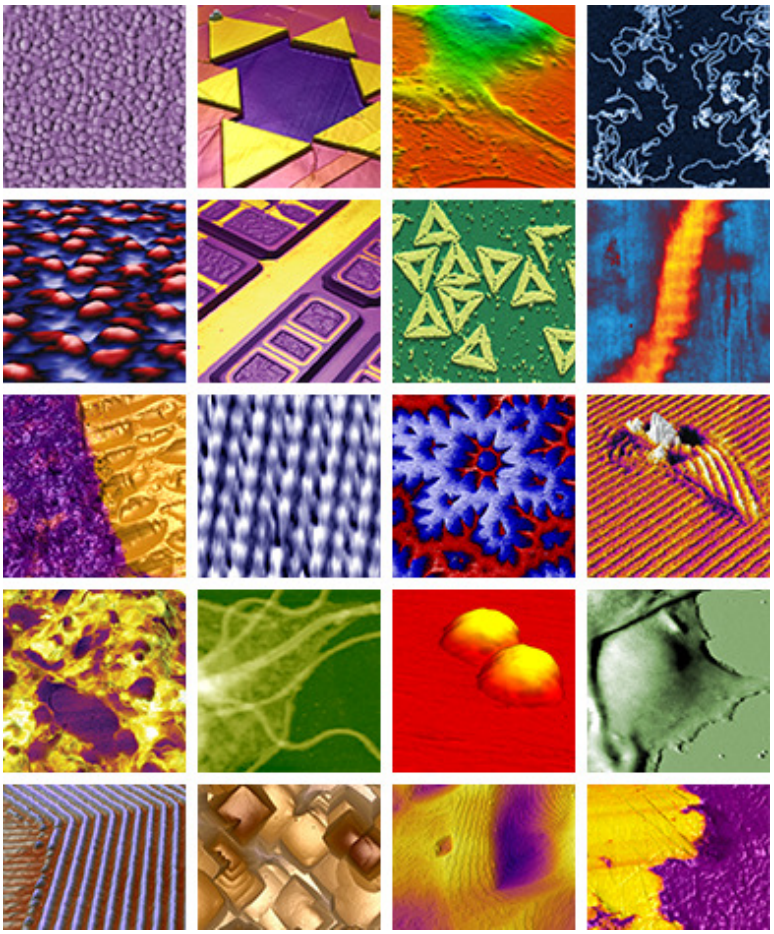
SPM Applications Guide

USER GUIDE 3

Applications Guide



Including beta (complete, reviewed) chapters.

Version 16, Revision: A-2053

Dated 09/28/2018

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 are:

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, Nanolithrography, 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 15.

Getting Help There are many ways to get help with your Asylum Research instrument, and it is always free:

- go to https://support.AsylumResearch.Com. Here you will find FAQ articles, software downloads, manual downloads, and a user forum which puts you in touch with thousands of other AR SPM users.
- e-mail us at support@asylumresearch.com
- call us at +1-805-696-6466. During US west coast business hours you will get a human being to speak with. After hours you still have a good chance of catching one of our scientists. Within the US you can call our toll free number if you wish (1-888-472-2795). If you are outside the US timezones, call your local asylum office or distributor.
- If necessary we can initiate a remote session and have one of our scientists operate your AFM over the internet.

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 what chapter and section your 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. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

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

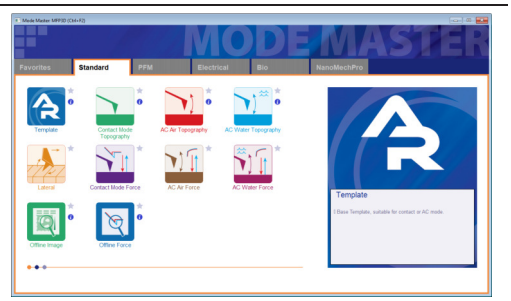
Contact Mode imaging is sufficiently commonplace that a tutorial has been developed for it. If you own an MFP-3D family AFM, please follow *MFP-3D User Guide, Chapter: Tutorial: Contact Mode Imaging in Air*. If you own a Cypher AFM, please follow *Cypher User Guide, Chapter: Tutorial: Contact Mode in Air*. This will familiarize you with the basics of loading the cantilever, sample, and such. Once everything is adjusted and the cantilever is ready to engage above the sample surface, you can switch over to the sections that follow here in order 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

Based on the previous section, it is assumed that:

- The cantilever is close enough to the surface 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 window.
   - If not, click the Mode Master button at the bottom of the screen: .



2.

Select Mode:

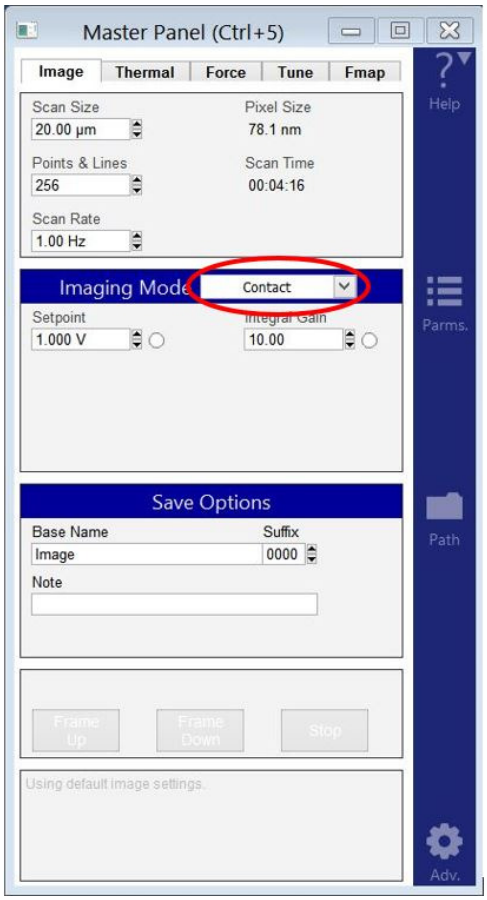
- Select *Standard tab* ▷ *Contact Mode Topography*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

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):
  - MFP-3D: 8 - 10
  - MFP-3D Extended Z: 2-3
  - 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 to the right of the Master Panel.

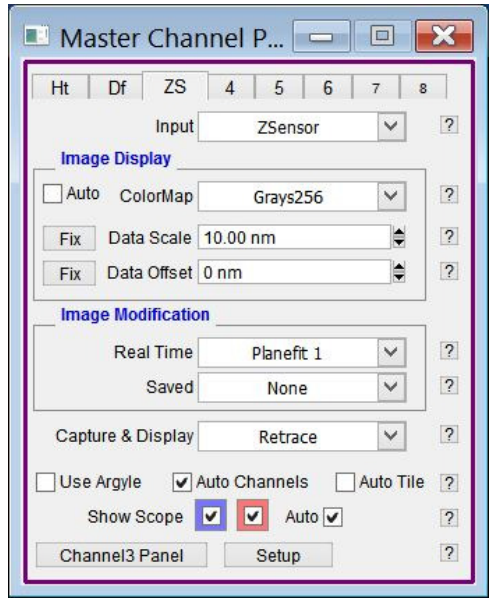
Note Parameters highlighted in orange will be mainly used to fine tune imaging parameters when scanning.



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 examined before scanning commences. 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.

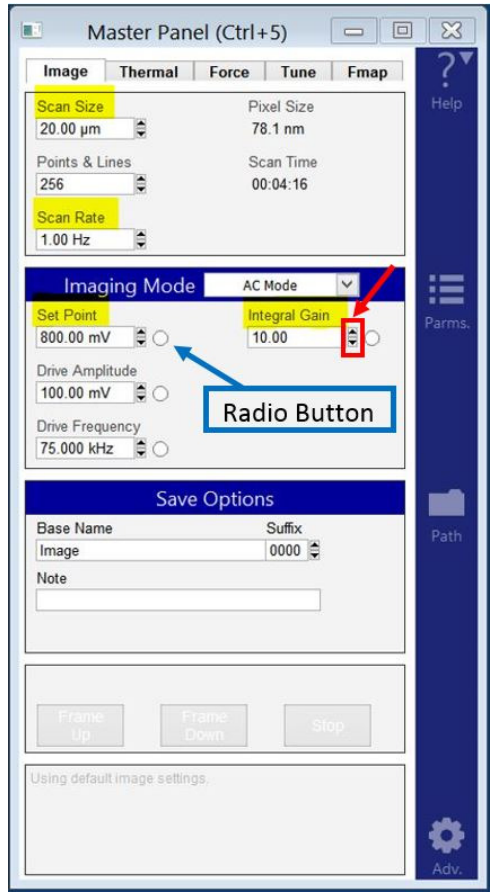
Mode Master: Most of what was described in this section is automatically set by the Mode Master when *Contact Mode* is selected from the standard modes.

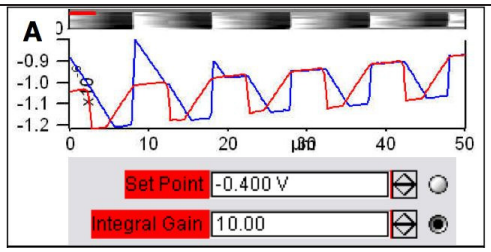
1.3. Optimize Imaging Parameters

1. Click the 'Frame Up' button on the Image Tab. After a brief moment, imaging will begin. Tune the parameters with Set Point, Integral Gain, and Scan Rate. Use the arrow clickers to right of setvar windows to adjust parameters.

2.

- 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 which 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.





Determining Image Quality:

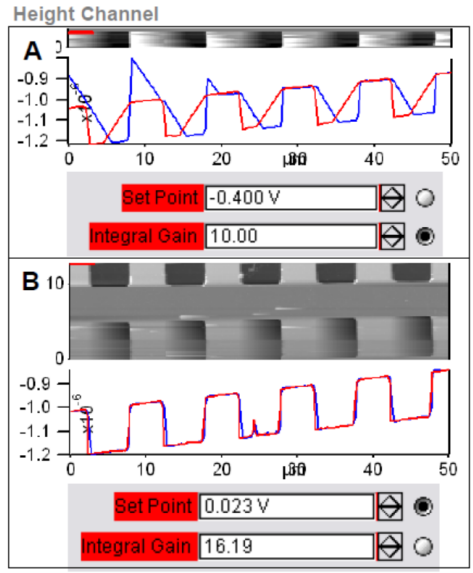
3.

- 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). Then you 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**.

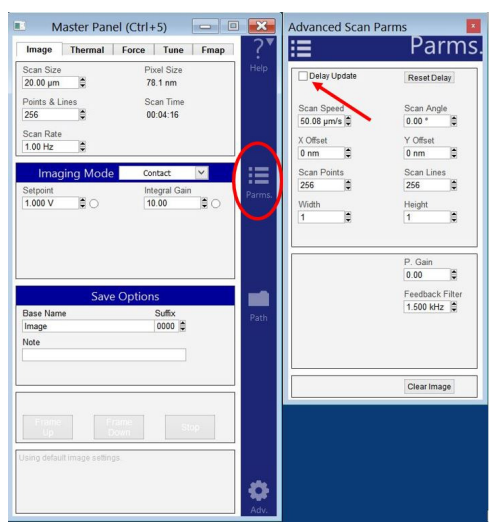
4.
- The Setpoint voltage and Integral gain should first be adjusted to achieve good tracking. The picture to the right shows an example of imaging a calibration grid, with corresponding parameters from the Main tab below.
- Panel A) shows the initial tracking of the tip upon engagement with a Setpoint voltage of -0.4V when the free air voltage was about -0.45V.
- Panel B) shows that with an increase in the Setpoint force and a slight increase in the Integral gain, tracking is improved greatly.



Note Don't 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 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 0 degrees because of the shape of the tip at the end of the cantilever. Some cantilever manufacturers compensate for the slight angle the cantilever is mounted relative to the surface by having the front and back angles of the pyramid at different angles.

6.
- The *Delay Update* checkbox allows the user to change the parameters during a scan, which will take effect at the end of that scan.
- During the period before update, the parameters changed will be highlighted in a orange color.
- 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 by the image on the right.



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

A Most imaging parameters in the main tab of the main panel (See 3) will 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, then any changes you make to parameters above that box will only update 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 nice 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. 2021, DATED 07/09/2018, 12:21. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

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 (See **??** on page **??**), 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 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 278 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.

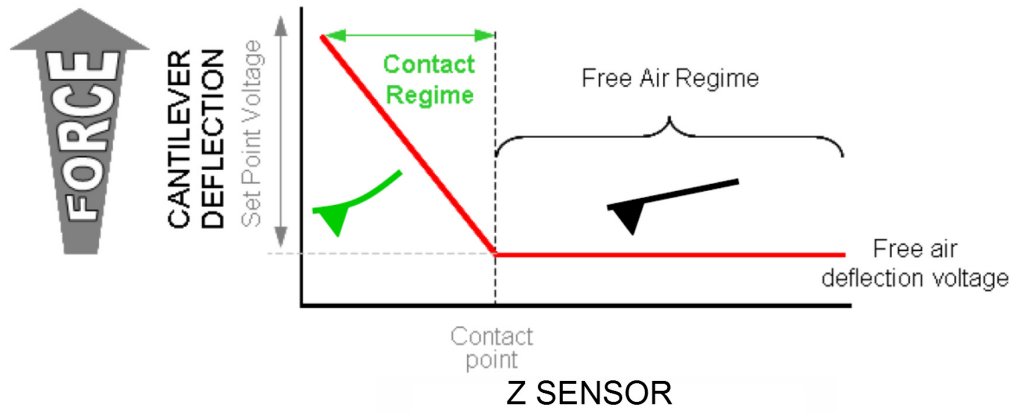


Figure 2.1.: Qualitative depiction of Cantilever Deflection vs. Z Sensor.

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 full extended, and the deflection is still below the required setpoint. What this means is 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. As the cantilever goes over the sample, voltage to the Z actuator will change due to the feedback. The Z actuator is made from piezo electric 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. The sensor is much more accurate but introduces some noise to the measurement. Over large ranges this noise is neglible 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 maintaing a constant deflection but not perfect. Sudden changes in the sample topography cause the Z feedback to either under or over compensate. The *Deflection* channel is therefore a measurement of the error signal. This channel can be useful for detecting features that don't show up well on the other channels.

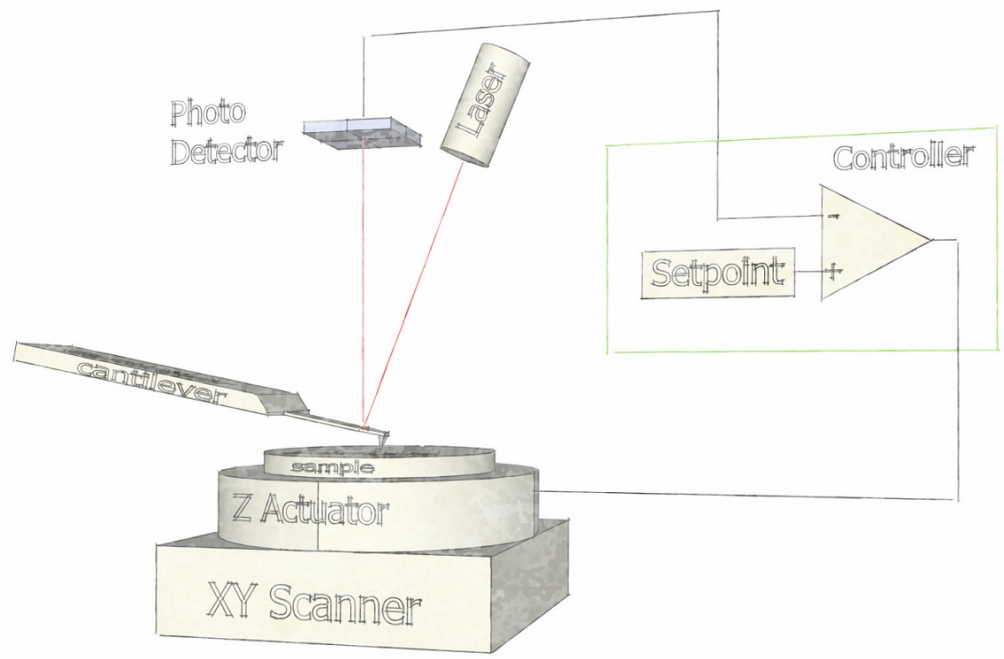


Figure 2.2.: Contact Mode AFM. Z actuator feeds back to keep cantilever deflection at a given setpoint.

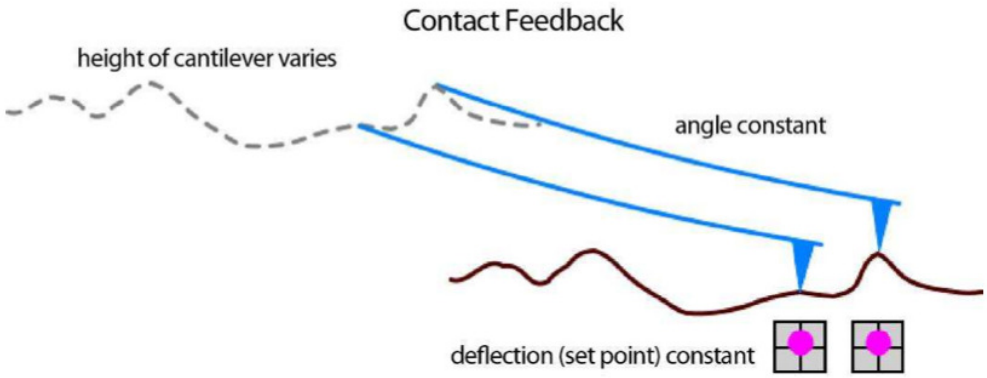
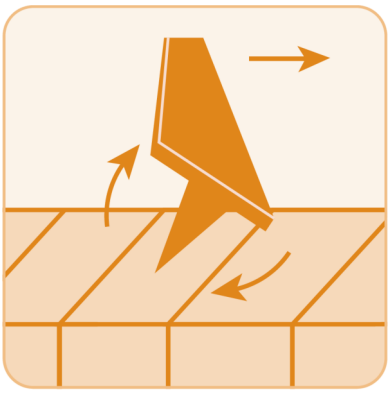


Figure 2.3.: Contact Mode tracking of the surface. Feedback keeps the cantilever deflection constant.

3. Lateral Force Microscopy

CHAPTER REV. 2014, DATED 05/09/2018, 18:29. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



Chapter Contents

3.1. Lateral Force Imaging

3.1.1. Introduction

Lateral Force Microscopy (LFM) can easily be performed on both of 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 photodector that measures the cantilever deflection, except that "left" and "right" halves of the photodector are differenced to measure the lateral deflection (twist) of the cantilever.

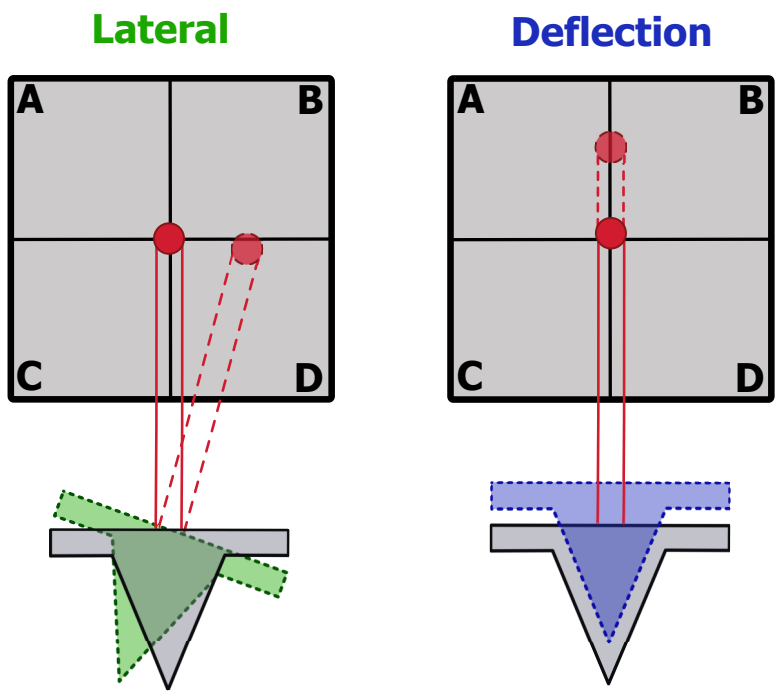


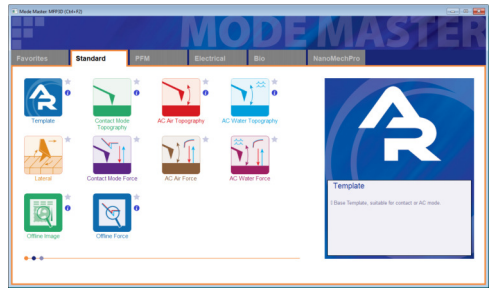
Figure 3.1.: The Deflection signal versus the Lateral signal on the photodetector.

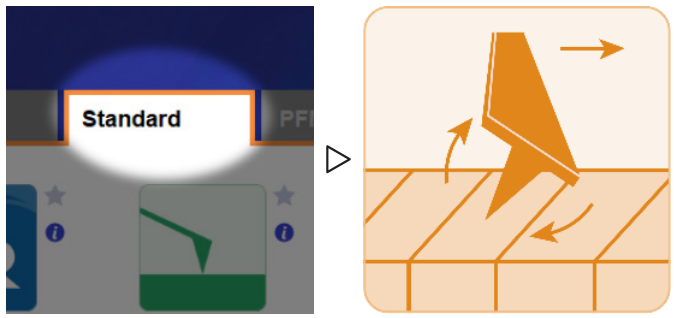
3.1.2. How to Guide

It is assumed the user is already familiar with Contact Mode AFM (1). 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, and such.

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 now re-arrange 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 will cover this technique separately. (See 3.2)

3.1.3. Zeroing the Lateral Signal

Note	LFM will produce 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 photodector 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:

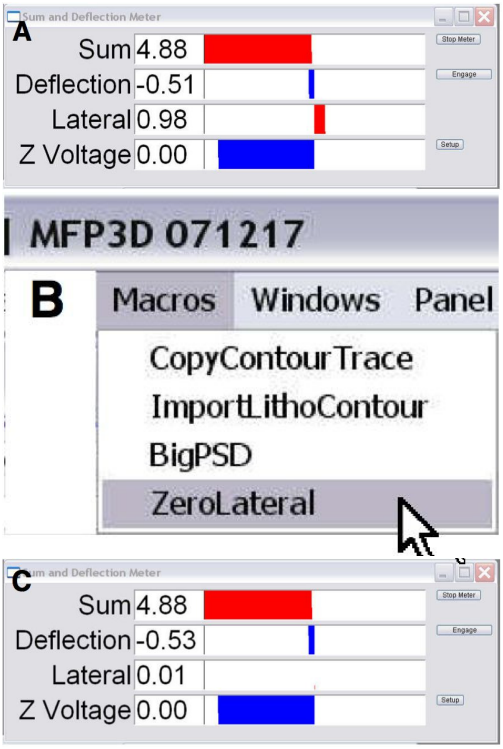
3.1.3.1. MFP-3D Classic/Origin/Origin+

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 wherever 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 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 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 in to the Head to mechanically center the laser spot on the photodetector.

3.1.3.3. Cypher S/ES/VRS

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. Please see

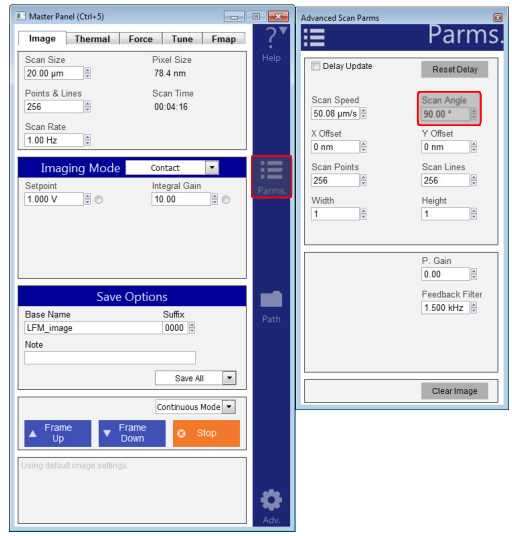
3.1.4. Setting the Parameters

1.

Scan Angle

- Loading **Lateral** mode automatically sets the *Scan Angle*to 90°.
- To check this, select 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°.

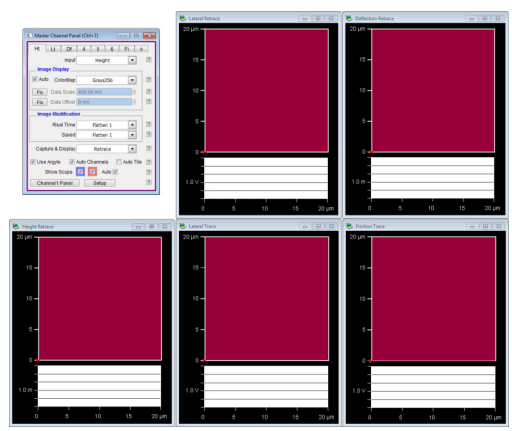


2.

Channel Configuration

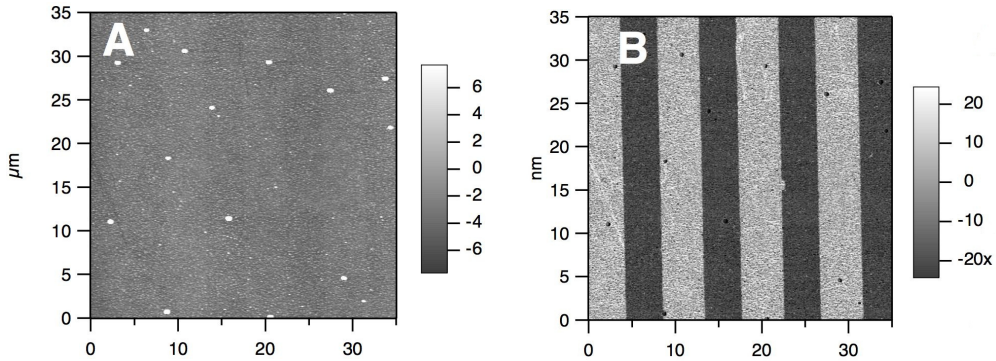
- Loading **Lateral** mode automatically configures the *Lateral* channel in the *Maser Channel Panel.*
- 5 channels are automatically displayed: **Height, Deflection, Lateral: Trace, Lateral: Retrace, and Friction: Trace**

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 shows a couple images from a sample which has bands of alternating molecules adhered to its surface. This was achieved by micro-contact printing. Note that Height image in Figure 3.2a 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, however, shows great contrast, indicating a difference in tip-sample frictional forces between these two molecular species.



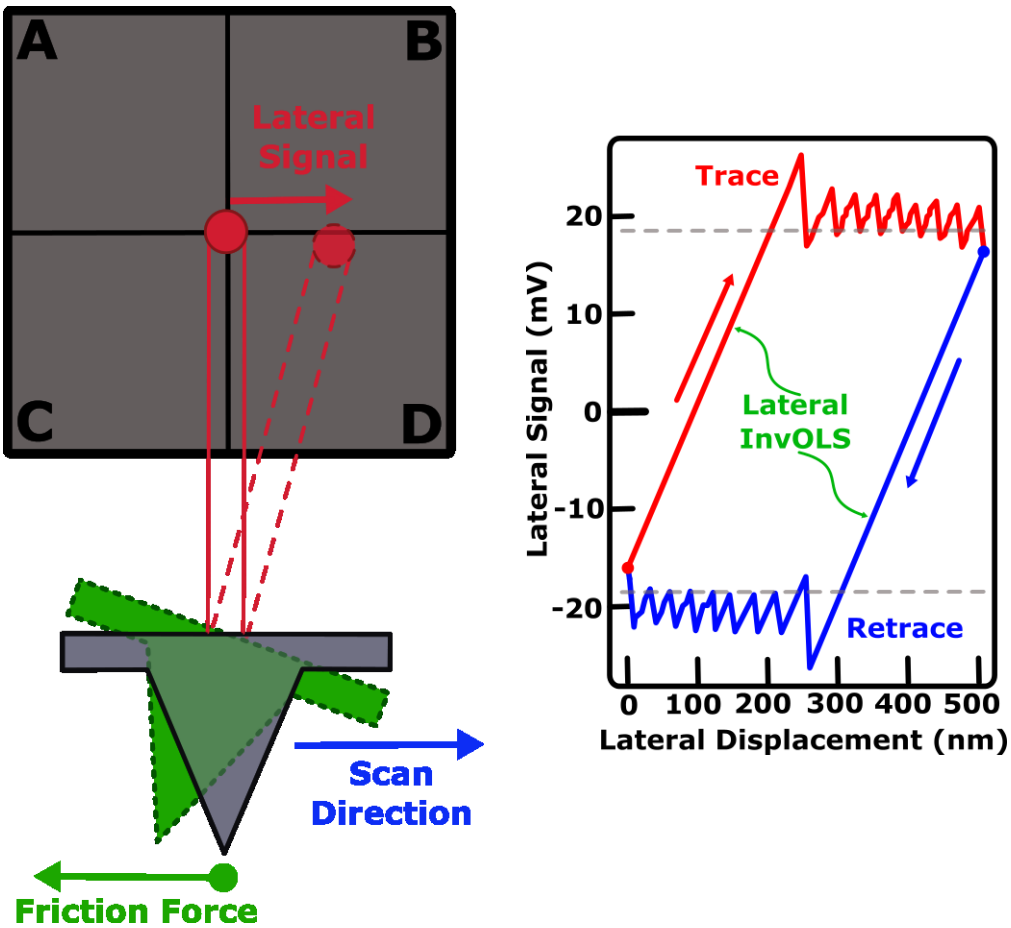
(a) Height Image **(b)** Lateral Image

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 sensivitiy (Lateral InvOLS) in order to quantify the friction measurements, a bit like a Deflection force curve might be used.



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

Figure 3.3.: An example of a standard friction loop measurement.

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 3.3b, 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. Needless to say, 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.

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 3.3b).

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]

[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)

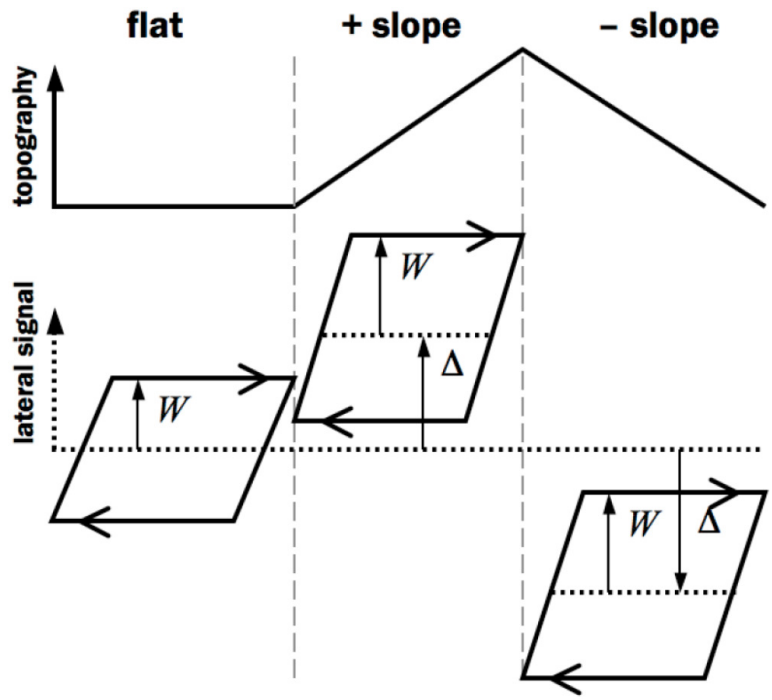
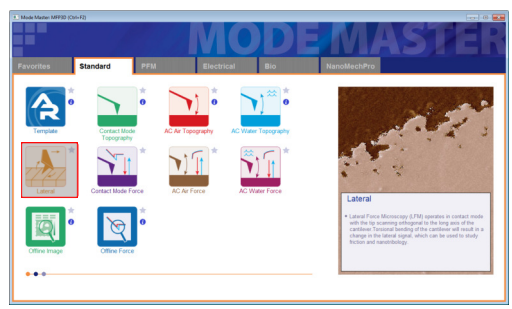


Figure 3.4.: Friction loops on flat, inclined, and declined surfaces at a given applied load. The half-width (W), representing the frictional response, only slightly varies, while the offset (Δ), due to the surface tilt, varies substantially. The load dependence of W and Δ are used to determine the lateral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

3.2.3. How to Guide

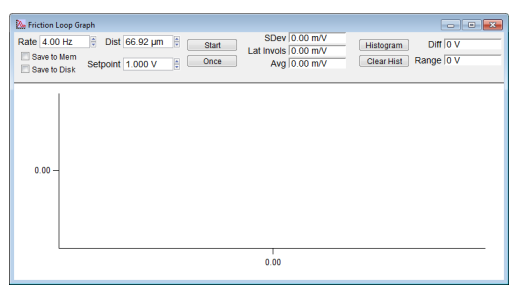
1. **Mode Master**

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



2. **Friction Loop Panel**

   - The Friction Loop panel has a few parameters to control:
     - **Rate:** Tip scan rate
     - **Dist:** The distance the Y-piezo will ramp
     - **Setpoint:** The tip deflection/normal force



3.3. Friction-Load Maps

3.3.1. Introduction

A 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. I can better understood and interpretted 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 3.5) and make relative comparisons of changes in friction between tip and sample.

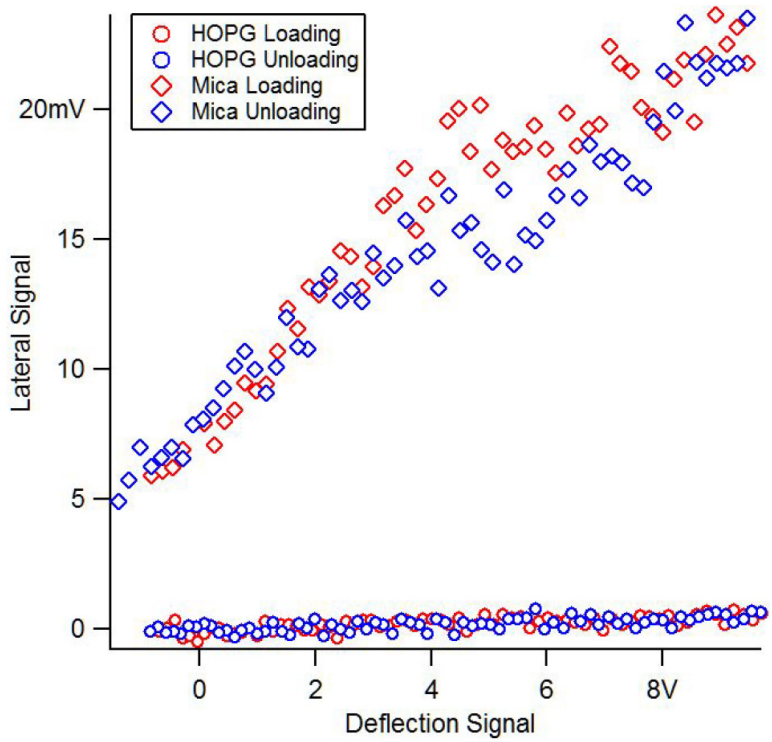


Figure 3.5.: Uncalibrated, relative comparison of the COF for HOPF and Mica

Everything considered, a Friction-Load Map is essentially a Friction Loop (see 3.2) in combination with a Force Curve (see 18). In this technique, the cantilever is rastered back and forth across a single line like in a Friction Loop, however the normal force is also incremented after each completeld line up to some maximum value ("loading"); and then decremented in the same manner ("unloading"). Both the Lateral signal and Normal signal are monitor simaltaneously, which utimately constitute the data for a friction-load map.

