fit on the final step of the k determination. Wheel the tip several hundred microns up, if possible, and the peak should be sharper (for an example, see Section 20.3.5 on page 291).

18.2.8.2. Experimental

1. **InvOLS / k Changes:** Choosing a cantilever that is appropriate for the measurement is important. In general, the spring constant of the probe should be close to the effective spring constant of the sample (i.e., for every nm of cantilever deflection, there should be a nm of sample indentation).

2. **Thermal Drift:** It is common once a system is set up to let it equilibrate for 30 minutes or more. When operating in fluid, this process can take longer. This will be especially common in labs having a large variation in the room temperature.

3. **Sample Prep:** It is important to have the substrate well affixed. Avoid two-sided tape, as it can introduce drift and creep into the experiment. This includes copper and carbon tape. An alternative that is removable is silicone vacuum grease, First Contact (R) from photonic cleaning solutions, and silver paint from Ted Pella.

Thermal Equilibration

Work in Fluid **If the sample allows (i.e., doesn't swell). Do this to avoid large adhesion (i.e., jump to contacts), which will make the point of contact determination even more subjective.

IF Working in Fluid When acquiring force curves in fluid, which is common, it is a good idea to let the system equilibrate for 30 min to a couple of hours. The most dramatic deflection drift will occur in the first 20 minutes, but drift will continue to occur as the thermal gradients approach equilibrium.

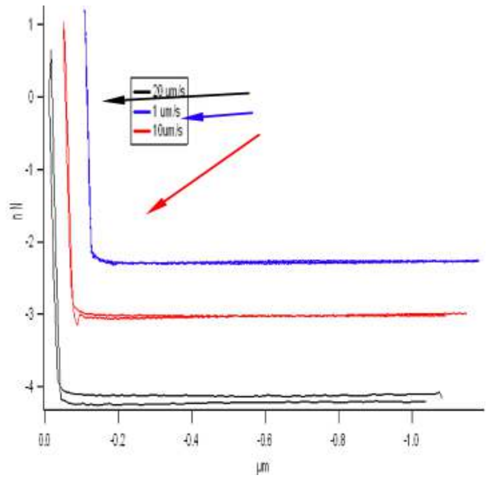
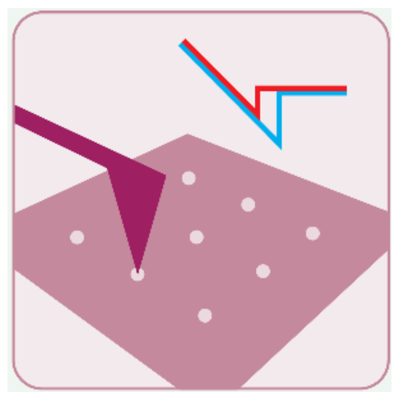
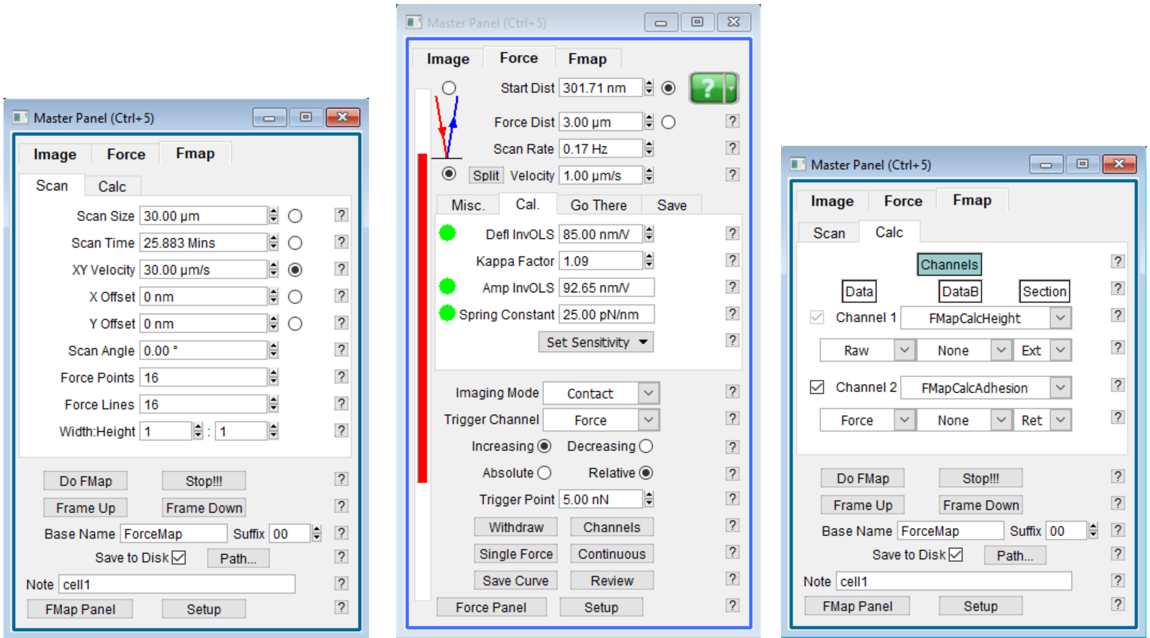


Figure 18.17.: Hydrodynamic effects can occur in fluid with fast force curve acquisition cycles.

18.3. Force Mapping



Force Mapping is used to acquire an m×n array of force curves to create an "image" of the surface. It is essentially a 3D map of the tip-sample interactions.



(a) Fmap tab showing Scan sub-tab parameters

(b) Force tab showing Cal. sub-tab and Triggering parameters

(c) Fmap tab showing Calc sub-tab parameters for defining Data Channels per respective axis

Figure 18.18.: Force Map (FMap) tab

The third tab in the Master Panel called "FMap" (Figure 18.18a on page 249) allows the acquisition of Force Maps, also known as Force Volumes, on Asylum Research AFM's.

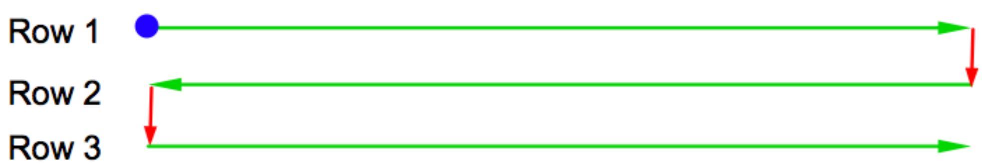


Figure 18.19.: Curve Collection

As shown in Figure 18.19 on page 249, the curves are collected in a 'Frame Down' raster fashion (starting point blue dot) in an "across the fast scan, move to next slow scan line, across in fast scan" iteration. The opposite is true is when doing a 'Frame Up' Force map.

Note For MatLab users, exporting is an important consideration for post processing due to the way the files are exported.

The FMap tab has two sub-tabs with similar controls for imaging, plus new data channels appropriate for Force Maps. The force parameters used in the Force Maps must be entered in the Force tab. The Help menu defines these parameters pretty well:

- **Scan** Defines the XY scan area and operates similar to the main Tab (Figure 18.18a on page 249).

- **Calc** Defines what channels will be displayed. See description above (Figure 18.18c on page 249).
  - The Data column displays captures the X axis data.
  - The Data B column displays/captures the Y axis data.
  - The Section column allows you to extend or retract a portion of curve to be analyzed for the map.

The lower portion of the FMap tab is for capturing data, similar to the Main tab.

The 'Continuous Maps' checkbox collects force maps continuously in the same scan range region until told to stop.

18.3.1. Procedure for Force Map Acquisition

Since force maps may take minutes to hours to acquire, depending on the sample and parameters necessary for quantitative measurements, it is a good idea to acquire several individual force curves (ideally at different locations) and analyze them using either the Analyze tab or Elastic tab in the master Force Panel.

To acquire and analyze several individual force curves:

1. Define whether to acquire Force Curves in Contact mode or AC mode.
2. Define the *Scan Size* for the Force Map. If acquiring a force map directly after imaging an area, the *Scan Size* field is automatically filled in from the *Scan Size* field on the Main tab of the Master Panel.
3. Define the *XY Velocity*, i.e. the translation between points in XY. This is similar to scan rate. If you are uncertain what to use the default value of 100nm/s is a good starting point.
4. Adjust the *Scan Angle* of the tip as needed. This parameter is identical to normal scanning (i.e., if 0°, then the x-axis will be parallel to the length of the cantilever; 90° will be normal to the length of the cantilever). If you have already captured an image, you most likely want to keep the scan angle the same.
5. Define the number of *Force Points* and *Force Lines*. These two parameters are quite important. A lot of points and lines will result in long scan times, often hours long, which may not be ideal for a living sample. You may also want to limit the number of points and lines so that the tip isn't indenting in the same area of the sample more than once. Too few points, however, typically won't give you enough for statistics. You will need to optimize *Scan Size*, *Force Points*, and *Force Lines* for each sample and measurement.
   a) The *Scan Time* is how long it will take to acquire the Force Map based on the defined parameters (all the XY translations and time to acquire each force curve).
   b) The *Width:Height* is the aspect ratio of the scan area, just as in imaging.
6. Define the force acquisition parameters in the Force tab. The software uses these parameters for the Force Map. The main parameters to adjust include:
   a) Force Distance (*Force Dist*): make sure that the Z Piezo will pull the cantilever away from the sample surface enough so that when the tip moves to the next position it won't ram into the surface.

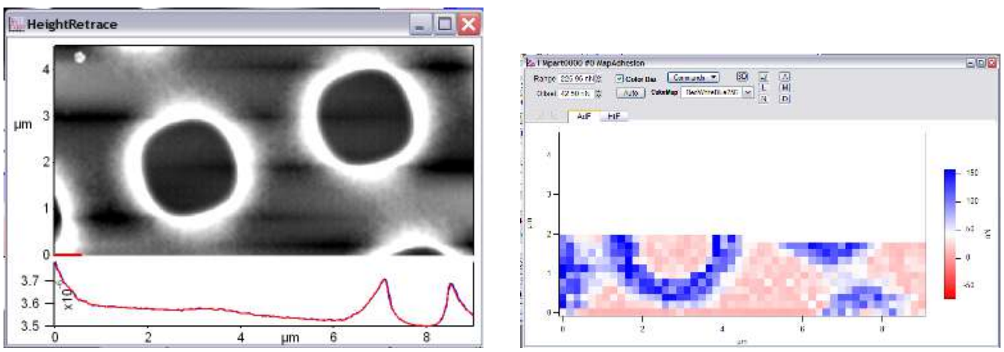
b) *Velocity*: many samples (soft, biological, viscoelastic) show a velocity dependence effect on modulus (i.e., the fast the tip indents into the surface the higher the calculated modulus) so it is important to select a velocity that won't artificially increase modulus.

c) *Trigger Channel* and *Trigger Point*: Although there are many options if the cantilever has been calibrated, it is important to select a channel other than raw Defl (i.e., DeflVolts). Force is most commonly used, as this is what is displayed when applying a contact mechanics model to the data (Figure 18.18b on page 249).

7. Select the appropriate data inputs in the Calc sub-tab. The defaults are Height and Adhesion. You can also swap out "Adhesion" for a "2-Point Modulus Fit". But it is important to note that the full fit cannot be done in real time; you will have to wait until after the entire Force Map has been acquired before using the Elastic tab.

A window, looking like an MFP-3D 'Display Window', appears during acquisition, with tabs correlating to the channels selected in the Calc. sub-tab (see Figure 18.20 on page 251).

8. Click 'Do Scan', 'Frame Up' or 'Frame Down' to begin Force Map acquisition.



(a) (b)

Figure 18.20.: Screen shots of real time force map acquisition. Pixels are updated after each subsequent acquisition.

18.3.2. Cantilever Based Indentation

Cantilever-based indentation probes compliance of materials, but it can be subject to error due to the ploughing effect of the probe as the piezo pushes the probe tip into the surface. The error can be reduced by using small indentation values.

This section will introduce:

- Most basic concepts of nanoindentation of materials.
- Empirical considerations regarding the cantilever/ tip selection.
- How to navigate the AR indenter panel software.
- Some examples of various materials from the AR user base.
- Tip material is important because it must have a much larger modulus than the material to be indented.
  – Silicon tips can be used for many biological and polymeric materials but may not be effective on harder materials.

– Diamond (solid or coated) and silicon nitride tips are widely available. Spheres or various modulus can be glued to levers for using Hertzian contact mechanics (also see Section 18.3.3 on page 255).

It is also important to mention the delicate interplay between elastic and plastic deformation in the material being indented (elastic will recover from the removed load, while plastic does not). The indenting probe pushes down into a material; reaches some trigger point and retracts. The primary difference between a simple force curve and an indentation is the use of the indentation panel (*AFM Controls > Indenter*), which gives better control of the indentation. Indenting on a plastic material leaves an indent depth (hf) in the material smaller than the total indent depth (h). This can be described as a simple power law relation:

$P=a(h-h_f)^m$

Where:

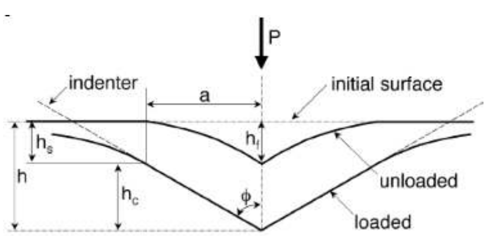
P is Load; typically in the force dimension (N).

a and m are empirically determined fitting parameters associated with indenter shape.

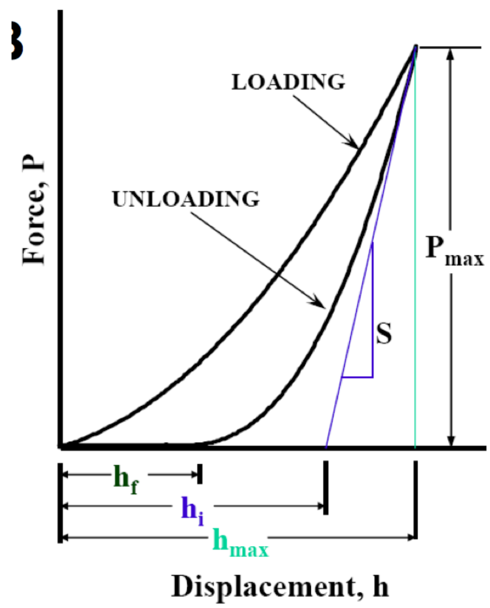
h is the total indentation depth.

h_f is the final displacement after complete unloading, and also determined by fitting.

There are various other h's depending on the fitting model being used.



(a) Various indentation depths (h) in a material that deforms plastically



(b) Typical load/unload curve for a material that has some plastic deformation

Figure 18.21.: Qualitative schematic commonly used in nanoindentation to illustrate the difference between maximum indentation (h) and indentation depth after load is removed (h_f). (Figure adapted from Oliver, W.C., Pharr, G.M., J. Mater. Res. 2004, 19 (1) p3-20.)

General qualitative responses of various materials under applied load can be seen in Figure 18.21 on page 252. Panel A shows a fully elastic system; there is no hysteresis between the load and unload components, indicating that the material fully recovered while as the load was removed.

Panels B and C show various amounts of plastic deformation (less and more, respectively), based on the degree of hysteresis between load and unload components. Panel D shows some material response/rearrangement event occurred during the *unload* cycle; Panel E shows some material response/rearrangement event occurred during the *load* cycle. Panel F shows that material is moving significantly at the initial unload, indicative by the negative slope of the unload component.

Note To obtain curves like this with the MFP-3D, the Indenter Panel needs to be used (Section 18.3.2.1 on page 254).

References:

- Vertical indentation-
  - Oliver, W.C., Pharr, G.M., "Measurement of hardness and elastic modulus by instrumented indentation: Advances in understanding and refinements to methodology", J. Mater. Res. 2004, 19 (1) p3-20. (REVIEW)
  - Gong, J., Miao, H., Peng, Z., "Analysis of the nanoindentation data measured with a Berkovich indenter for brittle materials: effect of the residual contact stress ", Acta Materialia 52 (2004) 785–793.
  - Mencik, J., "Determination of mechanical properties by instrumented indentation", Meccanica (2007) 42:19–29. (REVIEW)
  - W.C. Oliver and G.M. Pharr, J. Mater. Res. 7, 1564 (1992)
  - VanLandingham, M.R., "Review of Instrumented Indentation", J. Res. Natl. Inst. Stand. Technol. (2003), 108, p249-265. (REVIEW)
  - Schuh, C.A., "Nanoindentation Studies of Materials", Materials Today 2006 9 (5) p32-39.
  - VanLandingham, M.R., Villarrubia, J.S., Guthrie, W.F., Meyers, G.F., Nanoindentation of Polymers: An Overview", Macromol. Symp. 2001, 167, p15-43. (REVIEW)
  - Fischer-Cripps, A.C., "A review of analysis methods for sub-micron indentation testing", Vacuum 58 (2000) 569-585. (REVIEW)
  - Fischer-Cripps, A.C., "Critical review of analysis and interpretation of nanoindentation test data", Surface & Coatings Technology 200 (2006) 4153 – 4165. (REVIEW)
  - Mosesonm A.J., Basu, S., Barsoum, M.W., "Determination of the effective zero point of contact for spherical nanoindentation", J. Mater. Res. (2009) 23 (1) p204-209.
- Cantilever based:
  - Lin, D.C., Dimitriadis, E.K., Horkay, F., J. Biomech. Eng. "Robust Strategies for Automated AFM Force Curve Analysis—I. Non-adhesive Indentation of Soft, Inhomogeneous Materials", (2007) 129, p430-440.
  - Lin, D.C., Dimitriadis, E.K., Horkay, F., J. Biomech. Eng. "Robust Strategies for Automated AFM Force Curve Analysis—II: Adhesion-Influenced Indentation of Soft, Elastic Materials", (2007) 129, p904-912.
  - Lin, D.C., Horkay, F., "Nanomechanics of polymer gels and biological tissues: A critical review of analytical approaches in the Hertzian regime and beyond", Soft Matter 2008 4 p669-682. (REVIEW)
  - Masterson, V.M., Cao, X., "Evaluating particle hardness of pharmaceutical solids using AFM nanoindentation" International Journal of Pharmaceutics 2008 362 p163–171.
- Some useful web sites that provided some understanding:
  - http://www.its.caltech.edu/~ae244/Lecture10_110107.pdf
  - www.csm-instruments.com/en/webfm_send/42/1
  - http://www.microstartech.com/

18.3.2.1. Indenting Procedure

Before indenting, the following steps should have occurred:

1. Load tip, align SLD on cantilever, and zero PSD.
2. Bring the probe into piezo range of the sample.
3. Confirm the *Imaging Mode* is set to "Contact".
4. Find InvOLS and cantilever spring constant. This can be done with the GetReal software, or with a force curve and the thermal method. (18.3.5)

Software Operation: There are two ways to approach cantilever-based indentation in the MFP3D software, as follows:

1. Use the Force tab of the Master Panel exclusively; this is exactly the same as standard force curves. Trigger points can be used as loads; dwells can be used as holds or creep studies.
2. Use the Force Tab in conjunction with the Indenter Panel. With this approach, the Force tab can be used to apply a very small trigger point (i.e., 1 nm deflection trigger), then the cycle defined in the activated indenter panel applies the load until it reaches its end and completes the retract curve.