Note that the Deflection signal in 3.6 looks like a lot like 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 of of the surface. You can create a similar Friction-Load Map by simply 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, pull off, and at negative loads.

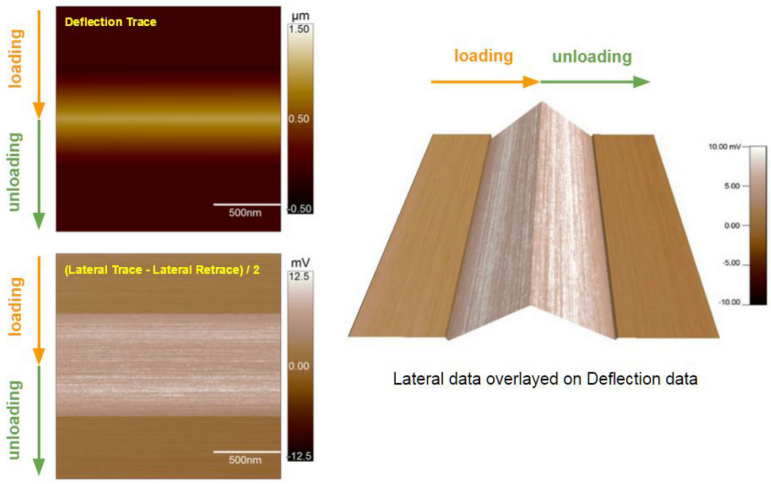


Figure 3.6.: An example of a typical Friction-Load Map dataset

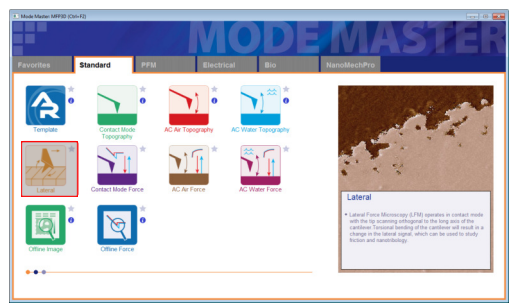
3.3.2. How to Guide

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

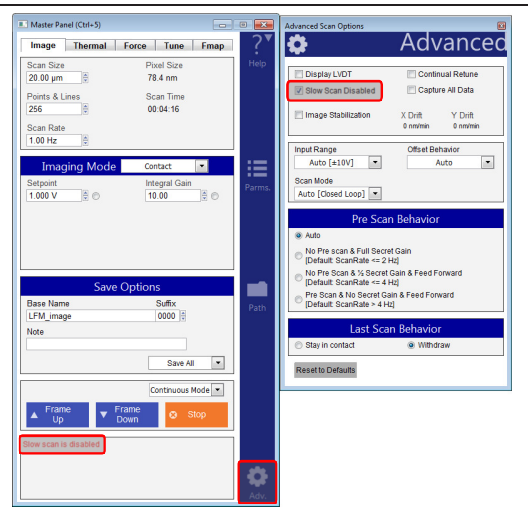
- When the **Mode Master** window appears, choose the **Standard** tab.
- Select the **Lateral** mode icon. All the necessary panels for Friction Loop operation will now automatically load and arrange on the desktop.



2.

Disable Slow Scan

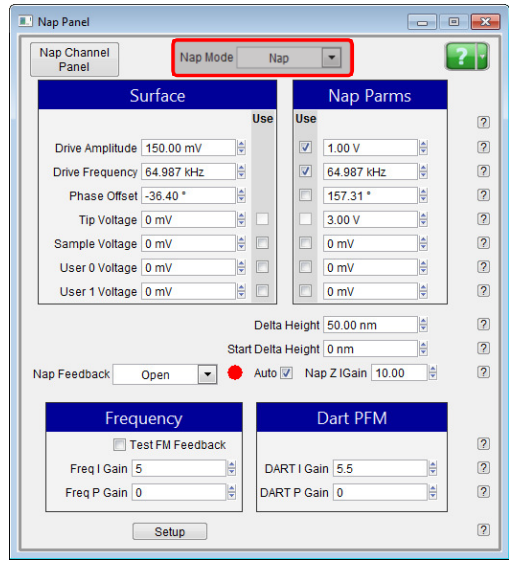
- In the **Master Panel**, select the **Adv.** tab.
- Select the checkbox for **"Slow Scan Disabled."** This will cause the AFM to scan the same line repeatly when the AFM scan is begun.



3.

Nap Panel

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

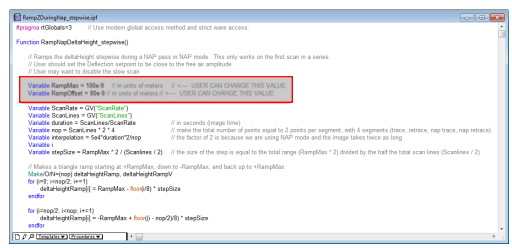


4.

Launch the .ipf

- Launch the **RampZDuringNap_stepwise.ipf** by double clicking the file icon.
- To 2 variables for the user to adjust are: `RampMax` and `Offset`.

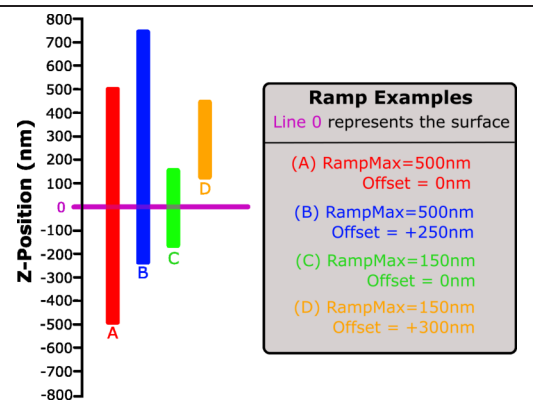
Note This file hijacks what *Do Scan/Frame Up/Frame Down* does. Instead of topographic imaging, it will lift the Z-piezo away from the surface to a defined initial position, ramp a defined stepwise amount towards the surface while scanning a line, then retract to the initial defined position.



5.

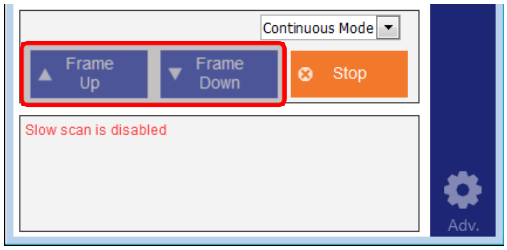
Adjusting the Variables

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



6. Once you have set these 2 variables, you must *Compile* the **RampZDuringNap_stepwise.ipf** by clicking the 'Compile' button at the lower left of the .ipf procedure window. Once compiled, this button will no longer be visible. To learn more about compiling Igor procedure files, please read the Igor manual.

7. Select 'Frame Up' or 'Frame Down' in the **Master Panel** to begin the Friction-Load Map.

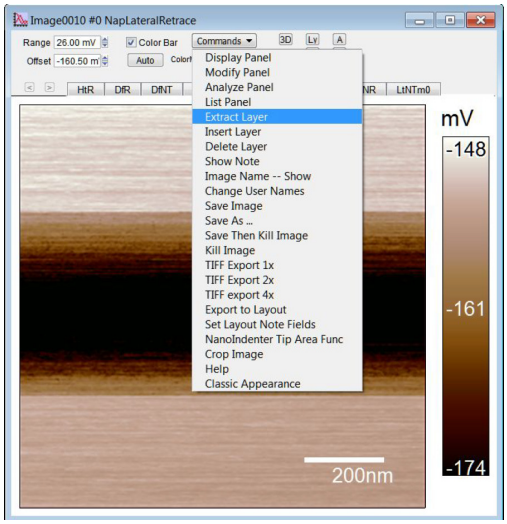


3.3.3. Data Processing

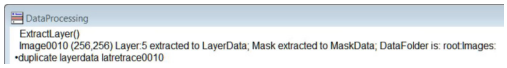
Friction-Load Map data generally require a bit of processing to understand well. This section will step through a typical procedure.

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*.



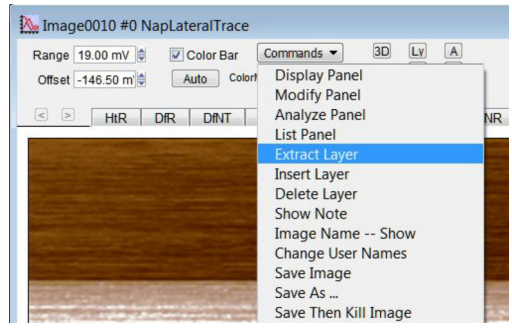
2. In the Igor command line (CTRL+J), copy the *LayerData* wave to a new wave using the `duplicate` command. In the example here, that new wave is given the name "*latretrace0010*".



3.

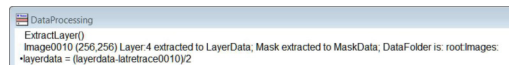
Trace Layer

- 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.

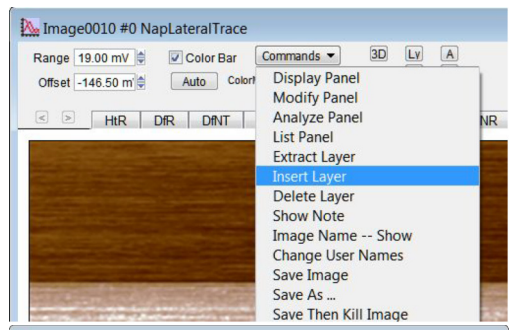


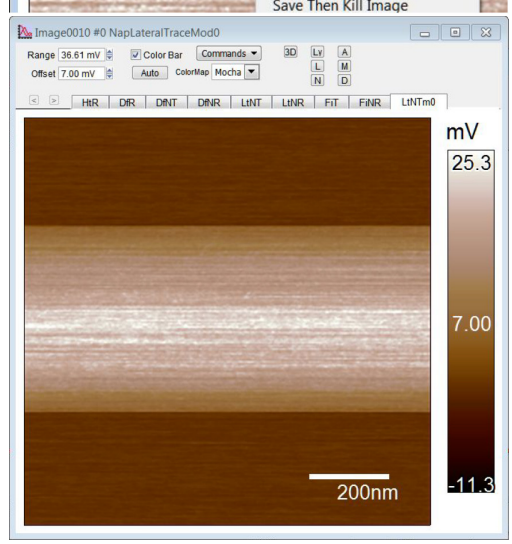
5.

Insert the Layer

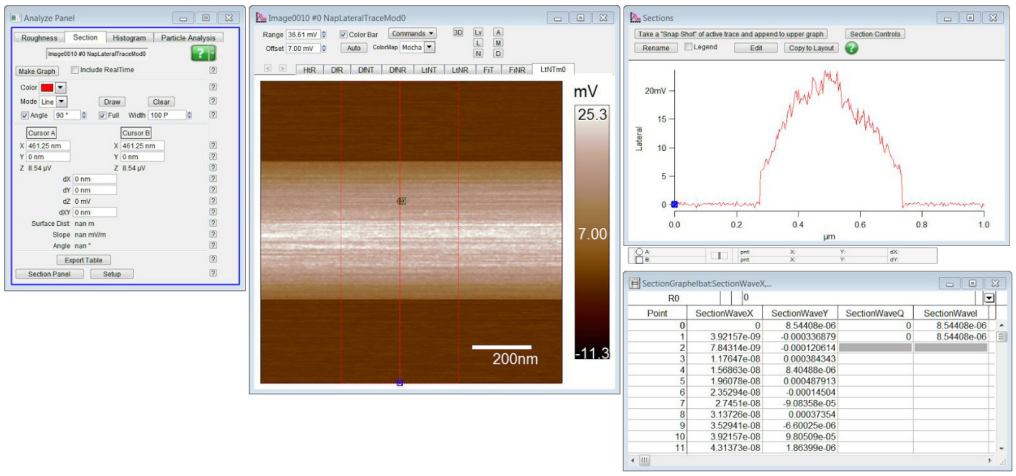
- Navigate to *Commands>Insert Layer* at the top of the image window.
- This will insert the result of that subtract (*LayerData* wave) as a new layer into the Friction-Load Map .ibw file.
- In this example, this inserted layer is named **NapLateralTraceMod0 or LtNTm0** in the tab.

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





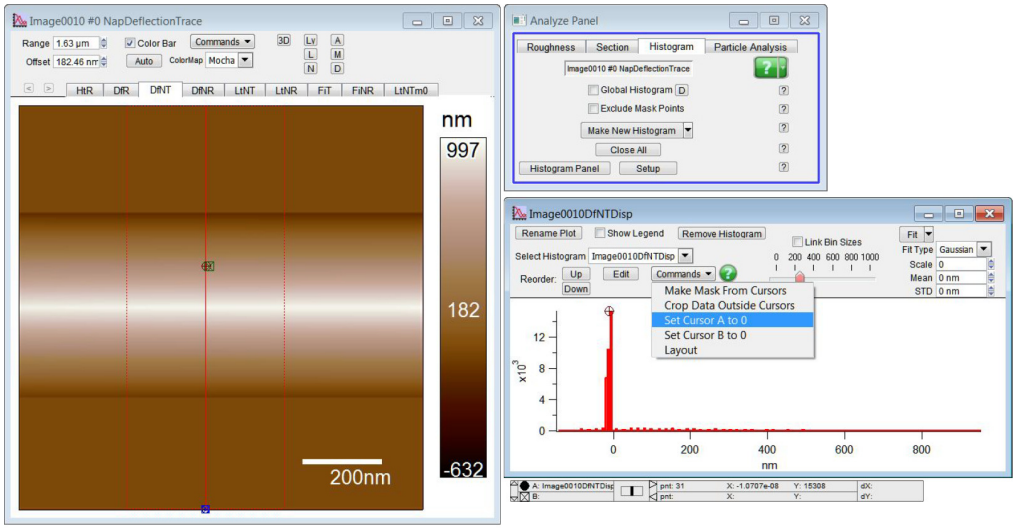
6.



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 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.

7.

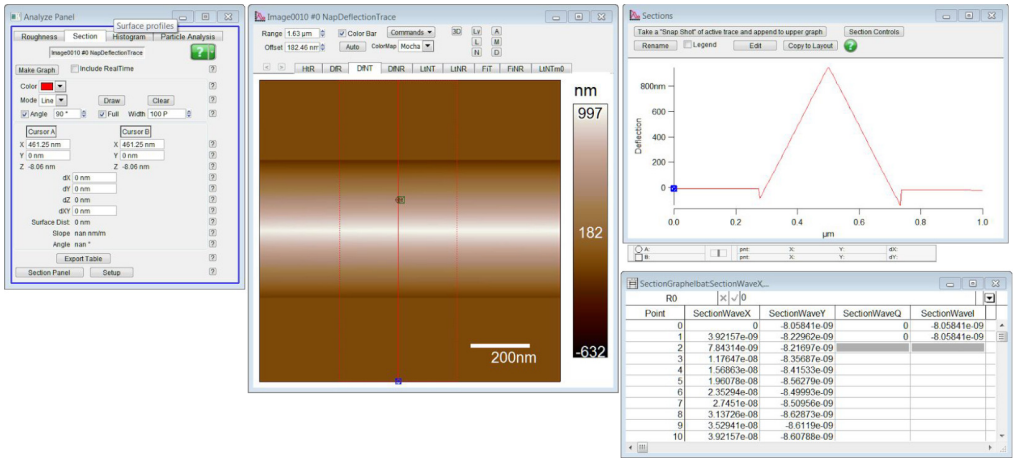


Deflection Offsets:

- 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 will remove any offests 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 offsetted **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 is only necessary if you wish to perform quantitative comparisons.

10.

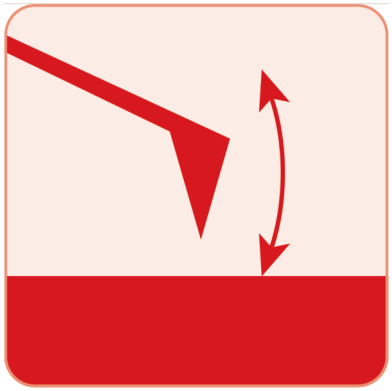
Graph the Data

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

Note You may wish 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. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



Chapter Contents

4.1. Introduction

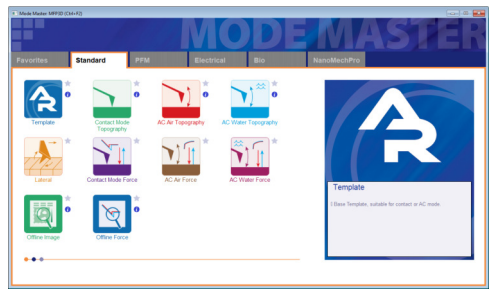
This section assumes you are already capable of starting the imaging process If this is new to you then:

- 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. In case there are differences in how the software interacts with our different instruments, we'll address that as necessary.

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* ▷ *AC Air Topography*
   - The screen will now re-arrange 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. AutoTuning

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 a little more about that now in 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*. This pulls the tip from the sample.

Note These instructions are for repulsive mode AC imaging.

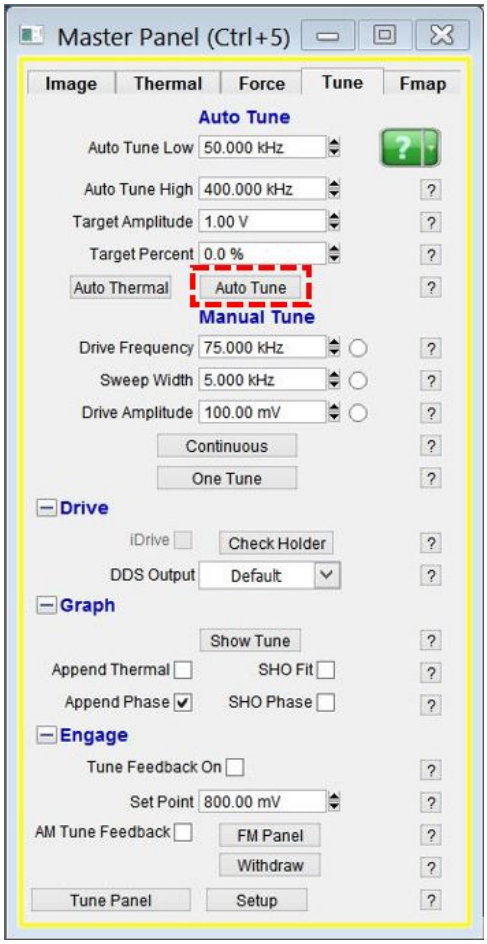


Figure 4.1.: Tune tab

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 & scanning. See Section 5.3 on page 48 for further description of repulsive mode.

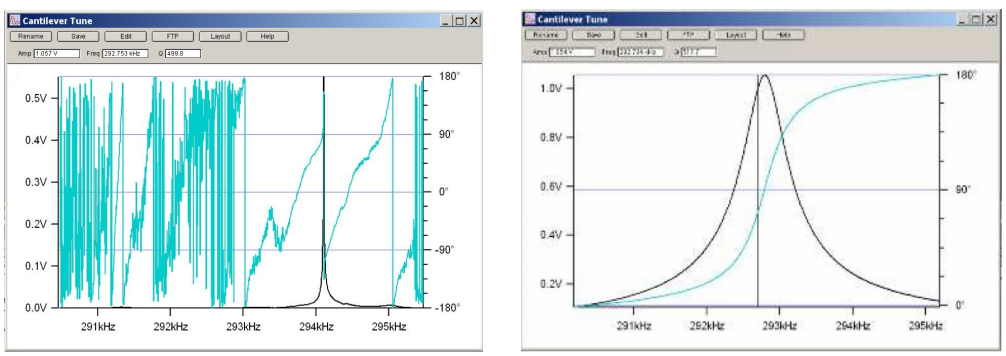
4. The default *Auto Tune Low* and *High* values for air imaging are typically 50 kHz to 400 kHz, respectively, accommodating most fundamental drive frequencies for common commercially available AC Mode cantilevers.

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 tune graph, similar to the one in Figure 4.2 on page 37, will appear. The first tune will most 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:

- Picks a *Drive Frequency* at the specified *Target Percent* (default of -5%).
- Adjust 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

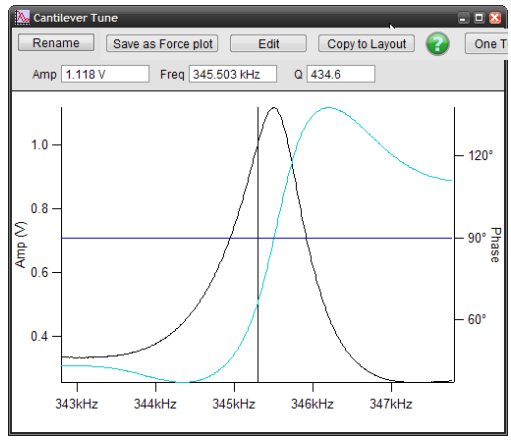
If you see a message about the drive amplitude being too low, see **??** on page **??**.

If you do not know the resonant frequency of your cantilever, see Chapter 21 on page 290.

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, how to adjust gains, how to interpret the images and figure out what is going wrong. It may be written to be MFP3D centric, but it covers the basics, and even if you have a cypher, a lot of what is in there is still very useful, you just may need to adjust the values of the gains and rates a bit for your cypher.

4.6. Net Attractive & Repulsive AC Mode

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 it 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 large range of its oscillation. This is much more difficult to do and can not 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. Since it can be tricky when you actually want attractive mode imaging to occur, here are some simple steps to help keep the tip in attractive mode.

You can tell you are in attractive mode when your phase signal is > 90°.

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 Qgain

Higher Q resonance peaks favor attractive mode imaging. Q control gives some control.

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.

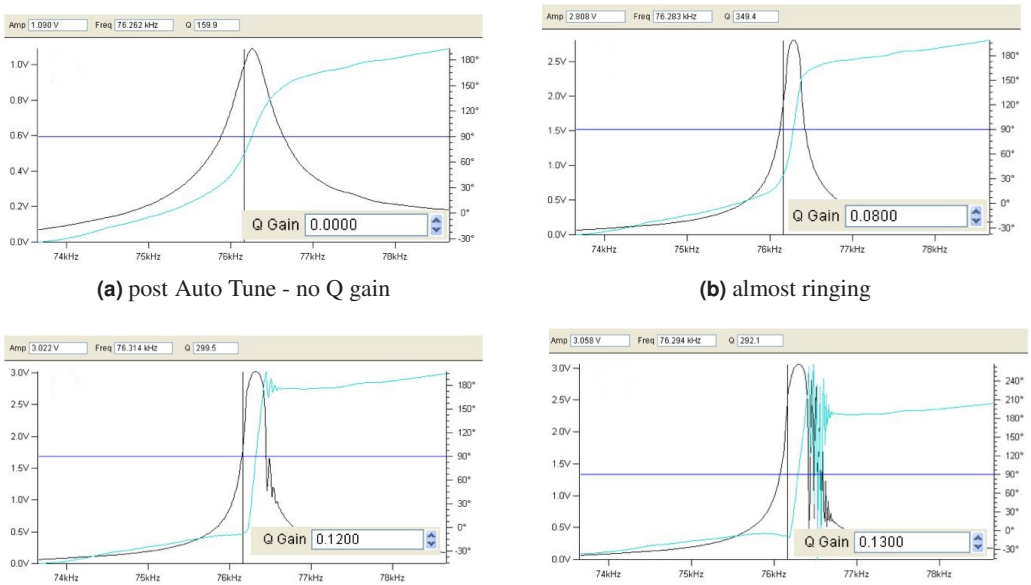
Figure A shows the last tune of an Auto Tune; no Q gain is added.

Figure B shows the response after a small 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 going even higher, showing even more ringing in the amplitude and phase data.

Notice with additional Q gain, the amplitude response will go 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

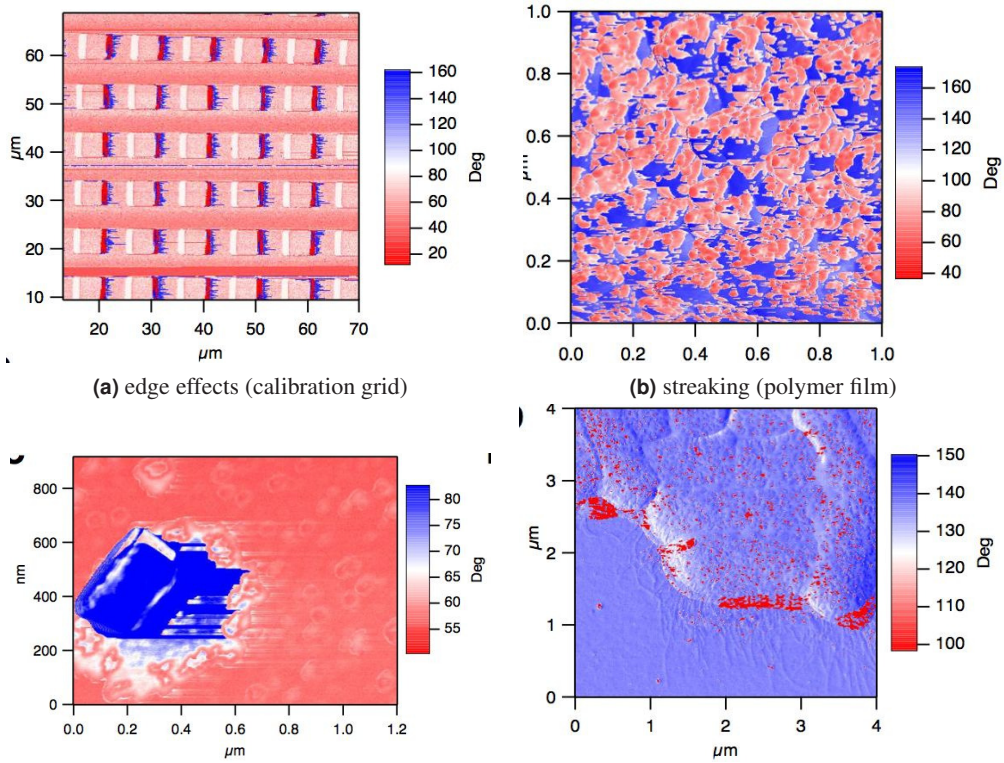
Figure 4.3.

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° (4.6.1) 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 it is desired to be in attractive mode, reduce the drive amplitude then the setpoint. 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.



(a) edge effects (calibration grid)

(b) streaking (polymer film)

(c) polymer particle

(d) bacterial film

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 - here are a few things that 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 4.5). 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.
- The tip could be 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. The Set Point Based Imaging Method

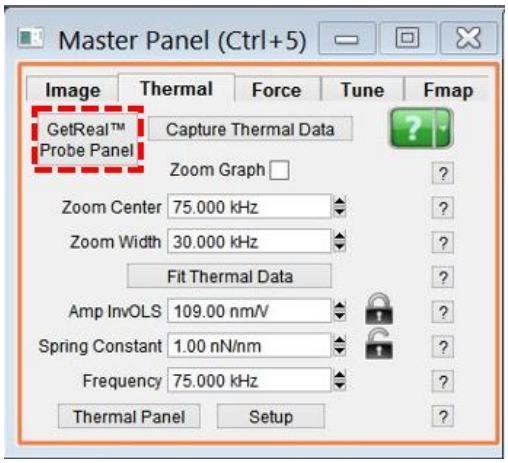
Set point based imaging is an iterative method of determining scanning parameters. Formerly, the procedure involved setting an arbitrary free air amplitude and adjusting the set point accordingly. In this alternative method, a set point is chosen based on the estimated roughness of the sample surface and InvOLS of the cantilever. Once this has been determined, the set point as 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 set point to maintain a stable image.

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

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.

1. **Getting Started**

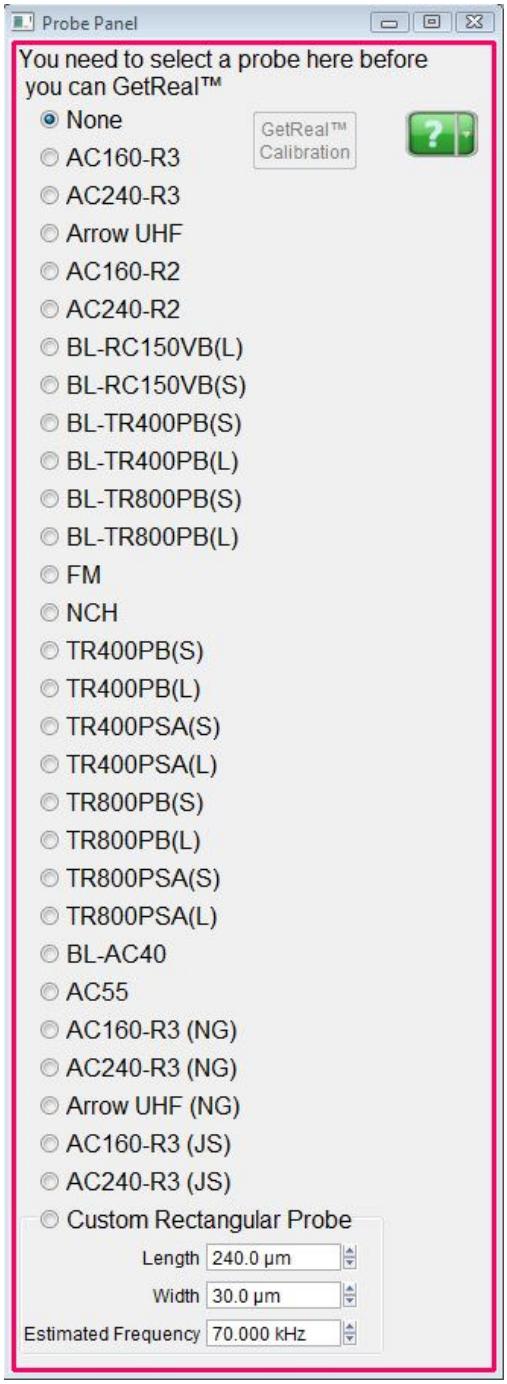
   - Make sure the cantilever is engaged on the surface of the sample. For instructions on how to do this for the MFP3D, see **??** on page **??**. For the Cypher, see **??** on page **??**.
   - Click on the Thermal tab of the main control panel.
   - Click *GetReal Probe Panel*. The panel will open.



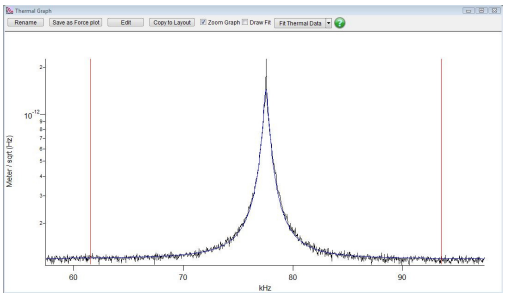
2.

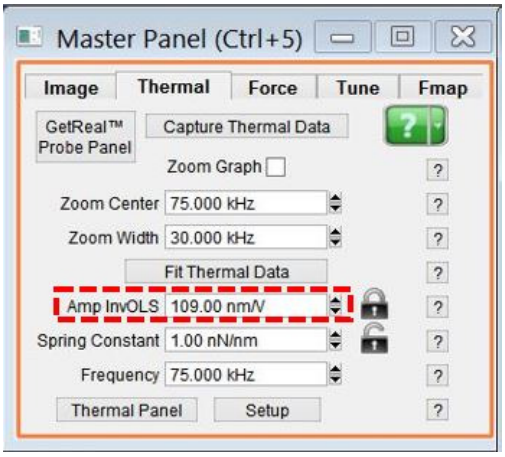
Initiating Cantilever Calibration

- You will see a list of different cantilevers. Select the cantilever you are using.
- It is also 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 forums 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 some time, a graph of the calibration will display. It should resemble the figure on the left.
- Once the cantilever has been calibrated, a value will appear in the Amp InvOLS field. This represents the amplitude in nm/V.

4.

Note "InvOLS" stands for "Inverse optical lever sensitivity." However, the number is the reciprocal of the optical lever sensitivity rather than its inverse.

5.

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.

6.

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.

7.

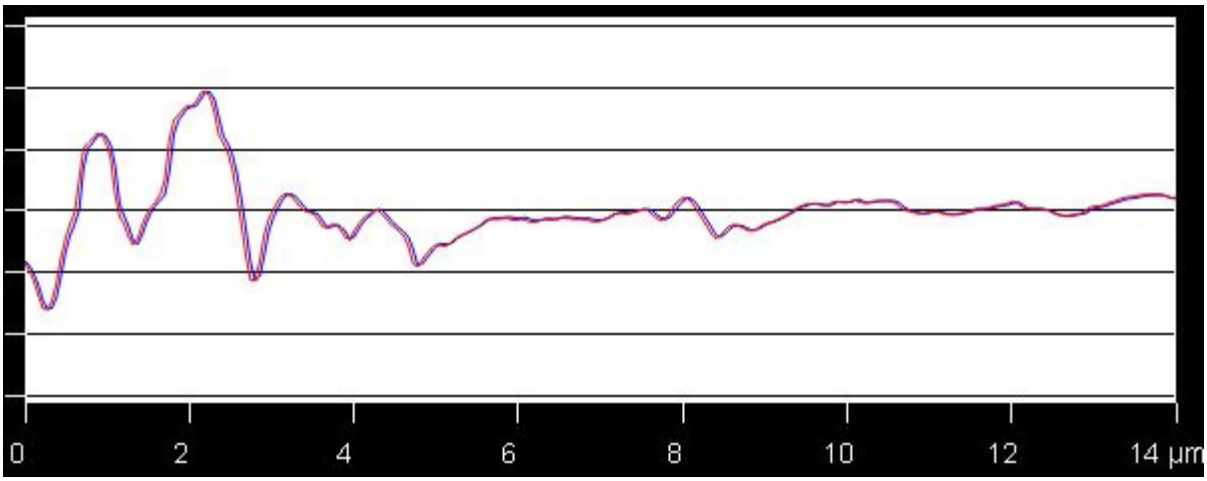
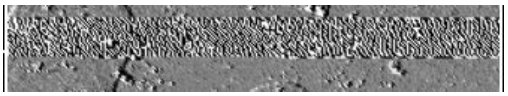


Figure 4.6.: An example of closely tracking trace and retrace lines

Tip Better Tracking

8. 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. 2021, DATED 07/09/2018, 12:21. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

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 5.3. 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 motion

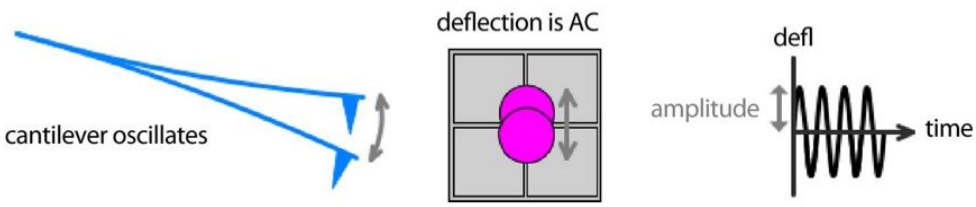


Figure 5.1.: An oscillating cantilever leads to an oscillating signal on the photodetector.