The AR Indenter Panel

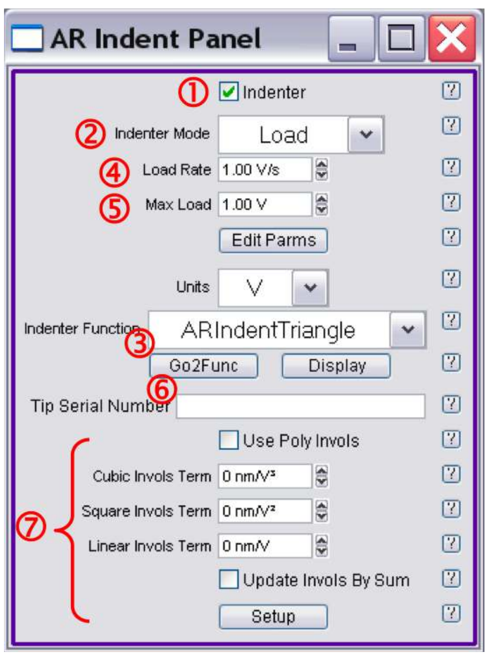


Figure 18.22.: AR Indent Panel. Numbers correspond with steps below.

1. When the 'Indenter' check box is activated, the AR Indent Panel uses Closed Loop Z-sensor to apply the load (after the initial force trigger used to find the surface).
2. The *Indenter Mode* dropdown menu allows you to select important options regarding the feedback:

    a) *Load*: Uses force or deflection feedback to control the load.

b) *Displacement*: The motion of the Z-sensor signal or how far the probe chip (the part you pick up with tweezers) moves during the indentation.

c) *Indentation*: The Z-sensor signal minus the deflection. This indicates how far the probe tip pushes into the sample surface.

3. The *Units* dropdown allows you to choose between Volts, Distance (m), or Force (N).

4. *Load Rate* can now be chosen in a comfortable dimension.

5. Maximum load (*Max Load*) can also be selected.

6. The *Indenter Function* dropdown lets you choose between triangle (voltage/loading) waves, add a dwell (for creep), or use the AR Function editor for custom routines (very flexible) AC and DC modes. Specific options include:

a) *ARINdentTriangle*: Linear loading and unloading at the same rates.

b) *ARINdentTriangle2*: Linear loading and unloading at different rates.

c) *ARINdentTriangleDwell*: Linear loading and unloading at the same rates, with a hold (dwell) in between

d) *Function Editor*: Allows custom waves to be built, including static and dynamic load and hold applications (see Section 18.3.4 on page 258 for more about the function editor).

7. Disregard these: The *Tip Serial Number* and 'Use Poly Invols' is for the Vertical Nanoindenter Flexure Module accessory option.

18.3.3. Colloidal Probe Microscopy

Colloidal probe microscopy is a force spectroscopy technique in which a spherical probe is mounted onto a cantilever and used (in most cases) to push on a soft material. The sphere allows Hertzian contact mechanics to be applied to the analysis.

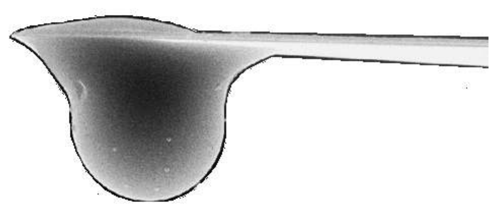


Figure 18.23.: Colloid Lever Side View

Some reviews and useful papers regarding colloidal probe microscopy include:

- Bonaccurso, E., Kappl, M., Butt, H.-J., Current Opinion in Colloidal & Interface Science, 2008 13 107-119.
- Vezenov, D., Noy, A., Ashby, P., J. Adhesion Sci. Technology 2005 19 (3-5) p313 – 364.
- Leite, F.L., Hermann, P.S.P., J. Adhesion Sci. Technology 2005 19 (3-5) p365 – 405.
- Christendat, D., Abraham, T., Xu, Z., Masliyah, J., J. Adhesion Sci Technology 2005 19 (3-5) p149-163.

- Tormoen, G.W., Drelich, J., J. Adhesion Sci Technology 2005 19 (3-5) p181 – 198.
- Kappl, M., Butt, H.-J., Particle & Particle Systems Characterization 2002 19 (3) p 129-143.
- Lin, D.C., Horkay, F., "Nanomechanics of polymer gels and biological tissues: A critical review of analytical approaches in the Hertzian regime and beyond", Soft Matter 2008 4 p669-682. (REVIEW)

Some assumptions for acquisition and Hertzian analysis include:

- Load $\propto \partial(\text{depth})^{m}$, where is a function of the geometry & the elastic properties (Young's modulus and passion ratio) and *m* represents the shape of the pressure distribution.
- Is fully elastic.
- Pressure distribution same a spherical indenter shape.
- Radius of spherical indent < radius of sphere
- Frictionless
- Bodies large compared to volume under contact.
- Isotropic
- Spherical indenter contacting flat surface.
- Cantilever spring constant is NOT similar to material being pushed (want cantilever k much larger).
- Elastic indenter much stiffer than surface

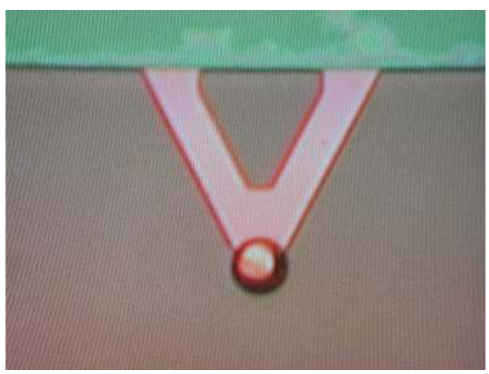


Figure 18.24.: Colloid Lever Top View

Note On cantilever calibration with spheres, it is usually more challenging to calibrate the cantilever spring constant (via thermal method) with an affixed sphere, due to the increased surface area (especially in air). An example of large surface forces acting on a sphere can be seen in Figure 18.25 on page 257; notice the large jump to contact and adhesion to surface before snapping back to free air.

There are two ways to avoid large attractive forces that will affect the virtual deflection calibration:

- *If working in air* Calibrate virtual deflection and the thermal tune far off the surface (several hundred microns) to avoid long range charges between the sphere and surface that premature the deflection cantilever.
- *Work in fluid* Although the much lower Q of cantilever can make fitting the curve from thermal method more challenging.

For acquisition, some considerations:

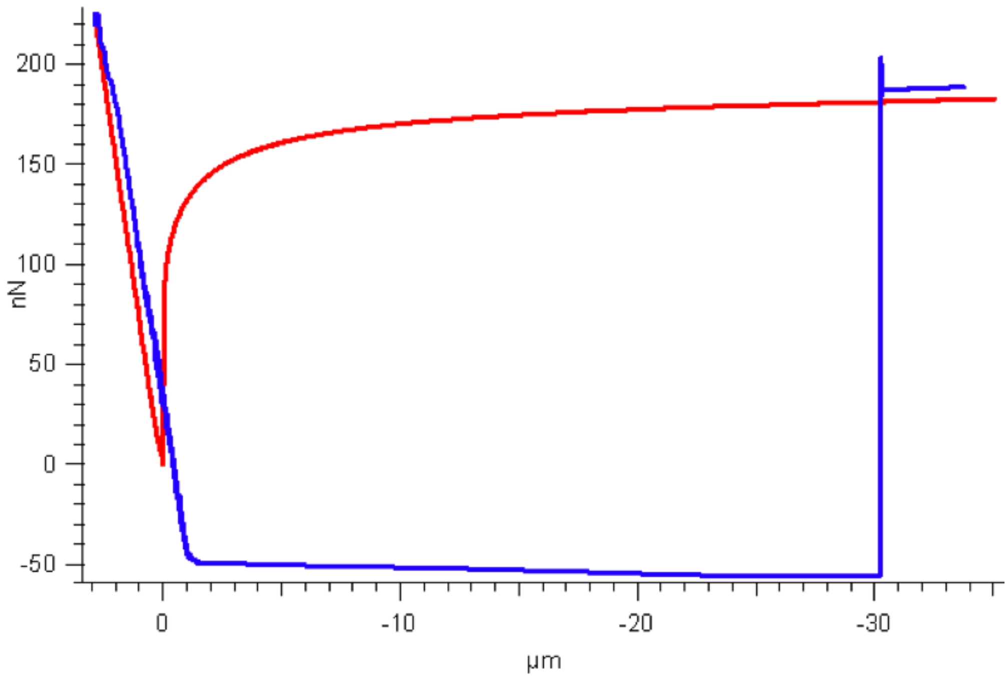
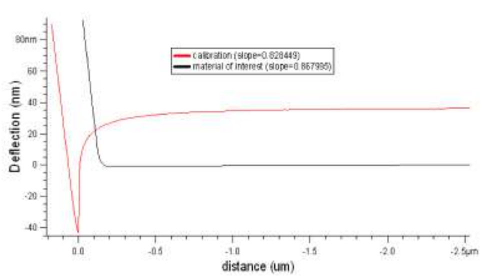
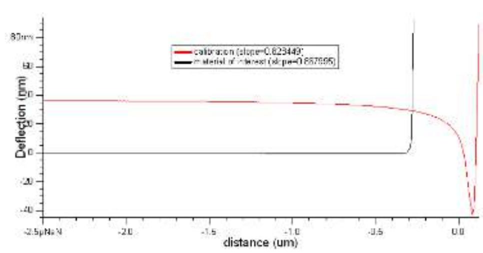


Figure 18.25.: Large surface forces can act on a sphere affixed to cantilever

- Do not indent more than a quarter of the diameter of the sphere affixed to the cantilever; this is for the Herztian mechanics-based analysis.
- Choose the cantilever spring constant so that it can apply a load to the sample, rather than (fully) deflect with the load. This can be determined from the Force vs. Indentation plot: If the contact regime has a slope that goes to infinity, then the lever is likely fully deflecting under the load, rather than deforming the sample. Figure 18.26 on page 257 shows an example of this. In the left graph, it is not clear that the lever is fully deflecting. However, when force is plotted against indentation (or separation), the slope looks like a right angle to the free air (non-contact) portion of the curve, as shown in the right graph.



(a) Force vs. distance



(b) Force vs. Indentation (deflection (PSD signal) is subtracted from the LVDT signal.

Figure 18.26.

Colloidal probes are available two configurations:

- Commercially available from NovaScan (Iowa), sQube (Germany), and probably some other vendors.
- Can be home-made in the lab, especially if your AFM has some sort of bottom view optics.

Note Tipless levers are also available from most vendors.

Regarding sphere materials, things to consider are:

- Material's thermal expansion coefficient is an important consideration. (Borosilicate has a favorable one.)
- Material's modulus (don't want it too soft to act as another spring in the system).
- Mono/poly dispersity (i.e., as close to sphere shape as possible).
- Material charge: Most polymers and glass products are charged, which can contribute to attractive/repulsive forces.

18.3.4. Dynamic (AC) Force Spectroscopy

Dynamic force spectroscopy acquires force curves while in AC mode. Dynamic force spectroscopy is done by ramping the z piezo while oscillating the AFM probe. AC mode force curves show the interaction of the oscillating probe as it approaches, begins tapping, and often deflects against the surface of the sample (see Chapter 5 on page 46).

As the drive amplitude is varied, the degree of net attractive and net repulsive interaction the tip has with the surface can change. Figure 18.27 on page 258 shows various AC force curves acquired on mica at varying drive amplitudes. Notice the amount of net repulsive and net attraction the tip has with the surfaces varies with distance from the surface.

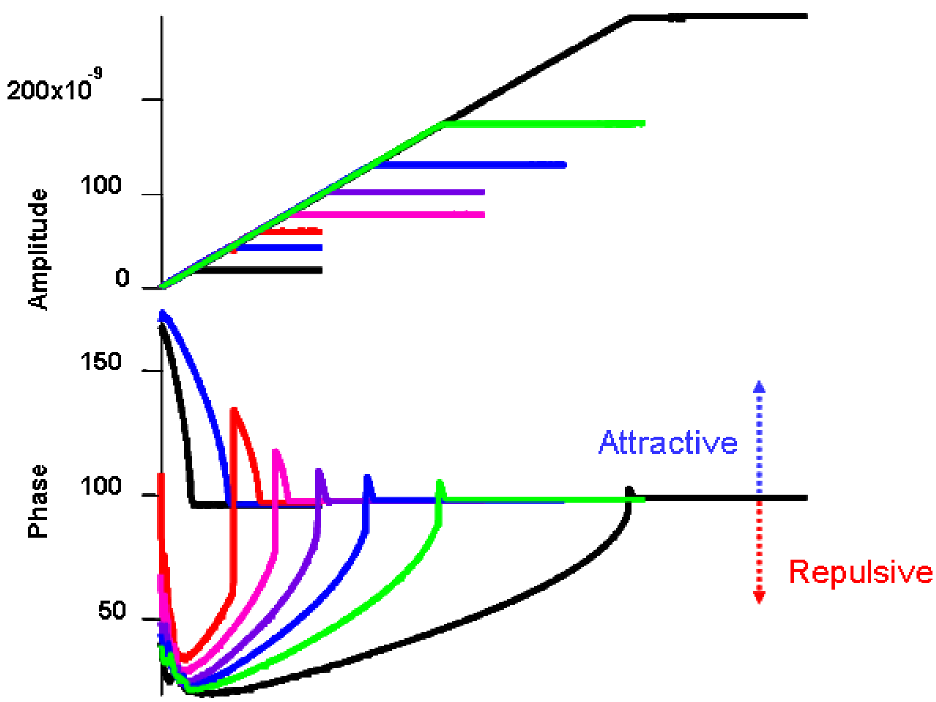


Figure 18.27.: AC force spectroscopy

Figure 18.28 on page 259 shows some "point and click" AC force curves defined from a phase image of water-based latex paint (Sherwin Williams).

- The sample was imaged with an Olympus AC 240 (k~1.6N/m; ~70kHz) in air in repulsive mode; the free air phase was ~64 ° (depicted by orange dashed line).
- The force curves were acquired at constant (drive) amplitude (same as the image), and the trigger point was 5 nm to reduce the amount of phase bi-stability (mode-hopping) under applied load.
- There are clear differences on the surface. Notice that points 2 and 4 (black and blue, respectively) are similar during the approach (extension) curves; there is an initial attraction to the surface, then repulsion; and at some point, when the tip becomes closer to the surface, it flipped back to attractive mode.
- Point 3 (red) never goes into the repulsive regime.

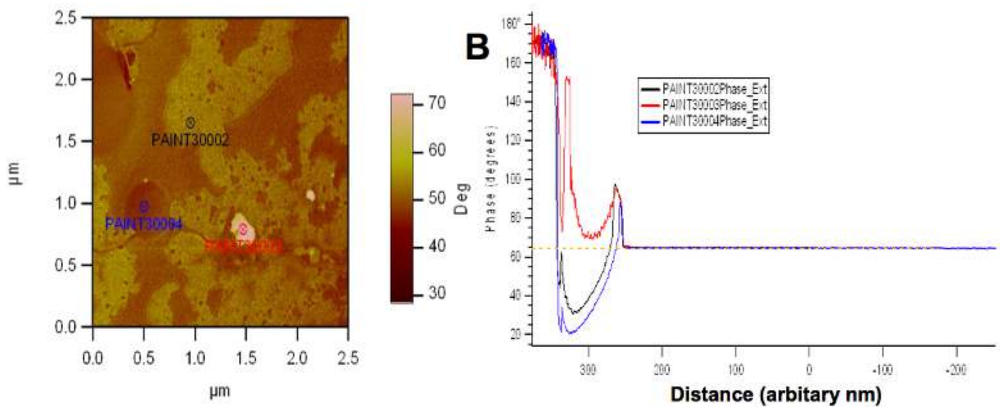


Figure 18.28.: AC “point and click” force curves on heterogeneous sample. A) Phase image of paint sample with location of user-defined force curves; B) individual AC force curve Phase vs. distance (Z)–orange dash line indicates Free Air Phase values of ~64 degrees. (Yes, that is the di color table on the 3D.)

18.3.5. The MFP-3D Function Editor

The MFP-3D software has a “Function Editor” that is very useful for applying custom made waveforms for force spectroscopy, indentation, and various electrical techniques.

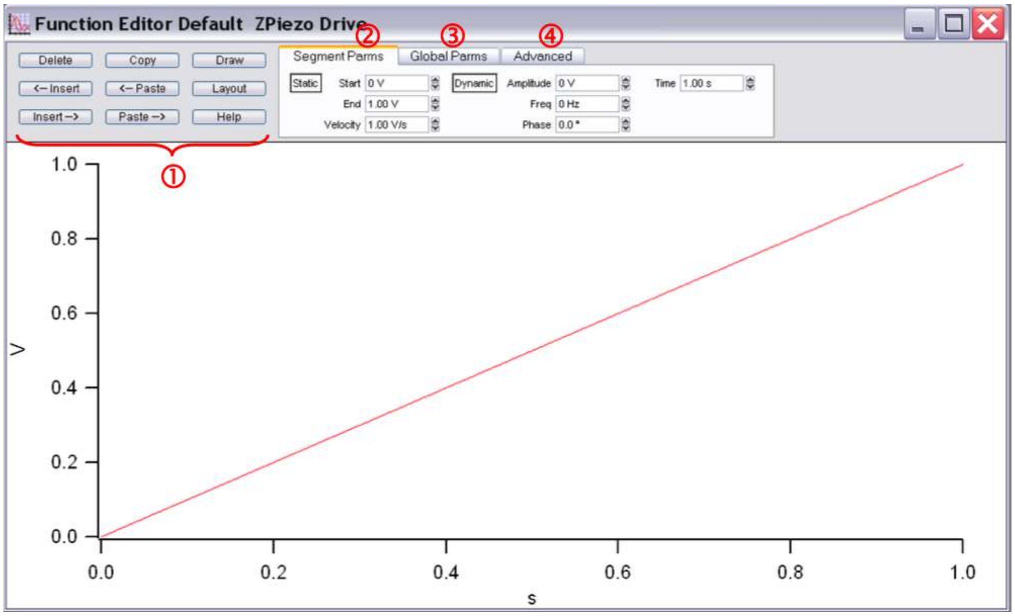


Figure 18.29.: The AR Function Editor allows custom wave forms to be applied to the Z piezos or tip sample biases for advanced force spectroscopy experiments.

To open, click 'Function Editor' in the Indenter Panel. A panel like the one shown in Figure 18.29 on page 260 will appear.

1. Use the buttons to the right of the panel which allow the segments to be copied then pasted to the left or right of the copied segment.

   'Insert' Inserts a segment to the left or right of the forward-most (red) segment, having the same constant values as the red segment its attached to.

   'Paste' Pastes a copied segment; will have the same parameters as copied segment.

   'Copy' Copies a segment. Segments must in activated (red) with mouse click to copy them

   'Draw' Allows 'free hand' line segments that are strung together.

   'Layout' Dumps image of function to an Igor Layout.

   Whichever segment is displayed as red is the forward most segment to be manipulated via the buttons or setvars.

2. **Segment Parms** sub-tab:

   'Static' Used for DC voltages applied to the piezo (stack). The *Start* and *End* piezo voltages and *Velocity* can be user-defined.

   'Dynamic' Used for AC voltages applied to the piezo (shake); the *Amplitude*, Frequency (*Freq.*), and *Phase* setvars can be adjusted accordingly. Useful for oscillating the tip for viscoelastic materials.

3. **Global Parms** sub-tab:

   Scales and offsets the signals in the function generator.

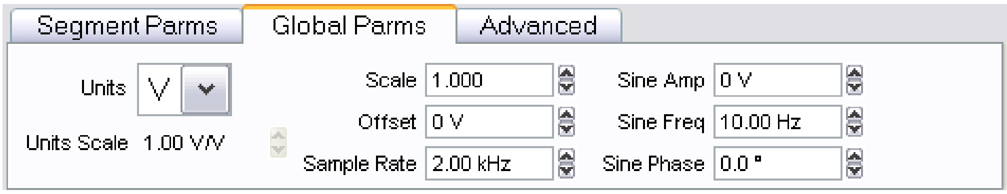


Figure 18.30.: Global Parms Subtab

'Units' Pull-down allows choice between Volts (applied to piezo) or meters (accurate with calibrated InvOLS). This can also be useful if using the AR Function Editor for applying electrical biases to tip or sample if doing an electrical technique (not described here).

'Units Scale' Will give the sensitivity (InvOLS) displayed if meters selected for Units.

'Scale' Allows the generated function to be scale be some setvar factor; axes will rescale.

'Offset' Offsets the signal on the Y axis.

'Sample Rate' Frequency (# of pts/sec) at which the data is collected.

'Sin Amp' Allows the sinusoidal amplitude to be scaled.

'Sine Freq' Allows the frequency of the Sine signal to be scaled.

'Sine Phase' Offsets the Phase from 0 to some other user defined value.

4. **Advanced** sub-tab:

   Allows you to save functions for future use and to define what the function will drive.

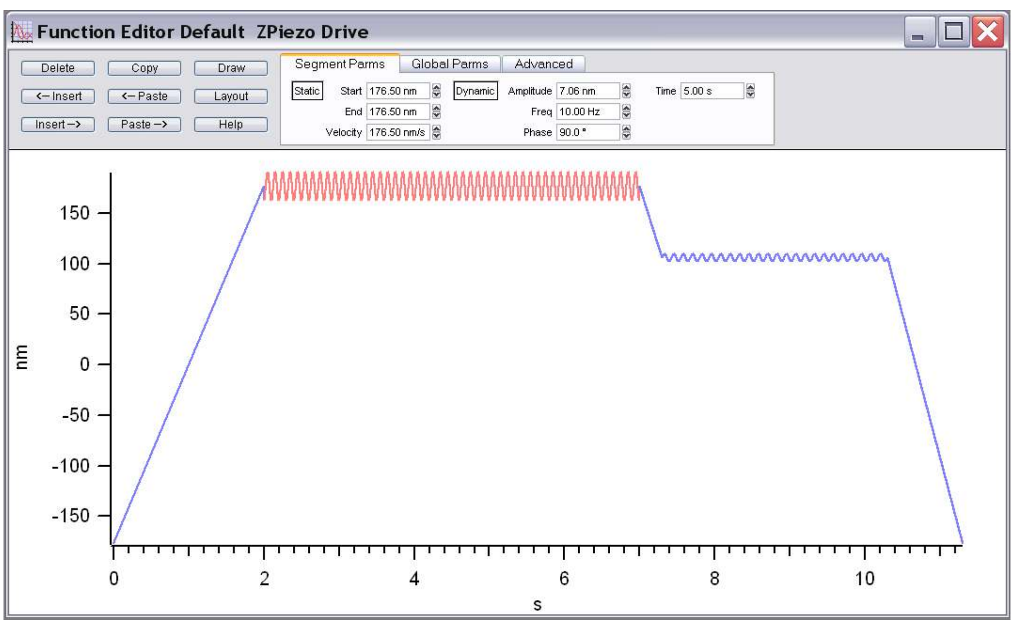
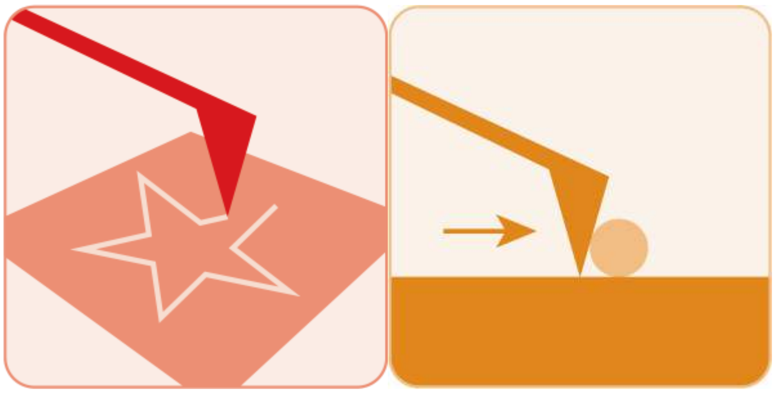


Figure 18.31.: An example custom waveform

Figure 18.31 on page 261 shows a simple function generated in the Function Editor. Many segments were added and set to tell the cantilever to deflect to 176 nm, hold for 5 seconds with an oscillation of 10 Hz with 7.06nm peak to peak amplitude at 90° phase, pull back a bit and hold again for 3 s at 8 Hz with 31.52 nm peak to peak amplitude 0°, and then return to a negative deflection.

19. MicroAngelo^{TM} Lithography & Manipulation ...



CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

The closed loop X-Y-Z positioning of the MFP-3D and Cypher systems is ideal for accurate and complex scanning probe-based lithography and nanometer-to-micron scale tip manipulations. Typically, an area of the substrate is first imaged nondestructively (in AC mode or gentle contact) to determine what the surface looks like, then mouse strokes representing the tip's path(s) can be added to the image using the MicroAngelo software. Images can be imported and scaled appropriately, setpoint voltage (or tip bias) ranges defined for the execution, and scripting performed.

The following instructions and examples assume you are using an MFP-3D AFM that is set up with desired tip and sample and is engaged on the surface.

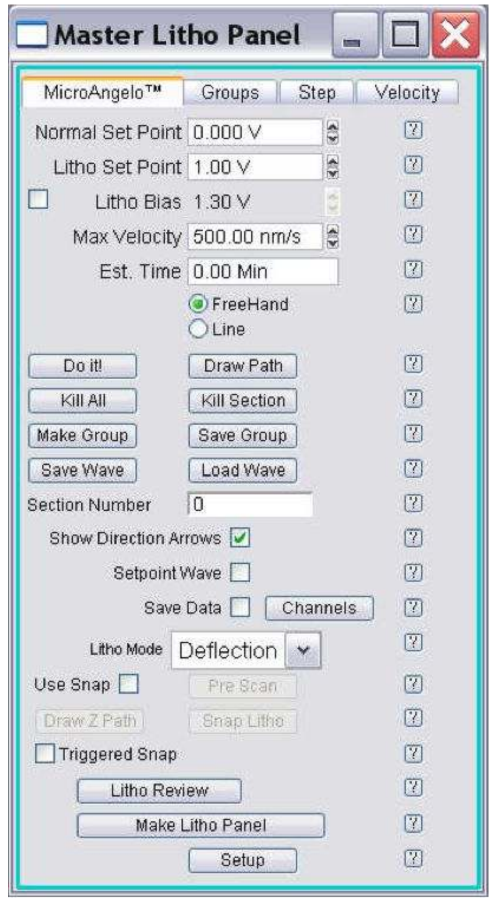
19.1. Introduction to Software Panel

To open the Master Litho Panel and image the substrate:

1. In the main menu bar of the AR SPM software, open the Lithography panel by choosing *MFP Controls > Litho Panel*.
The Master Litho Panel consists of four tabs including MicroAngelo, Groups, Step, and Velocity, as shown at right.

   - **MicroAngelo** tab: Allows you to draw lines on the image representing tip paths, determine the set point during the manipulation and the set point between paths (kind of like a retraction set point), make groups of paths, define constant velocity during the path, determine mode (contact or AC), apply constant tip bias (if performing an oxidative or reductive manipulation, and other features of this window will be described later in this procedure text.
   - **Groups** tab: Allows you to import an image, rescale a saved path (or group of paths) that you have previously drawn, or select between saved groups in memory. There are slider bars that allow X& Y rescaling preserving the aspect ratio; offsetting the group in X and/or Y; and group rotation.
   - **Step** tab: Allows you to make arrays of points by defining number of points, spacing between points, dwell times (& ranges), and tip voltages (& ranges, if applicable).
   - **Velocity** tab: Varies the tip velocity along the path based on the pixel color on an imported image.

   Note The Help menus are excellent for using the MicroAngelo software. To access, click on the question mark to the right of every parameter or button in the software panels.

2. Image the substrate to determine an area suitable for lithography or the manipulation. Depending on your substrate material or application, you can have the spring constant determined prior to the manipulation to be aware of the amount of force applied at a given set point voltage.

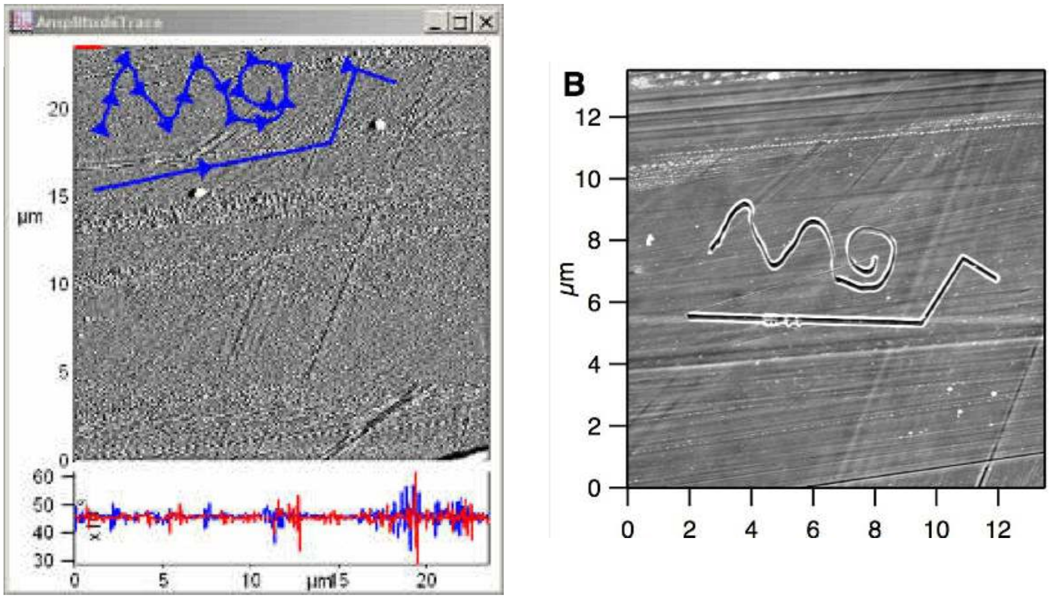
19.2. Drawing lines

19.2.1. Basic Operation

MicroAngelo allows the user to draw linear or freehand lines representing tip paths on an image window. Multiple lines can be grouped together, offset, rotated, and rescaled, as needed.

1. In the MicroAngelo tab of the Master Litho Panel, activate the 'FreeHand' or 'Line' radio button, depending on the type of line you want to draw.
2. Click the 'Draw Path' button. Notice the button now reads 'Stop Draw', the freehand and line selections become faint because they can no longer be switched with each other, and a crosshair cursor appears when the mouse is on one of the image channel windows.
3. Draw a path on the image window with the mouse cursor. If drawing more than one path (with Line tool or FreeHand tool), left-click at each segment and double-click when finished drawing a line segment.
4. Paths can be grouped together. If you want to save the pattern for future use, click 'Make Group', then 'Save Group', and name it in the dialogue that appears. Notice this name will now appear in the list in the Groups tab.
5. If you want to switch between the FreeHand and Line tools, click 'Stop Draw', select the one you now want to use, and click 'Draw Path' again. Notice the drawn line turns from red to blue after clicking 'Stop Draw'.
6. For scratching-based lithography or manipulations, change the the *Litho Mode* to "Contact". (You can also instead change the *Imaging Mode* to "Contact" on the Master panel.)
7. Select a *Normal Set Point* that will not damage the surface while the tip is moving between paths. In Contact mode, a deflection value that is the same as the free air deflection (or more negative than that value) won't damage the surface between litho paths.
8. Determine and select a *Litho Set Point* value that will apply enough force to perform the desired lithography result (scratch, oxidative/reductive, diffusion based direct write, etc.) or have enough force to perform the manipulation without riding over what is intended to be pushed.
9. Click 'Do It!' to execute the litho event. Notice this button label changes to 'Stop Litho'.
10. The tip will be withdrawn when completed with the lithography/manipulation execution.
11. Selecting the *Show Direction Arrows* checkbox will show the direction the path will traverse and become apparent on the image. To adjust the number of arrows on the path pattern, double-click the path. A dialogue appears which allows you to reduce or increase the number of arrows, similar to how plots are customized in Igor.

12.



The figures above show a simple set of lines drawn with MicroAngelo: one was by FreeHand, the other by Line. The two paths were grouped by clicking the 'Make Group' button, then the 'Save Group' button, followed by a dialogue asking what to name the group. The Free Air deflection (from Sum & Deflection Meter) was -0.57V, so the *Normal Set Point* was defined as -0.5V, while choosing the *Litho Set Point* as 0.5 V. The *Velocity* was 600 nm/s.

- This same approach can be applied for sample manipulation via the tip.
- If you rescale the image/ scan area to view results, you will probably have to kill the section by clicking the 'Kill All' button.

19.2.2. Using the Setpoint Wave Checkbox

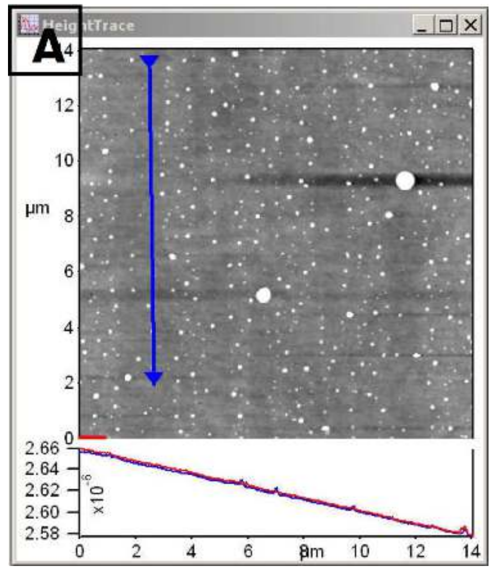
You may use the 'Setpoint Wave' checkbox in the MicroAngelo tab to systematically vary the Set Point.

1. When 'Setpoint Wave' is checked, a new dialogue appears that allows Set Point voltage ranges to be applied along an individual line/path, in either a linear or staircase
2. waveform. Note that it is not able to distinguish between objects in a group.

3.

Draw the path:

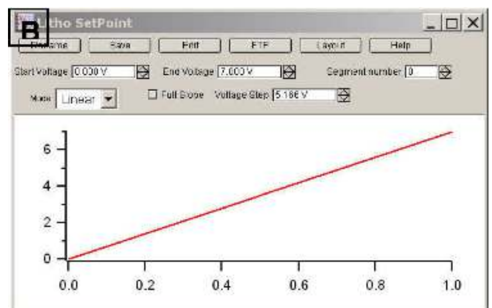
- After imaging the surface in AC mode, draw the path by clicking the 'Draw Path' button, followed by the 'Stop Draw' button.



4.

Set the point wave:

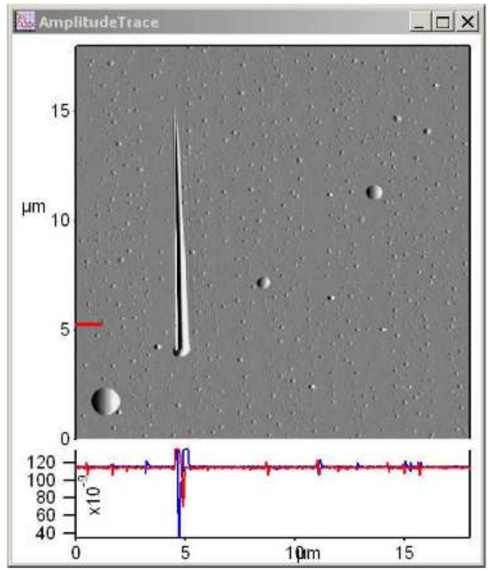
- Check the 'Set Point Wave' checkbox, which brings up the Litho SetPoint window.
- The control parameters at the top of this window allow you to define a start and end Set Point voltage, as well as define whether that ramp is stepped linearly or as a step function.
- In the example shown at right, the Set Point range was from 0 V to 7 V with a linear ramp wave. The *Voltage Step* setvar value is insignificant when using linear waveform ramps; it only applies when using step wave forms.



5.

Execute the event:

- Click the 'Do It!' button to execute the MicroAngelo event.
- The image at right shows the results of this ramping Set Point (applied force). As expected, with increasing Set Point voltage/ force, the tip plows further into the polycarbonate surface as it traverses the defined path, seen as a feature increasing in width.
- The path was defined so that the tip would experience increasing torsion as the pattern was traversed (to demonstrate what is described in the next section).