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 really quite small and that nearly any figure you see in any paper, book, or manual, will greatly exaggerate the amplitude of oscillation.

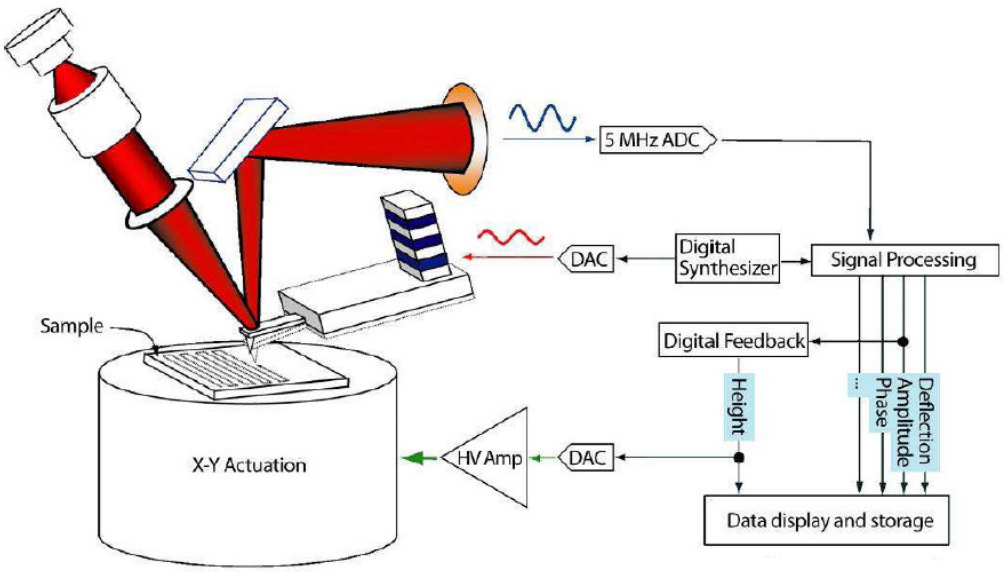


Figure 5.2.: Basic diagram of AC mode imaging.

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

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 nanovariations in sample mechanical properties. While previous sections explain a lot

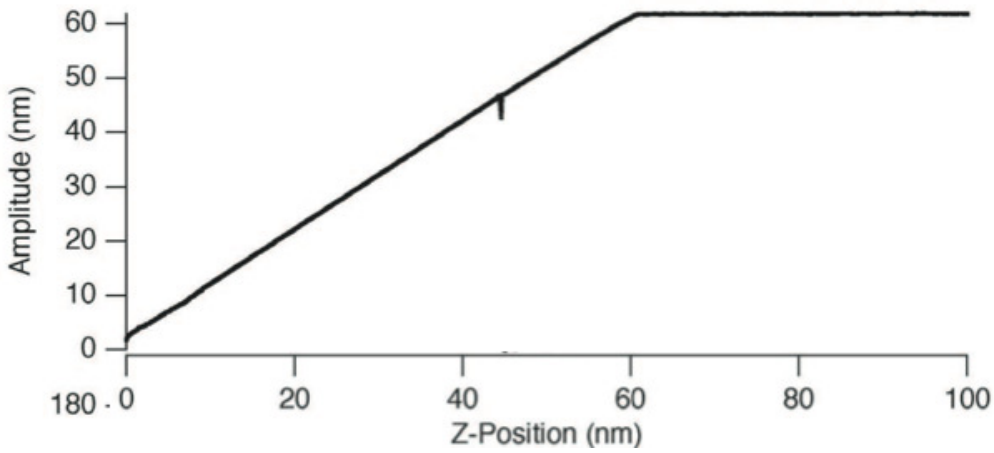


Figure 5.3.: Tip oscillation amplitude as it approaches the surface. The small glitch in amplitude is explained later, see 5.4.

about AC mode imaging in terms of amplitude, it is only half the story since 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

Lets revisit Figure 5.3 on page 48, and this time we'll show the phase signal as well.

Again we'll start at the right hand side of the graph. The cantilever is driven nearly at resonance. The amplitude is 60 nm, the z-position is 100 nm. The tip does not touch the surface, even at it's 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 during each oscillatory cycle it spends near the surface. The closer we move the cantilever to the sample, the more attractive force experienced, 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

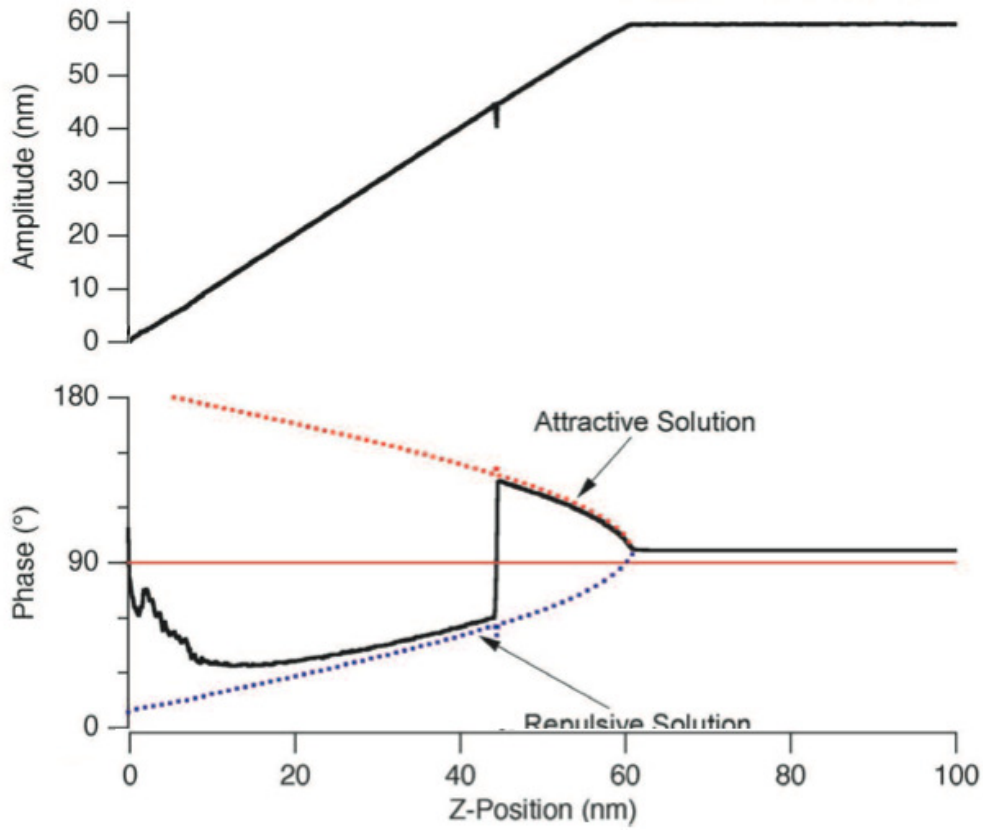


Figure 5.4.: Cantilever amplitude and phase as a function of average tip-sample separation.

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

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 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 of 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.

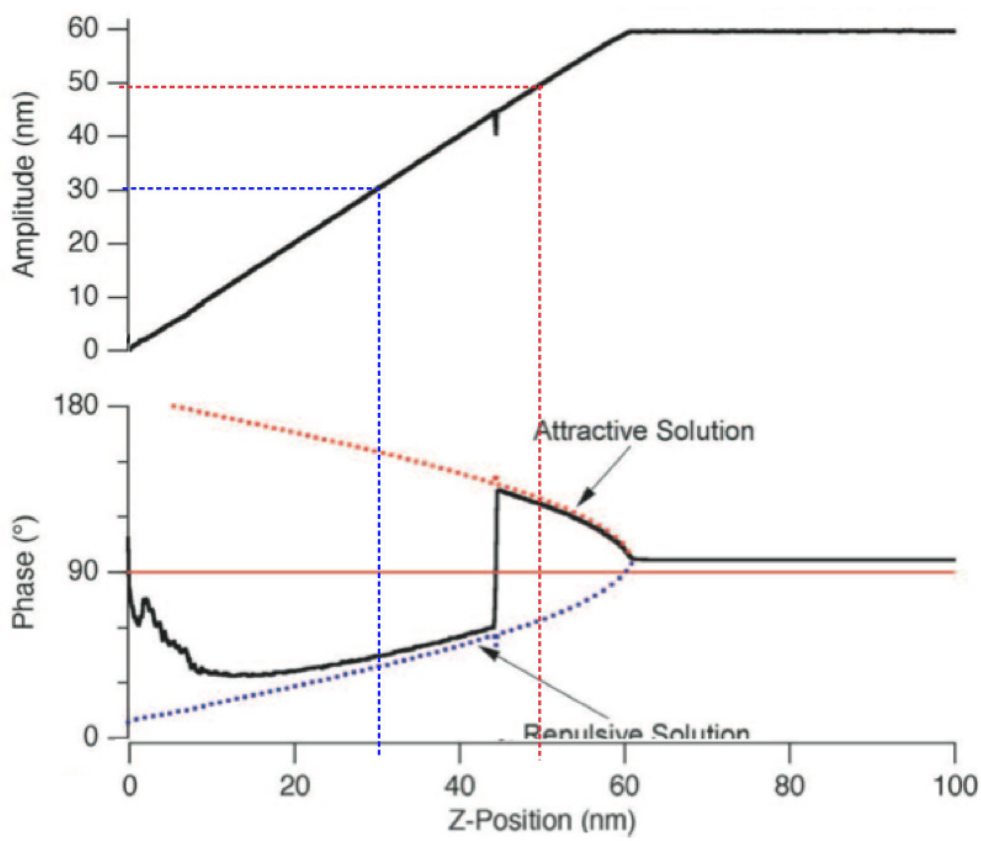


Figure 5.5.: AC mode feedback setpoint of 50nm (red dashed lines) gives attractive mode imaging, a setpoint of 30nm (blue dashed lines) gives repulsive mode imaging.

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 from the corresponding phase curve that 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 pretty good rules of thumb for imaging in purely attractive mode, purely repulsive mode, or something prone to switching between the two.

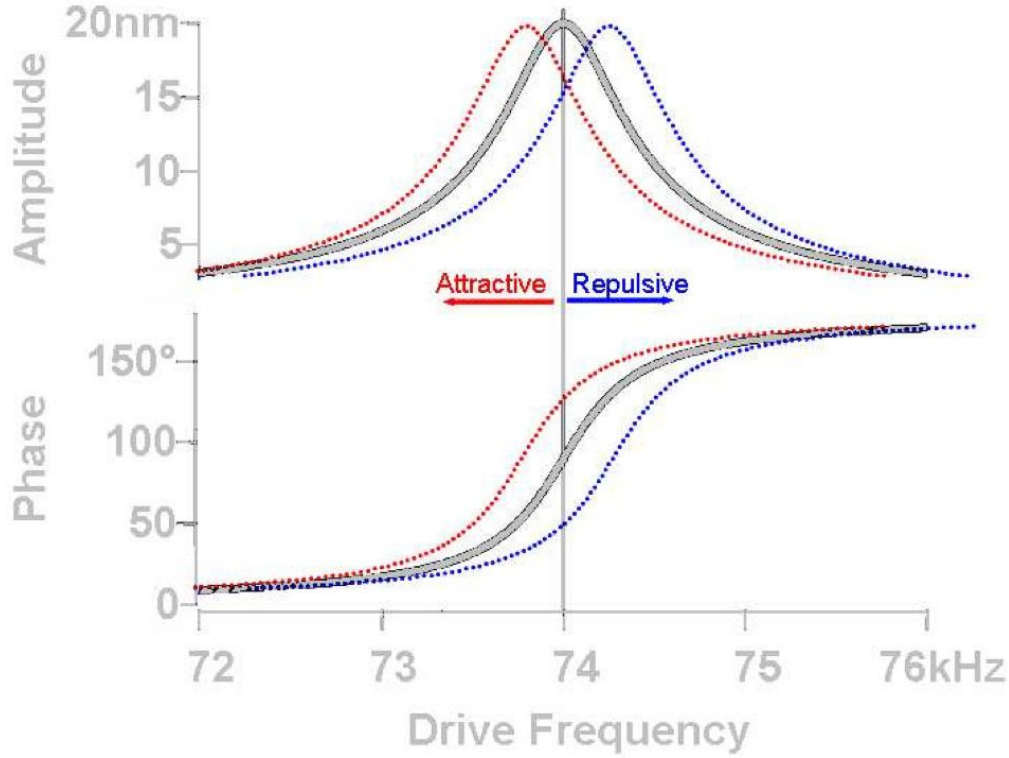


Figure 5.6.: A shift in resonance frequency, f_o (at constant amplitude) as the tip interacts with surface results in phase shift of the AC signal: Decrease in f_o results in phase increase (attractive mode), while increase in f_o results in phase decrease (repulsive mode). The colors represent the color 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/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 of Figure 5.7 on page 52 show for that cantilever and sample bigger means > 150 nm and smaller means < 50nm.

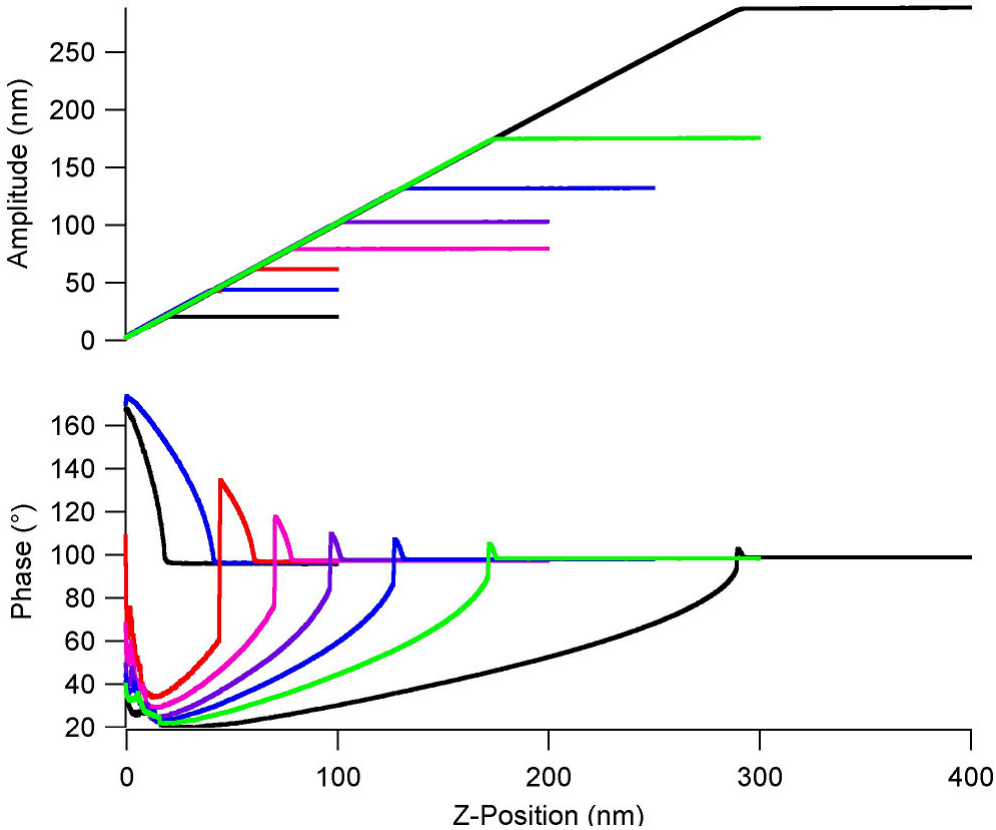


Figure 5.7.: A family of AC force curves for different free amplitudes.

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 4.6.2.3 .

5.3.2.5. Sample Attractive Forces

You typically don't have much control over this, 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. 2021, DATED 07/09/2018, 12:21. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

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

Chapter Contents

[1] *Asylum Research, Santa Barbara, USA.*
[2] *Center for Nanophase Materials Sciences (CNMS) at Oak Ridge National Laboratory, Oak Ridge, USA.*

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 piezo- 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™.

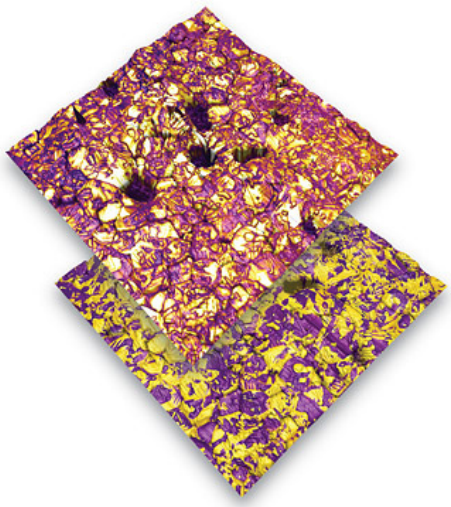


Figure 6.1.: PFM amplitude channel overlaid on AFM height (top) and phase image overlaid on height (bottom) of lead zirconium titanate (PZT), 20 µm scan.

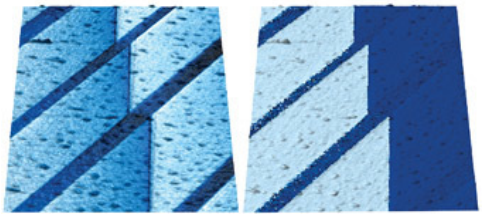


Figure 6.2.: PFM amplitude overlaid on AFM topography (left) and PFM phase overlaid on topography (right) on (100) oriented BaTiO3 single crystal (from Castech Crystals). The amplitude and phase image show 90° and 180° domain walls in BaTiO3. 10 µm scan courtesy of V. R. Aravind, K. Seal, S. Kalinin,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

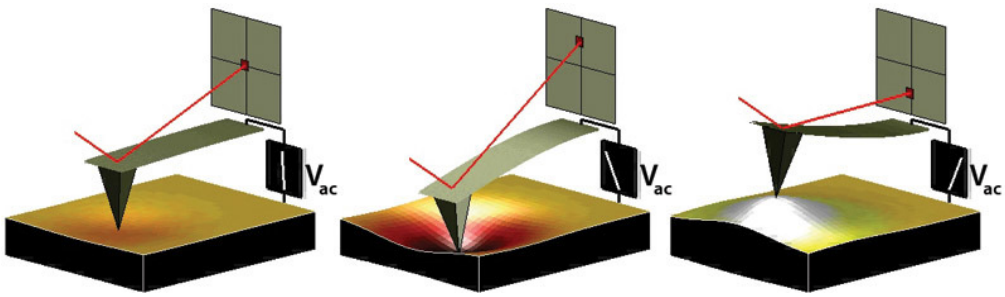


Figure 6.3.: Depiction of PFM operation. The sample deforms in response to the applied voltage, which in turn, causes the cantilever to deflect, which can then be measured and interpreted in terms of the piezoelectric properties of the sample. Image courtesy S. Jesse, ORNL.

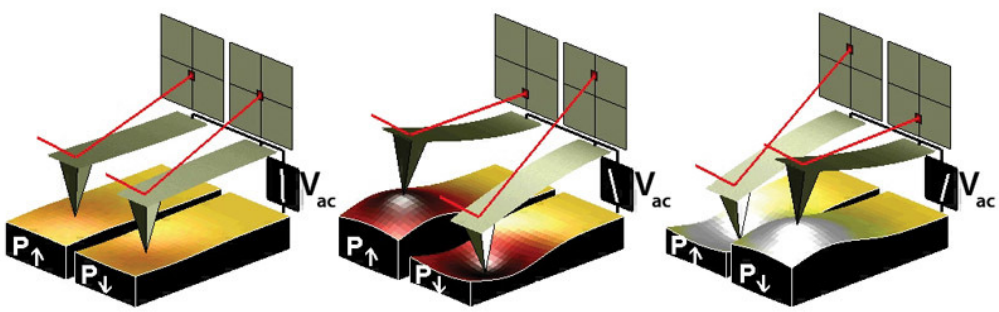


Figure 6.4.: Sign dependence of the sample strain. When the domains have a vertical polarization that is pointed downwards and a positive voltage is applied to the tip, the sample will locally expand. If the polarization is pointed up, the sample will locally contract. The phase of the measured response is thus proportional to the direction of the domain polarization. Figure 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 No. 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 $\varphi = 180°$. 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], since 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 No. 2, ISSN 0003–6951.

[8] Kalinin, Sergei V. et al., Vector piezoresponse force microscopy. Microscopy and Microanalysis, 12 JUN 2006 No. 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 No. 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 No. 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 No. 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 equation 6.6 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.

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.

[12] Jesse, S/Baddorf, AP/Kalinin, SV, Dynamic behaviour in piezoresponse force microscopy. NANOTECHNOLOGY, 17 MAR 28 2006 No. 6, ISSN 0957–4484.

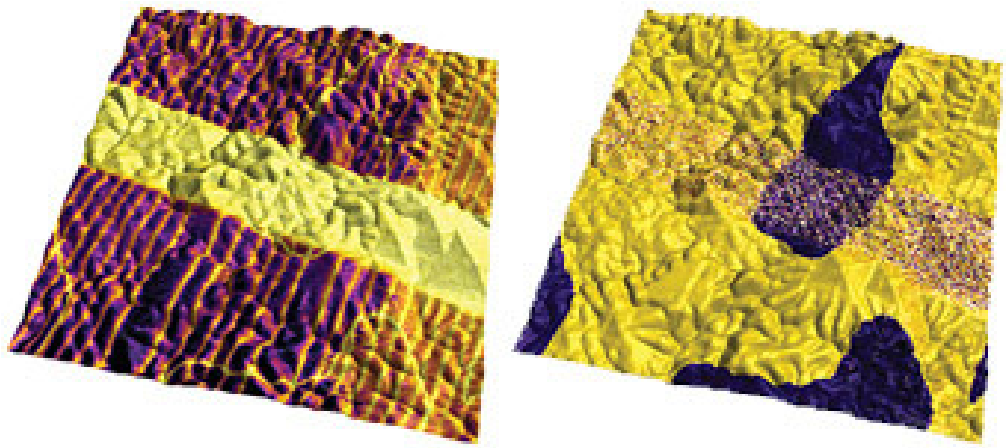


Figure 6.5.: Vertical PFM amplitude overlaid on AFM topography (left) and PFM phase overlaid on AFM topography (right) images of lead titanate film, 5 µm scan. Images courtesy of A. Gruverman and D. Wu, UNL. Sample courtesy H. Funakubo.

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 – of 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 (see sections below on DART and BE) 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 below.

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

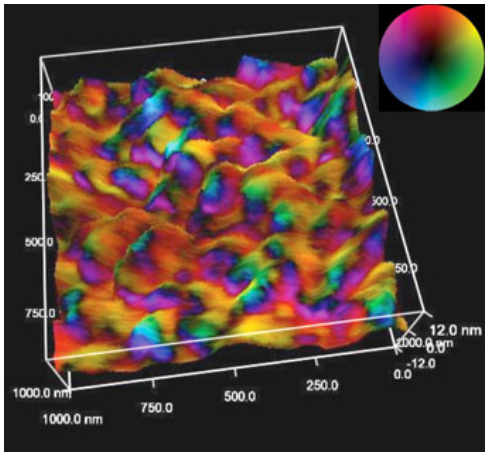


Figure 6.6.: BST film with vector PFM overlaid on AFM topography, 1µm scan. Image courtesy of C. Weiss and P. Alpay, Univ. of Conn., and O. Leaffer, J. Spanier, and S. Nonnenmann, Drexel University. Color wheel indicates PFM vector orientation.

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.

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 in order to differentiate 90° and 180° domain switching in $PbTiO_3$ thin films[16].

[13] Eng, LM et al., Nondestructive imaging and characterization of ferroelectric domains in periodically poled crystals. Journal of Applied Physics, 83 JUN 1 1998 No. 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 No. 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 No. Part 2 Suppl. S, ISSN 0947–8396.

[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 No. 21, ISSN 0003–6951.

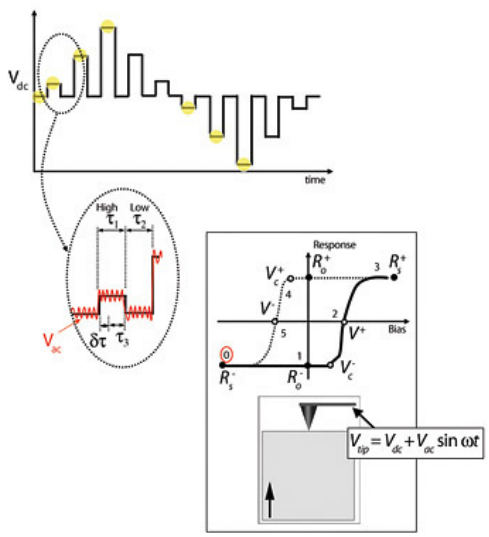


Figure 6.8.: Switching spectroscopy PFM diagram (see text for discussion). Reused with permission from Jesse, Baddorf, and Kalinin, Applied Physics Letters, 88, 062908 (2006). Copyright 2006, American Institute of Physics.

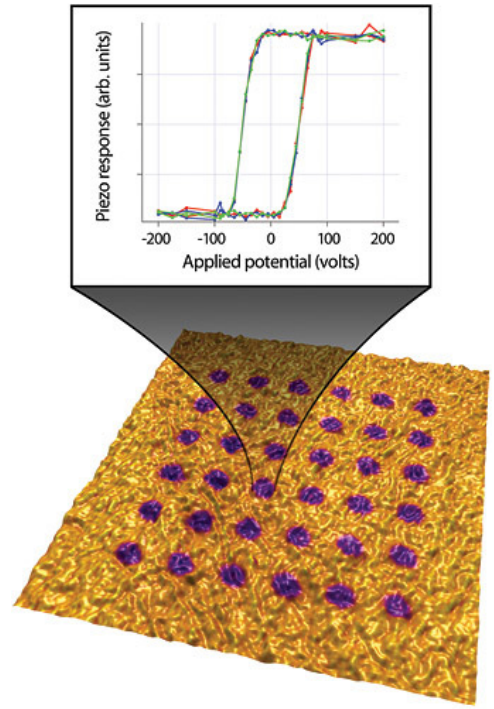


Figure 6.9.: Rendered topography of a $LiNbO_3$ sample with the PFM signal overlaid on top, 4μm scan.

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 which are oriented predominantly normal to the plane of the film, whereas regions that appear magenta-blue or light green possess polarizations which 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 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 ferro-electric 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 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.

[17] Jesse, S/Baddorf, AP/Kalinin, SV, Dynamic behaviour in piezoresponse force microscopy. NANOTECHNOLOGY, 17 MAR 28 2006 No. 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 No. 3, ISSN 1476–1122.

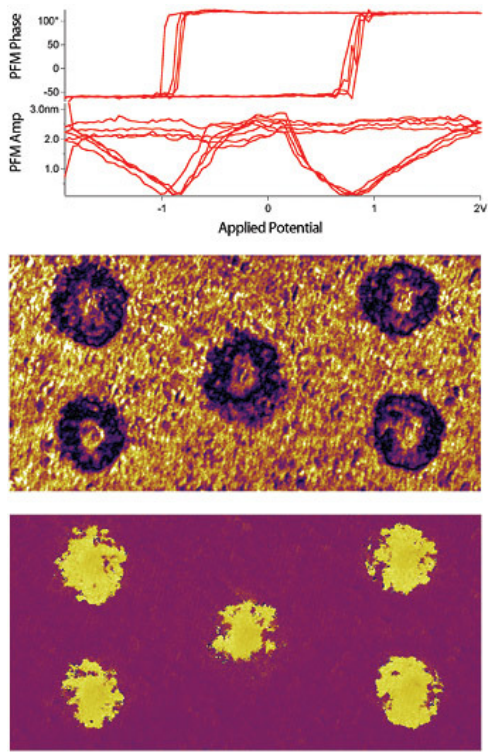


Figure 6.10.: Sol gel PZT sample where local hysteresis loops were measured and displayed (representative phase and amplitude loops shown at top). After the switching spectroscopy measurements, the area was imaged, the DART amplitude (middle) and phase (bottom) are shown, 3.5µm scan.

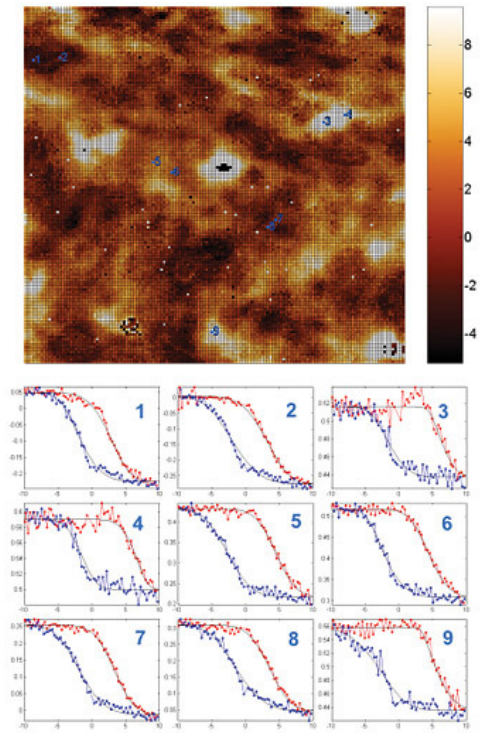


Figure 6.11.: SS-PFM and hysteresis loops of capacitor structures. Data courtesy K. Seal and S.V. Kalinin, ORNL. Sample courtesy P. Bintacchit and S. Trolier-McKinstry, Penn State Univ.

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-

- 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}\approx$100 pm/V for PZT, 10 pm/V for $LiNbO_3$) allowed direct imaging and spec-

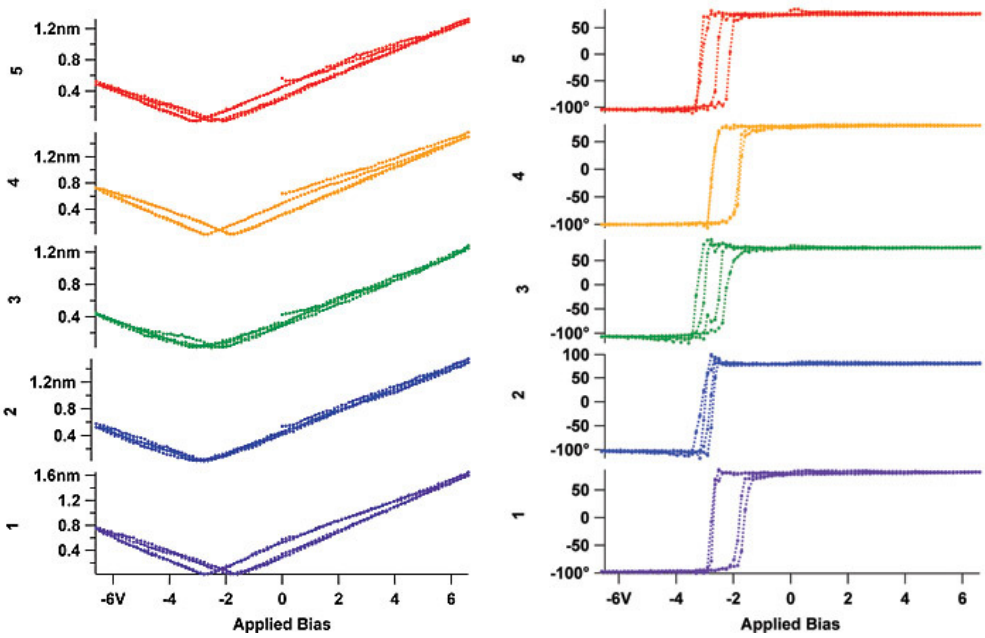


Figure 6.12.: Amplitude (left) and phase (right) hysteresis loops measured at five different locations on a PZN-PTi thin film.

troscopy 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, as required to prevent damage to biological systems, as well as stray electrochemical reactions[19].

The resonant frequencies are determined only by the weakly 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

[19] Frederix, PLTM et al., Assessment of insulated conductive cantilevers for biology and electrochemistry. NANOTECHNOLOGY, 16 AUG 2005 No. 8, ISSN 0957–4484.

[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 No. 1, ISSN 0021–8979.

[21] Garcia, R/Perez, R, Dynamic atomic force microscopy methods. Surface Science Reports, 47 2002 No. 6-8, ISSN

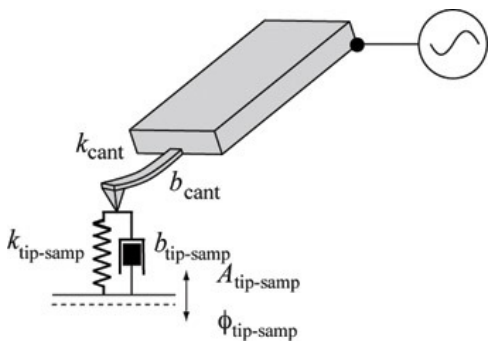


Figure 6.13.: In PFM, the cantilever voltage is modulated, usually at some fixed frequency. This causes the sample to distort at some amplitude and phase. Mediated by the contact mechanics, this drives the tip which, in turn, is monitored by the AFM sensor.

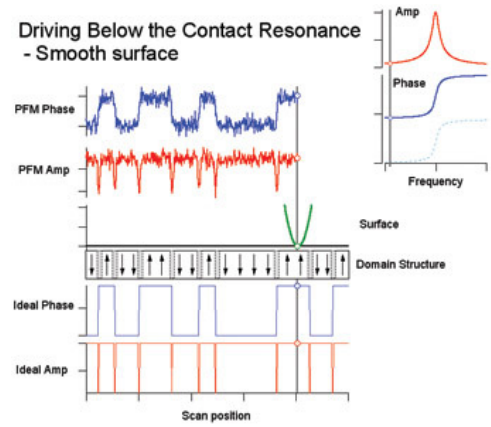


Figure 6.14.: This figure shows the ideal and measured PFM response of an idealized tip (green) scanning over a smooth surface (black line below the "tip"). The domain structure of the ferroelectric sample is shown below the surface where the arrows correspond to the sample polarization direction. The 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.

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

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

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

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

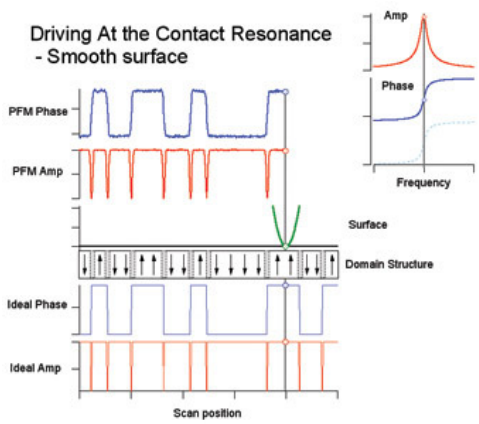


Figure 6.15.: This figure shows the same situation as described in Figure 6.14 on page 65, except that here we are using resonance enhancement to boost the small PFM signal. The inset frequency tune in the upper right corner shows the drive frequency. In this case, since the Q-factor of the resonance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

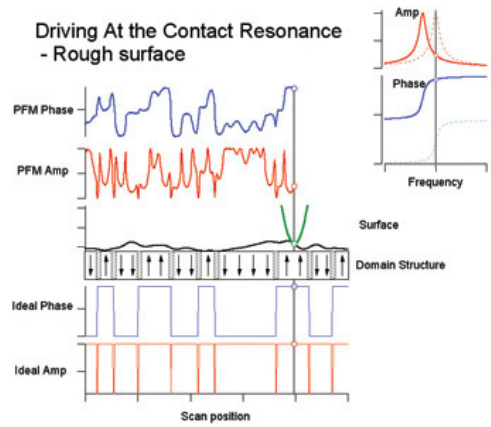


Figure 6.16.: This figure shows a practical limitation of using the contact resonance as the drive frequency. In conventional PFM systems, the contact resonance can change by 10-30 kHz over the course of imaging a rough sample. Typical cantilevers have a full-width half max of 4-10 kHz meaning the phase 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 piezo response in the sample surface ($A_{\text{tip-samp}}, \varphi_{\text{tip-samp}}$). 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 ($k_{\text{tip-samp}}$) will typically change significantly, which in turn,

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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}\approx$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 since they are 180° out of phase. However, these experimental conditions are very rare.

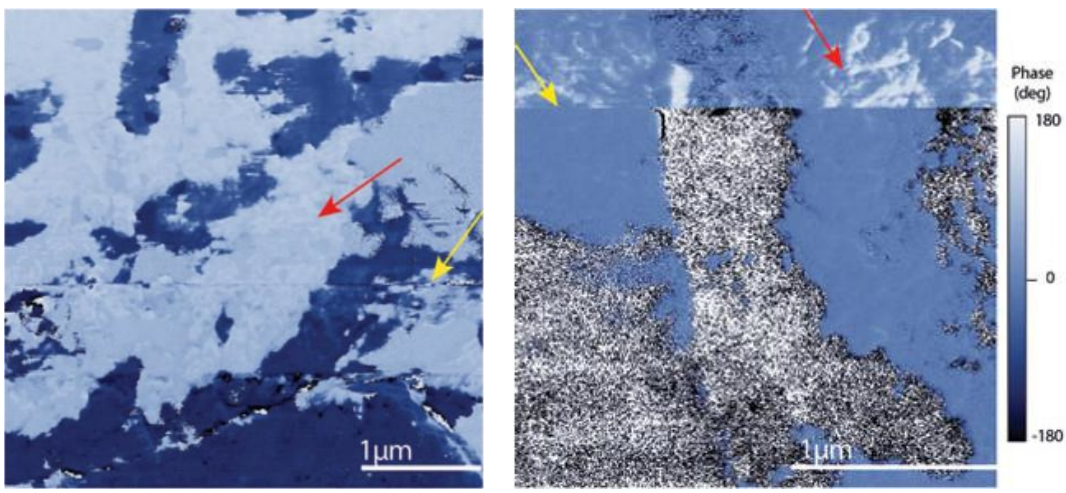


Figure 6.17.: PFM phase channel on a polished PZT sample. The cantilever was driven near the contact resonance to enhance the SNR. There is significant crosstalk between the sample topography and the PFM signal. Red arrows indicate "roughness" where the contact stiffness changes the measured phase. The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

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.

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 con-

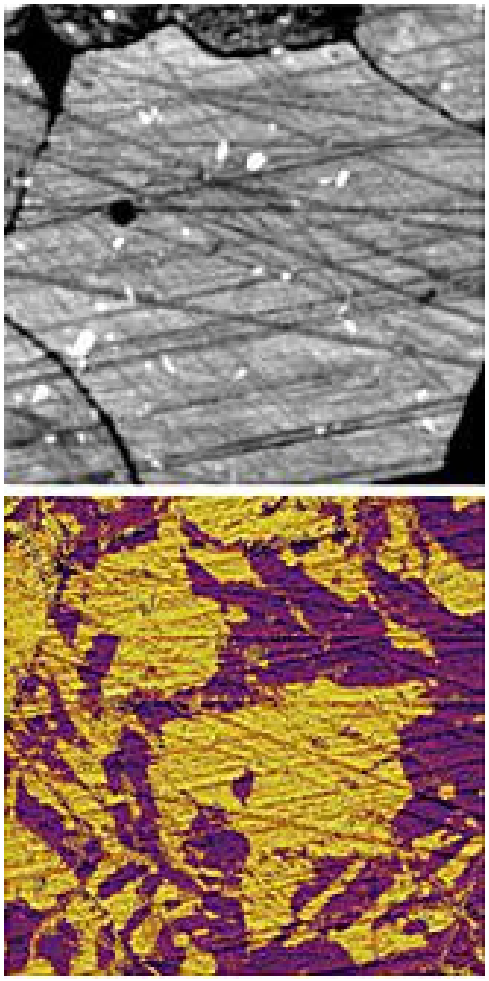


Figure 6.18.: PZT showing crosstalk, 14 µm scan.

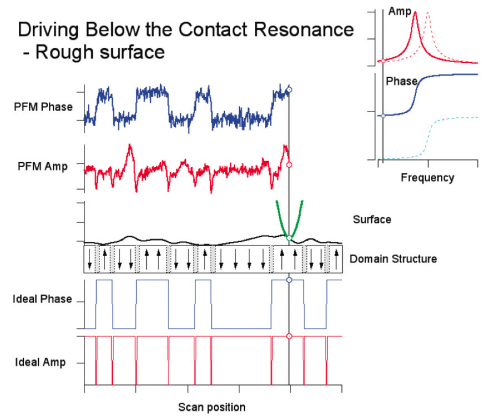


Figure 6.19.: Driving below contact resonance with conventional PFM. Here, the cantilever is driven well below the contact resonant frequency. The effects of surface roughness are minimized, though still visible in the measured PFM amplitude. However, this reduction in crosstalk comes at the high price 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.

sequence, 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 therefore can 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, since 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 that probably originates 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 in particular 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

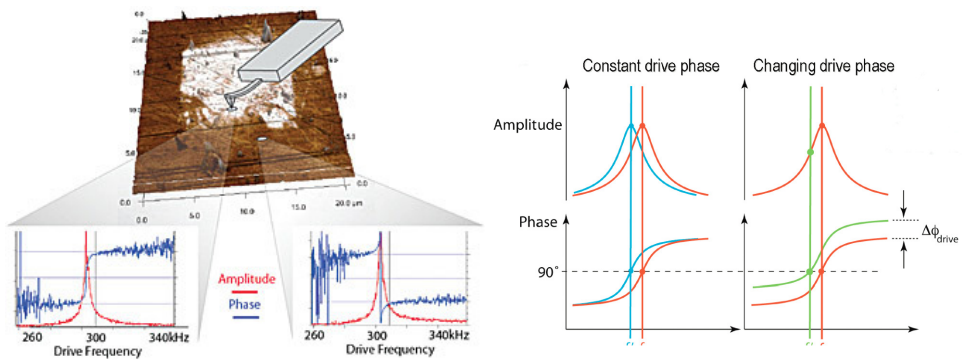


Figure 6.20.: For domains with an antiparallel (180°) orientation, conventional PLLs drive the PFM frequency away from resonance. (Top) Amplitude, red, and phase, blue, cantilever response over antiparallel domains. In the measurement, phase is offset by 180° over anti-parallel domains (see curves on 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.

Figure 6.20 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 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

[22] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007 No. 47, ISSN 0957–4484.

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, requires that we continually adjust the drive frequency to keep it at the contact resonance. If one 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 No. 47, ISSN 0957–4484.

[24] Kalinin, Sergei/Jesse, Stephen/Proksch, Roger, Information acquisition & processing in scanning probe microscopy. R&D MAGAZINE, 50 AUG 2008 No. 4, ISSN 0746–9179.

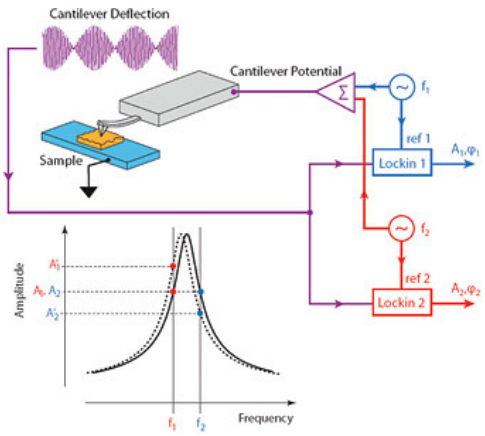


Figure 6.21.: Schematic diagram of DART, showing a drive phase independent feedback signal.

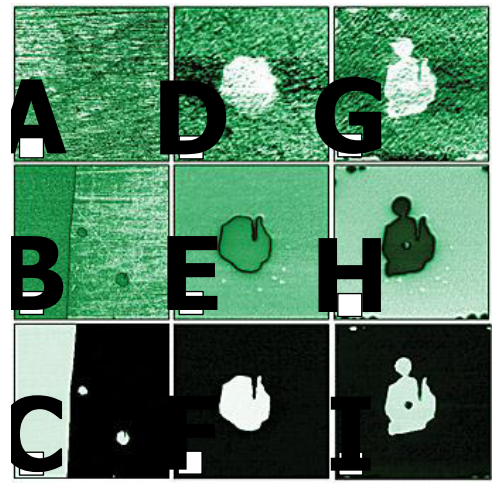


Figure 6.22.: (A), (D), (G) Resonance frequency, (B), (E), (H) piezoresponse amplitude and (C), (F), (I) piezoresponse phase images of antiparallel domains in lithium niobate. Shown are images of the (A)–(C) native domain structure, (D)–(F) an intrinsic domain and (G)–(I) domains switched by ±176V (locations 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

[25] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007 No. 47, ISSN 0957–4484.

[26] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007 No. 47, ISSN 0957–4484.

shown Figure 6.22A. The PFM amplitude and phase images show a macroscopic 180° domain wall and two inversion domains which 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 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 Band Excitation 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.

Figure 6.25 reprinted with permission[30].

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.

[27] Rodriguez, Brian J. et al., Dual-frequency resonance-tracking atomic force microscopy. NANOTECHNOLOGY 18 NOV 28 2007 No. 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 No. 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 No. 43, ISSN 0957–4484.

[30] 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 No. 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 No. 43, ISSN 0957–4484.

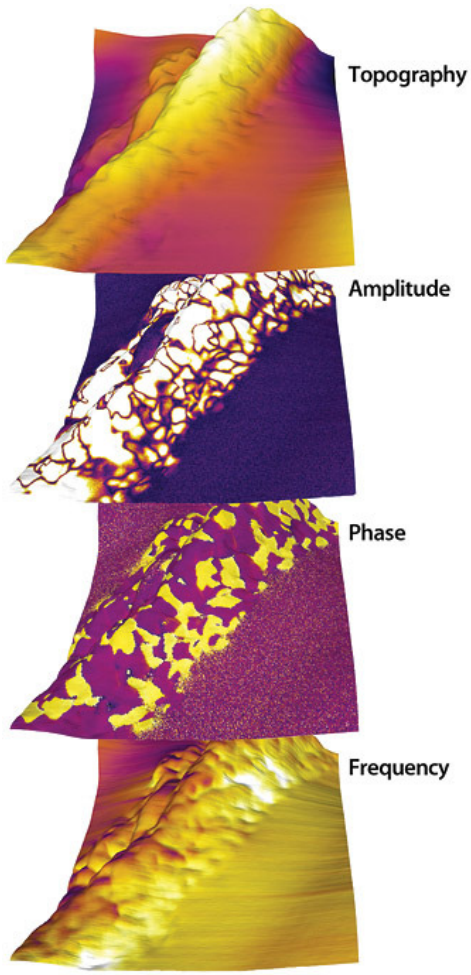


Figure 6.23.: PFM of multiferroic $BiFeO_3$ nanofibers, 1 µm scan. Collaboration with Shuhong Xie, Xiangtan University, China and JiangYu Li, University of Washington.

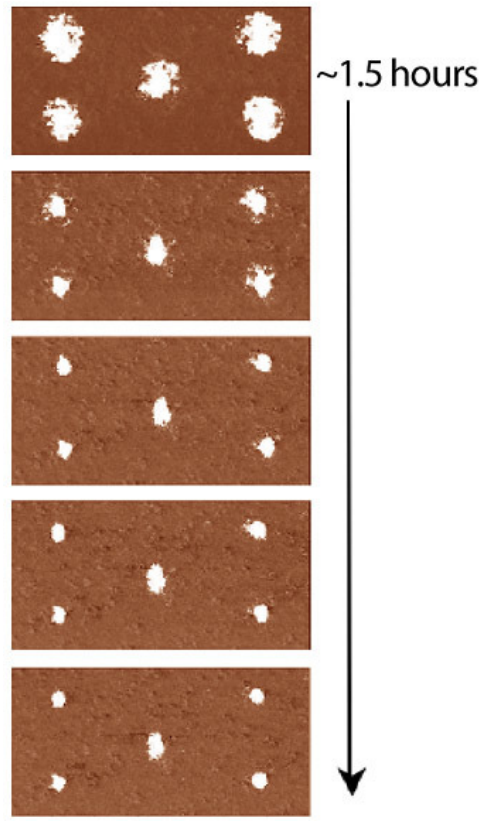


Figure 6.24.: Stable imaging using DART allows relaxation studies. This series of images shows the relaxation of sol-gel taken at different intervals for approximately 1.5 hours. 3.5 µm scan.

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

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

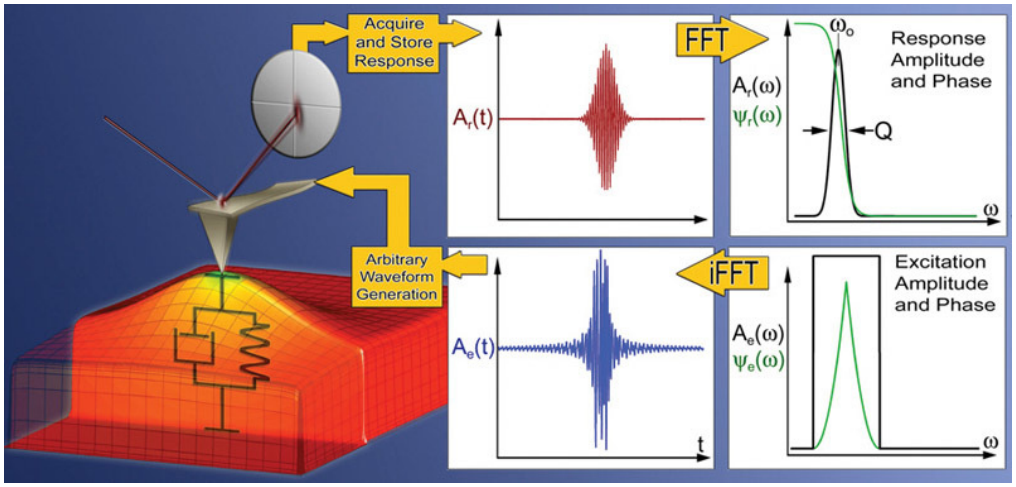


Figure 6.25.: Operational principle of the BE method in SPM. The excitation signal is digitally synthesized to have a predefined amplitude and phase in the given frequency window. The cantilever response is detected and Fourier transformed at each pixel in an image. The ratio of the fast Fourier transform (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, that are plotted to yield 2D images, or used as feedback signals.

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 cross-talk, 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.

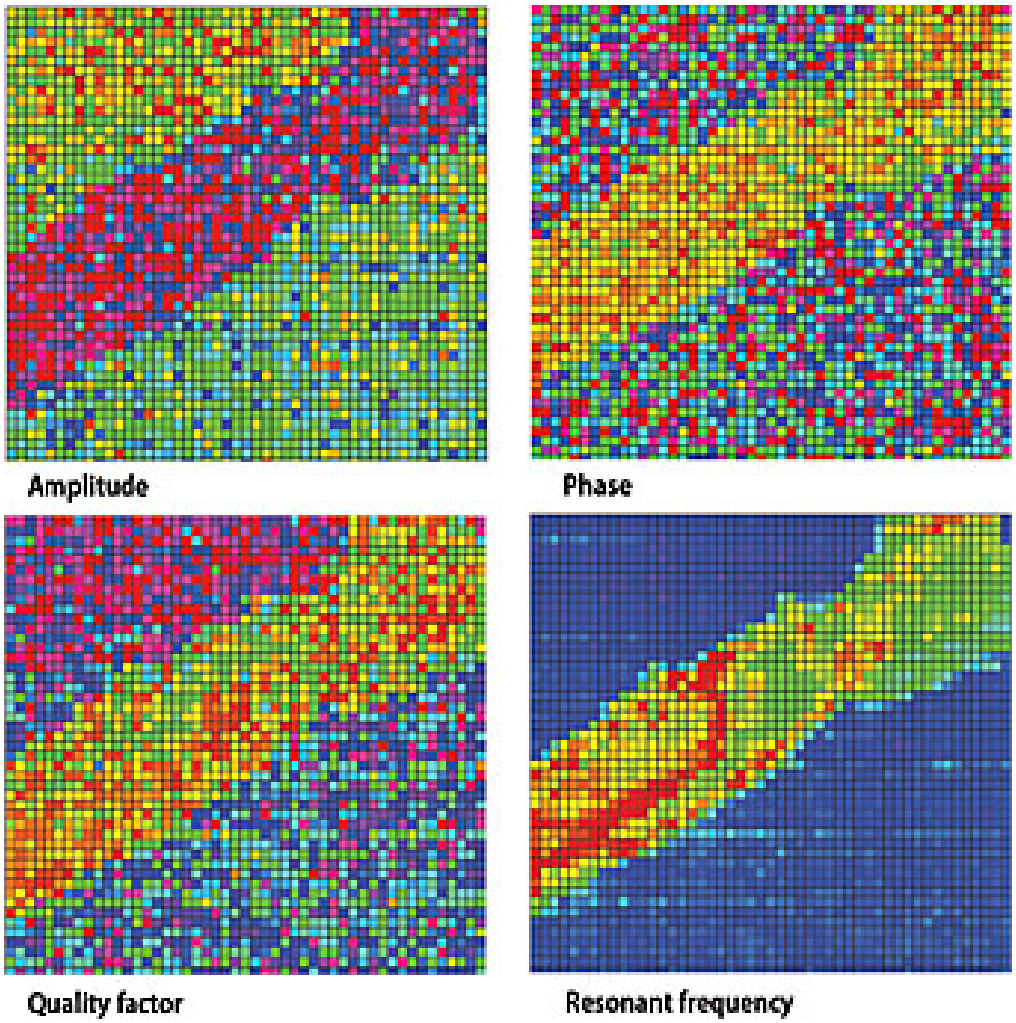


Figure 6.26.: Amyloid fibril (bovine insulin) on mica imaged in water using BE-PFM technique, 250nm x 250nm. Image courtesy of G. L. Thompson, V. V. Reukov, A. A. Vertegel, M. P. Nikiforov, Clemson University, Dept. Bioengineering, and S. Jesse, S. V. Kalinin, Oak Ridge National Lab.

6.6.2. High-Speed PFM (HSPFM)

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 No. 7, ISSN 0003–6951.

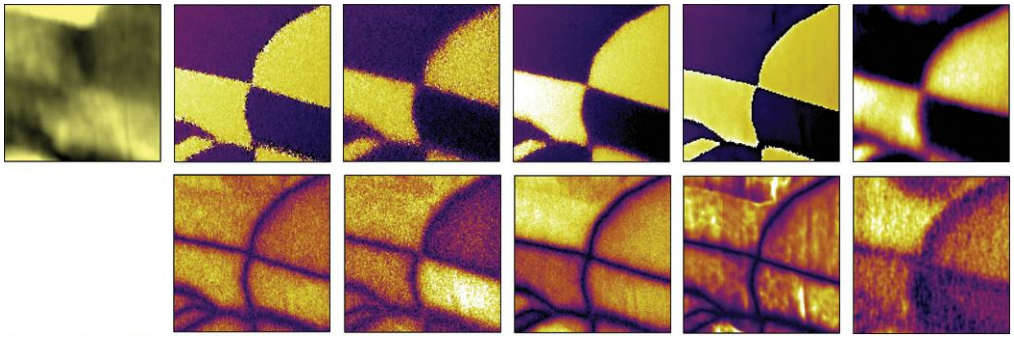


Figure 6.27.: High Frequency PFM using Asylum's fast photodiode on a ceramic PZT sample at different frequencies (phase left, amplitude right) – below first resonance (top row) and at cantilever resonances (all others) using a MikroMasch NSC 35B cantilever. 1µm scans. Image courtesy of K. Seal, S. Kalinin,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

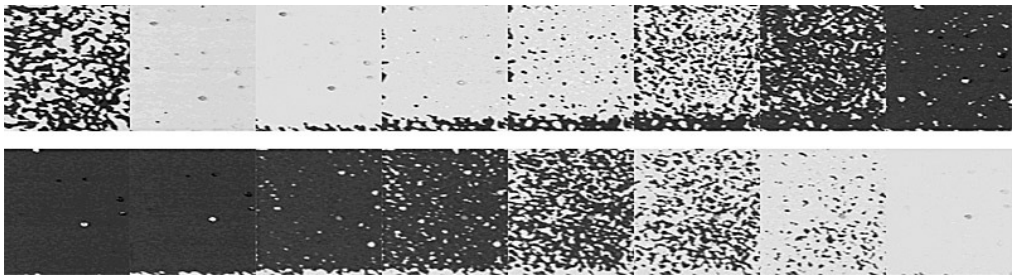


Figure 6.28.: This image sequence (left to right, top to bottom) is excerpted from a movie of 244 consecutive High Speed PFM images (4 µm scans) depicting in situ ferroelectric memory switching. For the first half of the movie, the tip is biased with a positive DC offset throughout the measurements. 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 and, 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.

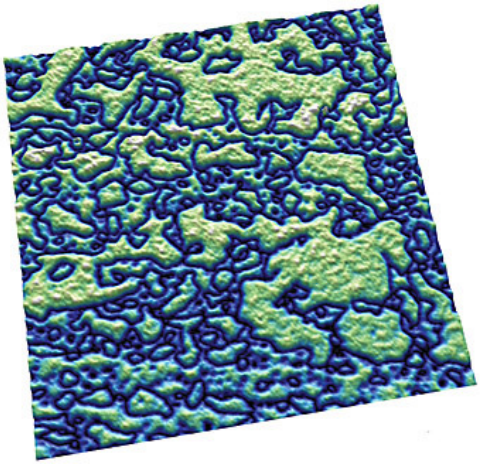


Figure 6.29.: (001) domains in a PZT thin film, 3.8µm scan. Image courtesy N. Polomoff and B. D. Huey, University of Connecticut Institute of Materials Science. Sample courtesy R. Ramesh, UC Berkeley.

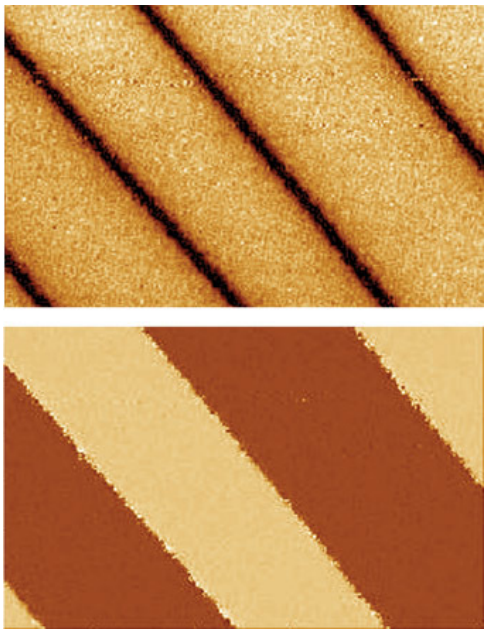


Figure 6.30.: PPLN amplitude (top) and phase image (bottom) acquired with the MFP NanoIndenter, 50µm scan.

The ability to probe forces and directly image the piezo response 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, measuremed 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 No. 3, ISSN 0884–2914.

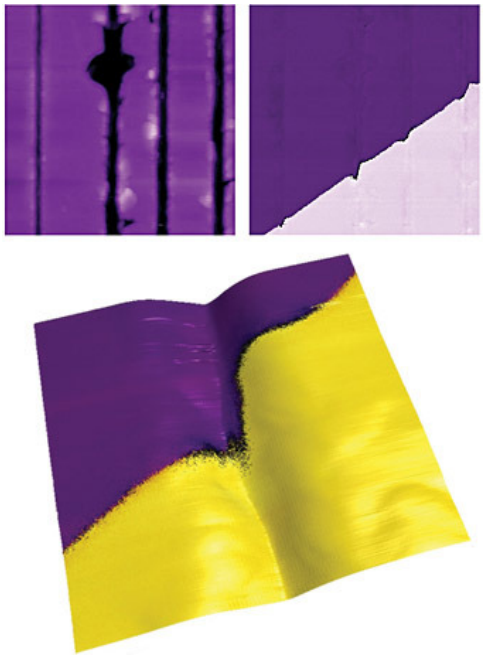


Figure 6.31.: Surface topography of PPLN after it has been purposefully scratched with different loading forces using the NanoIndenter, 10µm scan (top images). 1µm scan (bottom).

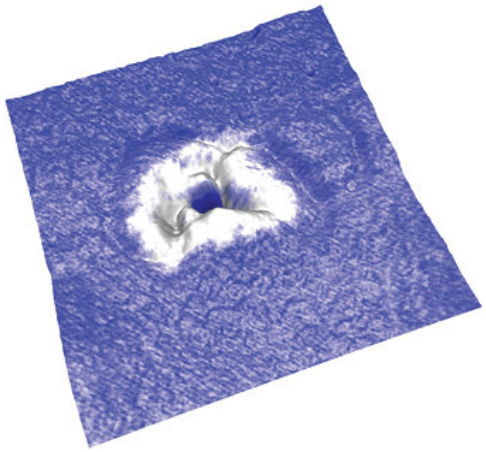


Figure 6.32.: Zoom of the top surface of a red blood cell. The surface shape was rendered to show the topography while the phase channel is overlaid on top to show piezo response. A small sub-micron region on top (white) of the cell exhibited a much different piezo response than the surronding cell surface. 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

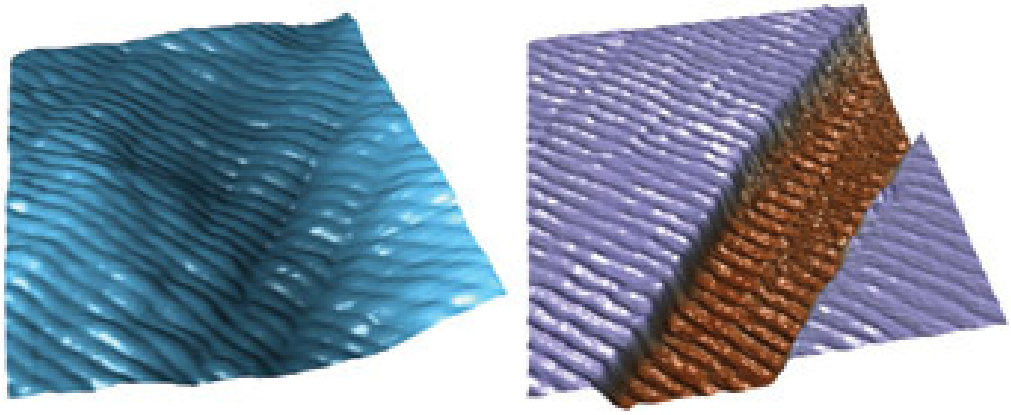


Figure 6.33.: Topographic (top) and PFM phase (bottom) images of collagen fibers, 1.4μm scan. Image courtesy D. Wu and A. Gruverman, UNL. Sample courtesy G. Fantner.

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 piezo response.

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 No. 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 No. 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 No. 1.

[38] Giannakopoulos, AE/Suresh, S, Theory of indentation of piezoelectric materials. Acta Materialia, 47 MAY 28 1999 No. 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 No. 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 No. 3, ISSN 0141–8637.

[41] Kalinin, SV/Karapetian, E/Kachanov, M, Nanoelectromechanics of piezoresponse force microscopy. PHYSICAL REVIEW B 70 NOV 2004 No. 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 No. 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 No. 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 No. 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 No. 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 No. 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 No. 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. InEncyclopedia 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 No. 22, ISSN 0003–6951.

[52] Kalinin, Sergei V. et al., Local bias-induced phase transitions. MATERIALS TODAY, 11 NOV 2008 No. 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 in order 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.

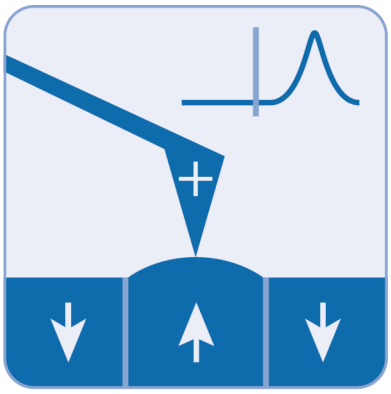
Switching Spectroscopy Mapping A quantitative measurement that reveals local switching characteristics for real-space imaging of imprint, coercive bias, remanent and saturation responses, and domain nucleation voltage on the nanoscale.

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)

CHAPTER REV. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



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.

In the following, we describe a method of using the contact resonance of the cantilever 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 lockin amplifier, the amplitude and phase of the response can be measured.

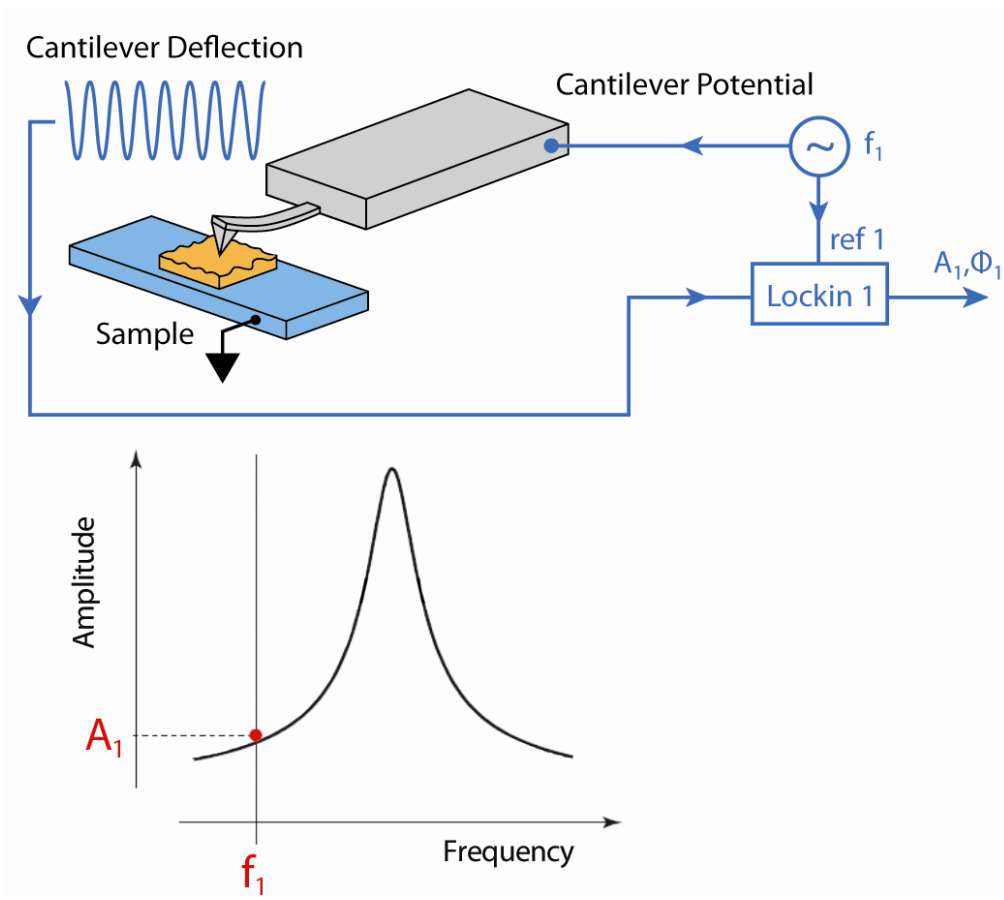


Figure 7.1.: A diagram of Single Frequency PFM

The use of contact resonance to boost small piezo signals is more complex than might be expected at first examination 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, 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 **??**.

7.1. Choosing a PFM Cantilever

Cantilevers for PFM should generally 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.

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 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-NCHR	800-1,200 kHz

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

7.2. Single Frequency PFM

It is assumed in this instruction set that the user is proficient with basic AFM operation. This technique uses the standard cantilever holder for low-voltage PFM or a high voltage option for your microscope.

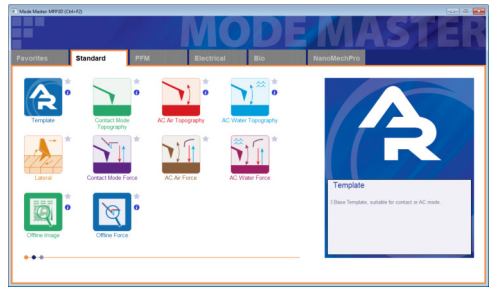
7.2.1. Sample and Cantilever

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 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* ▷ *Single Freq PFM*
   - The screen will now re-arrange and 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 put 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.

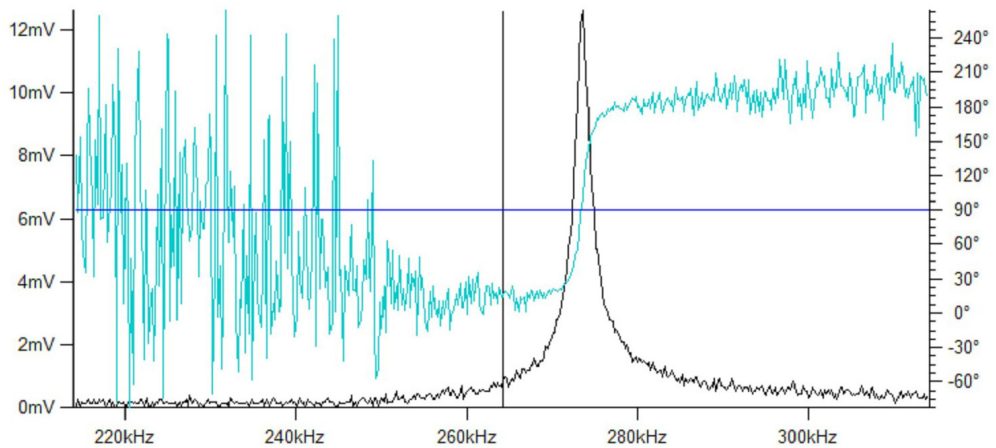
7. **Engage the Surface**

   - 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 on *Set Drive Frequency*. Your goal is to select a frequency close to but not on the peak to allow enough amplification but to avoid effects by changes in contact resonance.

4.



Center Phase

- Click on *Center Phase Offset* and put the resonance at 90°.