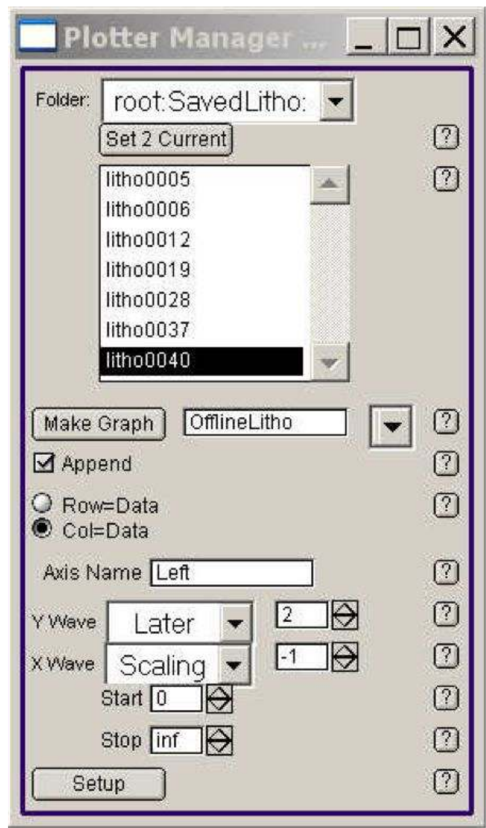
19.2.3. Save Data Checkbox

This 'Save Data' checkbox allows you to view the deflection and lateral signals during the event.

1.

Viewing the event signals:

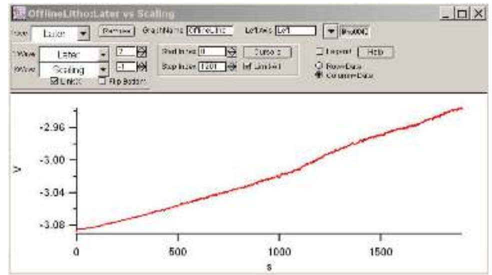
- To view the deflection, lateral, and time signals during the manipulation event:
  - *Before* executing the event (before clicking the 'Do It!' button), click the 'Save Data' checkbox.
  - *After* the event, click the 'Litho Review' button to open the Plotter Manager Panel, similar to the one seen in the example at right. This panel shows a list of litho events performed and saved in Igor's memory.
- Choose the event you want to see the saved data for during the event.
- Choose X & Y wave desired from the respective dropdown menus.



2.

- Click the 'Make Graph' button to view the data.
- A plot appears, similar to the one at right. In this case, the lateral data is shown, representing the torsion on the cantilever during the lithographic event.

Note The lateral signal during the manipulation is limited by the bandwidth of the ADC used for the lateral signal (~100 kHz). The bandwidth on the deflection signal uses the Fast ADC at 2.5 MHz.



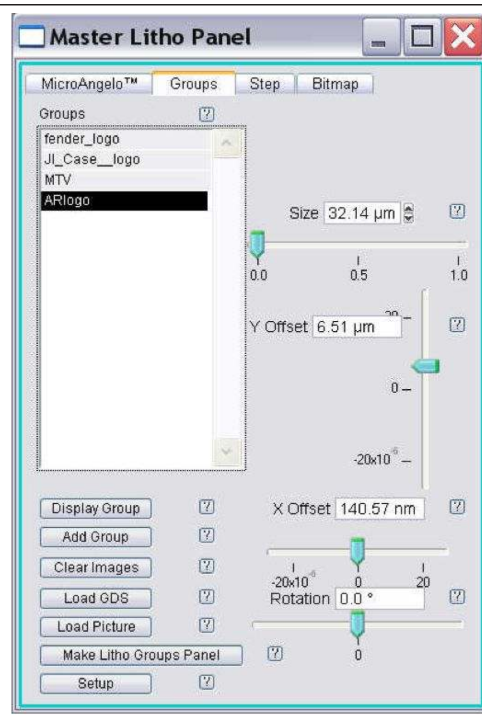
19.2.4. Importing Images as Patterns

Importing an image to use as a group in MicroAngelo is a very straightforward task. Many image files can be imported, such as tiffs, jpeg, bmp, etc., as well as GDS (graphic design system) CAD drawing files.

1.

Load the image:

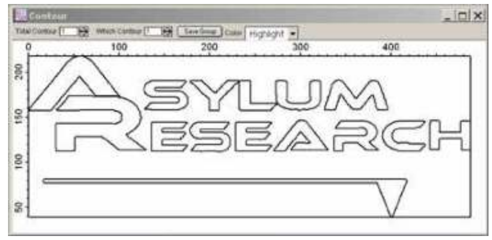
- On the Master Litho Panel, open the Groups tab.
- Click on the 'Load Picture' button (or 'Load GDS', for CAD files).
- Select the stored file that you want to use from the browse dialogue that appears.



2.

Review the contour image:

- The image loads, and Igor immediately opens a window with a contour of the loaded image. (The screenshot at right shows the results of loading the AR logo.)
- The Contour image defines the edges of the imported image as the path the tip will traverse during the manipulation.

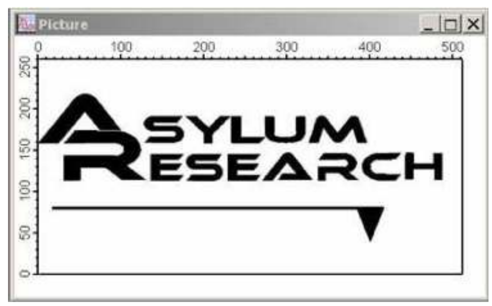


3.

Contour window parameters:

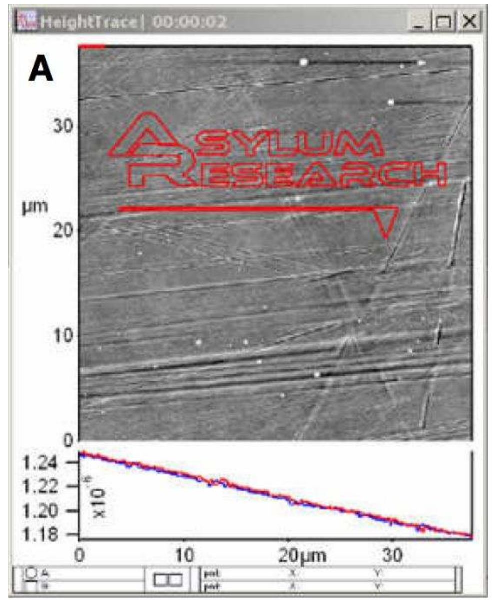
- At the top of the Contour window, notice that there are parameters you can adjust. These parameters are discussed further towards the end of this section. For now, this simple image will be contour value '1'. The parameters include:
  – *Total Contour* has something to do with edge effects; it's similar to a threshold in the mask function of the Modify panel.
  – *Which Contour* chooses which contours will be included in the group ultimately used as the path.
  – *Color* is a dropdown menu used to color the different contours.

Note Contour effects are more pronounced in imported images that are grayscale or have color. It's best to try it on your own.



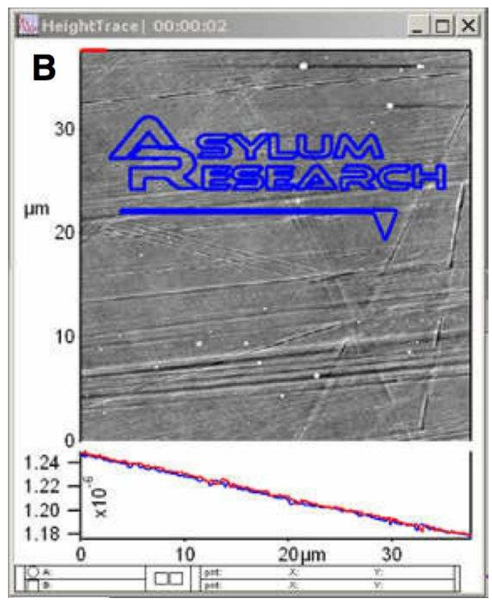
4. **Save and display the group:**

- Click the ‘Save Group’ button to add the group to the list (In this example, it is called “ARlogo”, as shown at right.)
- Highlight the group you want to use.
- Click the ‘Display Group’ button. This displays the group (colored red) in the image window, as shown at right.



5. **Position and add the group:**

- Position the group where you want it within the image area using the X and Y offset slider controls, along with rotation and size slider controls.
- Click the ‘Add Group’ button, which makes the group pattern blue in the image window, as shown at right.



6. Go back to the MicroAngelo tab.

7. Define the *Normal Set Point* and *Litho Setpoint* voltages and tip velocity.

   - Choose a *Normal Set Point* voltage value that doesn’t damage the surface between paths.
   - Choose a *Litho Setpoint* value that will induce enough force to scratch the surface.
   - Choose a tip velocity that will be at a rate sufficient enough to do what is intended.

8. If the manipulation requires an oxidative or reductive potential, click the ‘Litho Bias’ checkbox and define an appropriate bias to the tip.

9. Make sure “Contact” is selected as *Litho Mode*, if that is the mode you want to use. (Incidentally, if you change the Imaging Mode in the main tab of the Master panel, it will be updated here as well.)

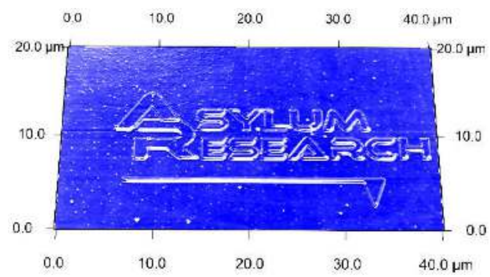
10. The ‘Show Directions Arrow’ checkbox, if selected, will show the direction the path will traverse, which becomes apparent on the image. To adjust the number of arrows on the path pattern, double-click the path, and a dialogue comes up allowing you to reduce or increase the number of arrows, similar to how plots are customized in Igor.

11. When all parameters are defined, click the ‘Do It!’ button.

12. During the manipulation, a red dot representing the location of the tip, based on the values from the X,Y LVDTs, is shown on the screen. You’ll also notice the ‘Draw Path’ button now shows what section of the group path is traversing. If you click this button during the litho procedure, it won’t do anything.

13.

- At the end of the patterning, the tip is withdrawn.
- In the pattern example shown in step 5 above, an AC 160 Si cantilever was used to scratch a polycarbonate surface. The resulting scratch can be seen in the image at right.



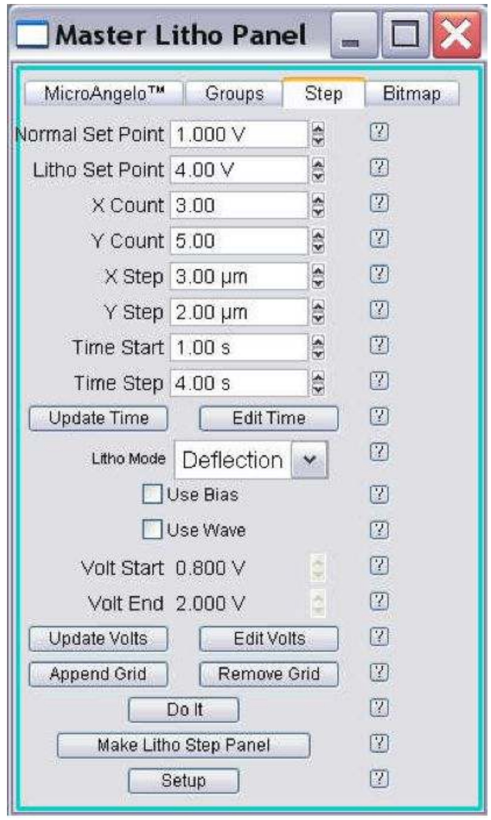
19.2.5. Creating Array Patterns: The Step Tab

Arrays can be created using the Step tab. This tab can have the tip dwell at each subsequent array point for a user-defined time or apply a different force. Both functions can be systematically increased, or custom values can be manually entered.

19.2.5.1. Creating simple arrays

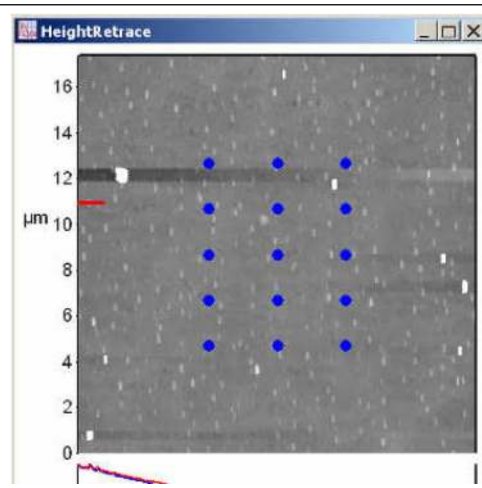
1.

- Choose the number of points you want in the array with *X Count* and *Y Count* setvar values.
- Choose the distance you want between points in the array, in both X and Y dimensions using *X Step* and *Y Step* setvar values.
- The *Time Start* setvar value is the amount of time the tip will be in contact with the surface (dwell) at each point in the array. If you want to have the tip have the same dwell at each point, enter “0 s” for the *Time Step* value.



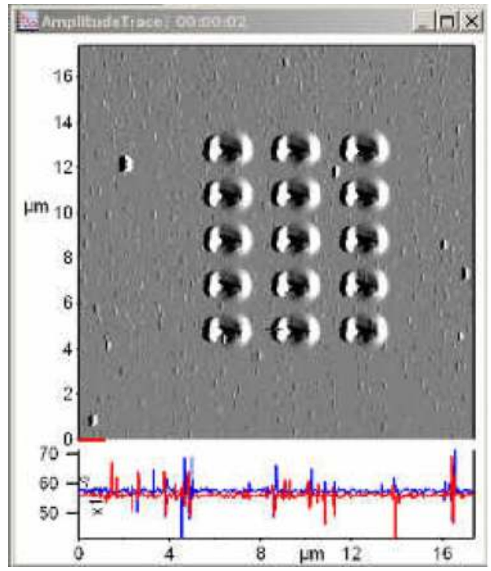
2.

- Click the ‘Do It’ button to fabricate the array pattern. Notice the Sum and Deflection Meter deflection values reaching the defined Set Point during the indents.
- Point 1 in the array starts with the lower left, moves to the right, and then moves to left array in next row.



3.

- The array will be centered in the image window. The XY offsets in the group tab don't move the array to a user-defined area in the image (like it does when using a pattern).
- In the example shown at right, a 3 x 5 array was created using the parameter values shown in Step 1. Large forces were applied using a rather dull tip, resulting in the poor-quality image below.



Varying Tip Dwell Time at each Array Point The amount of time the tip stays in contact with the surface at each array point can be independently or systematically varied using the Steps tab.

- To systematically vary the dwell time at each subsequent point in the array:
  1. Define a *Start Time* value which represents the amount of time the tip dwells at point 1.
  2. Define a *Time Step* value: this is the amount of additional time spent at each subsequent point.

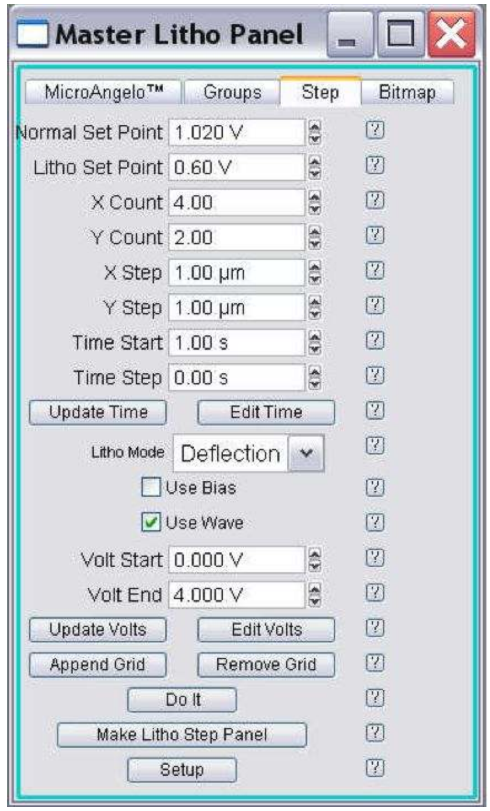
     For example, if you want the tip to spend 3s longer at each subsequent point, enter "3s" in *Time Step* (with *Time Start* being "1s"). Point 1 (lower left of the array) would spend 1s, Point 2 would spend 4s, Point 3, 7s, etc. Make sure to click the 'Update Time' button to ensure this change takes effect.
  3. The individual time values at each point can be viewed in spreadsheet form by clicking the 'Edit Time' button.
- To independently vary the dwell time at each subsequent point in the array:
  1. When a more custom variation is required, click the 'Edit Time' button.
  2. This brings up a spreadsheet where you can manually change the time at each respective point.

Varying Tip Set Point Voltage at Each Array Point The amount of applied force the tip imparts to the surface the surface at each array point can be independently or systematically varied using the Steps tab. This can be helpful when doing a series of indents at different applied forces in an array.

1. Check the 'Use Wave' checkbox. The system then disregards the *Litho Set Point* value in the MicroAngelo tab.

2. To systematically vary the applied force at each subsequent point in the array:

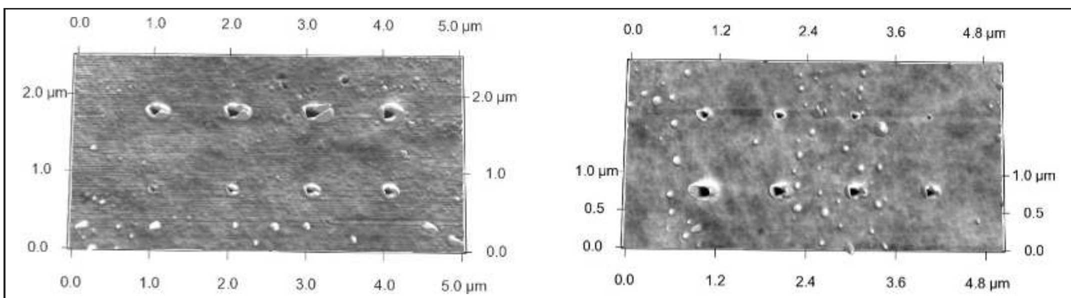
- Define a starting Set Point voltage in *Volt Start*.
- Define an end Set Point voltage in *Volt End*.
- Click the ‘Update Volts’ button to save and activate the changes.



3. The individual Set Point values at each point can be viewed in spreadsheet form by clicking the ‘Edit Volts’ button.

4. Click the ‘Do It’ button. The deflection at each array point can now be monitored in the Sum and Deflection Meter panel.