7.2.4. Imaging

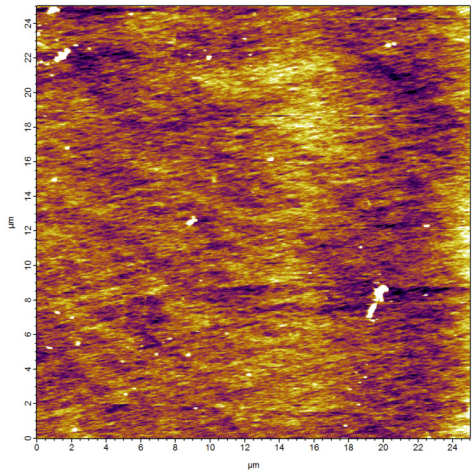
At this point, you're ready to image. Since PPLN the domains are typically many microns across, it can be advantageous to scan a larger size. Also note – you will have more reproducible results if you scan at 90°.

1. Hit *Do Scan* to start imaging.

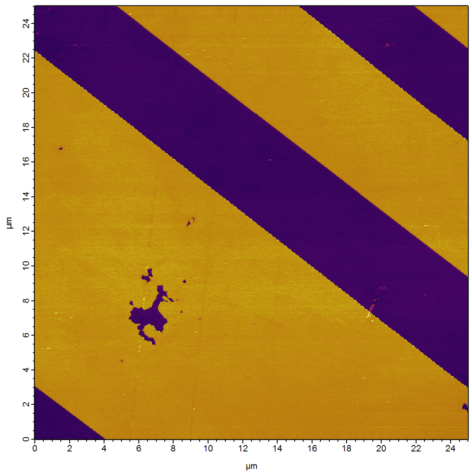
Inpsecting data

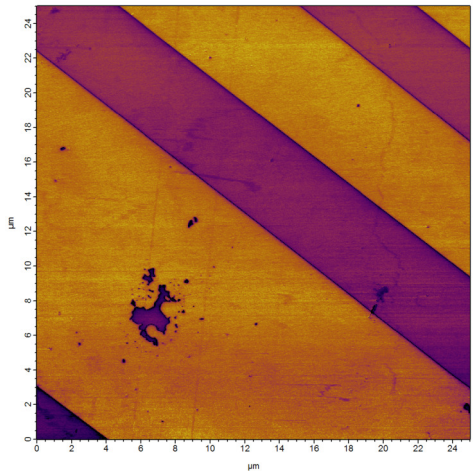
- If all is well at this point, you should see some 4-10µm periodic signals on the amplitude, phase and frequency channels corresponding to the poled domains in the PPLN sample. You can also adjust drive voltage to optimize images.

Clockwise, starting from the right, Topography, Amplitude, and Phase.



2.



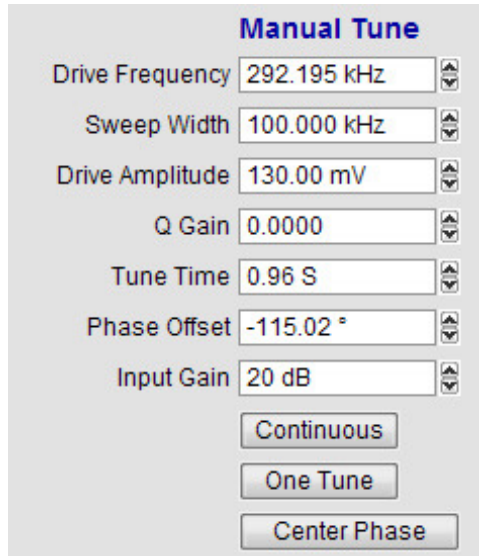


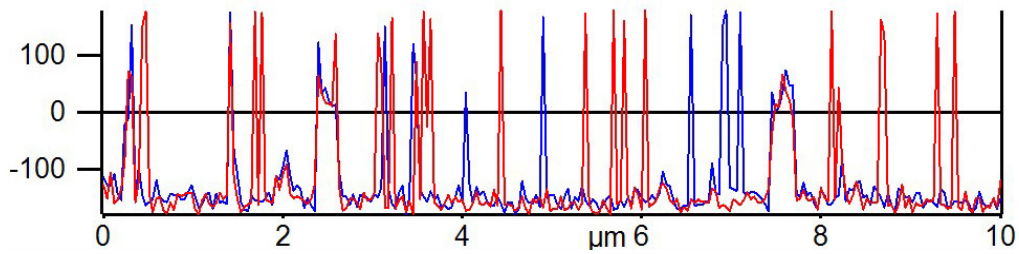
3. A common problem is the loading force being too small. If you have tried adjusting the drive voltage and still do not see good domain contrast, try increasing the loading force. Click *Stop* and decrease the deflection with the PD knob (or PD motors on Cypher), then retune on the surface. The contact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

4.

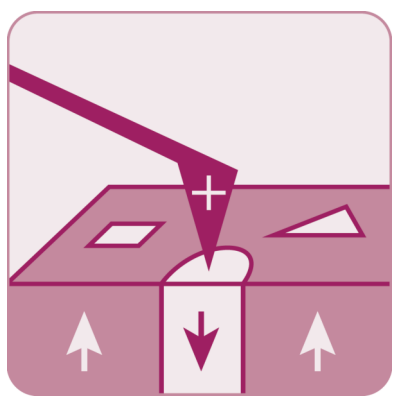
Phase Wrapping

- If phase wrapping occurs (PFM phase goes below -180º or above 180 º) you will see white spots in the phase image. The scope trace below shows these spots as peaks.
- Adjust the *Phase Offset* during scan from the 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 **??**.

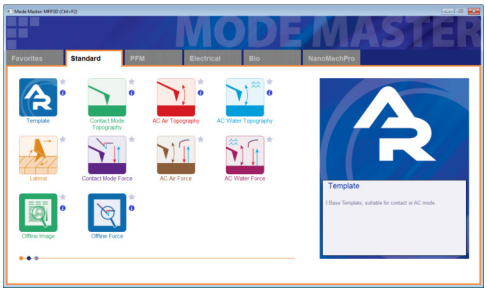
In the following example, lithography is done on a Sol Gel PZT using the High Voltage PFM option. Using the High Voltage holder, a bias of 13.2V was used to guarantee polarization reversal.

7.3.1. Software Instructions

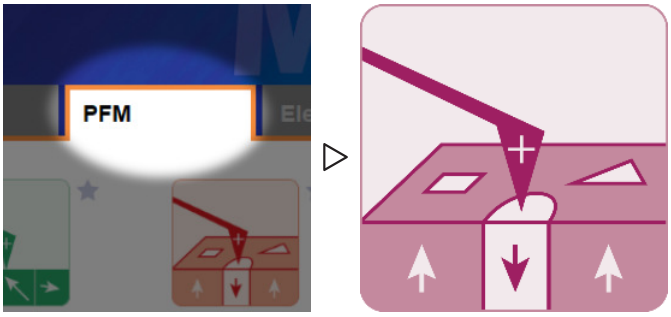
1.

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.



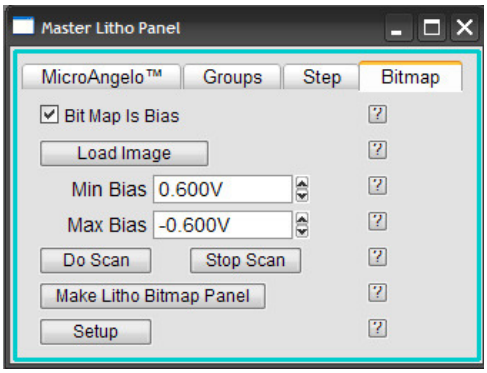
Select Mode:

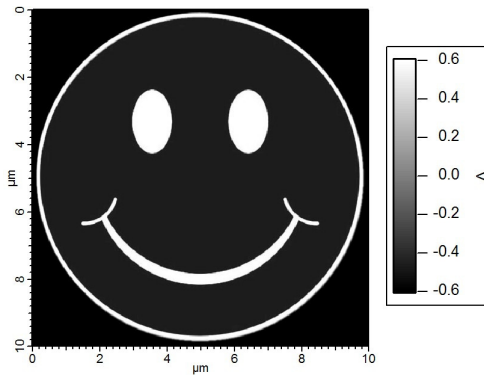
- Select *PFM tab* ▷ *Litho PFM*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

3.

Loading Bitmap Image

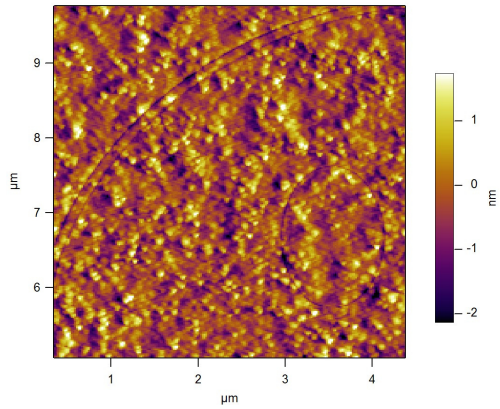
- Click *Load Image* and select a file. The bit-mapping lithography requires a 256 x 256 or 512 x 512 image. A file named *Smile.jpg* is used for this example.
- You will need quicktime installed to load the graphic into Igor `http://www.apple.com/quicktime/download/`.
- You can download this jpg from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





4. Click *Do Scan* to start the bitmap bias lithography.

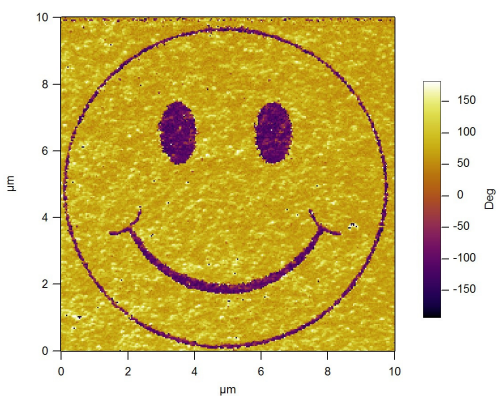
5.

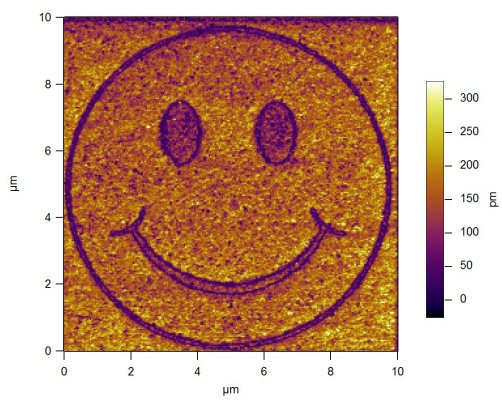


Writing Bitmap

- 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 in the above right image shows the pattern appearing in the deflection during a lithography scan, a good sign that lithography is working

6.



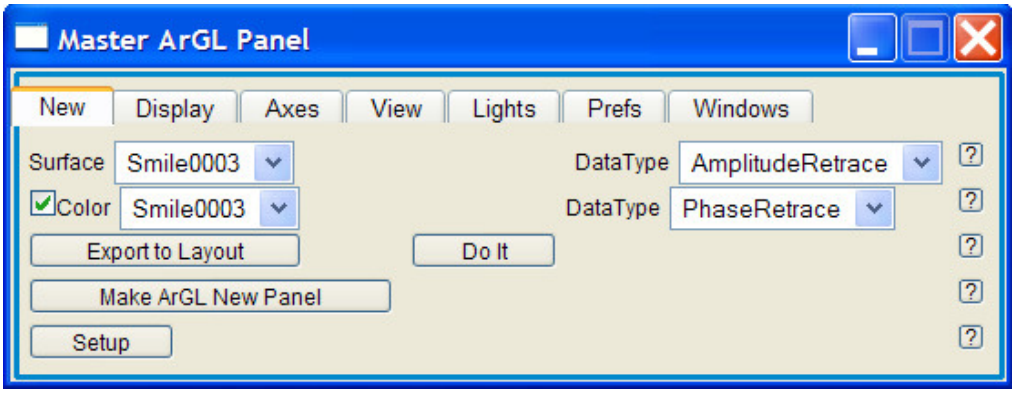


- Switch to the tune panel and tune for single frequency PFM and image as normal. Left image is Amplitude, right Phase.

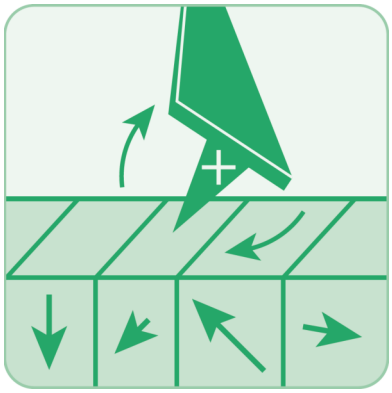
7. **3D Overlay**

- A nice 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*.

Right, 3D Overlay image, bottom 3D Panel.
See also AR Software Manual:Argyle 3D:Overlaying Data on a Surface.

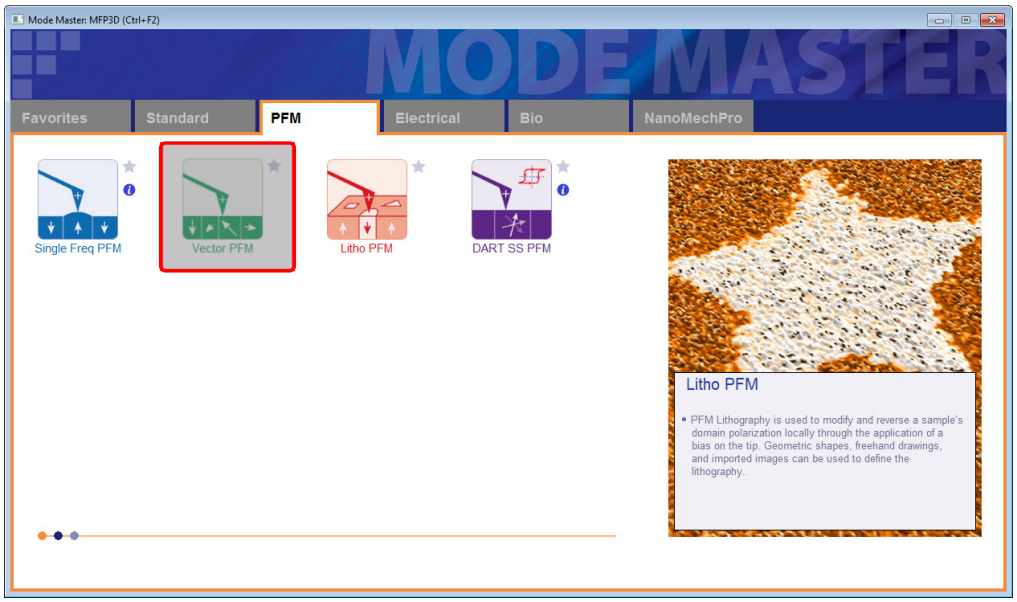


7.4. Vector PFM



Lateral PFM (Vector PFM) is a two-pass technique (Chapter 10 on page 134) 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.

1.



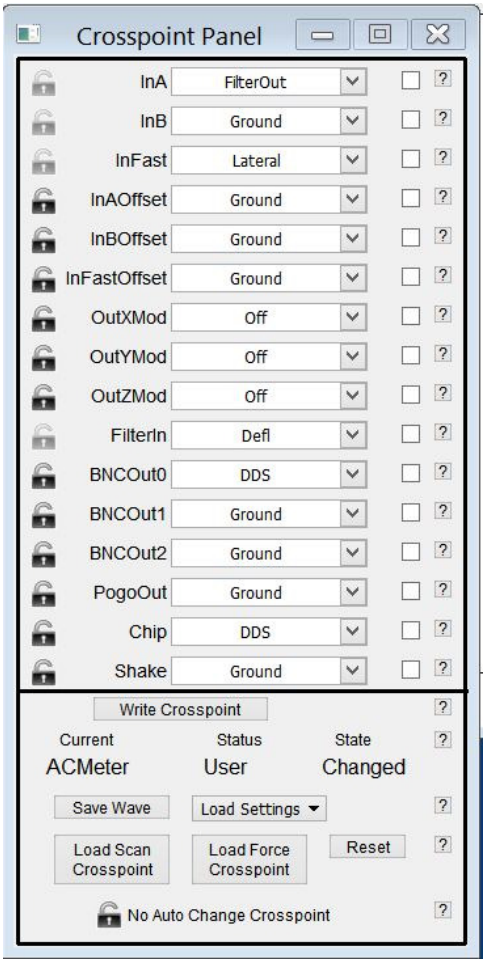
Mode Master

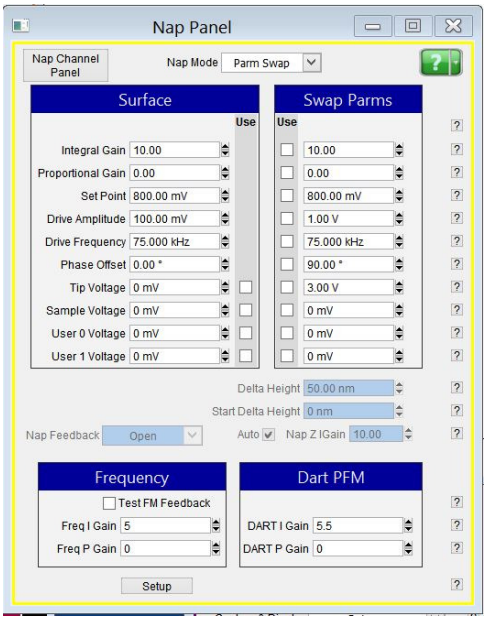
- When the **Mode Master** window appears, choose the **PFM** tab.
- Select the **Vector PFM** mode icon. All the necessary panels for Vector PFM operation will now automatically load and arrange on the desktop.

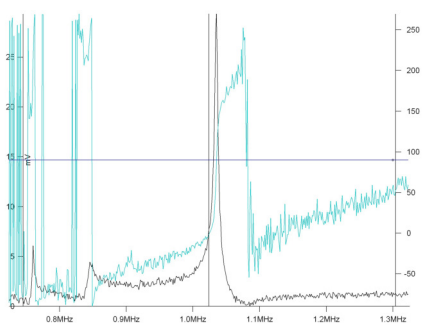
Lateral Tuning MFP-3D

- To tune for the lateral signal, the cross point switch must be manually changed to *InFast* ▷ *Lateral* , click *Write Crosspoint* and select *No Auto Change Crosspoint*
- Engage and tune as normal, the resonance frequency is usually around 1MHz.
- Manually copy the *Drive Frequency, Drive Voltage and Phase Offset* from the Surface parameter fields of the Nap Panel to the corresponding Parm Swap fields. The surface values will be changing in the next step.
- Make sure to unlock the crosspoint.

2.



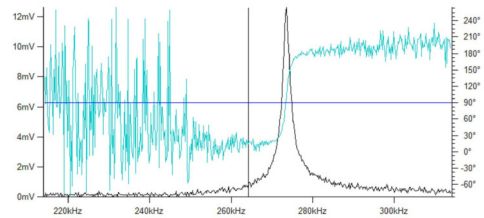




3.

Vertical Tuning

- To tune the vertical deflection signal, change *Infast* to *ACDefl*, click *Write Crosspoint* and make sure *No Auto Change Crosspoint* is not selected.
- Type in the *Drive Amplitude*, hit *Engage*, *One Tune* and 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.

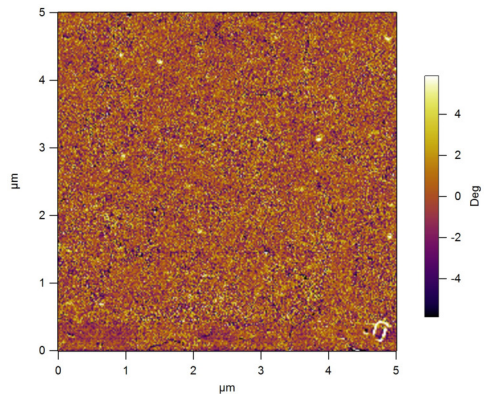


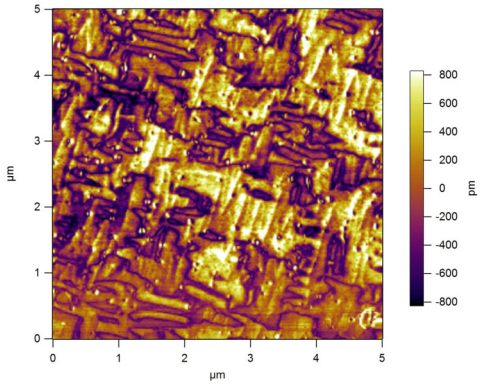
4.

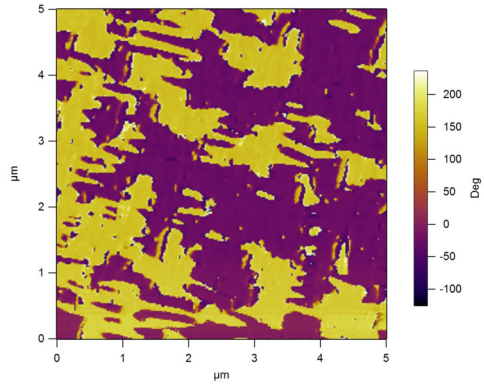
Scan

- Hit *Do Scan* to start imaging.

Clockwise, starting from the right, Vertical Phase, Lateral Phase and Lateral Amplitude.

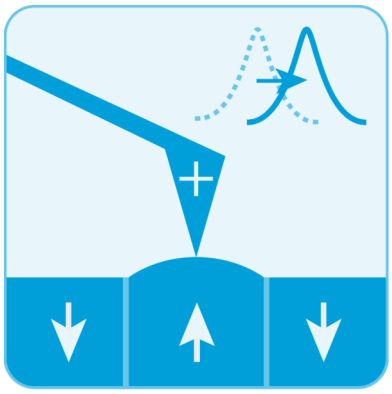






8. Piezo Force Microscopy (PFM) using Dual AC Resonance-Tracking (DART)

CHAPTER REV. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



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, 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 will go through:

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 No. 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 No. 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 No. 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 100 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.

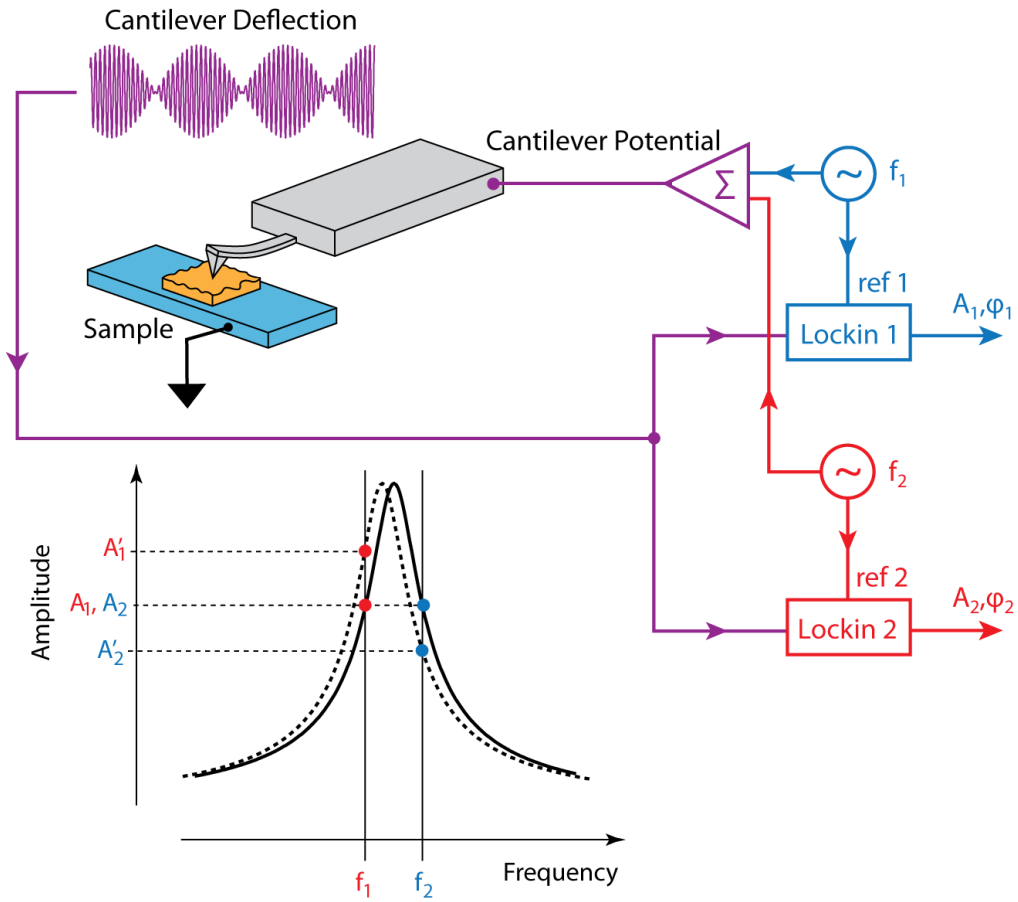


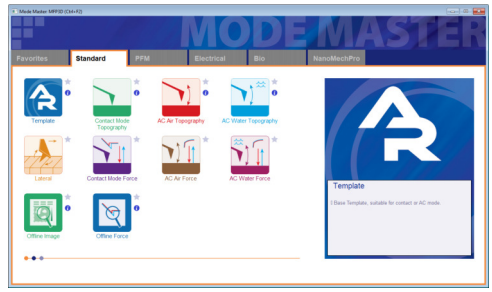
Figure 8.1.: Dart Operation

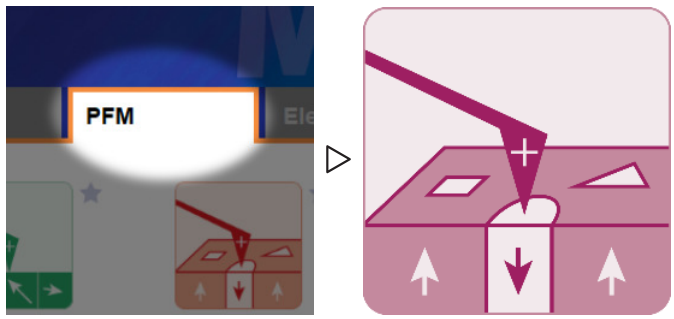
8.2. Choosing a cantilever and starting a DART template with ModeMaster

It is assumed in this instruction set that the user is proficient with basic AFM operation. This technique uses the standard cantilever holder for low-voltage PFM or the HV option.

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 will now re-arrange and present all the controls necessary for this type of AFM imaging.

Note Loading **DART SS PFM** mode will also load the template for point switching spectroscopy (SS PFM) measurements. You may not need to perform this for PFM with DART imaging, but will cover this technique separately (see Section 8.4 on page 107).

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 generally 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

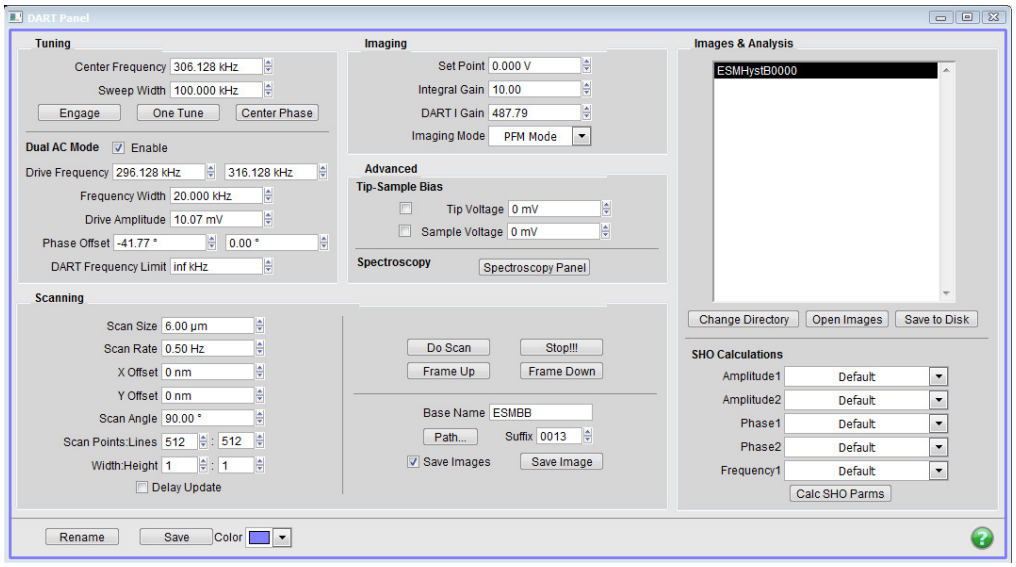


Figure 8.2.: The DART panel.

Note	These instructions are for software version 13 or later. 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 starting the AR AFM software from scratch, or by selecting *File ▷ new AFM template* from main menu bar.
- From the Mode Master window click on *PFM.*
- From the next window click on *DART-SS-PFM* (See Section 8.2 on page 100).

Note If the Mode Master does not appear, click [ctrl+F2], or select *User Settings ▷ Mode Master* from the main menu bar.

2. **The Dart Panel:**
   - The panel in Figure 8.2 on page 102 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 Area* of the DART panel:
     - Set the drive amplitudes to 10 mV, which 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):
     - Typically, you will want to record *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.

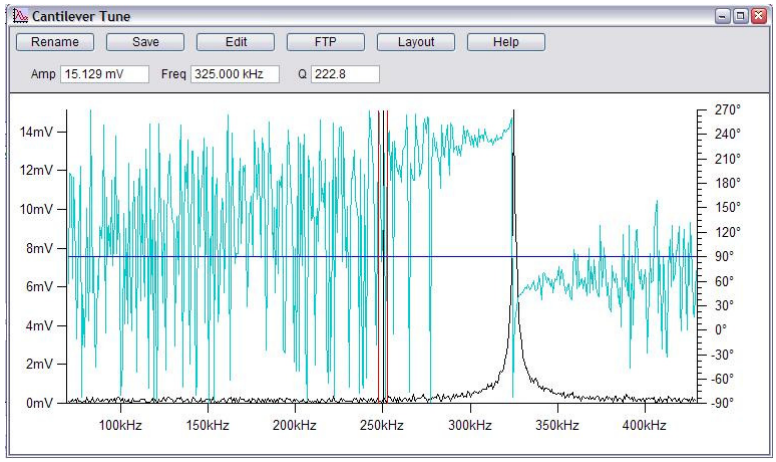
7. Maximize the sum, and then put deflection at approximately -0.5V. We typically operate at a setpoint of 0V to take advantage of the optional 10x gain on the photodetector signal (very useful when measuring the tiny motions in PFM).

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.
   - MFP3D Only: Take care to insure 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.

9.

Engaging the Surface

- Hit 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 for contact resonance for a few different cantilevers. We'll assume you are starting with an AC240.

Note	To find the contact resonance of an unfamiliar cantilever, do at thermal tune off the sample surface (Master Panel->thermal tune tab->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 in Figure 8.2 on page 102 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.
- Hit the *One Tune* button and you should see a peak.

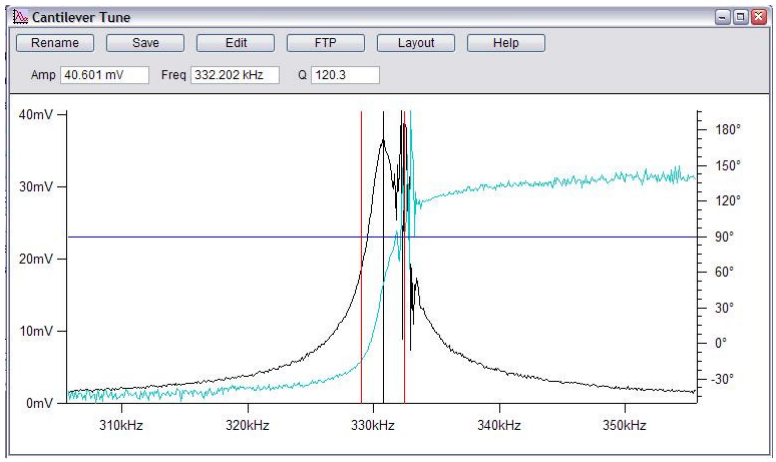
Note If no clear peak is visible, as in the graph above, try widening the sweep width and gently increasing the drive voltage. For an AC240, once the drive goes above ~5 volts you may start to degrade the tip quality. For PPLN and lead zirconium titanate (PZT) samples, 200mV is usually sufficient to see the peak but 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 click on *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 lockins and means things are functioning normally.

12. In the tuning portion of the DART panel click on *Center Phase*, this sets the resonance at 90°. You should see *amp1* and *amp2* 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, so if in doubt, tune again!

13.



Successful tuning result

- The shape of the curve is nice, the amplitude at the two drive frequencies are between 10 and 20mV. The squiggles in the amplitude and phase around the second frequency are from interference between the first and second lockin as the first lockin 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

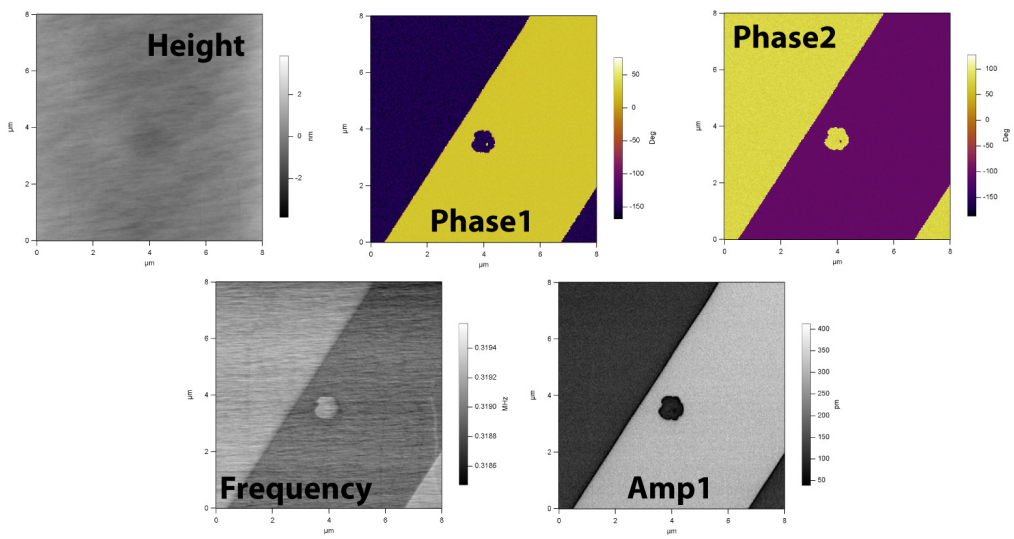
- In the Scanning section of the DART panel 8.2 we find it useful to start with a reasonably large scan range (perhaps 20 by 20 µm).
- Set the scan angle to 90° (generates more reproducible results).

15.

Set Imaging Parameters and Start Imaging

- Hit *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 figures 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

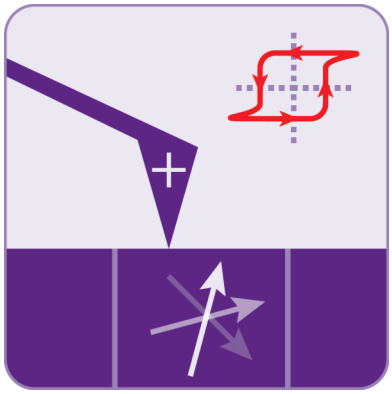
- Right-Click on one of the images and choose *Fix All Scales*.
- Depending on the balance of electrostatic and piezo forces, you may see more or fewer signals split between the amplitude and the phase channels.
- If frequency tracking is smearing or trace and retrace look very different, the *DART I Gain* should be increased. Don't be afraid to play here, 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 hit *Stop*, 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)



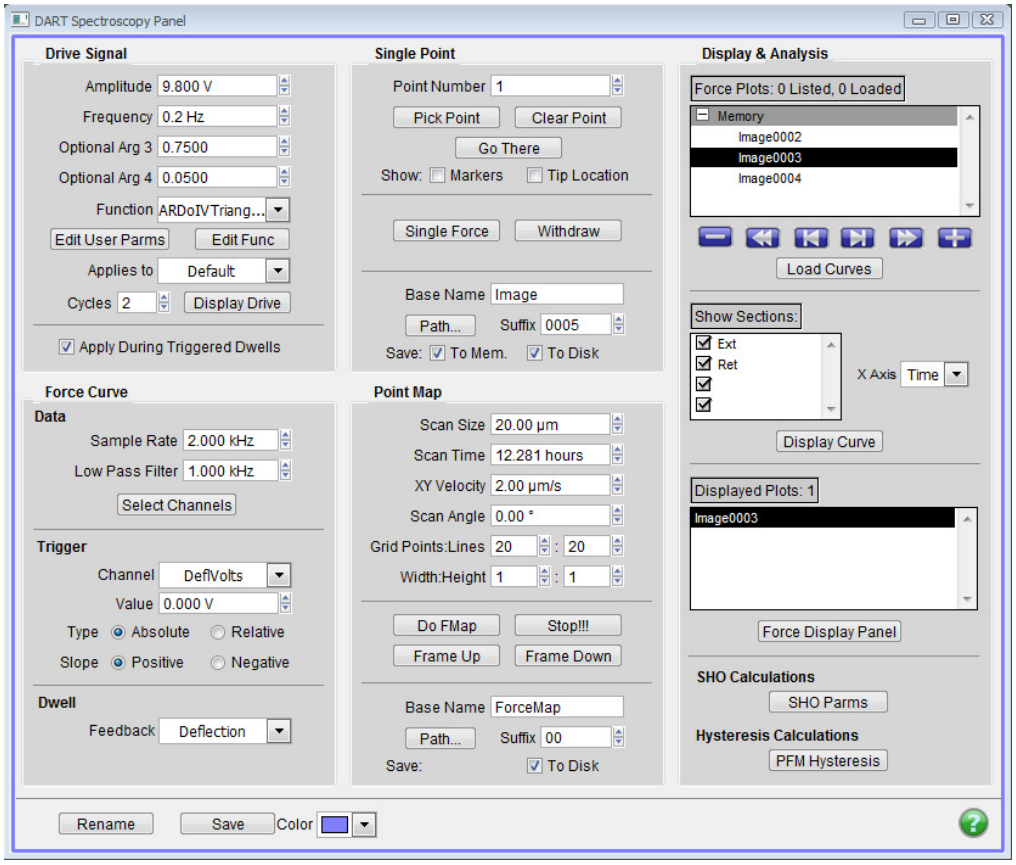


Figure 8.3.: The DART Spectroscopy Panel

In the following, we will measure point hysteresis loops. We will 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 which 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 109 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 2 times the oscillator drive amplitude when calculating this. It is generally 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 some examples see Figure 8.5 on page 109.

[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, with 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, you will need a high voltage option to see hysteresis loops. With the standard configuration you will not see the loops.

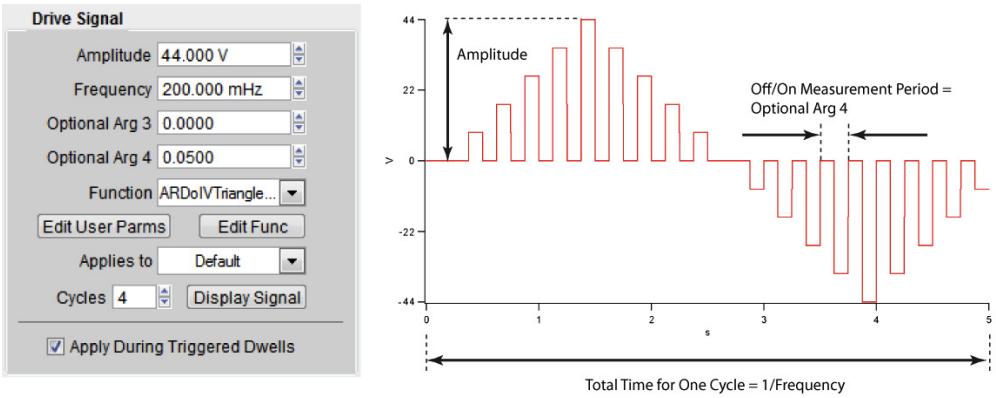


Figure 8.4.: PFM drive waveform parameters

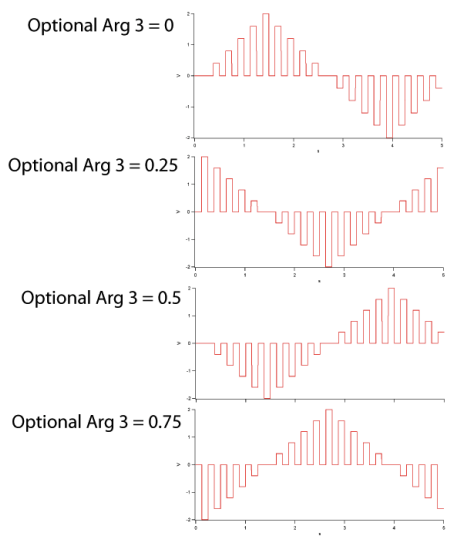


Figure 8.5.: Some examples of the effect of Optional Argument 3

8.4.1. Dart PFM Spectroscopy Example

1. The *DART I Gain* for spectroscopy should be smaller than values used for imaging. A good number to start with is 10.
2. On the Spectroscopy Panel panel (Figure 8.3 on page 108), go to the *Drive Signal* group of controls.

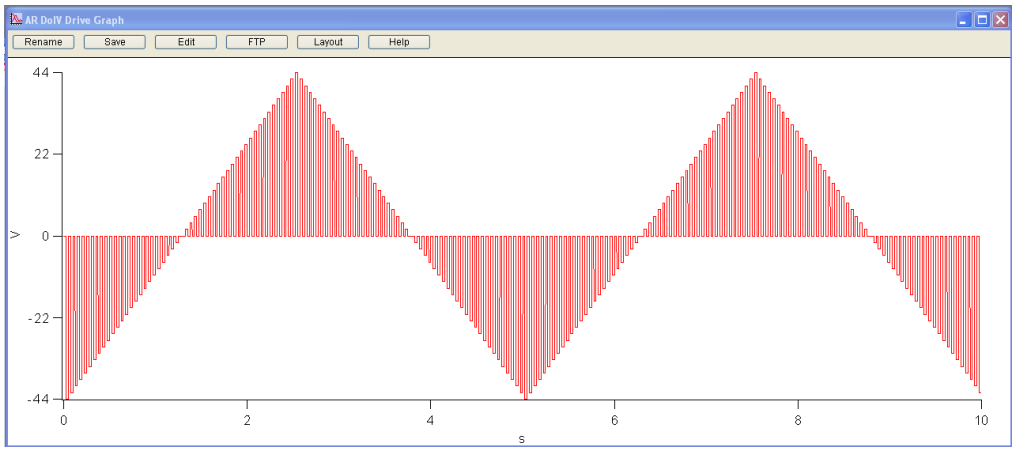


Figure 8.6.: A typical scan waveform for measuring in-field and remnant hysteresis loops for SS-PFM.

3. Click on the *Display Drive.* This brings up a voltage versus time display for the loop measurements. This is useful for ensuring you are applying the proper waveform.
4. Select the *ARDoIVTriangleSquare* waveform in the *Function* pull down menu. This waveform will generate applied steps with a ramping maximum value. It will generate 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* group of controls. For measuring hysteresis loops, we use some of the machinery used in measuring force curves and force volumes.
   a) In *Data* section, by default, Amplitude1, Phase1, and Frequency are selected versus Time. You can check this by clicking *Select Channels* .
   b) *Trigger* is set to *DeflVolts* and should have a absolute value set to the setpoint that was used for imaging, typically zero volts.
   c) *Dwell Feedback* is set to Deflection, this will enable the feedback loop keeping the cantilever deflection constant during the hysteresis loops.
   d) Now select a point or points for the hysteresis loops. Select a reference image by clicking on it to bring it to the top.
      i. Click on the *Pick Point* button.
      ii. On the reference image, position the round cursor on the location for your hysteresis loop.
      iii. Click on *That's It* to finalize your choice.
      iv. Repeat for as many locations as you wish. You can also select additional points this way after you start measuring hysteresis loops.
      v. *Show Markers* to bring up the locations and indices of the various locations you've selected.
      vi. Check the show *Tip Location* checkbox. This should 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 on *Single Force*, the data displayed in the Force 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:

a) Go to *Display and Analysis*.

b) Select the data wave 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.

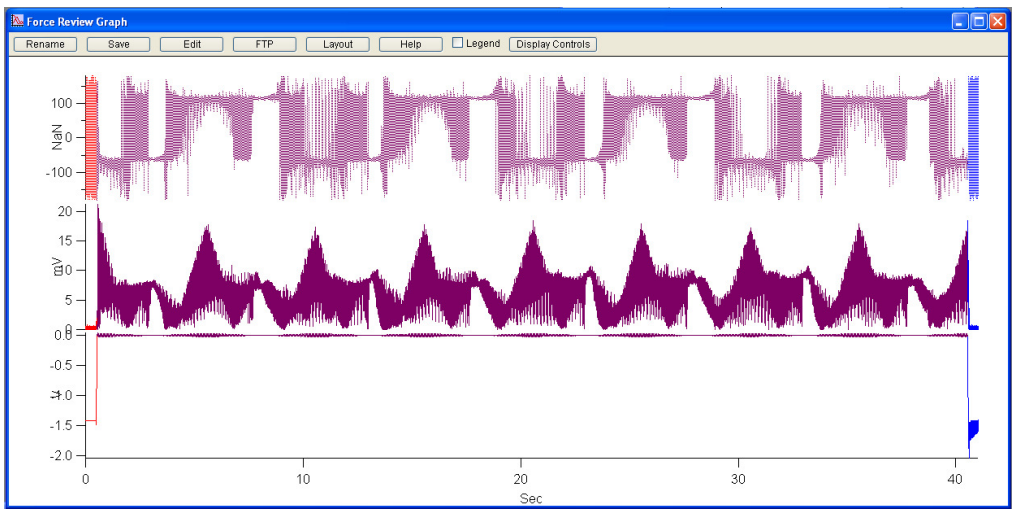


Figure 8.7.: The SS-PFM raw data set. The phase1, amp1 and deflection traces for an 8-cycle, 40 second hysteresis loop. The details of the switching are difficult to discern from the time data and need to be processed to produce hysteresis loops.

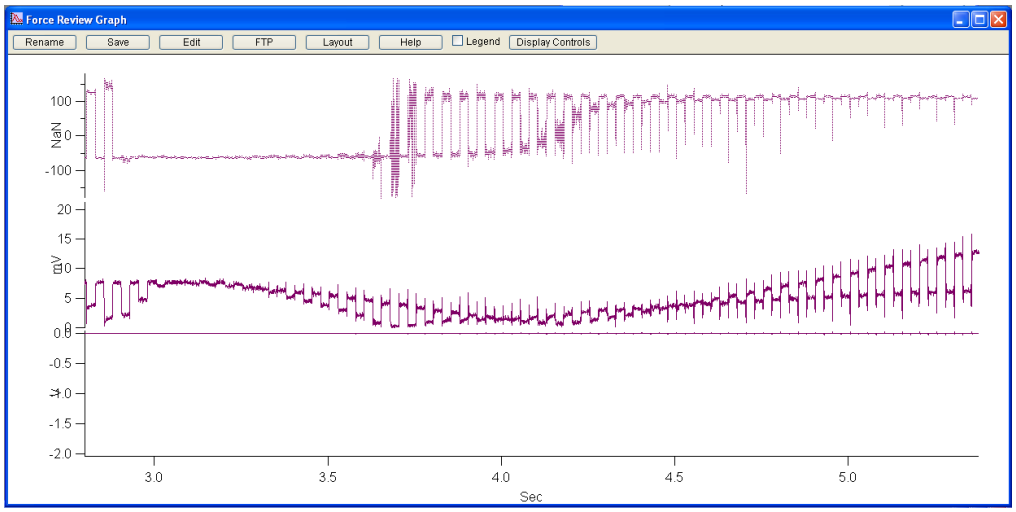


Figure 8.8.: Zoomed in region from Figure RawData. Here, the individual steps from the stepped SS-PFM method of[8] are evident. Repeat the above steps as many times as you would like, adjusting the drive parameters as you wish. If you notice noise during the higher voltage portions of the curves, you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

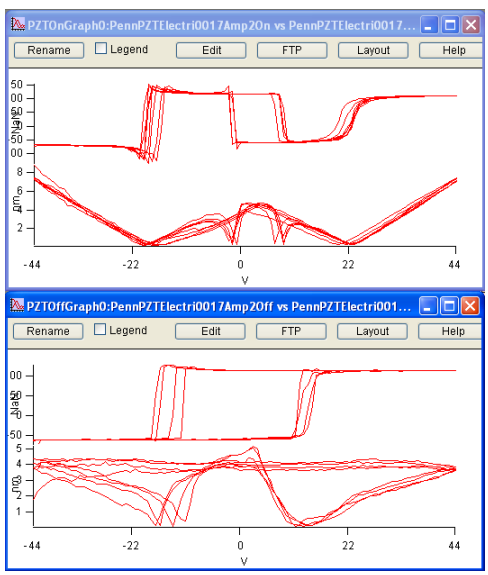


Figure 8.9.: The applied ("On", top graph) and remnant ("Off", bottom graph) hysteresis loops obtained from the processed time waves of figure 8.7 and 8.8. The phase loops are on the top of each window and the amplitude "butterfly" loops are on the bottom.

8.4.2. SS-PFM Array Measurements

1. Start with single point measurements as described above. This will allow you to adjust parameters and optimize the conditions for your sample and tip.

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

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

c) As in the above 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 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's a problem, click the *Stop!!* button, make adjustments to the parameters and start again.

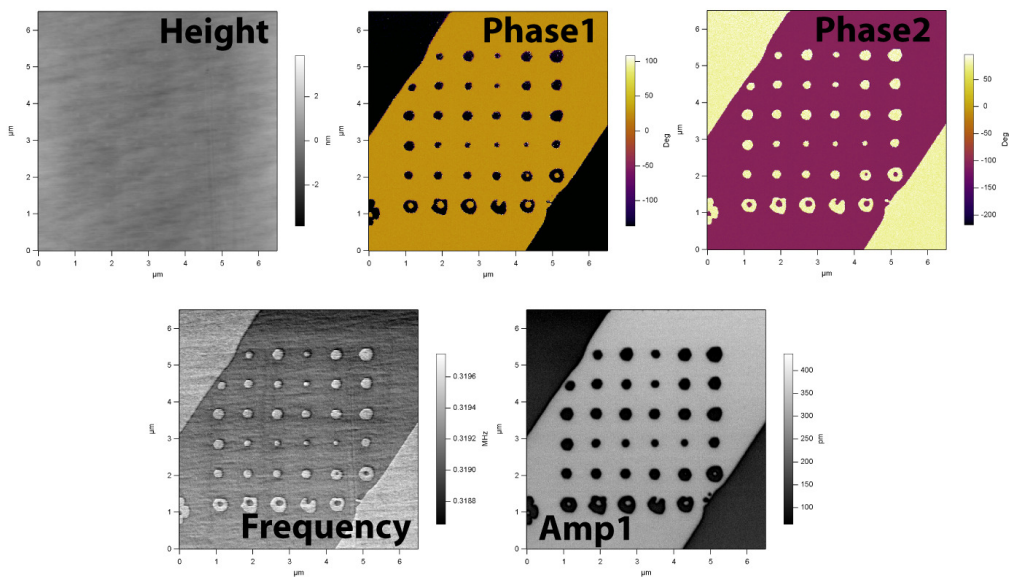


Figure 8.10.: DART PFM image of PPLN after a 6 by 6 array of hysteresis loops have been measured.

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 it is likely the tip will 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 lead to instabilities in the frequency feedback loop.

8.5.2. Image Size

Set the image size to a small value (typically 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, which can potentially have a large effect on the contact resonant frequency.

8.5.3. Feedback Gains

One big 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 should have distinct features correlating to topography or material properties. The amplitude should show signal within different domains, and should 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, and 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 if the tip is damaged, or if the *DART I Gain* setting is too small, it is possible for the resonance peak to move outside of the two drive frequencies. Once 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 the 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. In Minnesota, if you don't like the weather, wait five minutes. 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 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 little 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 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 115.

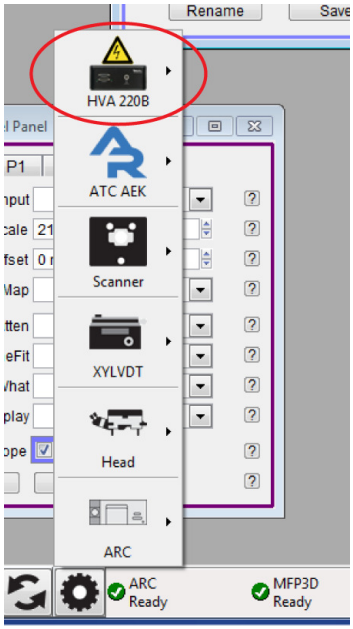
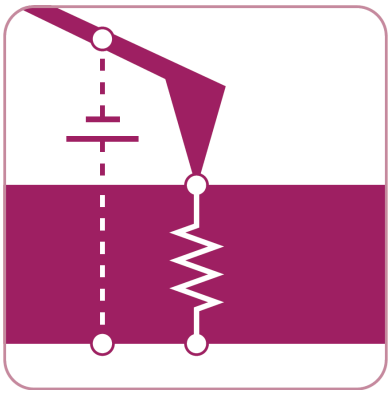


Figure 8.11.: Testing communication with the HVA using the SmartStart Device interface. The HVA icon indicates that the HVA is present and active.

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, 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 help.

9. Conductive AFM (ORCA)

CHAPTER REV. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



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.

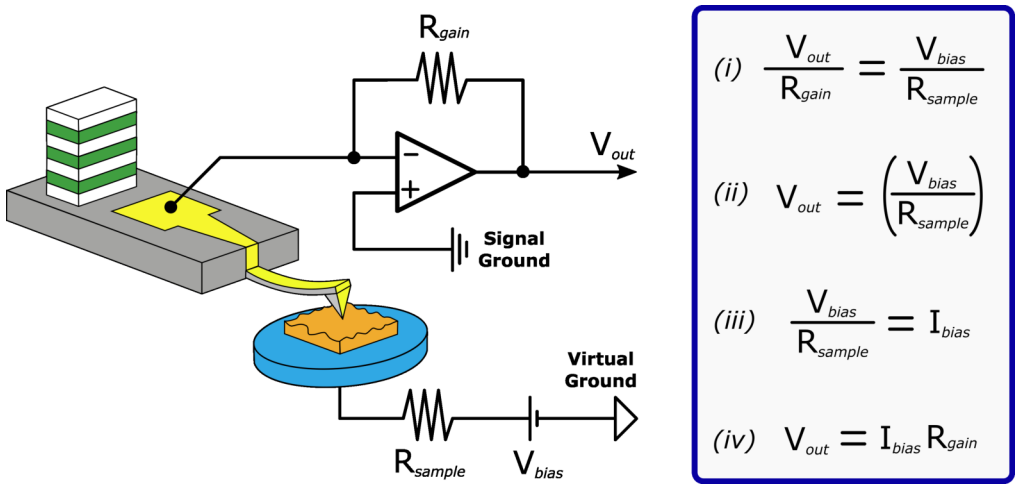


Figure 9.1.: Schematic of CAFM with relevant equations. Current is passed through the tip and into a transimpedance amplifier, converting the current to a voltage.

9.2. Video Tutorial

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

9.2.1. Terminology

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

Subcatigories: 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

In 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*

These chapters will 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 this 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.

- 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 perform pretty close to each other. The Tungsten Carbide and Ir probes last a little longer. For Polymer samples or soft materials these are fine. radii are typically 50nm
- Conductive diamond coated probes. Use a diamond-like carbon film coated onto a Si probe. Available from Nanosensors. These 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. More expensive than most probes but last a long time. Forms 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 loose its ability to conduct.

9.5. Repeatability in CAFM

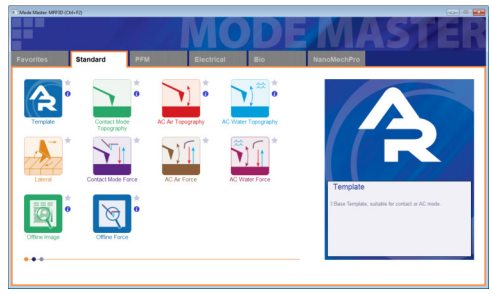
- **Tip change**, this is the most common. Metal coated probes are infamous for chipping, breaking, and otherwise acting in an obstinate manner. This normally shows up as a systematic asymmetry in the current to topography comparison. In other words, the current will appear on only one side of all the 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** is 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** is 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 are think the circuit is fine.
- **Silicon Oxide**, this one really belongs in the previous item, but it is so common, 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 insure you don't have a metal-oxide-semiconductor (MOS) contact in your circuit.

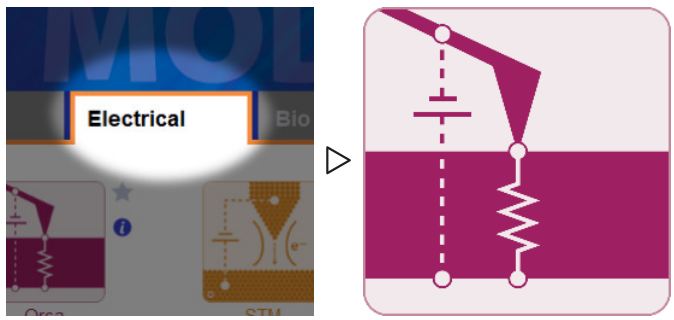
9.6. Current Imaging

9.6.1. 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: .



2. 

   Select Mode:

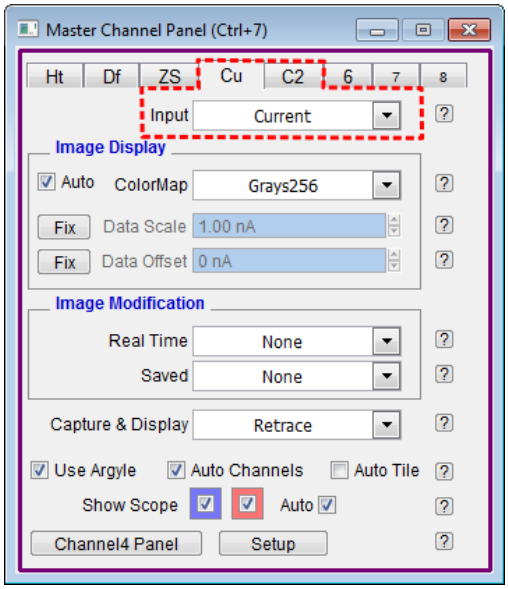
   - Select *Electrical tab* ▷ *Orca*
   - The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

   Note The ORCA cantilever holder must be first 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 appropriate manual for set up instructions.
4. Set the AFM to operate in **Contact mode** .
5. Set *Scan Angle* to 90º.

6. **Open the Channels**

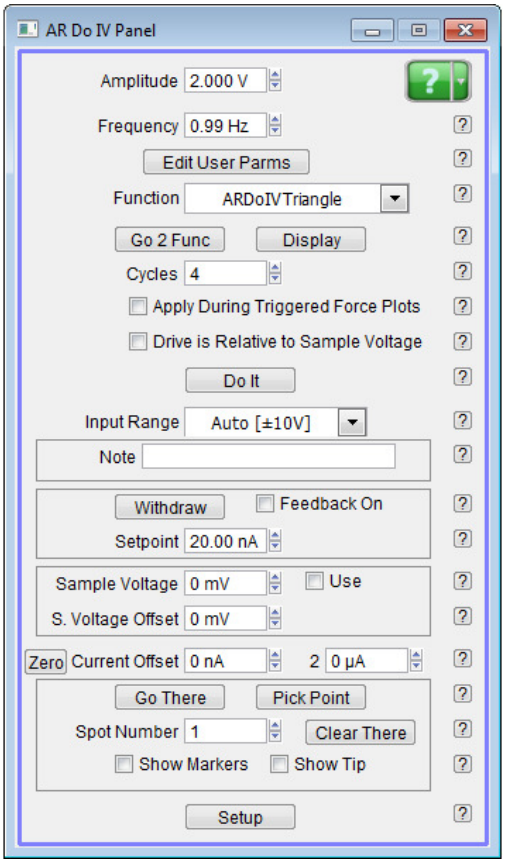
a) Select **Current** as one of the channel *Inputs* under the Master Channel Panel (*AFM Controls ▹ Master Channel Panel (Ctrl+7)*). For the Dual-gain ORCA holder you will have two channels instead, **Current 1** and **Current 2**.



7. Select **Current** as one of the channel *Inputs* under the Master Channel Panel (*AFM Controls ▹ Master Channel Panel (Ctrl+7)*). For the Dual-gain ORCA holder you will have two channels instead, **Current 1** and **Current 2**.

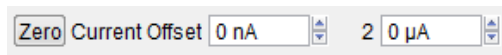
8. **Do IV Panel**

- Open the **Do IV Panel** (*AFM Controls ▹ Do IV Panel*). This panel is where you will control the majority of the electrical-based paraments in your experiment.



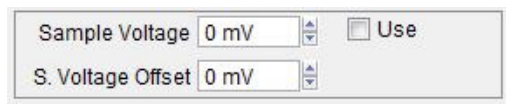
9. **Current Offset**

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



10. **Applying Voltage**

- Like the current, there are also often small, persistent voltage offsets between the AFM and the sample. These will 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 holdersample puck and the cantilever holder clip. Enter the opposite value measured in to the *S. Voltage Offset* parameter. A typically offset is between 10-100mV.
- Sample bias for ORCA experiments can be set with the *Sample Voltage* parameter on the **DoIV panel**.



11. When you first engage on the sample, you should start with a light contact force or low *Setpoint*, but may find that you will have to slowly increase the force. This will reduce 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.

12. 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.

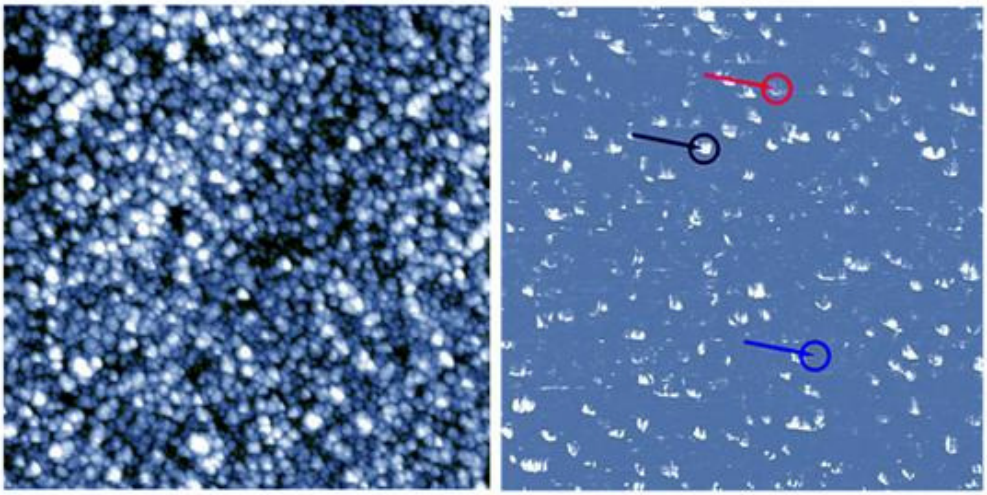


Figure 9.2.: This image is fairly typical of a thin film CAFM image. Note that tip shape artifacts are visible on the current image (left), but not on the topgraphic image (right). As the metal coating on the probe tip wears, or the tip is contaminated, these artifacts will be more prominent. Rocky Mountain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

I-V curves are done using the **Do IV Panel** which is opened from the menu *AFM controls ▷ Do IV Panel*. A description of all of the individual listings on the Do IV Panel can be found in the embedded Help file in the software by clicking on any question mark box to the right of the control in question. The Do IV Panel contains all of the controls that the user needs to:

- 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 a user may wish 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 Ohm-meter. 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 is comes about, because the number of conductive paths are 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 know, 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, it is also typically recommended that you first run I-V curves on the included test resistor to confirm everything is working properly, see[1].

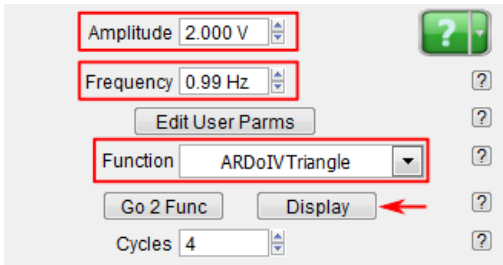
3. Engage the tip on the sample and acquire an image of your sample surface. For delicate samples that would be damaged or modified by Contact mode, AC mode is generally advised to take this image.

9.7.1.1. Setting up the I-V Curve

1. Set up the bias function you would like to execute on your sample.

2. **I-V Curve Functions**

   - I-V curves are most commonly treated like periodic functions. The pre-written functions are listed near the top of the **Do IV Panel** in the drop down menu labeled *Function*.
   - The pre-written functions include a: triangle wave, square wave, triangle-square wave, and sinusoidal wave. The user can also select *Function Editor* from the drop down menu to create their own custom form.

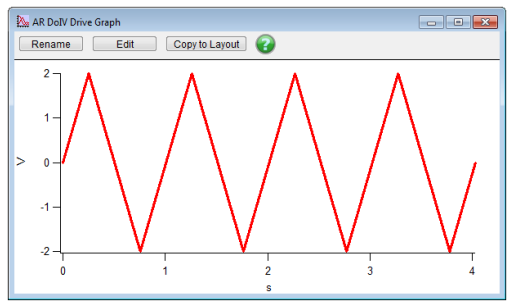


[1] *MFP-3D User Guide, Chapter: Conductive AFM (ORCA) Hardware..*

3.

Function Controls

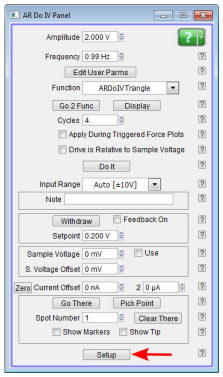
- The most convenient way to design your I-V curve is to display that function on a graph before you execute it. To do this, click the *Display* button below the *Function* drop down menu. This will bring up a graph of the voltage that will be applied to the sample as a function of time.
- You can adjust the *Amplitude* ,*Frequency*, and *Cycles* parameters of your function from the **Do IV Panel**. These will 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, then return to 0.
- *Frequency* will give how long one function cylce will last. For example, a 1 Hertz frequency will complete one full cycle in one second.
- *Cycles* will determine how many full cycles of your function are repeated per execution.

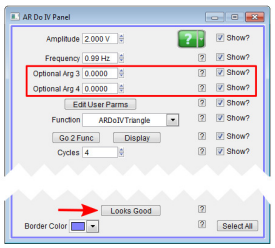


More Function Controls

4.

- If you need to adjust other parameters, such as including a DC offset in the curve or shifting the phase so that the bias is ramped from positive to negative or so that the ramp is only half of a curve, these can be adjusted using the parameters *Optional Arg 3* and *Optional Arg 4*.
- These parameters are hidden by default, but can be revealed by clicking on the *Setup* button at the bottom of the **Do IV Panel**. The "Arg" stands for "argument" and is will augment different things for the different pre-written functions.
- *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.





5.

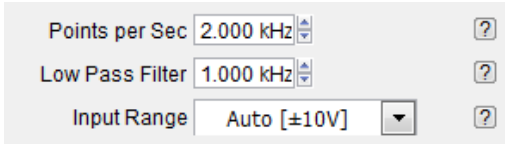
Function Averaging

- Checking the *Average* box will average each cycle of the periodic function to produce a single I-V curve. Use caution with this function, however, as often the current will change drastically over multiple I-V cycles. These 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.

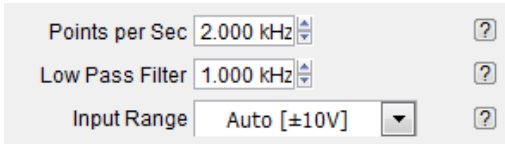
Function Frequencies

- 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 require DACs with much higher frequencies than 50kHz. To perform I-V curves above 20Hz, instead use a sinusoidal wave as the function .
- Additionally, for higher frequencies, you will typically need to increase the number of points present in the function. Do this by increasing the *Points per Sec* paramers on the **Do IV Panel**. Increase this parameter until the function wave looks smooth on the *Display* graph.



Function Filtering

7.
- 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 paramter 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 in the system.
- 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 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.



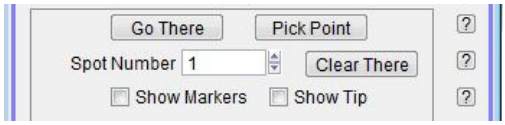
8. It is also possible to write custom functions with the **Do IV Panel**. Custom waves use the Waves from **User Parm Table** that can be brought up by clicking on the *Edit User Parms* button.

9.7.1.2. Picking Points for the I-V Curve

1. 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.

Setting Points

2.
- Click Show Markers and Show Tip
- Drag the red marker to the position you want.
- Hit *Pick Point*(first time) and *That's It*(subsequent times) for each location



Once you have set all the points you want to take I-V curves at, select the spot number you want your first I-V curve using the *Spot Number* field. Then 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.

When you are satisfied that you are in a good spot, enter a setpoint for the deflection either in the

ARDoIV panel or on the main panel under *Setpoint* , then check the box labeled 'Feedback on.' This will put the tip onto the surface and turn feedback on.

Now you are ready to get an IV curve. Simply click on the 'do it' button.

If you were using the 'Apply during triggered Dwells' box, instead of clicking *Do It*, go ahead and take a force curves.

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 the user is 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.

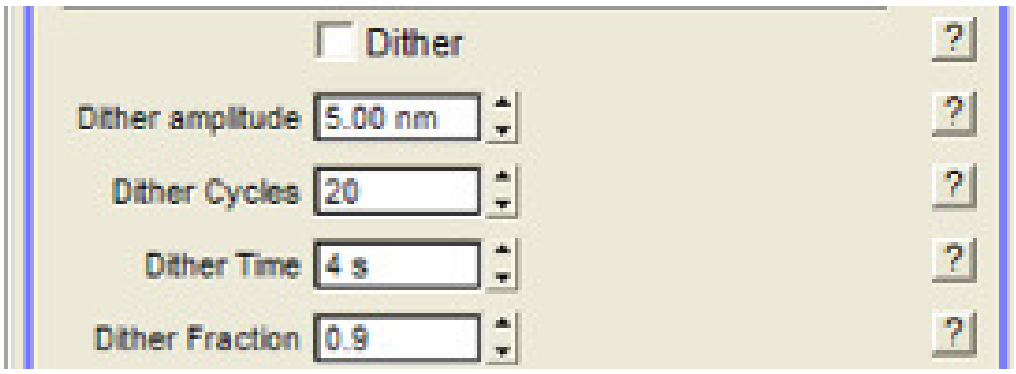


Figure 9.3.: [Dither] The dither function will create a spiral pattern the probe will trace out. This is intended to remove debris or to score the surface in order to get through oxides, thin films, or contamination on the sample surface.