5.



In the example above, two 2 x 4 arrays were produced by varying the Set Point voltage from 0 V to 4 V (left) and varied the Setpoint from 4 V to 0V (right). In both examples, the tip dwell time was constant at each point (Time Start = 1s; Time Step = 0s).

To independently vary the applied force at each subsequent point in the array:

1. When a more custom Set Point variation is required (in this case) click the ‘Edit Volts’ button.

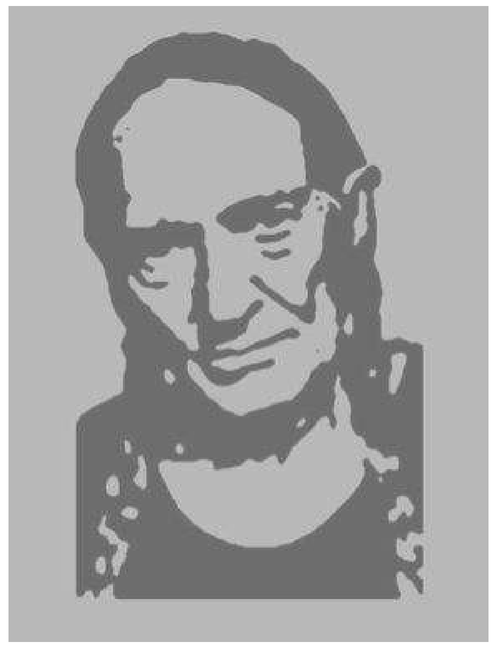
2. This brings up a spreadsheet where you can manually change the Set Point at each respective point.

19.2.6. The Velocity Tab

It is best to read the Help menu to learn more about the Velocity tab. Usually, it is used to draw images that have shading in them. An image is loaded, the colors are converted to gray scale: dark grays are patterned using the “Min velocity”, bright grays are patterned at the “Max Velocity”, and all grays in between are patterned at a velocity determined by the grayscale value and the velocity range entered into the value inputs.

This feature is good for diffusion dependent direct write scanning probe lithography techniques, among others.

(a) Velocity Tab



(b) Example of image patterned through the Velocity tab.

Part III

Spring Constant Calibration & Thermals

Part III: Who is it for? Succinct step by step instructions for the various methods of calibrating cantilever spring constants

Part Contents

20. Spring Constant Calibration

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

20.1. Introduction

Before you begin taking force curves on your sample, you will first need to determine the Spring Constant of your cantilever in order for those measurements to be quantitative, as the AFM only natively measures deflection/amplitude/force in units of Volts. There are many ways of accomplishing this; however, a few of the most popular methods are: the "New" Sader Method, the Thermal Method, and the "Old" Sader Method. We discuss all of these methods in this chapter.

The "New" Sader Method is perhaps the most accurate and convenient method at present. (See John Sader's 2012 paper titled, "Spring Constant Calibration of Atomic Force Microscope Cantilevers of Arbitrary Shape", for more details on this.) Nevertheless, the "New" Sader Method requires some factors that must be first determined experimentally for each specific probe type geometry you wish to use. At Asylum, we have already done this for many of the probes we carry in the Probe Store and that we use commonly day-to-day, and this list is always growing. Asylum has implemented aspects of this method in an automated calibration technique called 20.2™, which we cover in the next section. The extra terms for a given type of probe geometry can also be added by the user directly into the AFM software. If you are a member of the Asylum Research User Forum already, you can reach this thread here: Adding Cantilevers to GetReal. If you are not yet a member, you can join and go to the Asylum Research User Forum under the Customer Support section of the website, then search for "adding cantilevers to the GetReal™ calibration list".

If you do not wish to use the "New" Sader Method, the Thermal Method is often considered the next best method. While the "Old" Sader Method works well strictly for "ideal" high aspect ratio rectangular cantilevers, it can produce values that are as much as 100% different from the actual spring constant of the cantilever for geometries that deviate from this "ideal" case.

20.2. GetReal™ Automated Probe Calibration

GetReal™ calibrates your InvOLS and Spring Constant without ever having to touch the cantilever to the surface. This feature requires AFM software version 13 or later.

20.2.1. Video Tutorial

Consider watching this introductory video tutorial: Get Real Automated AFM Probe Calibration. (An internet connection is needed.)

20.2.2. How to Guide

In the **Thermal** tab, click the 'GetReal Probe Panel' button and select the lever you have loaded from the list. This loads several constants that describe that specific cantilever geometry. Then, click the 'GetReal Calibration' button, and the software begins to calibrate the spring constant using the plane geometry dimensions, the resonance frequency, and quality factor from the Thermal data. It will then do a reverse Thermal calibration to obtain the InvOLS, see Section 21.3 on page 297 for more details.

20.3. Thermal Method for Determining Spring Constant

Determining the Spring Constant (k) is a quick three-step procedure:

1. Correct for Virtual Deflection effects in the AFM hardware. (See Section 20.3.1 on page 283.)
2. Calibrate the relationship between cantilever deflection (measured in volts) and vertical cantilever motion. This is called InvOLS (Inverse Optical Lever Sensitivity) and is measured in nm/V. (See Section 20.3.2 on page 285.)
3. Withdraw tip and perform a thermal to determine the cantilever's resonant frequency. An algorithm computes the spring constant using the equi-partition theorem. (See Chapter 21 on page 293.)

This protocol can also be found in the Spring Constant Tutor: to view it in the software, select from the main menu bar: *AFM Controls > User Panels and Func > Spring Constant Tutor*. It contains everything you need to do to complete the k determination. (See Section 20.4 on page 291.)

Note If possible, it is often best to do the Spring Constant calibration in air, as it is easier to fit the higher Q response in air.

20.3.1. Virtual Deflection Correction

The virtual deflection is a mechanical coupling of the cantilever deflection signal with the Z-axis movement seen on MFP-3D AFMs; however, it is not present on Cypher AFMs. It is a result of the mechanical path not being quite perfect, creating a slight slope in the overall force curve. Although this may only be a few nm's over several microns of travel, if not corrected, it can skew the accuracy in the measurement and subsequent analysis of the force to be determined. The exact origin of it is still not fully understood, but it does depend on how the light is aligned on the cantilever as it travels through the Z-piezo range. By performing this step first, you will get a more precise estimate of the cantilever Spring Constant.

The virtual deflection correction essentially levels the free air part of the force-distance curve (i.e., constant deflection). This aids force-curve analysis because force measurements are relative to the baseline.

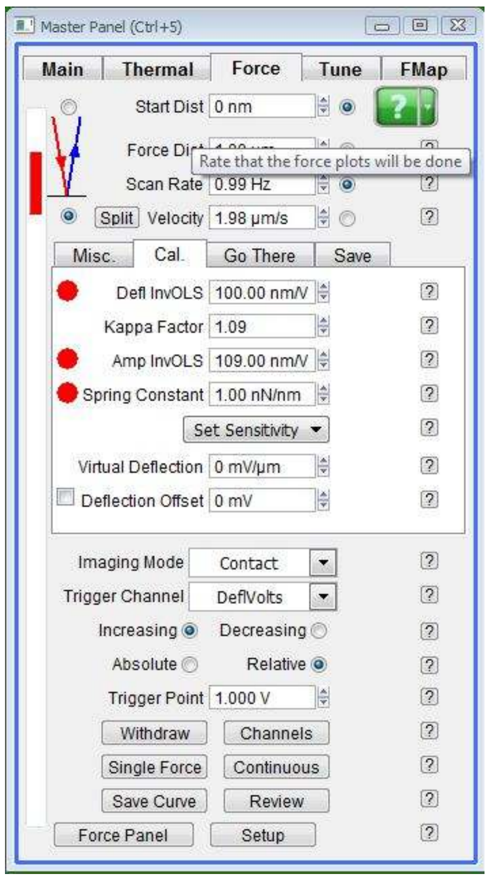
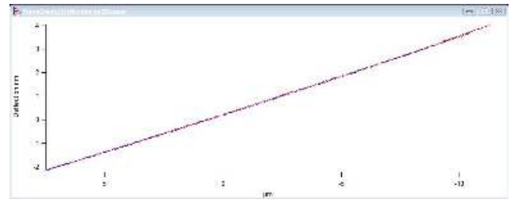


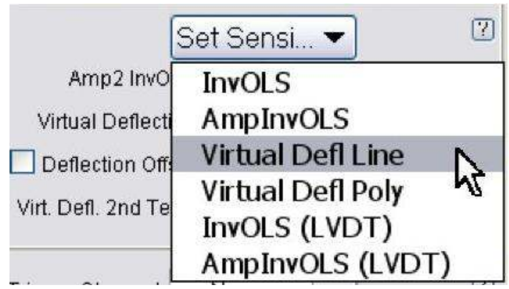
Figure 20.1.: The Master Panel > Force Tab > Calibration Tab.

1. You want the tip far from the surface (i.e., the tip will not contact the surface even when the Z-voltage is at 150 volts). This virtual deflection is a good thing to calibrate before engaging.
2. Set the *Trigger Channel* to "None".
3. Set *Start Dist* to "-inf" (or drag the red bar to the top).
4. Set the *Force Dist* to "inf" (or drag the red bar to the bottom).

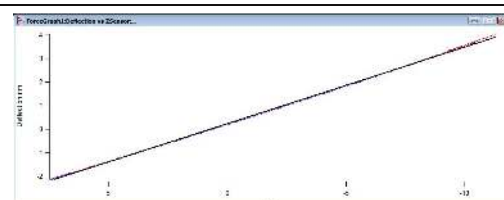
5. **Do a force plot:**



6. Right-click on the graph and select *Virtual Defl Line* (virtual deflection).
(Or, in the Cal Sub tab, click 'Set Sensitivity' and select *Virtual Deflection*.



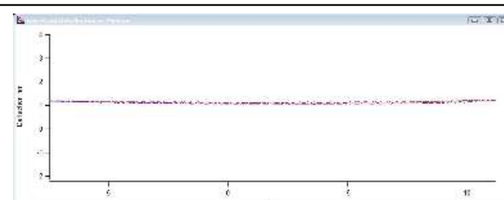
7. **Check the fit:**



8. You can fit a subregion of the force curve by using the Igor cursor (shown with Ctrl + i). Avoid doing second order poly virtual deflection on subregions of the curve, as they can greatly alter any data later collected outside of the region fit.

9. If you have an extended Z-piezo range MFP-3D Head, you may need to do a second order polynomial virtual deflection. The extra piezo stack seems to be able to move the virtual deflection in a nonlinear fashion.

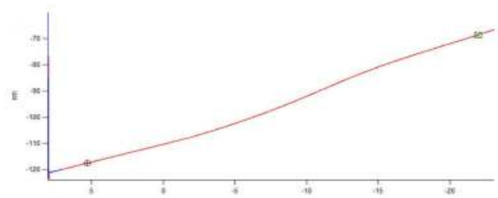
10. **Review:** The virtual deflection has now been corrected. The approach now looks level, as it should since it is not experiencing any deflection during the approach, in subsequent force curves.



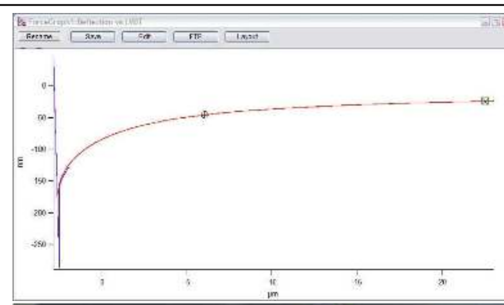
20.3.1.1. Tips and Tricks

Tip Extended Range MFP-3D Head

If you are using an MPF-3D extended Z range head (~28 µm Z range for early models and ~40 µm for current MFP-3D), the long approach of a force curve may not appear linear at all.
In this case you should select *Virtual Defl Poly* (in Step 6 on page 284) to account for the curvature of the approach curve.



Caution Is it critical that you do a full range force plot to calibrate the poly invols, as described above. Extrapolating a polynomial past the fit region can be very inaccurate.



Tip Eliminating Surface Charge Effects.

Certain flat substrates (glass, mica) have inherently charged surfaces. Combined with a soft cantilever (0.03 N/m), this can cause some odd non-linear deflections in the force curve approach.
Possible solutions:

- Choose a conductive surface such as freshly cleaved graphite (HOPG) or silicon on a conducting substrate.
- Place a static master ionizer near your sample, see Section 4.6.3.1 on page 41.

20.3.2. InvOLS

The objective of the second step is to measure the slope of the tip-sample contact region (called the inverse optical lever sensitivity or InvOLS), a parameter necessary for the spring constant algorithm. The InvOLS is not a measurement of the particular properties of the cantilever being used per se, but a measurement of the AFM sensitivity to deflection with the given laser spot location, cantilever, and photodetector combination as a whole. In the previous section, we eliminated the possible instrumental errors of this measurement that might otherwise contribute to cascading inaccuracies of the Spring Constant measurement.

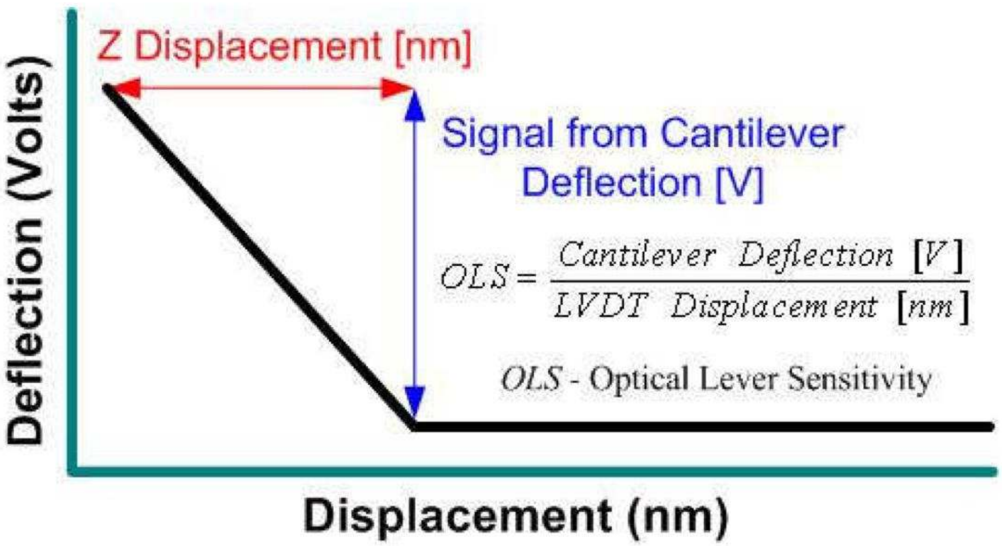


Figure 20.2.: Inverse Optical Lever Sensitivity measured from a force curve on a hard surface.

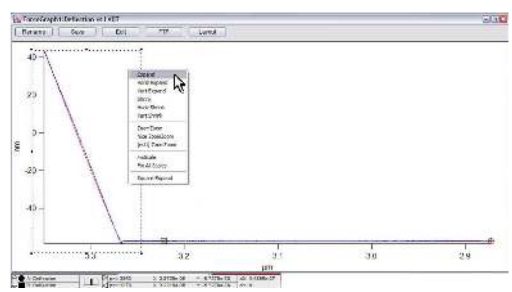
Figure 20.2 on page 286 gives a sense of what the optical lever sensitivity is, given in units of V/nm, and the software inverts it for the algorithm.

1.

Perform Force Curve:

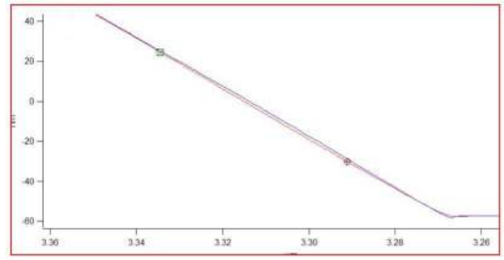
- Engage on the surface in Contact mode.
- Make sure the *Trigger Channel* is set to "DeflVolts".
- An absolute trigger of "1V" is a good place to start for most tips and samples.
- Click 'Single Force' (Ctrl +3) to do a force plot.
- Optionally click and drag a box around the sloped region of the curve, and then right-click to expand it for a better view.

Note If the virtual deflection correction is working properly, the non-contact region of the curve should be horizontal.



2. **Find Invols:**

- Right-click on the contact region at a reasonably large force and select *Calculate Invols*.
- It will show a fit to the deflection vs Z-Sensor and calculate Invols for you.



3. **[Optional] Fine Tune Cursor Positions**

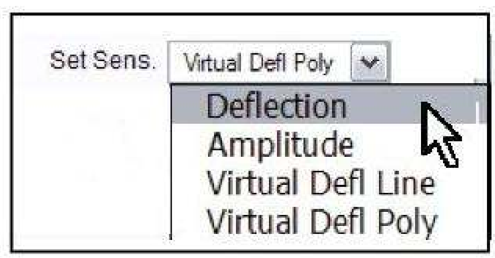
- You can move the A (circle) and B (Square) cursors after the fit to change the fit region.
- You can drag them each individually.

OR:

- Deactivate one of the cursors on the curve by going into the lower margin of that Igor window and clicking the open circle/square. (See image at right.)
- When it's deactivated, the cursor will turn black allowing you to finely position the other cursors with the arrow keys.

4. **Perform Line Fit:**

- Open the *Master Panel > Force Tab > Cal subtab*.
- Select *Defl InvOLS* from the 'Set Sensitivity' pull-down menu.
- A fit will be performed in the specified region, and the InvOLS will be updated.



20.3.3. Averaging InvOLS

The InvOLS can be slightly different from curve to curve. For more precise measurements of the InvOLS, statistical analysis can easily be performed.

1. At the bottom of the *Master Panel > Force Tab* (See Figure 20.1 on page 283), click the 'Set Up' button.

2. **[Optional]** Add the *Limit Cont* control.

- Check the *Limit Cont. to: Show?* checkbox.
- Click the 'Looks Good'
- Enter a value for how many force curves to be acquired in one spot, such as 100 curves.

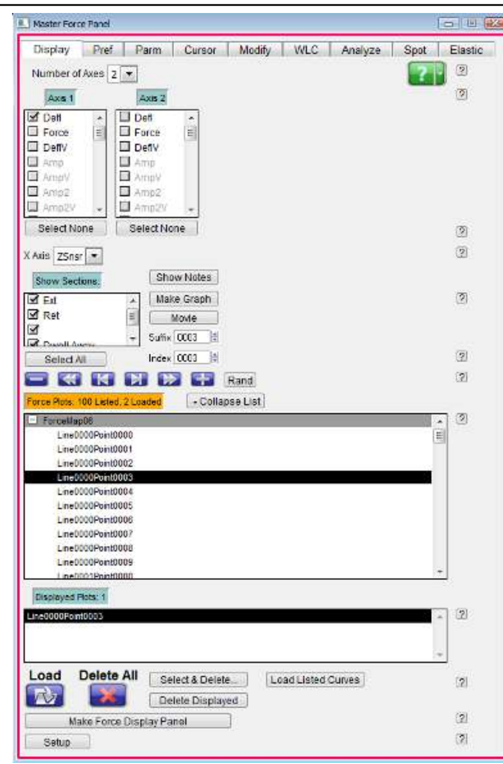
Note This allows a set number of force curves to be sequentially acquired at the same XY location without clicking the 'Single Force' button repeatedly.

3. Click the 'Continuous' button to acquire the force curves. This may take a few minutes, as each curve requires a second or two to process.

4. **Open the Master Force Panel:**

- From the main menu bar, select *AFM Analysis > Master Force Panel.*
- In this panel, select the Display Tab.
- Load the curves that you want to average the DC InvOLS on. (If you saved the curves to memory, they will already be loaded.)

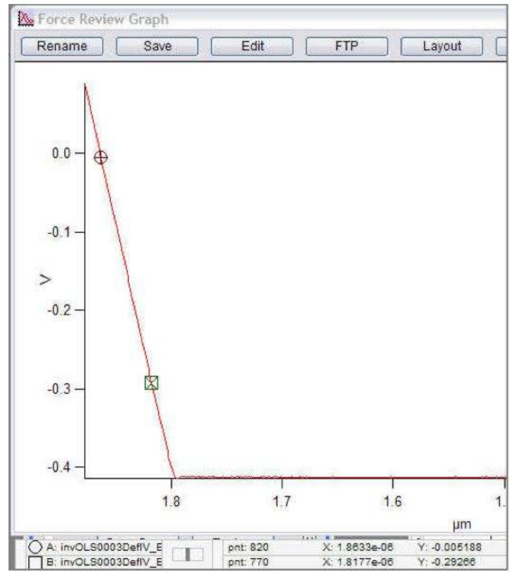
Note Notice the suffixes of these curves because they will have to be designated in the Analysis tab.



5. **Plot the force curves:**

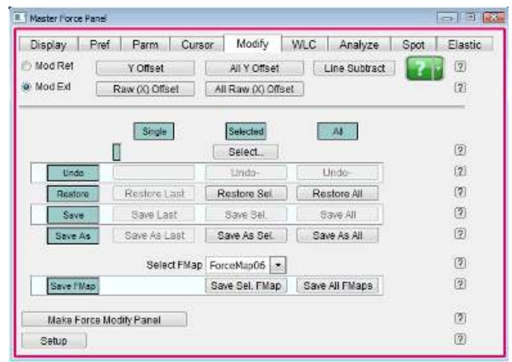
- Plot the force curves as Deflection volts versus ZSnsr.
- With the graph window selected, click Ctrl + i to show the information area on the bottom of the graph.
- Drag the cursors from this area onto the sloped part of the graph.

Note Take note of the Y-axis values. These are also used in the Analysis tab to designate the *Deflection Range* during the averaging.



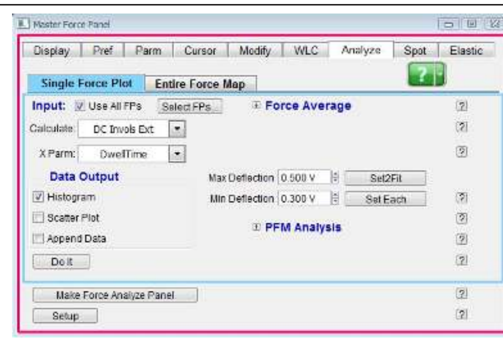
6. **Offset the data:**

- Go to Modify tab of the force review.
- Select 'Mod Ext' and click "All Y Offset".
- This offsets the data for deflection drift. The Invols analysis looks at the same voltage range, so deflection drift needs to be corrected.



7. **Prepare for Analysis:**

- Go to Analyze tab of the force review.
- On the *Calculate* dropdown, select "DC InvOLS".



8. **[Optional]** Click the 'Select FPs...' button to select which force plots you want to analyze.

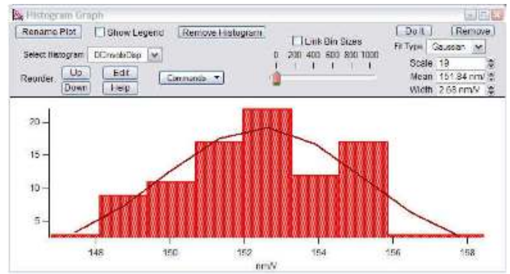
9. On the *Max Deflection* control, enter the value of the higher cursor.

10. On the *Min Deflection* control, enter the value of the lower cursor.

11. **Create the Histogram:**

- Check the *Histogram* check box.
- Click the 'Do It' button.
- A histogram should then be generated to show a distribution of InvOLS values.
- In the figure at right, a Gaussian fit was applied, although other fit types are available in the *Fit Type* dropdown.

Note The Mean and Width of the histogram fit is displayed, and that the Bin size can be changed using the slider bar.



20.3.4. Do a Thermal

Once you have your InvOLS calibrated, you can collect the thermal data needed to fit for the spring constant. Thermals are outlined in the next section Chapter 21 on page 293. Advanced note, you don't need to collect the thermal last. You can collect the thermal first; then when the Invols value is updated, the last collected thermal data is rescaled. You can then fit the rescaled thermal and obtain the correct spring constant.

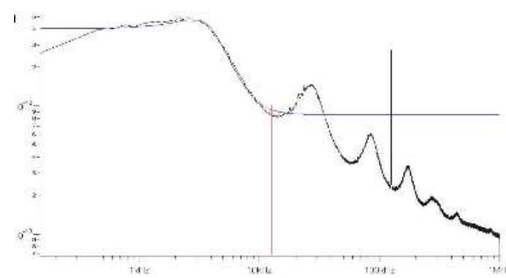
20.3.5. Tips and Tricks

Tip Thermal fitting in liquids.

Sometimes when using a soft or “floppy” cantilever in liquid, it can be difficult to determine the first resonance peak position when close to the surface. If you are fitting for the spring constant, it is probably best to do that in air when the liquid thermals are messy. But, sometimes, you can get cleaner thermals if you put some distance between the tip and sample, which can be done as follows:

- Take the frequency spectrum before engaging/taking force curve; perform a try fit once you have the InvOLS value.

  OR
- Disengage the tip and manually retract the tip a turn or two with the thumbwheel MFP3D (20 µm to 40 µm) or just move the tip up with the knob (Cypher) before taking the frequency spectrum. This seems to work much better, with a low number of iterations.



20.4. Spring Constant Tutor

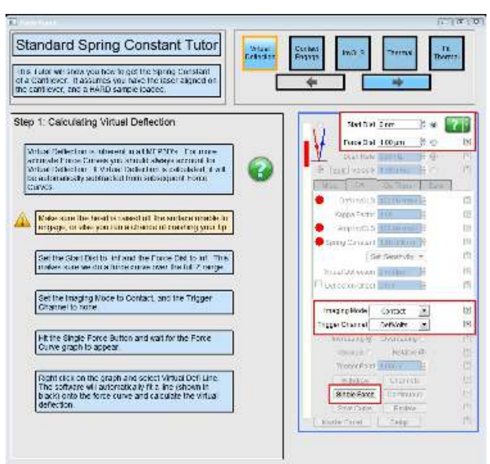


Figure 20.3.: Spring Constant Tutor

The Spring Constant Tutor allows all three steps of the Spring Constant (k) determination steps Section 20.3 on page 282 to be performed in a single software panel. This is particularly useful for beginners.

1. Go to *AFM Controls > User Panels and Funcs > Spring Constant Tutor*.
2. Steps run from left to right along the top. Each step has multiple sub steps that run from top to bottom on the left side.
3. Determine the virtual deflection from doing a full range force plot.
4. Engage the tip; notice the active Z-piezo voltage meter.
5. Determine the InvOLS using a *Trigger Channel* and *Trigger Point*. Notice the Deflection voltage is displayed because this force curve acquisition is done in Contact mode.
6. Thermal tune: First withdraw the tip, then click 'Do Thermal'.
7. Fit the fundamental resonant peak to complete the Spring Constant calibration.

20.5. "Old" Sader Method

The AFM software also has the option of using the "Old" Sader Method to determine the Spring Constant using the Igor command line. This technique uses the dimensions of the cantilever (in meters), the quality factor, and frequency (Hz) to back out a k value (N/m).

In the command line type:

```
Print kSader(w,l,Q,F)
```

Where `w` is the width, `l` is the length, `Q` is the q from the thermal tune and `f` is the frequency.

For example,

```
Print kSader (30e-6, 60e-6, 118.9, 67283)
```

results in:

```
k=0.338N/m
```

For more on Spring Constant determination, you may find the following references helpful:

1. J. E. Sader, J. W. M. Chon, and P. Mulvaney, "Calibration of rectangular atomic force microscope cantilevers," *Review of Scientific Instruments,* Vol. 70, 3967, 1999.
2. J. P. Cleveland, S. Manne, D. Bocek, P. K. Hansma., "A nondestructive method for determining the spring constant of cantilevers for scanning force microscopy," *Review of Scientific Instruments,* Vol. 64, pp. 403-405, 1993.

21. Thermals

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

Capturing a thermal spectrum consists of pulling back data at a high rate and doing a FFT to determine the spectral components. The MFP3D can run thermals up to 2 MHz and Cypher AFMs up to 32 MHz. These figures are based on sampling rates and anti-aliasing filters for the respective instruments.

Thermal Tunes are mostly used for a few specific tasks, including:

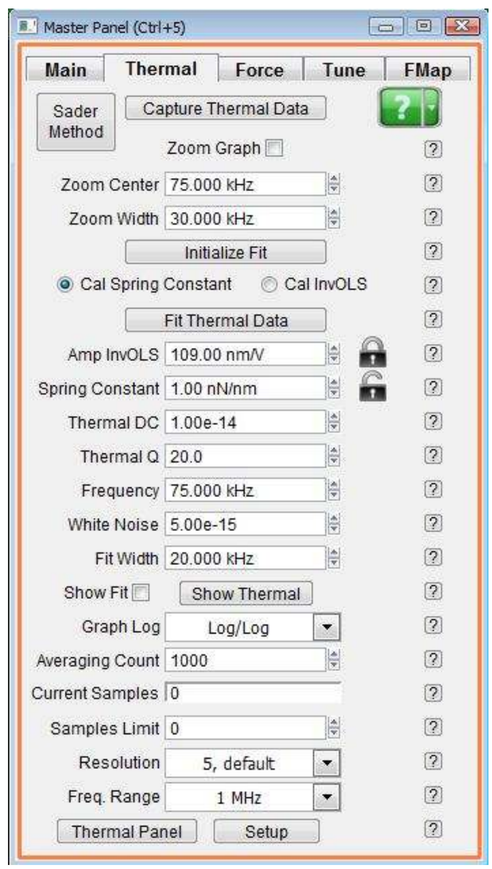
- Determining the cantilever Spring Constant OR InvOLS.
- Determining the frequency of a cantilever for drive frequency selection for AC mode in air or fluid, or higher resonance eigenmodes for DualAC imaging techniques.
- Determining resonant frequency changes if the tip has picked up material or has become broken.

21.1. Capturing the Thermal

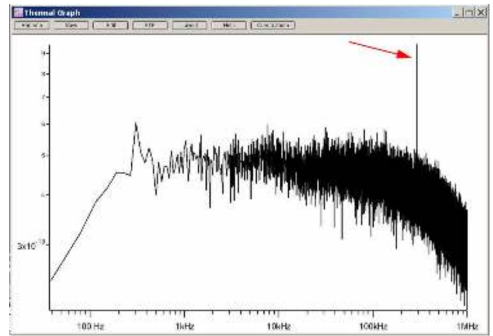
For the following procedure, we assume a probe is properly loaded, SLD aligned, and the photodiode (PD) zeroed.

To acquire a thermal tune of the cantilever:

1. Open the Thermal tab, located in the Master Panel (Ctrl + 5).



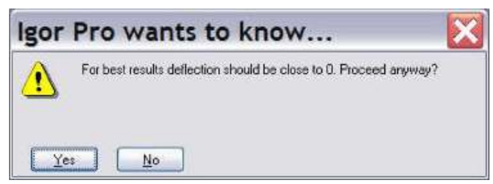
2. Click the 'Do Thermal' button (Ctrl+2). A power spectrum plot will appear, continuously averaging spectrums in real time.
In the example at right, an Olympus AC 160 Si cantilever in air was used. The higher Q of the cantilever causes the sharp peak in air. The red arrow points to the fundamental resonant frequency of cantilever.
Identifying the peak can take some practice, but the general rule of thumb is the highest lowest peak. When performing thermals in liquid, it is much more difficult to identify peaks. You should start out looking at thermals in air.



3. If the Deflection is not zeroed, a dialogue appears. To respond:

- On Cypher, there will be an option to zero the PD at that point.
- On an MFP3D (as shown at right), you can either respond 'No' and then zero the PD; or, if precision is not your top priority, click 'Yes' to continue.

Note The reason to zero the PD is to have a more linear response in their center.



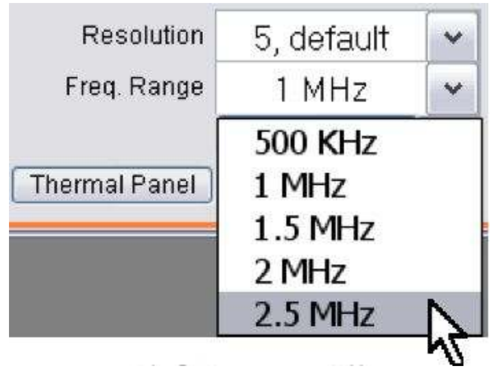
4. You can limit the number samples acquired. Generally, a couple dozen is sufficient for most tasks, unless the S/N ratio really matters. Notice the baseline is mostly noise, but the one sharp line around 300kHz is the AC160s resonant frequency (red arrow in 2).

5. Click the 'Stop' button (Ctrl+2) to terminate the collection of power spectrums.

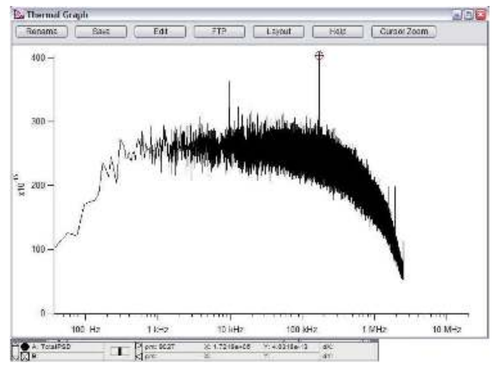
Resolution of the acquisition can be changed in the resolution dropdown menu of the Thermal tab. Larger numbers go faster (less resolution); while smaller values acquire slower but have less noise. The way the data is plotted (i.e., linear vs. logarithmic) can also be changed in the Graph Log dropdown menu.

Bandwidth of the Thermal tune can also be selected; the default parameter is 1 MHz for the MFP3D and 2 MHz for cypher. To change this to a larger or smaller value, click the Setup button in the Thermal tab, and activate the show checkbox for the *frequency range* at the bottom of the panel.

Use the Frequency Range pull-down menu to select other Thermal Power spectrum frequency ranges.



Be careful when selecting the 2.5 MHz Range on an MFP3D, as you can see all the way to Nyquist, the anti-alias filters may not have removed all of the aliasing: signals between 2 and 2.5 MHz may actually be from 2.5 to 3 MHz.



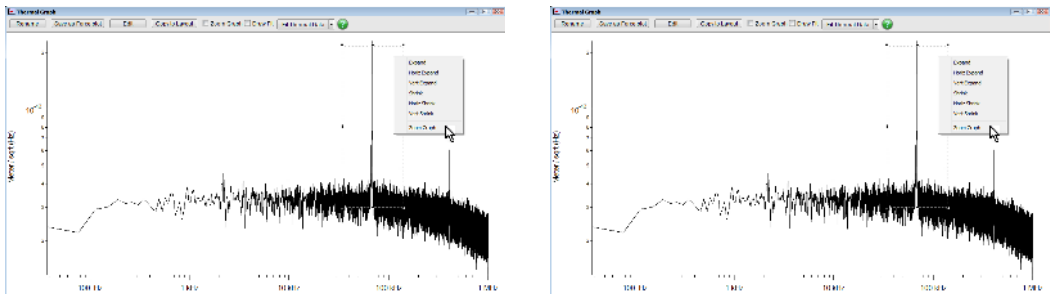
To save Thermal tune plots:

1. Click the Save as Force plot button at the top of the thermal plot. Note: This is a little strange, in that the PSD is saved as Deflection (what is really m/sqrt(Hz) is labeled as “m”), and you need to change the X-axis of the force plot to frequency to display it correctly in the force review.
2. The plot is sent to a Layout as a graphic with the copy to layout button at the top of the graph.

21.2. Thermal fitting for spring constant

1. You need to have calibrated AmpInvols before doing this stage, see Section 20.3.2 on page 285 for more detailed information.

2.



Select peak and fit thermal:

- Zoom into the area of the peak by left mouse dragging around the peak.
- Right-click the peak and select *Zoom Graph* (at the bottom). The result should look similar to the graph on the right.
- Click 'Fit Thermal Data'. The blue curve will appear.

3. The default setup is that AmpInvols is locked on the thermal panel see 1. This means when you do a fit, you are holding AmpInvols constant, and fitting for the spring constant of the lever.