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

The amplitude is the size of the spiral that will be traced with the tip. Cycles is the number of times the tip will turn within the circle to be dithered. The 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 that is created in a step-wise manner using the built in function generator. This allows the user 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.

To begin, select the function editor as the function on the DoIV panel. This will bring up a Function Editor window with a graph and some controls at the top. A default segment will appear. It 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.

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

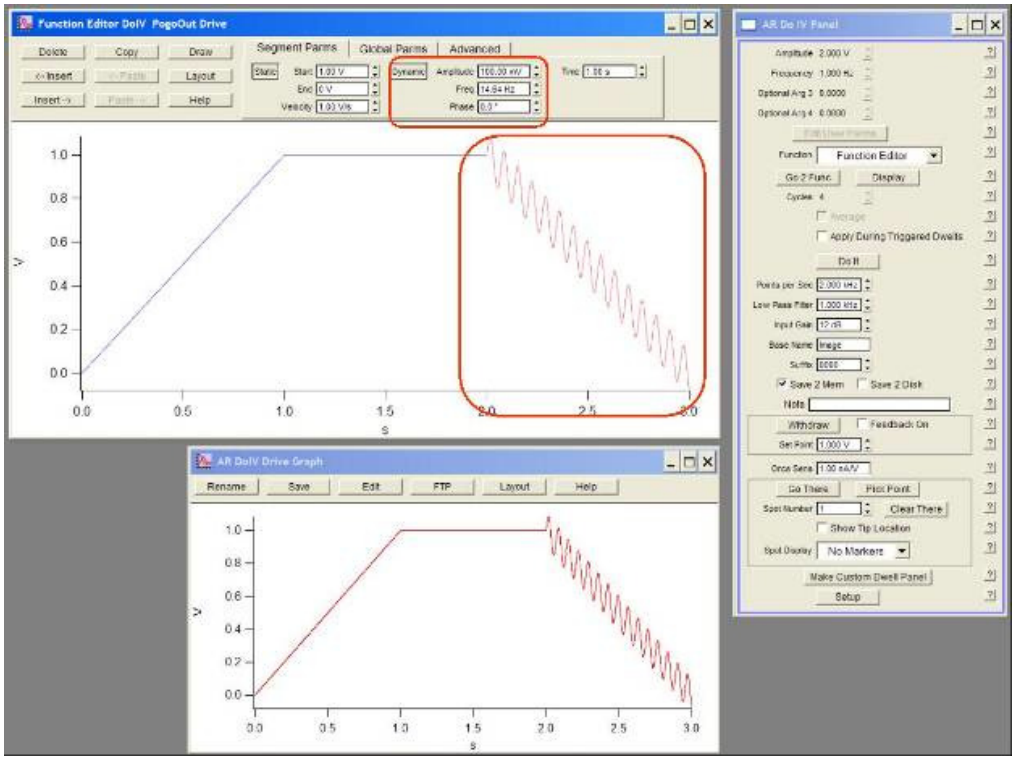


Figure 9.4.: [The Function Editor] The function editor can be used to make a variety of drive waves for current measurements. Insert new segments using the Insert button. Select different segments by clicking them. Each can be modified independently.

Under Global Parms, 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.

The tools on the upper left can be used to insert segments Delete, copy, paste, etc., it is even possible to draw segments by hand using the draw button.

Under the *Advanced*tab you can click the *Save* button to save the drive wave to use later.

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 force curve review panel.

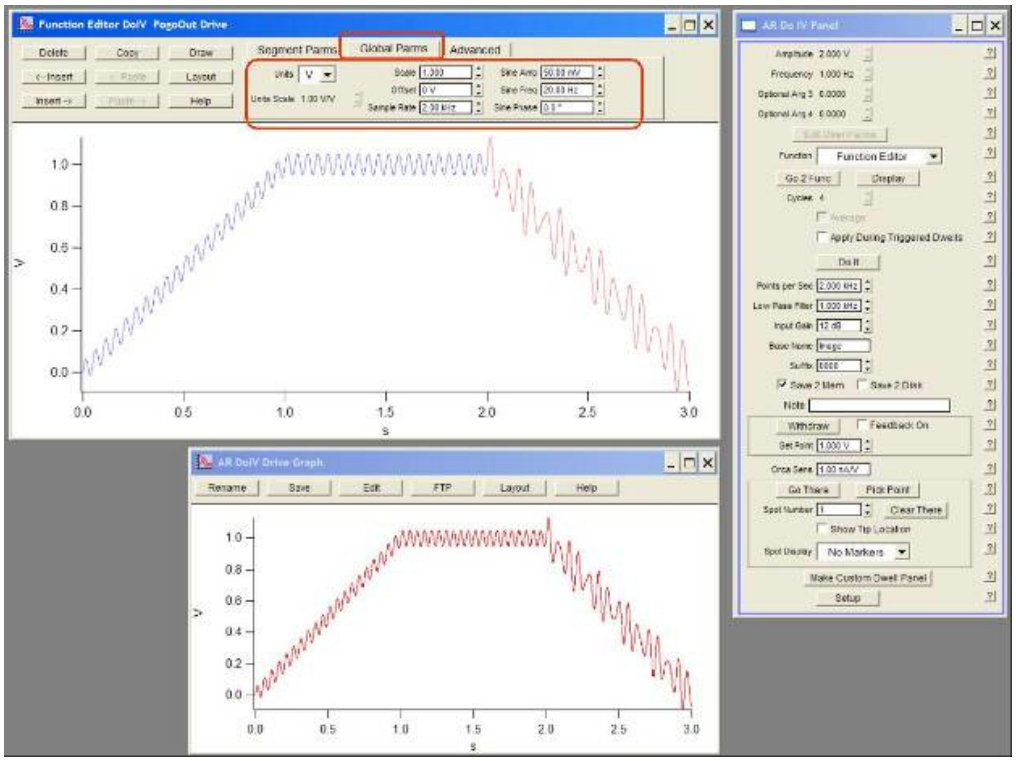


Figure 9.5.: [The Function Editor 2] Use the Global tab to modify all the individual segments at one time. Of particular use in this section is the Scale field. This allows you to scale up or down the entire wave function you have created. So if you had a custom wave from 0-1V with a dwel, scaling it by 5 would make it go from 0-5V.

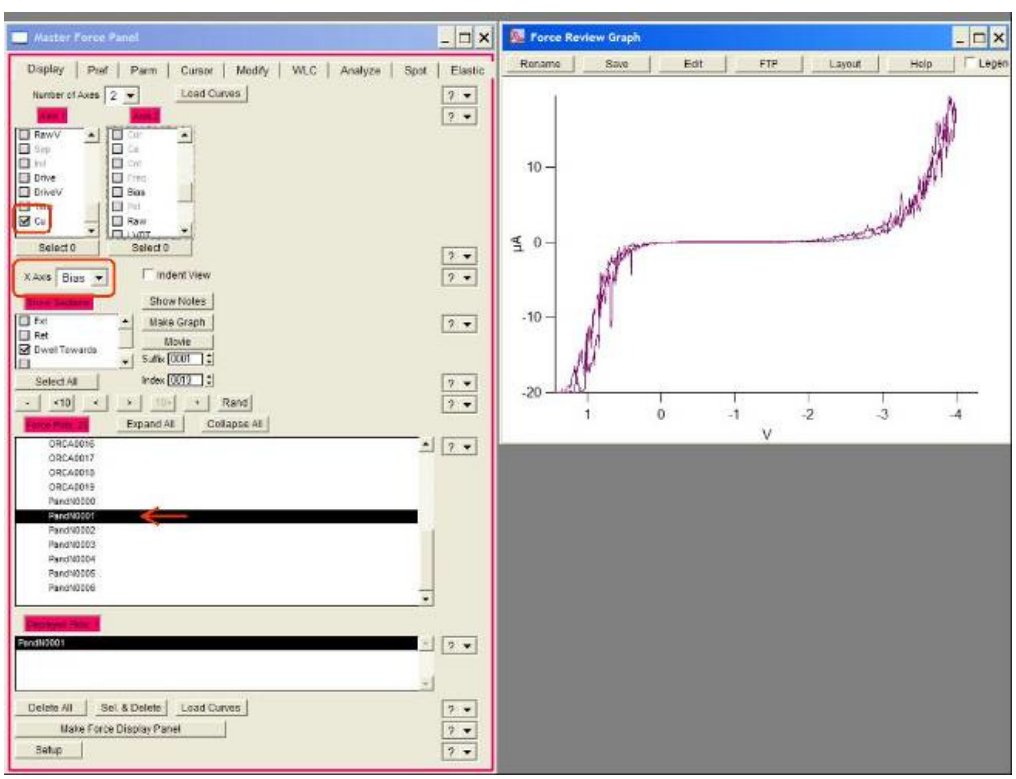


Figure 9.6.: [Current vs. Bias] To display IV curves, open the force review panel, view current (and current2 if you have a Dual Gain ORCA), Then set the x axis as bias, then select the dwell towards under the sections window. Click 'make graph' and the graph does not appear.

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 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).

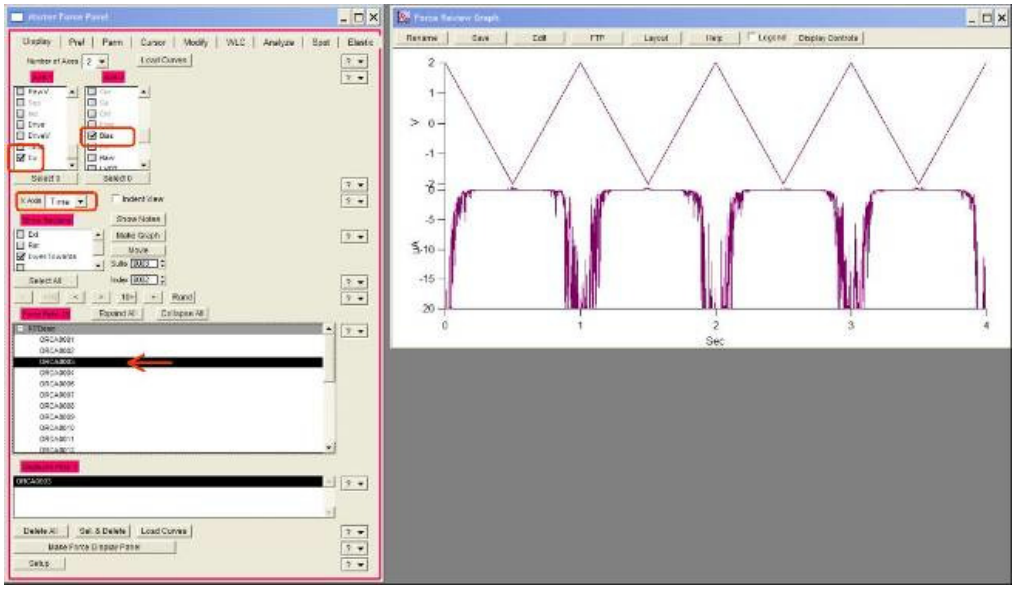


Figure 9.7.: [Current vs time] It is often useful to display the current vs time, particularly for samples that degrade or improve in conductivity with time. Si will oxidize as more current is passed through it if there is moist air near the surface.

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 select the last curve, then on the AR DoIV 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 will adjust the current offset to get zero current when there is no bias on the tip.

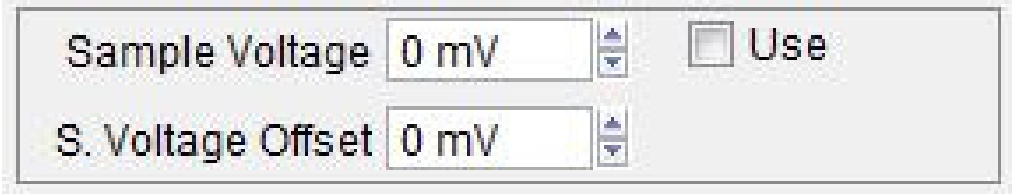


Figure 9.8.: [Current offsets]Bias and current offsets from the DoIV panel. These can be used to correct for small variations in the outputs of our DACs and the transimpedance current to voltage converter we use to measure current passing through the sample.

10. Nap Mode

CHAPTER REV. 2028, DATED 07/14/2018, 17:39. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

Chapter Contents

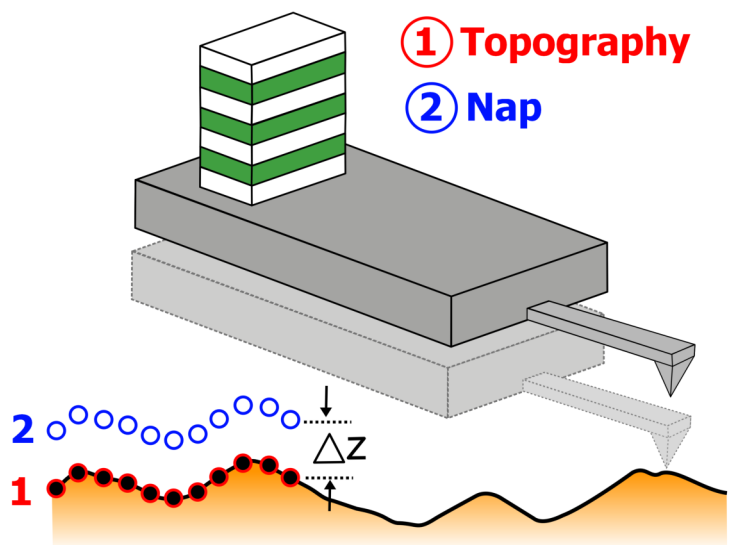


Figure 10.1.: During nap mode, the probe is first scanned across the sample surface, then raised to a delta height defined by the user. This allows for detection of long range forces between the tip and sample.

Nap mode uses two panels in addition to the normal controls for standard AC mode imaging. The first is the **Nap Panel**, which contains all the controls needed to run conveniental EFM, MFM, SKPM, and other two pass techniques. See Chapter 12 on page 145, Chapter 13 on page 153, and Chapter 11 on page 137 for more these techniques. The **Nap Panel** can be accessed by selecting *AFM Controls* ▷ *Nap Panel*(Ctrl + 9) in the menu. The second is the Nap Channel Panel, which 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 from the Nap Panel directly, or from the menu *AFM Controls* ▷ *Nap Channel Panel*.

Nap mode is a fairly simple technique in principal - 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, however 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

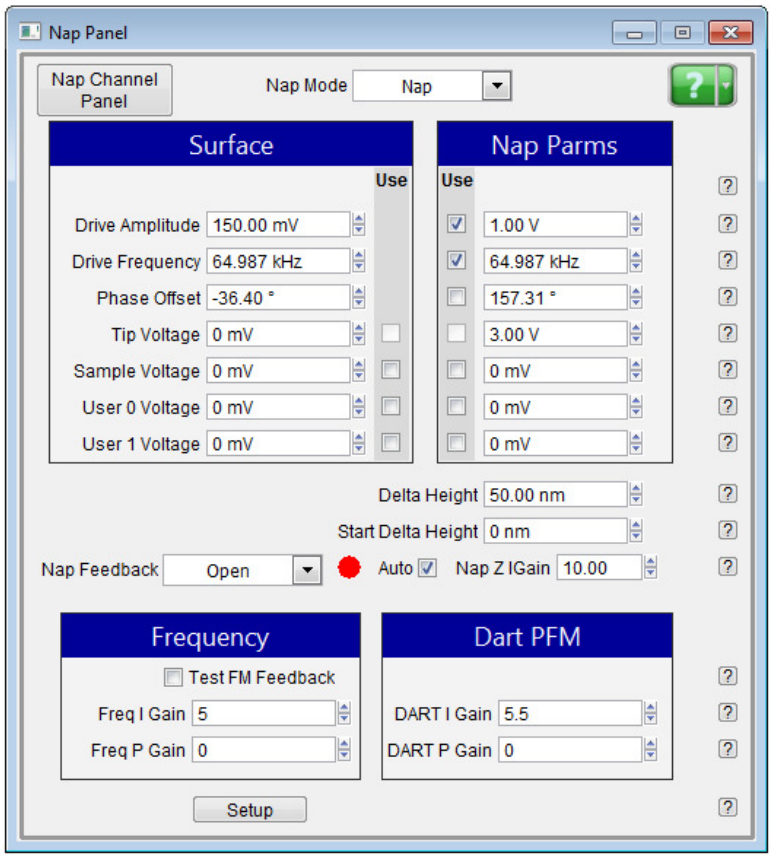


Figure 10.2.: Nap Panel, can be opened from the menu: *AFM Controls* ▷ *Nap Panel*(Ctrl + 9)

The Nap mode is set at the top of the **Nap Panel**, and can be set to:

- *Off*
- *Nap*
- *Parm Swap*
- *Snap*

Off: Nap mode off disables Nap mode, does a normal ordinary scan, with a single pass for trace and retrace.

Nap, standard Nap mode. The system scans 2 passed 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 **??** on page **??**, **??** on page **??**, and Chapter 11 on page 137.

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 2 different states; one value for the surface pass and a different value for the Nap pass.

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

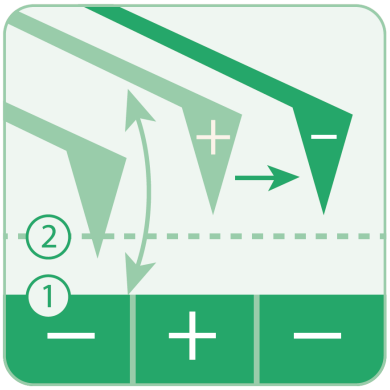
To select one of these, check the box in the column marked *use* to the left of each of the items in the parm swap column.

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. They 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. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



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. There a few synonyms for this technique:

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

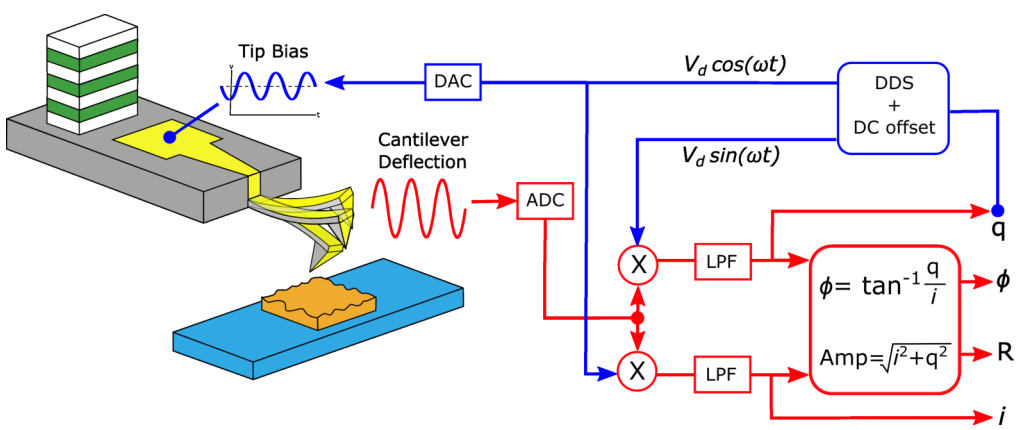


Figure 11.1.: Schematic showing how SKPM is performed on Asylum Research AFMs. The probe tip is driven electrically with an AC bias. The potential difference between the tip and the sample causes the probe to mechanically oscillate. These mechanical oscillations are then negated by a feedback loop on 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.

The 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 138 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 139.

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

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 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 seperate 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 - this is done exactly like a standard **AC Mode** scan line. The second pass, in this technique, has the tip raised a fixed distance above the determined topography and is used to measure the surface potential, seeChapter 10 on page 134 for more details on this method.

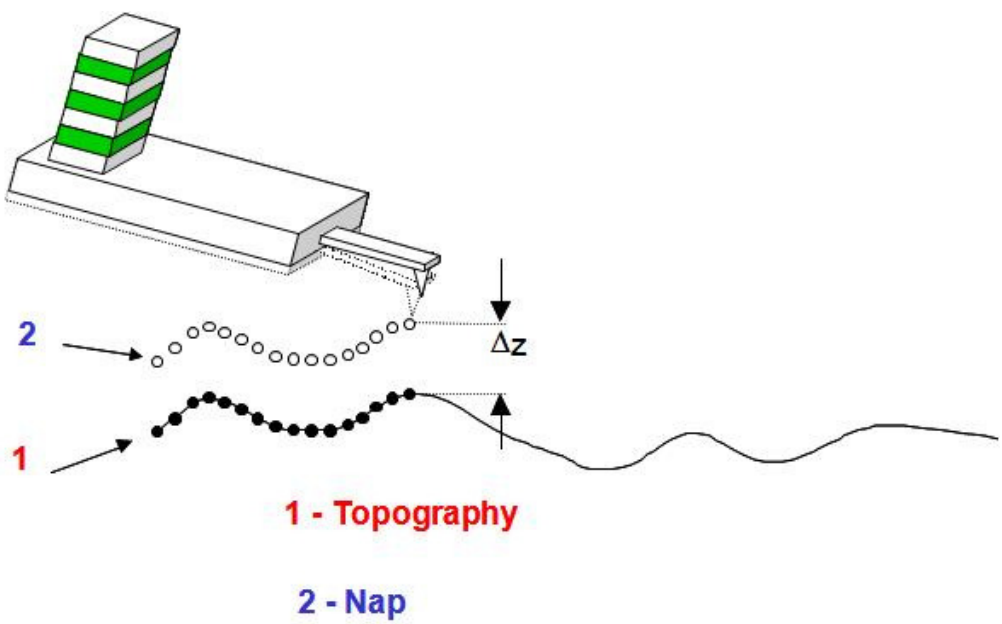


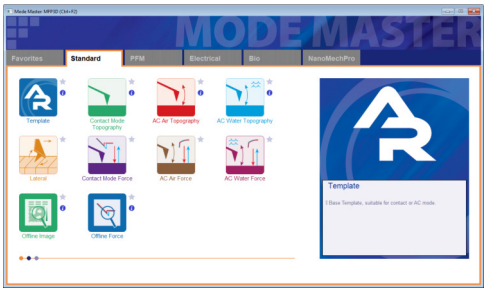
Figure 11.2.: Schematic of Nap Mode. For each scan line, the system captures the topography. The system then retraces that topography on the same line in order to keep a constant distance from the sample surface.

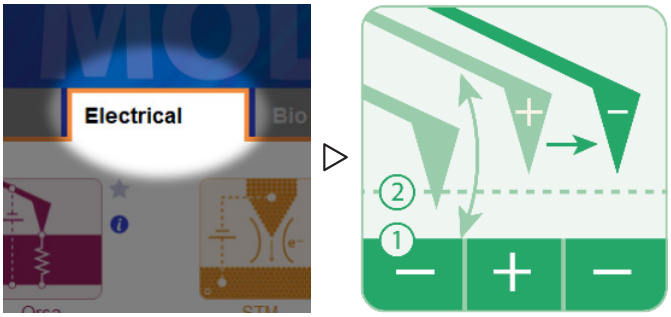
11.4. How to Guide

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 tab* ▷ *SKPM*
   - The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

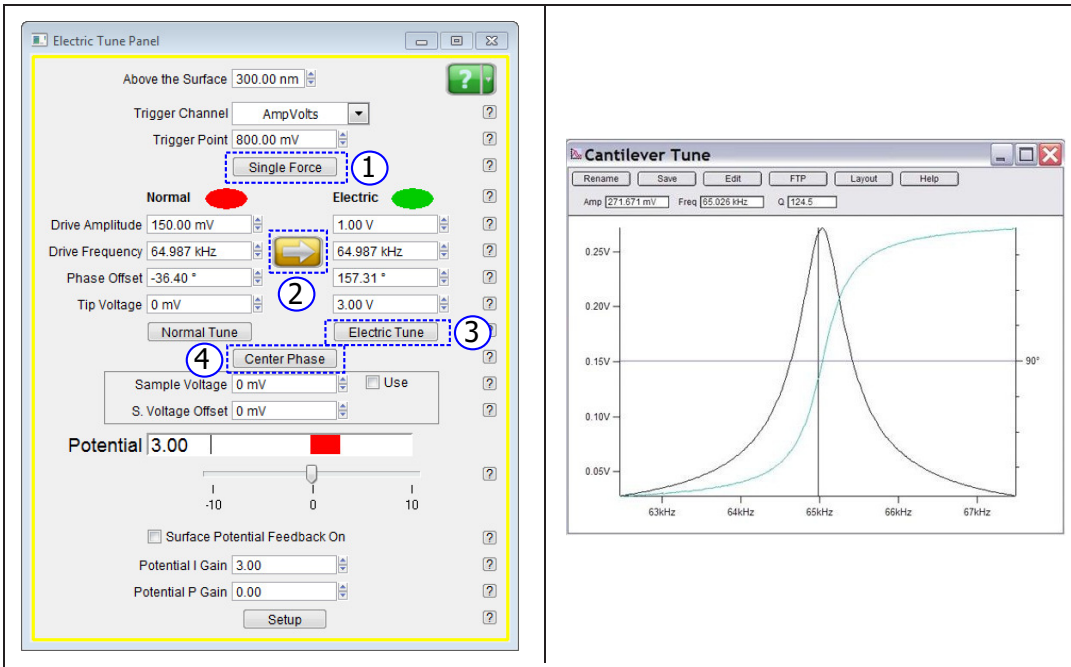
3. Mount and load the sample. Insulating and semiconducting samples need not be grounded, however metal samples should be grounded to the Scanner top plate to avoid arbitrary offsets in the potential that may mask the surface potential/work function between the tip and the sample.

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

5. Do an 'Auto Tune' on the cantilever to find the resonance frequency. Set the *Target Amplitude* to 1.0 V and set the *Target Percent* to -5%.

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. Often this electrical drive phase will 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**: 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*.

11. Click the 'Electric Tune' button. This will sweep an AC bias at the to 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 will set the phase properly so that the feedback loop can function properly. These buttons are shown in Step 8 on page 142.

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 collect an image. Typically, if the tip is at too high of a voltage, the Nap **Surface Potential** Channel will be railed either at positive or negative 8-10 V (depending on the *Drive Amplitude* voltage). If this occurs, set the *Tip Voltage* to a higher value and try again. Repeat as necessary.

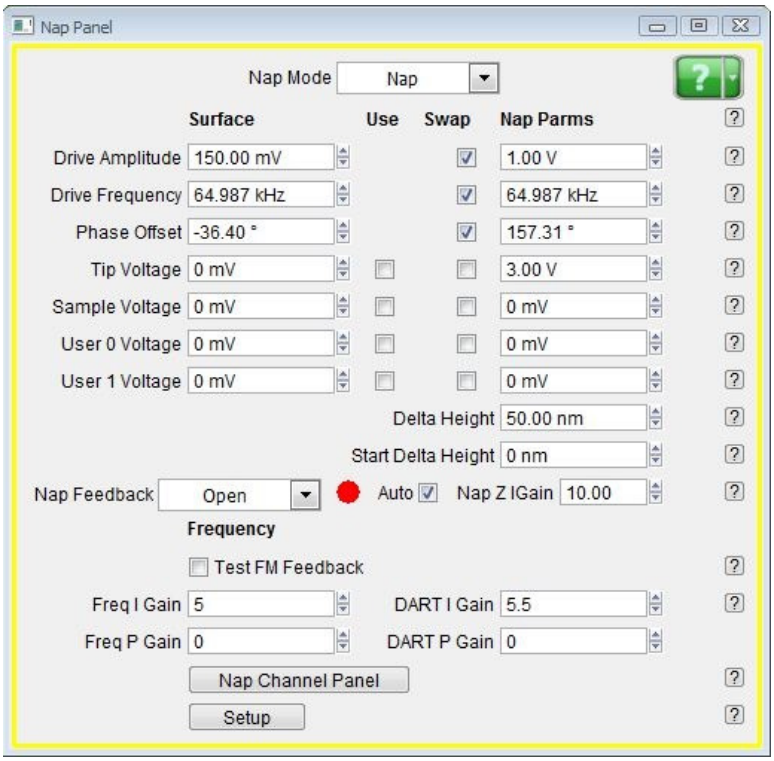


Figure 11.3.: Nap Panel configured for SKPM scan. The values in the paramers may vary depending on your tip and sample.

12.

13. Open the **Nap Panel** (*AFM Controls* ▷ *Nap Panel* (Ctrl+9)).

14. Select *Drive Amplitude*, *Drive Frequency*, and *Phase Offset* under the **Swap** column. See Figure 11.3 on page 143

15. Set the *Nap Mode* to "Nap".

16. Set the *Delta Height* parameter to 40nm. This will be lowered later if you want to try to get better lateral resolution during your image. If you are imaging a sticky sample, this might need to be raised more to get the tip off of the surface for the Nap scan.

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.

17. Open **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 143

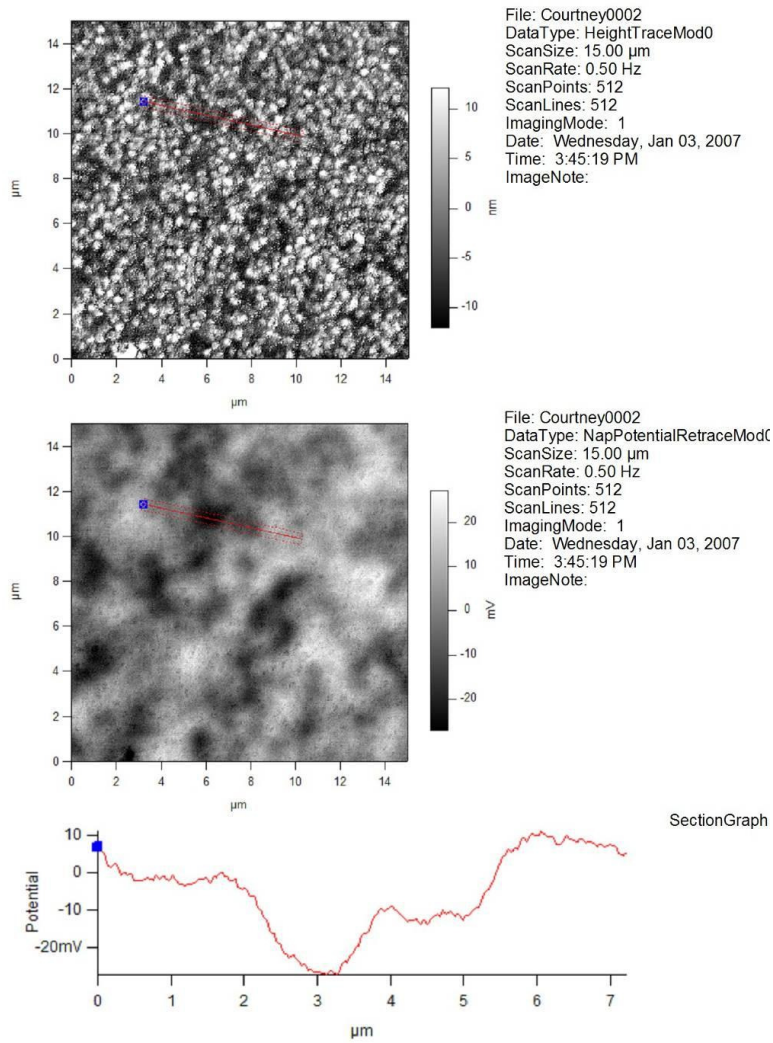
18. Make sure "Potential" is enabled as channel in the **Master Channel Panel** (Ctrl+7). Enabling this will automatically turn on the Potential feedback loop when scanning. Be sure that the Nap Potential channel (set on **Nap Channel Panel**) have Real Time and *Save*set to "None" under Image Modification.

19. 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. Typically, slower scan speeds 0.25-0.75Hz yield the best results in SKPM.

20. Begin the scan.

21. 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 1 for I Gain (integral) and 0.5 for P Gain (proportional).

22. To improve lateral resolution of the image slightly, try lowering the *Delta Height*.

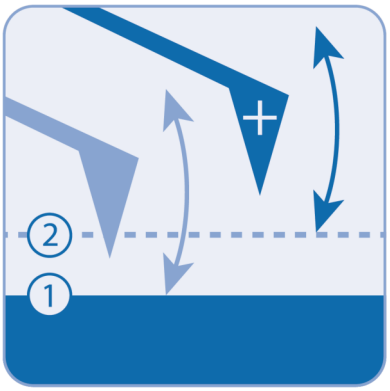


Sample Courtesy Andrew Lyon, Ga Tech

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.

12. Electrostatic Force Microscopy (EFM)

CHAPTER REV. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



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, the user can find areas under the tip that perturb the electric field between the tip and the sample.

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

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

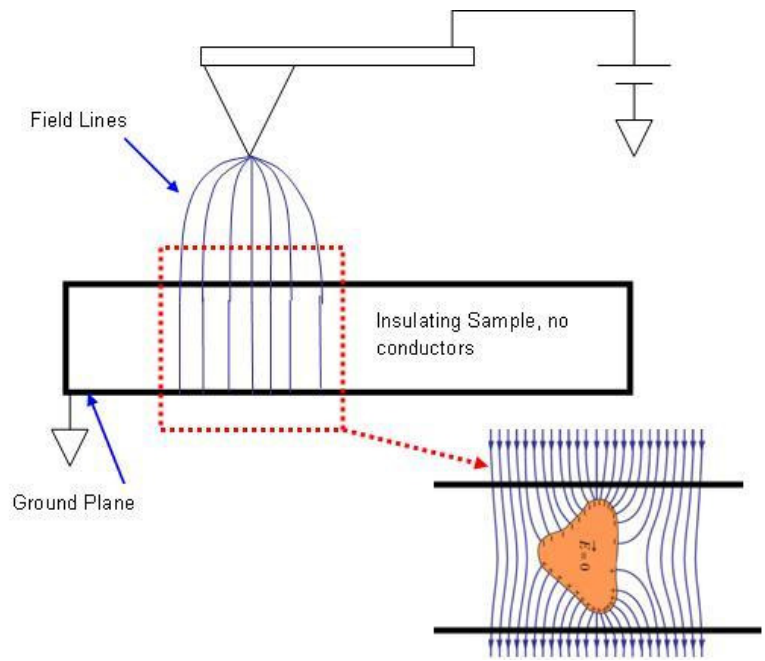


Figure 12.1.: This schematic shows how the introduction of a conductor into the electric field between the tip and sample can effect the field lines. The shift in the gradient due to the presence of the conductor causes a shift in the resonance frequency of the cantilever and therefore a shift in the phase and aplitude, 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 146), 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 147).