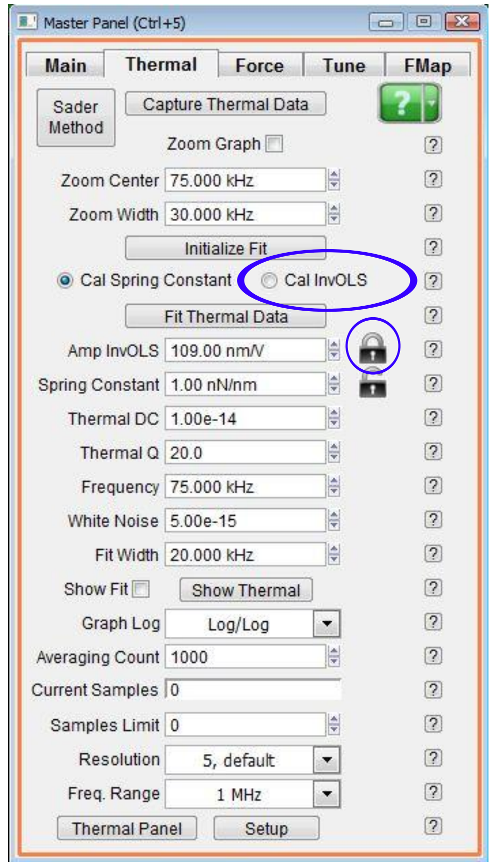
21.3. Thermal Fitting for Invols

This is mostly useful for recalibrating Invols after changing the sample or adding liquid. The basic idea is that you have calibrated the Spring Constant in air, on a clean hard surface. The Spring Constant should not change. Invols depend on numerous factors, such as temperature, spot position, liquid, etc.; if you know the Spring Constant, you can collect thermal data and discover what Invols value would give you the same Spring Constant.

General tasks for thermal fitting for Invols:

1. Calibrate Invols and/or Amp Invols in air on a clean hard substrate.
2. Collect thermal data.
3. Fit thermal data, fitting for spring constant.
4. Change sample or liquid.
5. Collect thermal data.

6. Click ‘Cal Invols’ so that Amp Invols is unlocked.



7. Click ‘Fit Thermal Data’. The Spring Constant will not change, and Amp Invols and Invols will be updated.

21.4. Thermal Tuning a Cantilever

Thermals work very well for determining the resonant frequencies, and ultimately drive frequencies, of cantilevers in fluid.

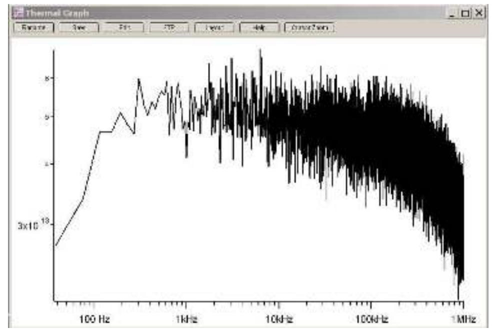
After you have fit the thermal peak, you can right-click it and select Move Freq, Phase, and Amp to Tune. This will set the *Drive Amplitude*, *Drive Frequency*, and *Phase Offset* for the cantilever drive based on the thermal fit.

You can do this operation from the right-click menu, from the ‘Fit Thermal Data’ button dropdown menu, or from the ‘Center Phase’ button dropdown menu located on the Tune panel.

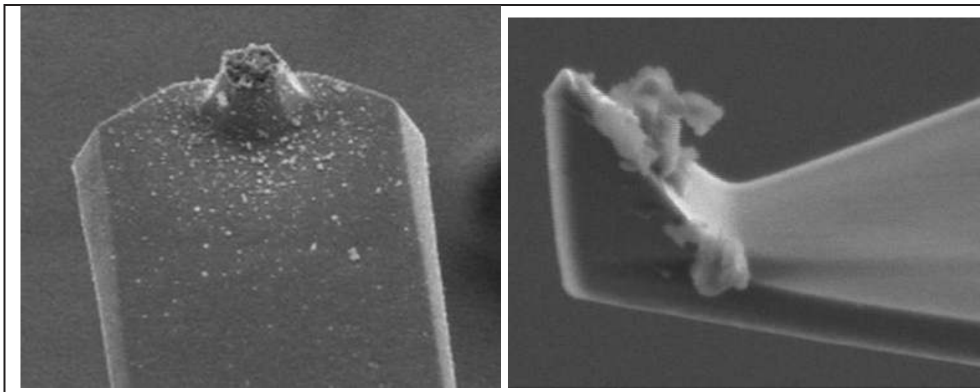
21.5. Thermals for Tip Checks

The thermal is also a very convenient way to check whether the light source spot is actually on the cantilever. When aligning spot via IR card, sometimes a nice sum is given when the spot is on the probe substrate. Thermals are a good way to double-check that the spot is on the lever.

1. The image at right is an example of a thermal tune when the SLD was on the back of the probe chip. A reasonable Sum voltage was displayed in the Sum and Deflection Meter panel.



2.



The images above show two cases in which a thermal is useful at determining what happened when the resonant frequency changes throughout an imaging experiment: If the tip breaks (lowering mass, increasing f_o), or if there is an increase in mass on the cantilever/tip from adhered debris (lowering f_o).

Part IV

Supplemental Information

Part IV: Who is it for? Extra applications tidbits you may find interesting along the way.

Part Contents

22. SPM Basics

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

Even though this user guide focuses on specific SPM applications techniques, it is still essential and of interest to understand the working concepts behind the **Scanning Probe Microscope** (SPM) to truly master it. A strong basic grasp of SPM will help you discern what data the software is displaying and what parts of the instrument the software is controlling.

This chapter covers very generic ideas on SPM. Each following chapter in this part of the user guide elaborates as necessary.

22.1. Basic Principles

The goal of basic scanning probe topographical microscopy is to take a very sharp tip and skim it over the surface of a sample, while keeping a log of the XYZ positions of where the tip has been. Usually a “flight pattern” is chosen so that an area of interest is covered in a regular fashion, in the same way a farmer might plow a field. Finally, a 3D plot of the XYZ log entries gives a nice topographical map of the surface.

A large-scale example is to create a map of the earth by flying a plane across its surface. With a radar altimeter, the pilot receives information to keep the plane at a constant distance above the terrain, while the GPS system continually logs location.

In both cases, the basic requirements to make the system work are (remember: tip = airplane and sample = earth):

- A way to move the tip in three dimensions.
- A way to measure XYZ position of the tip.
- A way to measure distance between tip and sample.

- A control system to drive the tip in the XY direction in a pattern to give good sample coverage.
- A control system to drive the tip in the Z direction in such a way as to keep the tip-sample distance constant.

In the case of the plane, the requirements are met by the plane's engines, flight controls, a GPS (Global Positioning System) receiver, a Radar altimeter, and the (auto) pilot. Say the pilot chooses to keep the plane 1000 meters above the earth while flying. This is known as the "set-point". An altimeter reading of 1005 meters indicates the earth is starting to fall away below the plane, and the pilot must correct it by descending to track the landscape. If the altimeter starts to read below 1000m, the pilot must ascend. The rules are so simple that an electronic circuit (autopilot) can be designed to climb the plane when the altimeter reads below the setpoint and to descend the plane when the altimeter reads above the setpoint. This is a very basic example of "feedback control".

22.2. The Scanning Tunneling Microscope (STM): A Simple SPM.

As an example of the SPM, we'll start with the Scanning Tunneling Microscope (STM), made famous for its Nobel Prize winning images of atoms on a surface. It is also the first historical example of a fully functioning SPM.

Here the five required items are:

Tip motion Given the relatively small distances over which the tip moves (usually microns down to angstroms), the preferred mechanical actuator is made of piezoelectric materials that expand with the application of voltage. 10s to 100s of volts usually cover that range nicely for a variety of commercially available piezo actuators. One controlled voltage source and piezo actuator per axis of motion does the trick.

Tip XYZ position measurement For very small distances of interest for STMs (usually 10s to 100s of atoms), the relationship between voltage applied to a piezo and the distance moves is very linear and reproducible. After calibrating once, applied voltage = position.

Tip-sample separation measurement The STM sample must be conducting. It has a voltage applied to it, and the tip is attached to a current measuring circuit which holds the tip at ground. The (tunneling) current between tip and sample is an exponential function of tip-sample separation and can be calibrated if necessary. Tunneling current value is directly related to a height above the sample surface.

XY tip motion control Computer-controlled voltage supplies drive the XY piezo positioners.

Tip-sample separation control Choose a current level, associated with a unique height above the sample surface, as the setpoint. The difference between that setpoint and the measured tunneling current is used to control the Z piezo positioner so that tip-sample separation remains constant during the XY tip scanning process.

While STM is older than AFM, the technique still has many uses.

22.3. The Atomic Force Microscope (AFM).

Where does the name "Atomic Force Microscope" come from? It is a force microscope, as it measures force between tip and sample, opposed to tunneling current with an STM. It is not required that the sample be conducting, one of many reasons why AFMs are much more commonplace today than dedicated STMs. An AFM is atomic, as it is capable of resolving atoms.

The list of basic requirements includes:

Tip motion Same as STM, though the range is usually larger, typically from 30-100 microns laterally and 5-50 microns vertically. Ultimate resolution is typically on the order of nanometers to fractions of an Angstrom.

Tip XYZ position measurement Due to the larger range, nonlinearities and hysteresis of piezo actuators no longer allow position to be derived from applied voltage. Linear, or at least non-hysteretic, sensors (such as the LVDT sensors in the Asylum Research SPM products) are required for proper position measurement. For flat samples, where Z motion is small, it can be acceptable to assume that Z piezo voltage is proportional to tip position. For rough samples, a linear sensor is necessary.

Tip-sample force measurement A sharp tip at the end of a micro-machined flexible lever (cantilever) is used to measure forces. The deflection of the lever is proportional to the force applied to the tip. A beam of light reflected from the back of the lever is typically used to measure the deflection.

XY tip motion control Same as STM, computer-controlled voltages driving piezoelectric actuators.

Tip-sample separation control Choose a fixed cantilever deflection, associated with a unique force between tip and sample, as the setpoint. The difference between that setpoint and the measured cantilever deflection is used to control the Z piezo positioner so that deflection remains constant during the XY tip scanning process.

The method just described is called “Contact Mode AFM”. Though not as popular as it once was, it is still a mainstay of AFM imaging. Please see Chapter 1 on page 6 for further discussion and how to peform Contact Mode imaging with the AR SPM software.

If Contact Mode imaging is not so popular anymore, then what has replaced it? The simple answer is probably AC mode imaging, where the cantilever vibrates (driven at or near resonance) as it moves over the surface. The net deflection remains zero, but the oscillation amplitude is affected by the presence of the sample. Hence, oscillation amplitude is chosen as the feedback parameter. (More information on this can be found in Chapter 5 on page 46.) There are at least a dozen commonly used imaging modes beside Contact and AC. These all get their own chapter in the user guide. Each chapter starts off with a little theory and then describes how it is put into practice in the AR SPM software. In some cases, special accessories are required, for which you can consult the user guide of your particular AFM instrument.

22.4. AFM Mechanics

22.4.1. About the Cantilever

AFM cantilevers come in many shapes and flavors with variations in size, tips, and spring constants. (See the Probe Store for an assortment.) Figure 22.1 on page 306 gives a typical example of a Silicon cantilever.

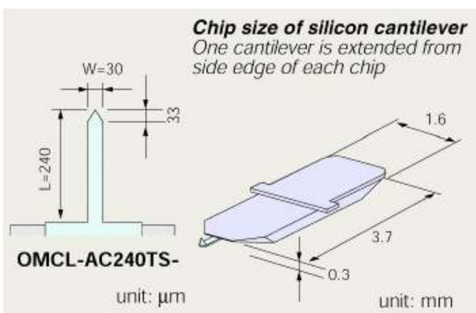
The three main components include:

Chip or handle The large piece of Silicon to which the cantilever itself is attached. Typically, about 3mm long and 1.5mm wide and about 0.5mm thick. Just large enough to grab it with tweezers and clamp it down in the AFM.

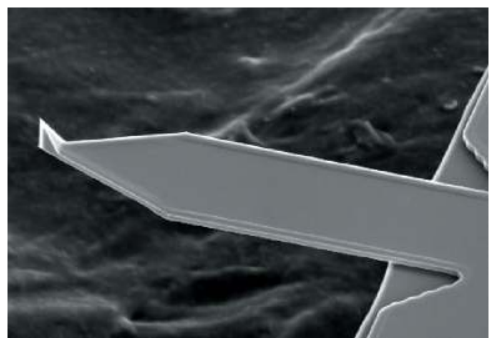
Lever or Cantilever The "diving board" protruding from the end of the chip.

Tip The pointy bit at the end which actually comes into contact with, or nearly in some cases, the sample.

Confusion in Naming	Note that different people will refer to the entire cantilever/chip object in different ways. Some will call the whole thing the tip. Some will call the whole thing the cantilever. In this document, we do our best to call the entire object the cantilever, with the sub-parts as the chip, lever, and tip. Context will usually make it clear if the word cantilever refers to the whole object or the lever itself.



(a) AC240 cantilever and chip dimensions



(b) AC160 SEM micrograph

Figure 22.1.: Cantilever Examples from Olympus

22.4.2. About the Lever Deflection Detector

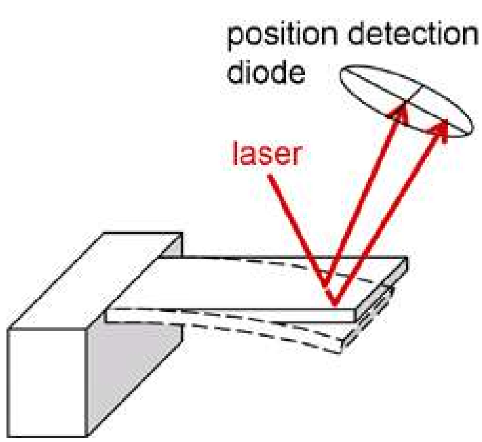


Figure 22.2.: Cantilever optical detection. The optical lever.

Detecting the cantilever deflection is typically done by shining a focused laser beam onto the back of the lever. Any bending of the lever will cause the reflected beam to move. This is illustrated diagrammatically in Figure 22.2 on page 306.

This detection mechanism is called an **optical lever**. Note that the word lever here does not refer to the cantilever, but the reflected beam which has a leveraging effect in that, for small angles of beam rotation, the longer the reflected beam segment, the more movement is incurred by the projected spot.

The detector itself is typically a **split photodiode** or **quadrant detector**.

Four individual photodiodes (basically a "solar cell" which outputs a voltage proportional to the amount of light hitting it) laid out as four square or rectangular quadrants with the beam aligned dead center have the following properties:

Sum The sum of the electrical signal generated by all four quadrants gives you the beam intensity. This tells you something about how much light is being reflected from the lever.

Difference The difference of two adjacent quadrants and two opposite quadrants tells you how far the beam spot has travelled off-center. A zero difference is a well-centered beam, assuming the beam spot is symmetric. Depending on which parts are differenced, it is possible to detect cantilever deflection and also cantilever torsion. The latter is useful for Lateral Force Microscopy or LFM (see 3).

Nomenclature	In this section, we talked about a **laser**. In actuality, this collimated light source is not necessarily a laser but can also be a Super Luminescent Diode, or **SLD**. This is a "laser-like" device with a shorter coherence length, which is advantageous in suppressing unwanted optical interference effects. We typically refer to this optical lever light source as "laser". The words "light source" could be used but may be confusing as there is also a plain white light source for illuminating the video image of the cantilever used for aligning sample and laser.

22.4.3. XYZ Positioning Mechanics

Motion in SPMs is typically accomplished by pieces of piezoelectric material that expand when voltage is applied. In Asylum Research AFMs, these so-called *piezoelectric actuators*, or simply "piezos", are used to push between a frame and a metal element that is attached with strong springs, usually thin bridges of solid metal called flexures. In parallel with each piezo is a linear sensor used to measure position accurately. Figure 22.3 on page 308 shows a diagram of such a device with a 1:1 action. By incorporating mechanical levers in the design, the motion of the stage can be larger than the motion of the piezo. In the Asylum Research MPF-3D AFM, the XY motion makes use of levers, while the Z motion does not.

Three such devices are required to achieve XYZ motion. Thus far we have spoken of the XYZ motion of the tip, but technically one speaks of **relative motion** between tip and sample. For instance, the MPF-3D AFM moves the sample in X and Y (laterally), but the tip in Z (vertically). The Cypher AFM moves holds the tip still but moves the sample in XYZ.

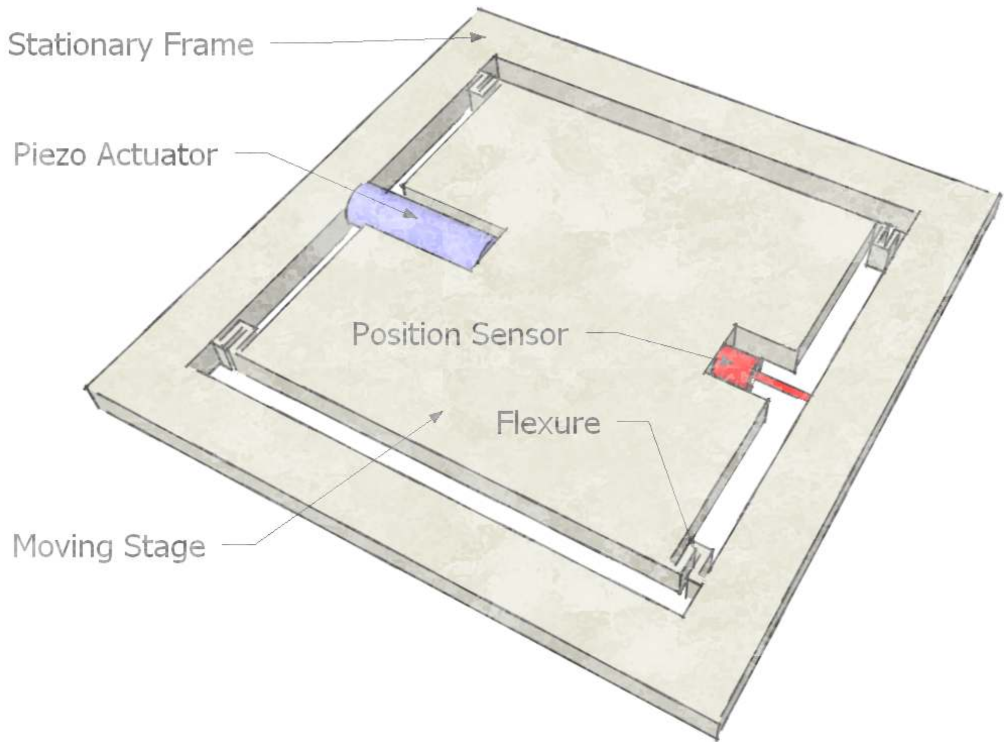


Figure 22.3.: Diagram of a flexure-based nano-positioning stage with position sensor. The frame, flexures, and stage are all cut from a single piece of metal.

22.4.4. A Basic AFM Diagram

Let put the pieces of the last two sections together in a rudimentary diagram of an AFM. 22.4 shows a sample mounted on a Z positioning stage that is, in turn, mounted on an XY positioning stage. Position sensors inside the positioning stages are not shown.

A collimated laser beam is focused on the cantilever, and the reflected beam is directed toward the split photodetector.

Not shown are:

- The rigid mechanical frame that holds the cantilever, laser, detector, and XY positioning stage fixed together.
- The mechanism that allows the cantilever to approach and retract from the sample.
- The laser aiming mechanism for centering the beam on the back of the cantilever.
- The reflected beam aiming mechanism for centering the reflected beam on the photodetector.
- A half-silvered mirror that allows for an optical (video) view down onto the sample and cantilever for identifying coarse sample features. This video view also helps when centering the laser spot on the back of the cantilever.
- Many electrical connections to the sensors and actuators.

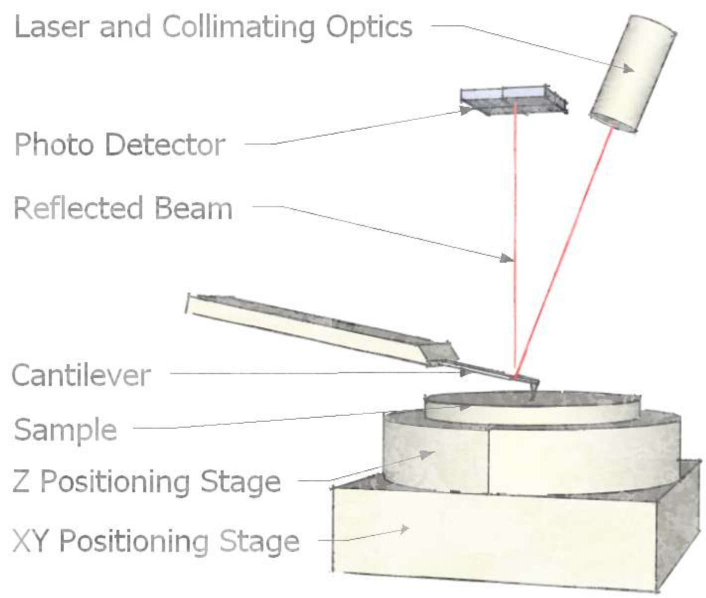


Figure 22.4.: A basic sample scanning AFM, such as the Asylum Research Cypher AFM. (A rigid frame to which the cantilever, detector, laser, and XY stage are attached is not shown.)

22.5. AFM Electronics

As you may have guessed, the voltages for driving the piezo actuators, the output voltages from the position sensors, and the photodetector need to be manipulated in real time in order for the instrument to work. Since desktop computers tend to too often be interrupted by things like slow hard drives and multiple programs vying for system resources, it is typical to attach a dedicated electronic controller directly to the AFM. It supplies the high voltages required to drive the piezo actuators and conditions the signals from the various sensors. In the case of the Asylum Research SPM controllers, the signals are digitized at a very early stage and most feedback and control operations required for XY sample scanning and Z position feedback between tip and sample are performed by reprogrammable real time computer processors.

The Asylum Research controller is extremely flexible in that it has a minimum of dedicated analog electronics, a crosspoint switch for routing nearly all input and output channels to all DACs and ADCs as required by a particular imaging technique. The digital processors can acquire new firmware on the fly for the digital filtering, measurement, and feedback controls required by a particular imaging scheme. It's not uncommon that five years after you buy your Asylum AFM, a routine software upgrade will unleash entirely new imaging modes.

In the chapters on various specific SPM imaging techniques, Figure 22.4 on page 309 will be redrawn with the necessary electronic connection and circuit elements required for the imaging technique.

23. Controlling the XY Scanning Motion

CHAPTER REV. 2437, DATED 09/04/2021, 17:03. USER GUIDE REV. 2437, DATED 09/04/2021, 17:03.

Chapter Contents

Read this chapter only if you are particularly interested in the minute details of how the SPM software specifically controls the XY-axes during scanning of samples.

Chapter 22 on page 303 discussed the notion of scanning a probe across a surface and reconstructing a topographical map of that surface. The scanning axes are divided into the “fast” scan axis and “slow” scan axis. In the aforementioned analogy of the airplane flying over the earth, the vehicle flies in the “fast” direction and, after every turnaround, slowly progresses sideways in the “slow” direction.

These are different than the X and Y axes of the scanner, in that a **Scan Angle** is applied to the fast and slow scans to determine what the drive is for the two axes of the scanner. However, in the display of the data on your computer screen, we display the fast scan direction as horizontal and the slow scan direction as vertical. For this section, we consider only 0° and 180° scan angles so that the fast scan direction is the same as the physical X-axis of the scanner, and the slow scan direction is the physical Y-axis of the scanner. Be aware that for 90° and 270° scan angles, the slow axis is the X-axis of the scanner, and the fast axis is the Y-axis of the scanner. Please refer to the hardware manual for your scanner as to which direction is X or Y.

Note	The MFP-3D scanner has a “nested” design. This means theY axis has to move the entire mass of the X axis as well as its own. The default motion at 0° scanning is fast for X (the less massive, nested part) and slow for Y (the more massive part). For line scan rates under 4Hz on the MFP-3D, there is not much noticeable effect due to the added mass in 90° scanning. The Cypher scanner is completely symmetric in X and Y and there is no mass advantage to scanning at 0° or 90°.

23.1. Fast Scan Direction

Figure 23.1 on page 311 shows the voltage applied to fast scan piezo as a function of time. This is a typical 1Hz scan, where the X position increases for the first half second (we call this “trace”) and then decreases back to the starting point for the second half ("retrace"). This fast scan axis is the X-axis of the scanner when scanning at 0° (or 180°), or the Y axis for 90° (or 270°). Regardless of scan direction, this motion is always the horizontal axis in the image displayed on the computer. The motion is repeated for every line in the image. For a 512-line image, imagine a sawtooth with 512 peaks.

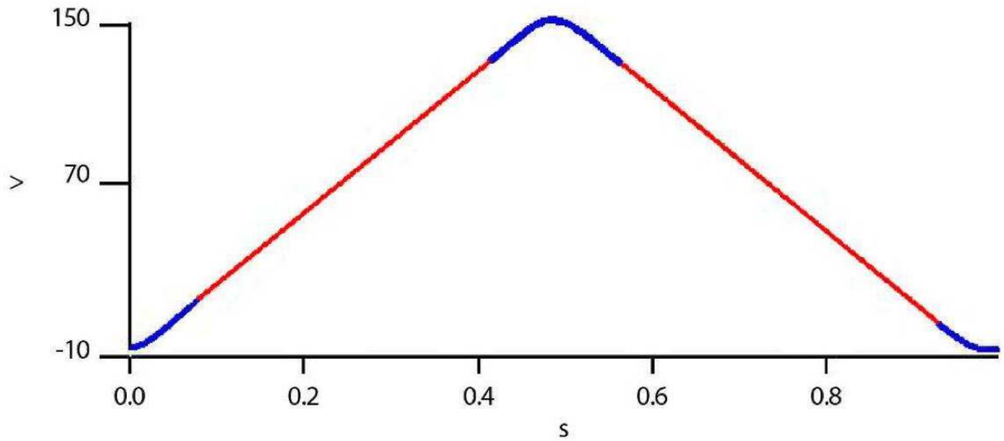


Figure 23.1.: Fast Axis X Piezo Drive (at 0°scan angle). Y-axis of graph is piezo voltage; X-axis is time. The rounded caps (blue regions) are called the "turn-around", where the fast axis changes direction. Data from the turn-around is not typically saved, though it is possible to access it for special applications.

23.2. Slow Scan Direction

The slow direction is the Y-axis of the scanner when scanning at 0° (or 180°). The scanner advances in the slow scan direction to access each successive scan line. Every time the fast scan direction completes a line, the slow scan advances its position. Slow scan comes in two flavors: Raster and Ortho.

Note	As of version 090909 and forward of the AR SPM software, Ortho scanning is now implemented. In the current version of the AR SPM software, both raster and ortho scanning are enabled. When imaging with less than 800 lines, Ortho scanning is used. For scans larger than 800 lines, Raster scanning is used, as Ortho scanning would require too much data storage in the Controller. This applies to both Cypher and MFP-3D AFMs.

23.2.1. Ortho Scanning

In Ortho scanning, the slow axis only moves during the turn-around of the fast axis, where data is not being recorded. Then, while the data is being collected, and where the fast scan is changing linearly, the slow axis is held constant. The straight "flight paths" of the tip are parallel and the turn-arounds are nicely rounded to minimize transient vibrations caused by scanner turnaround.

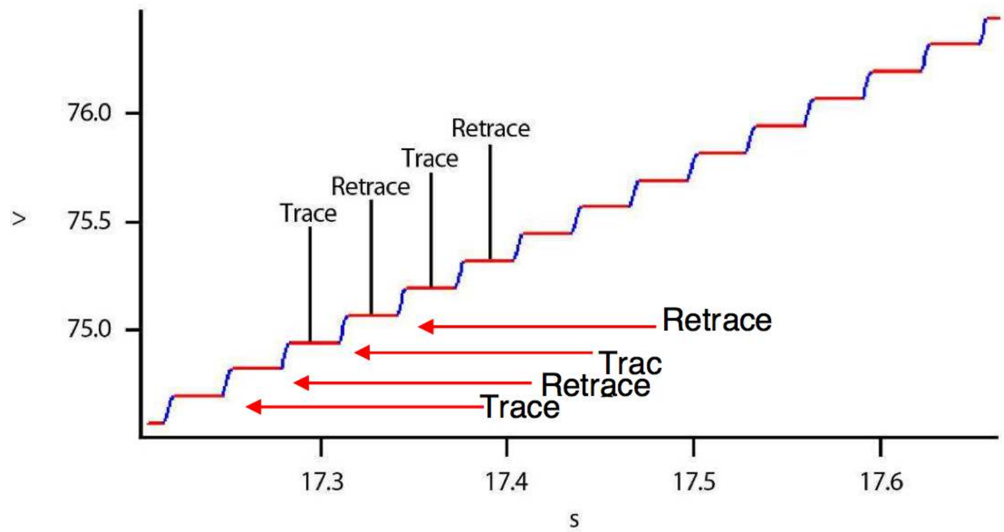


Figure 23.2.: Slow Axis of Ortho Scan. In the above image, the vertical axis is Y piezo voltage (scan angle 0°); horizontal axis is time. Axis limits are zoomed in to s how the same region as in preceding Figure (Slow Axis of Raster Scan). The blue sections denote turn-around regions. Approximately seven scan lines are shown here, each comprising two steps (one for Trace and one for Retrace).

Note	In Ortho scanning, the tip does not pass over the same point on the sample in Trace and Retrace. Instead, the Trace and Retrace images are shifted by one-half of a pixel in the Slow Scan direction. This is usually negligible but may be noticed on samples with steep features and images with a small number of lines.

23.2.2. Raster Scanning

In a Raster scan, the slow axis is simply changing linearly with time (Figure 23.4 on page 314) so that, even as the tip is moving along the fast axis, it is moving in the slow axis. The "flight paths" are not parallel, as is best seen by comparing Figure 23.3 on page 313 and Figure 23.5 on page 314.

In the early days, when AFMs were controlled by analog electronics, raster scanning was typical because it was easy to implement. There is an issue with raster scanning with the way the images are displayed. The positions of the pixels in the image do not exactly match the position of the tip where that pixel was collected on the surface, except for the middle column of pixels. This is because the rows of image lines are displayed as if they were all at the same slow axis position, but in reality, that position is changing as the fast scan is being collected (see Figure 23.5 on page 314). This effect is at most 2 * ScanSize/ScanLines, which is generally small enough to ignore. However, there are certain situations in which this effect becomes important, such as drift rate calculations. This effect also causes a slight rotation between Trace and Retrace images; in Figure 23.5 on page 314. Note that the set of scan lines for Trace is at a different angle than that for Retrace.

Even with digital controllers, open loop scanners cannot properly execute Ortho scanning due to piezo actuator creep, so raster scanning continues to this day. All Asylum Research AFMs have

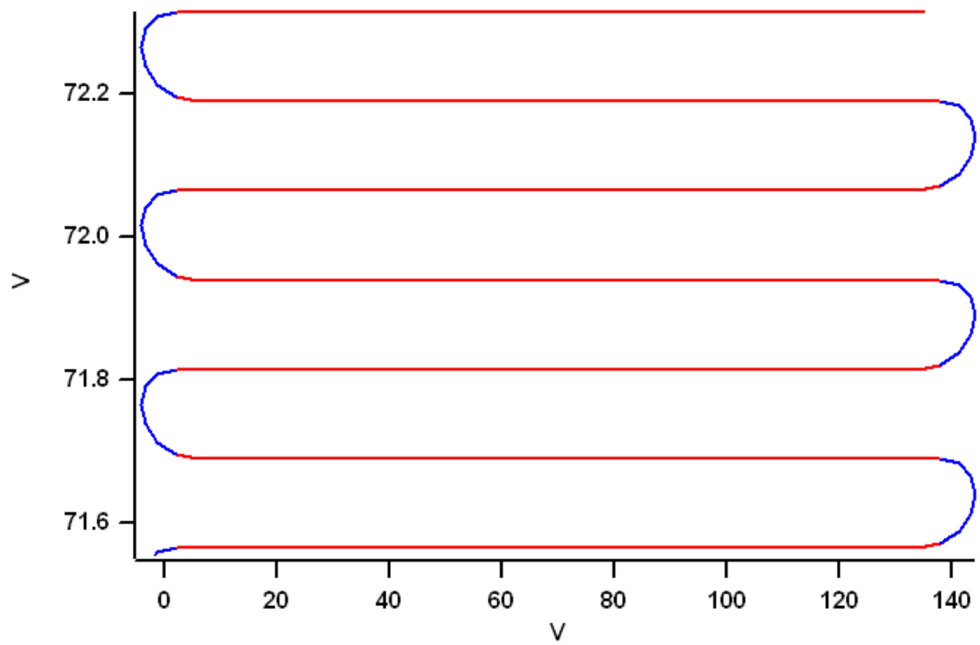


Figure 23.3.: Motion of the Tip Across Surface in Ortho Imaging. The X-axis is the fast axis drive, while the Y-axis is the slow axis drive. Three scan lines are shown. The blue sections denote turn-around regions.

closed-loop scanners and digital controllers and use ortho scanning almost exclusively. This mode eliminates the pixel position problems discussed (above) in raster scanning.

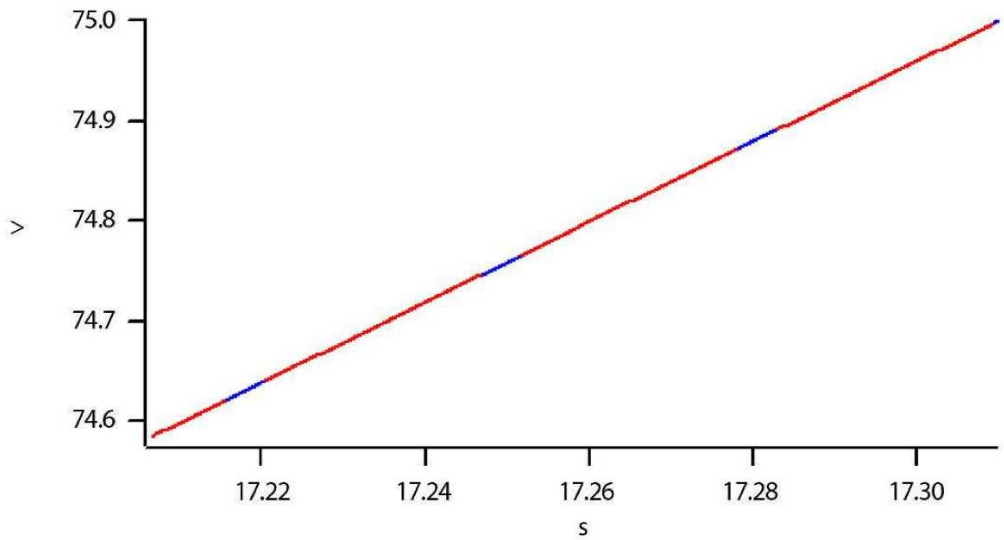


Figure 23.4.: Slow Axis of Raster Scan. Y-axis is Y piezo voltage (scan angle 0°); X- axis is time in image above. Axis limits are zoomed in to show the same region as in Figure. The blue sections denote turn-around regions.

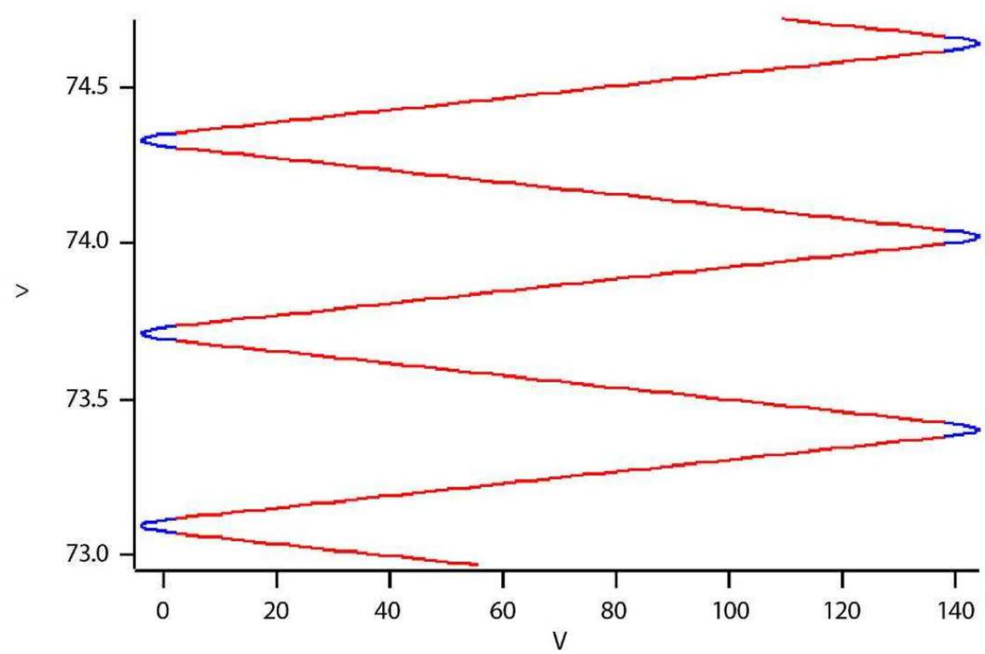


Figure 23.5.: X-Y Motion of the Tip across surface in raster imaging. The X-axis is the fast axis drive, while the Y-axis is the slow axis drive. Less than three scan lines are shown to better illustrate the concept. The blue sections denote turn-around regions.

Part V

Bibliography, Glossary, and Index

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