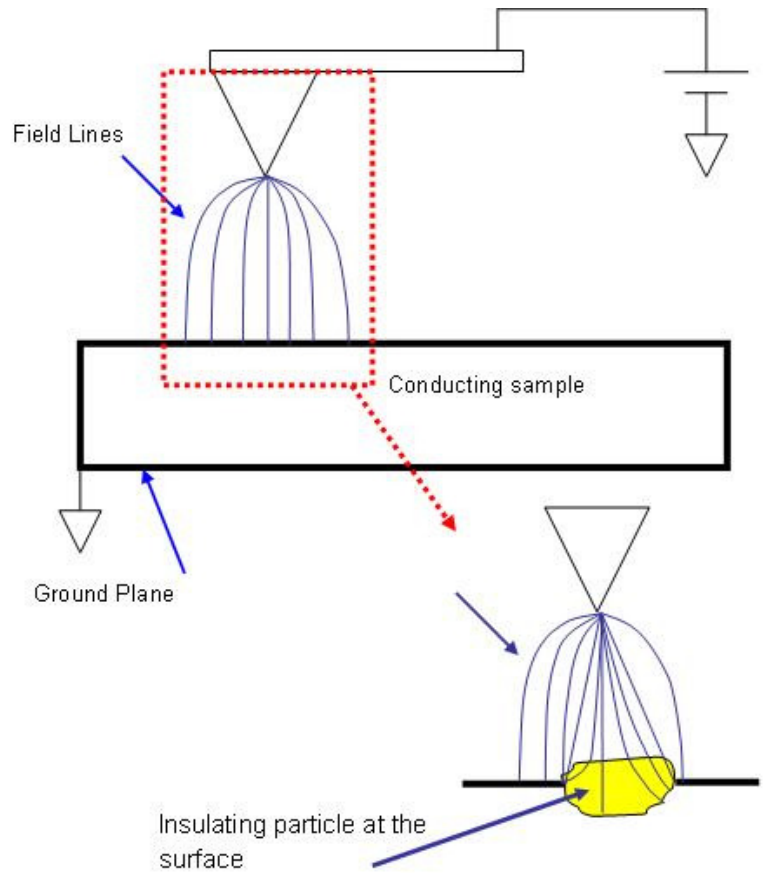


Figure 12.2.: Effects of introducing an insulator into a metal sample. EFM is generally not suitable for this sample configuration, as the field lines show little shift. Any shift would be difficult to discern from the changes in topography.

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 capacatiance, 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.

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

So 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 146, before the conductor is introduced into the insulating sample, the capacitor can be considered to be the tip toTheory 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 137.

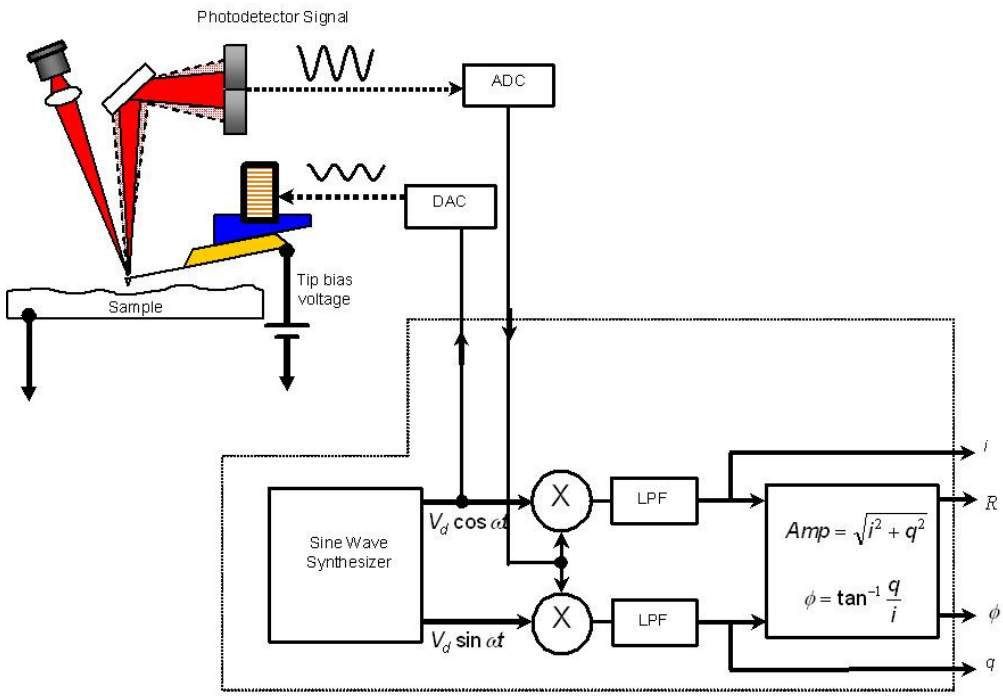


Figure 12.3.: Schematic of Electric Force Microscopy. The Probe is oscillated both during the main scan and during the nap scan. During the nap scan, the probe is lifted off of the surface, and a bias applied between the tip and sample to show the location of areas that are conductive or strongly charged.

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 134. This is done to maintain the separation between 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

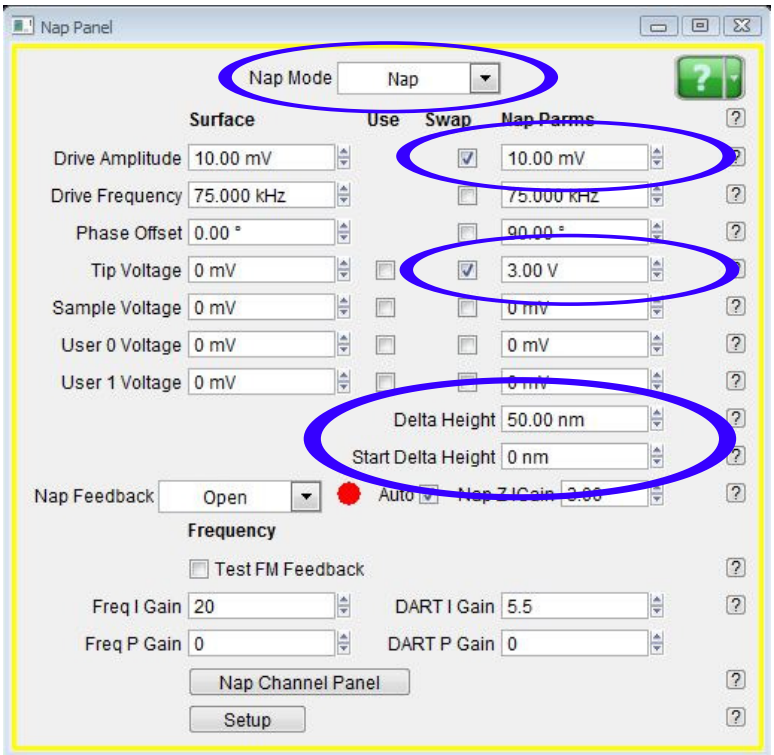
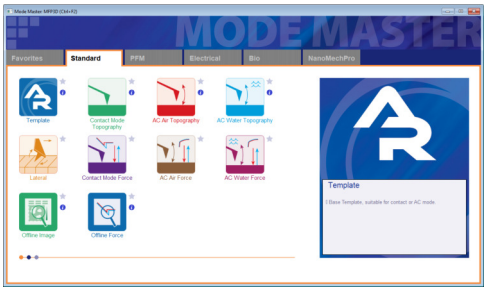
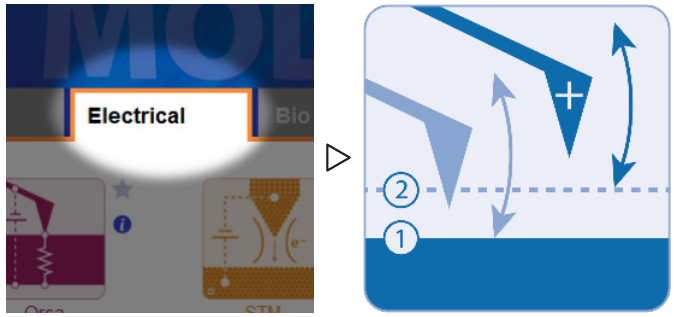


Figure 12.4.: Here the Nap panel is set for EFM operation. The relevant fields have been circled. The Use column in the middle left has check boxes that, when checked, cause that parameter to be used during the main scan. The swap column is has check boxes that, when checked, allow those fields to be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

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 tab* ▷ *EFM*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

3. Load the sample as normal, 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 generally preferable to have the sample grounded. This is because the field lines emanating from the tip will 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 is typically an A good first lever to try would be the electri lever, see also `http://www.asylumresearch.com/ProbeStore/APPLICATION?Entry=JustLooking#EFM,%20KPFM`; however, any conductive probe will do 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 will be maximized when the tip and the sample features are on the same order. If your features are larger than 100nm, though, 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), then open the nap channel panel.

6. Tune the cantilever as normal for an AC mode image. Once this is done, it is wise to approach and engage the surface in AC mode and 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 will be 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 parameter boxes under the parm swap column on the nap panel.

8. Next, set the delta height to the desired value. 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 149 shows the nap panel with typical settings for EFM.

9. Generally 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 will 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 details on this in chapter **??**.
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. By watching the amplitude in real time, the user 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 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, the user 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.

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 will adjust 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 wil 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 now result in a shift in the frequency. Further, any changes in amplitude will 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 details of this modeling is beyond the scope of this document (and currently it's author :-)).

How to implement FM-EFM

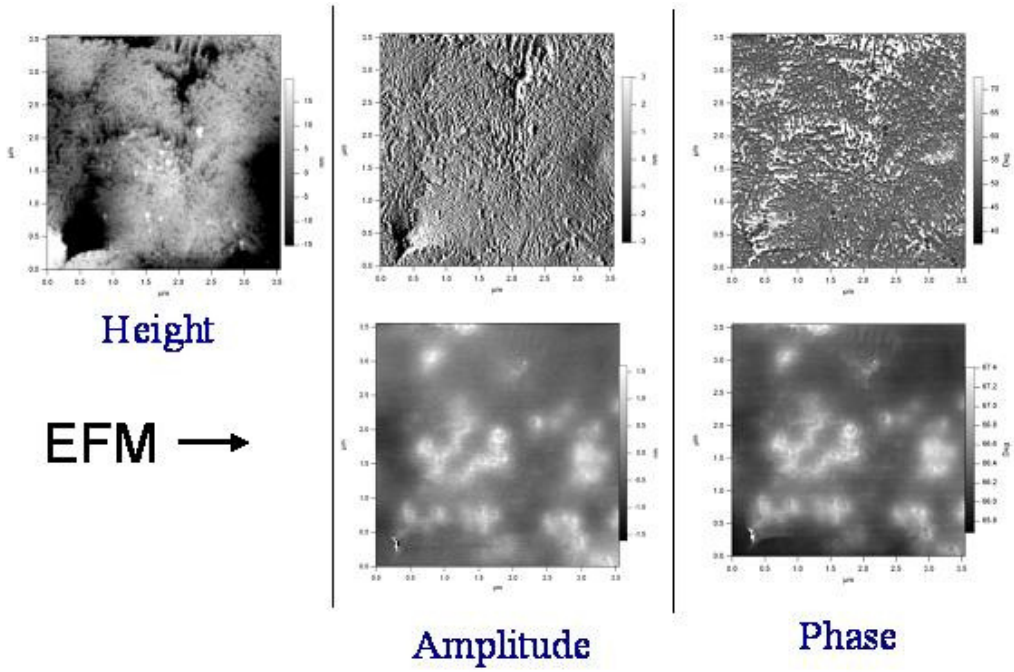
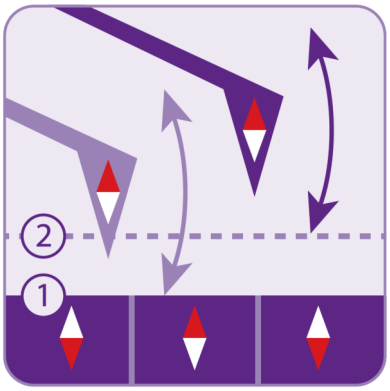


Figure 12.5.: EFM image of polypropylene with carbon black particles (CB) imbedded into it. The areas of contrast in amplitude and phase are due to the presence of CB particles either at or below the surface of the polypropylene.

1. Read through 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 will open the frequency channel in the nap channel panel as well. Check that the 'capture & desplay' 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, and click the tune tab on the Master Panel.
4. Set the Sweep Width to 5kHz, and 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. Ovserve the frequency and phase readouts on the sum and deflection meter, and raise the 'Freq I Gain'. Raise the value until the field is unstable, then lower it back to stability.
9. Ovserve the frequency and phase readouts on the sum and deflection meter, and 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. 2053, DATED 09/28/2018, 15:06.



Chapter Contents

13.1. Introduction

Magnetic Force Microscopy (MFM) is relatively simple in concept and operation, but often requires some considerable thought in interpretation. The technique oscillates the tip at its resonance frequency at an offset distance above the surface while raster scanning using a permanent magnetic probe tip. The magnetic field between the tip and the sample creates a force gradient between the two which in turn causes a shift in the resonance frequency of the oscillating cantilever. This shift in resonance also creates a shift in the phase lag between the drive and the response of the cantilever. By monitoring this phase lag, the user can visualize areas under the tip that perturb the magnetic field between the tip and the sample.

MFM is a two-pass technique. In the first pass, the normal topography profile is taken - typically in AC mode. On the second pass (referred to as "Nap mode"), that topography is recorded and the tip is then raised an offset distance above the surface. Phase data is collected to show the magnetically induced shift in resonance frequency. A general illustration of Nap mode is shown in Figure 10.1 on page 134. This is done to maintain the separation between the tip and the sample constant while collecting the MFM data.

Both 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 (typically 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 farmiarize 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:

- 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.
- Are familiar with the concepts of **Nap Mode**, covered in Chapter 10 on page 134.

13.2. Required Materials

- MFM probes (ASYMFM series cantilever or similar)
- Standard Cantilever Holder
- A rare earth magnet
- An MFM test sample (An old HDD or Floppy disk works great)

13.3. How to Guide

13.3.1. Preparation

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

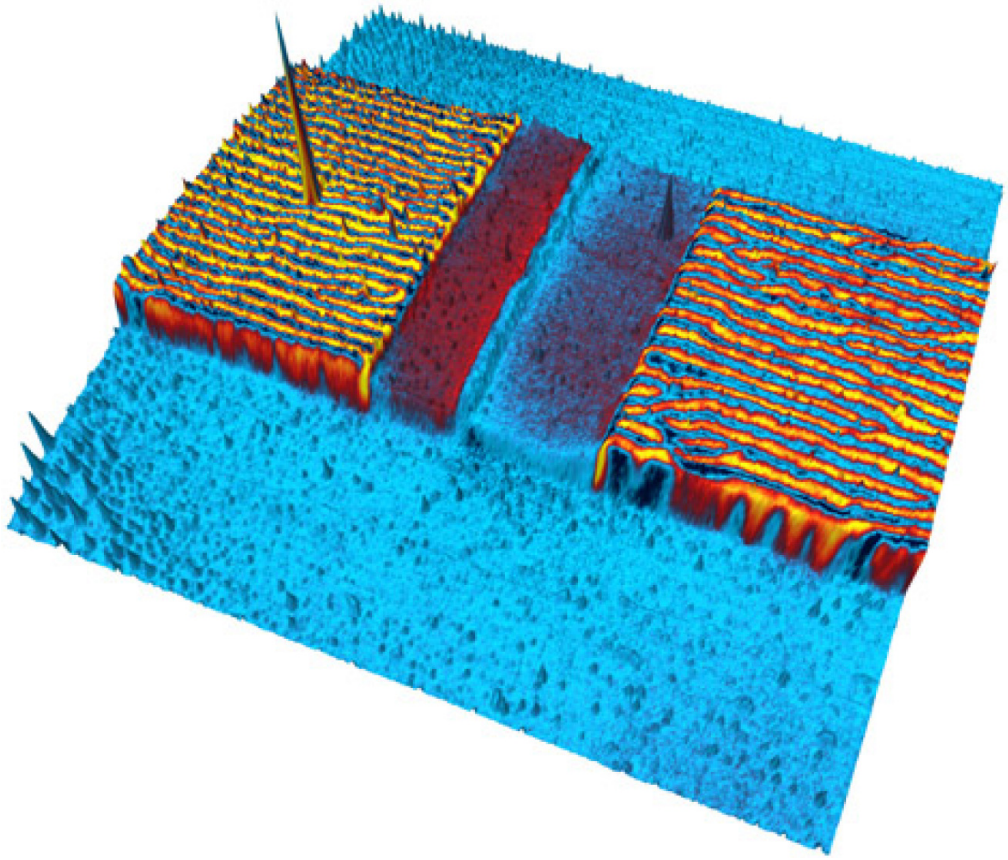
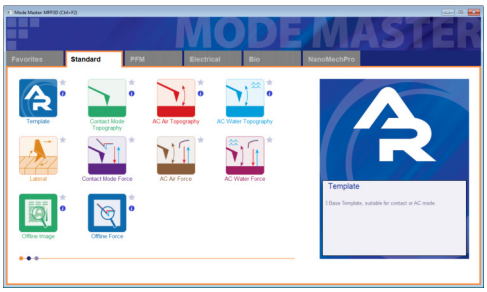


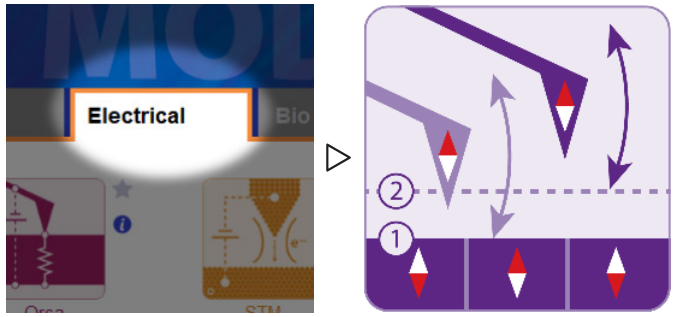
Figure 13.1.: The MFM signal showing the magnetic fields above an Iomega Zip™ 1 GB drive write head. The MFM phase signal was overlaid on top of the topography, 20 µm scan.

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

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: .



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

2. 

Select Mode:

- Select *Electrical tab* ▷ *MFM*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

3. The direction of the tip's magnetic moment will affect the image. Imagine a cantilever with a magnetic dipole tip $\vec{m} = m_x\hat{x} + m_y\hat{y} + m_z\hat{z}$, 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

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

4.

Magnetize the Cantilever

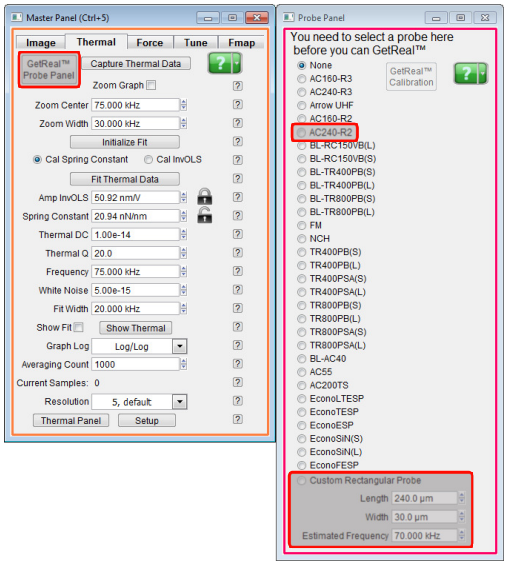
- To magnetize the tip along the z-axis of the cantilever, take one of the magnetic sample holders from the MFP-3D (or a comparable rare earth magnet):
- Load an MFM probe into the probe holder.
- Orient the magnet so that the side with the circles is facing the tip.
- Hold the magnet in one hand and 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, you do 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.

5. Load the cantilever holder and the sample into the AFM.

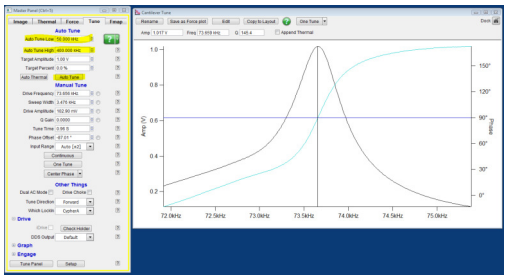
6. **Calibrate the Cantilever**

- Align the laser on the cantilever and zero the Deflection signal.
- If you are using an Asylum Research branded MFM probes, it is possible to automatically calibrate the probe using GetReal™.
- Open the **Thermal** tab in the **Master Panel** and select the **GetReal™ Probe Panel** button in the top-left of the panel.
- For Asylum Research MFM probes labeled without a revision appended (e.g. ASYMFM, ASYMFMHC, etc) select *AC240-R2*.
- For other probes, use *Custom Rectangular Probe*, and enter the dimensions and estimated resonance frequency of the probe.
- Click the **GetReal™ Calibration** button.



7. **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'.

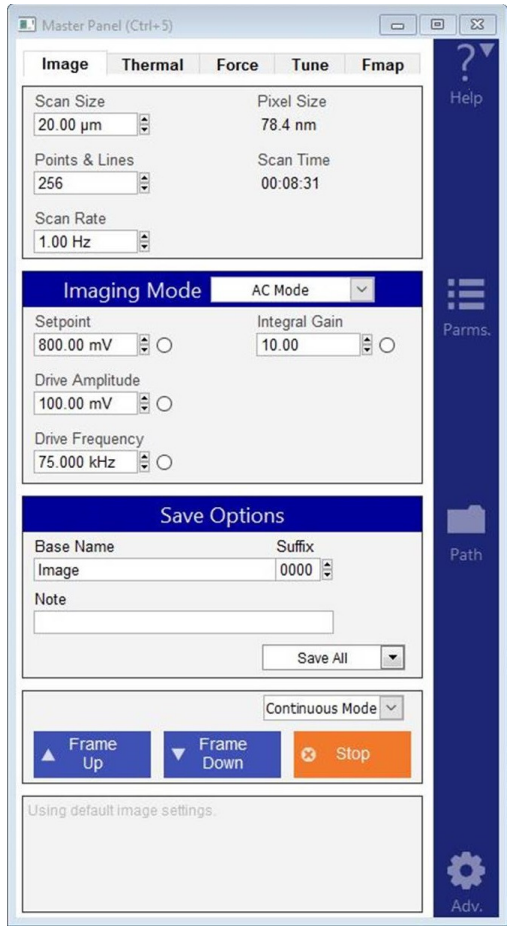


13.3.2. Imaging Parameters

1.

Master Panel

- 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 and find the Amp InvOLS. This value, multiplied by the amplitude setpoint, is the physical amplitude of the probe–typically **50-100nm**.
- Mechanical bandwidths on most MFM probes are typically around 500-700 pixels per second, so scan rates should normally be **0.5Hz to 0.75Hz**.
- The Drive Amplitude during the Nap pass scan should be **1/2 to 1/5** of the main Drive Amplitude. Higher Drive Amplitudes will give a higher signal to noise ratio, while smaller drive will give better lateral resolution.

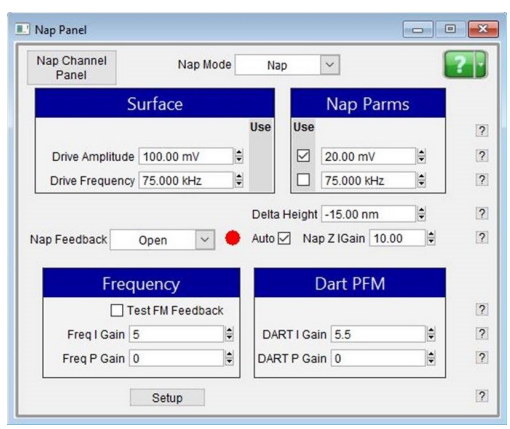


2.

Nap Panel

- The *Delta Height* on the **Nap Panel** should typically range from **zero to a negative value** - however, this while depend on the topography Drive Amplitude and the Nap Drive Amplitude. Generally, the Delta Height should be as low as possible without hitting the surface.

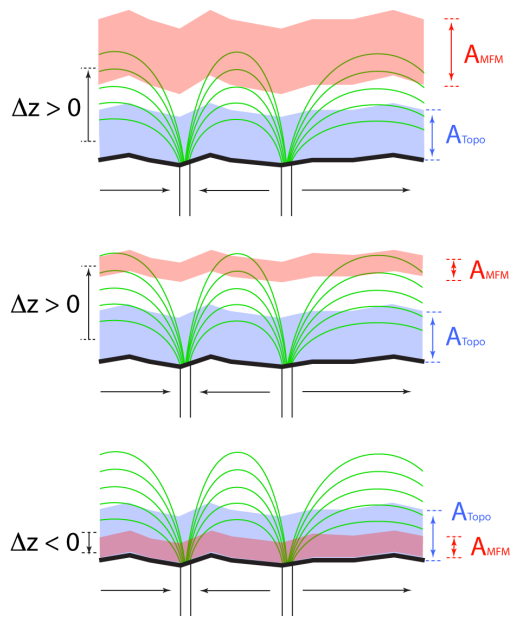
Note If the probe does begin striking the surface during the Nap pass scan (typically appearing as streaks), any MFM data will be obscured by the tip-sample interaction, and the Nap Phase or Frequency channel will resemble the main topography scan Phase channel



3.

Adjusting the Nap Pass

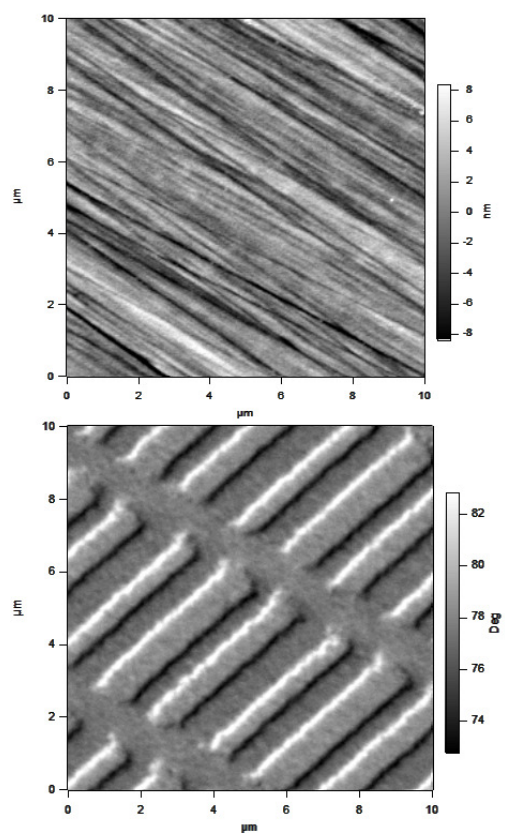
- When adjusting the *Delta Height* and *Drive Amplitude* of the **Nap** pass, there are two main principles to consider:
  - A smaller Drive Amplitude will allow the probe to be passed closer to the sample surface during the Nap scan; improving the lateral resolution. At some point however, this improvement 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 Amplitude during the Nap scan, but this results in a reduction in the lateral resolution.



4.

MFM images

- The MFM image should clearly show the magnetic domails independant of the topography and phase. In plane domains will show bright and dark contrast regions as shown in the image to the 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.

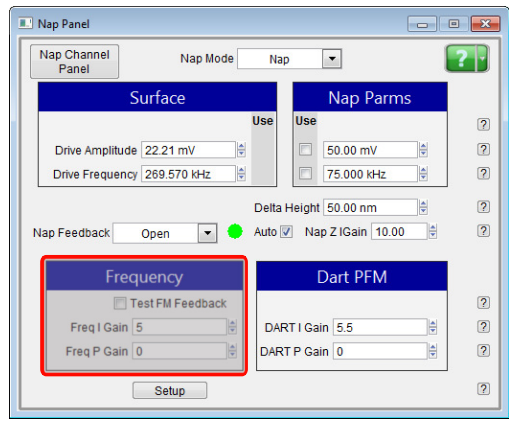


13.3.3. Optional: Frequency Feedback

Enabling the Feedback

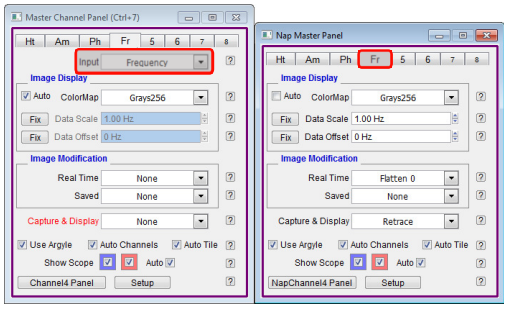
- It is also possible to set up a feedback loop during the Nap scan to track the resonance frequency using a phase lock loop (PLL). The goal and advantage of tracking the resonance frequency during the Nap pass is that it will allow the user to separate the phase and amplitude responses of the signal. 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.

1. - To enable and turn on a PLL, it is necessary to first decern 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 moduluation, and is another way to describe the PLL we are utilizing. Once the box is checked, raise the integral and proportional gains until the phase and amplitude in the Sum and Deflection Meter become unstable; the instability will be very apparent because the amplitude and phase signals will begin to oscillate uncontrollably. Back off the integral and proportional gains until the signals *just* stablize again.



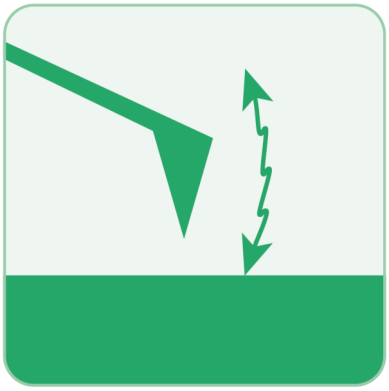
Opening the Channels

2.
- The Frequency channel must be opened during the Nap scan. On the **Master Channel Panel**, select one of unassigned channel tabs and set the Input to *Frequency*. The *Capture & Display* on the **Master Channel Panel** should by default be set to *None*. The Frequency *Capture & Display* setting on the **Nap Master Panel** should be set to *Retrace*.
- While imaging, the Phase data will now be an error signal channel in the frequency feedback loop (or "PLL"). Changes in the Amplitude will now reflect variations in the Q-factor of the cantilever. 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. 2042, DATED 08/31/2018, 16:24. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



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.
- A **High Freqency Cantilever Holder** is highly recommended, but not necesassry for lower frequency cantilevers.
- If Cypher AFM is equipped with **blueDrive**, this can be used in place of the High Frequency Cantilever Holder 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™ is a relatively new AC imaging mode offered both on the MFP-3D series and Cypher series of AFMs. The concept is simultaneously driving the cantilever at two of its flexural resonances, giving an AC waveform similar to the one depicted in Figure 14.1 on page 164.

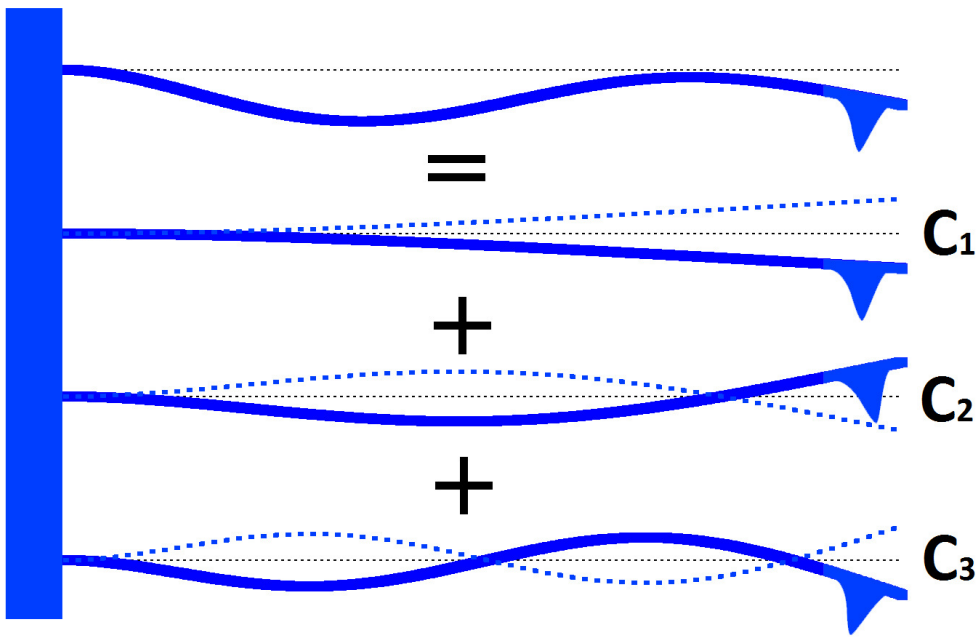


Figure 14.1.: Depiction of bimodal resonance operation.

The technique requires two independent lock-in amplifiers to accomplish this, 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 amplitudede 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.

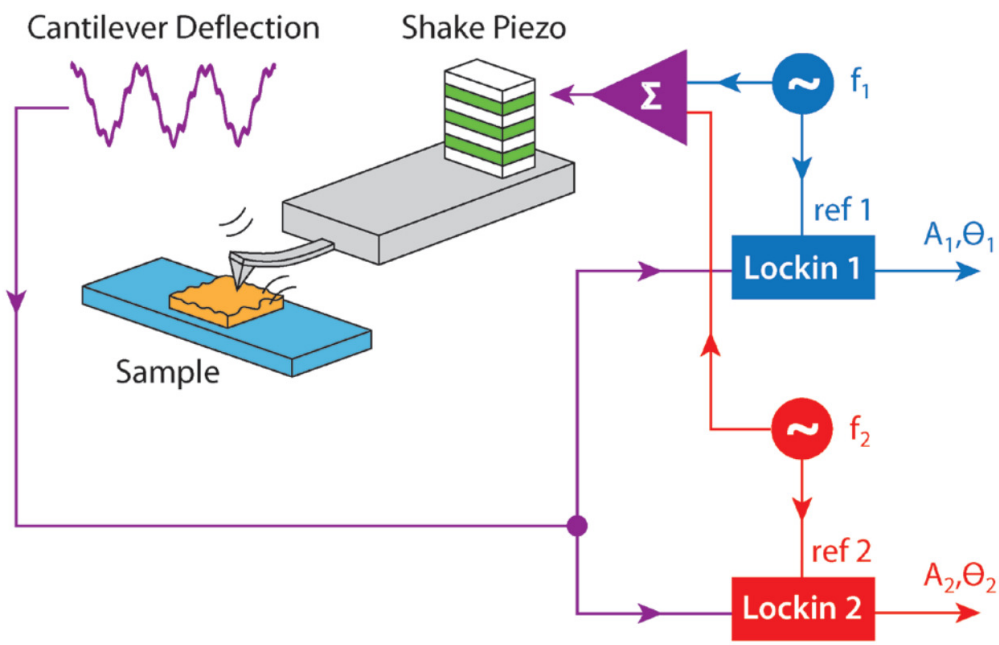


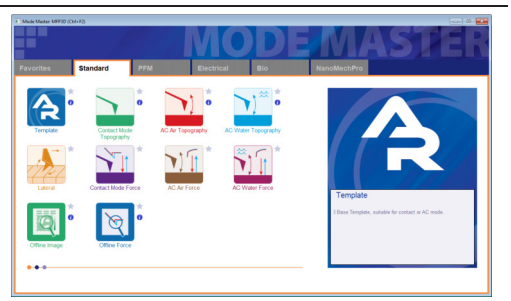
Figure 14.2.: In Bimodal Dual AC, the cantilever is both driven and measured at two (or more) frequencies. The sinusoidal "shake" voltage is a sum of voltages at frequencies f1 and f2. The cantilever deflection then contains information at both of those frequencies, as shown in the red curve. The amplitude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

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, the author has found that just a small amount of some water based latex paint on a glass microscope slide (allowed to dry) gives excellent contrast in the Amplitude & Phase images, and 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 ingredient that gives 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. 

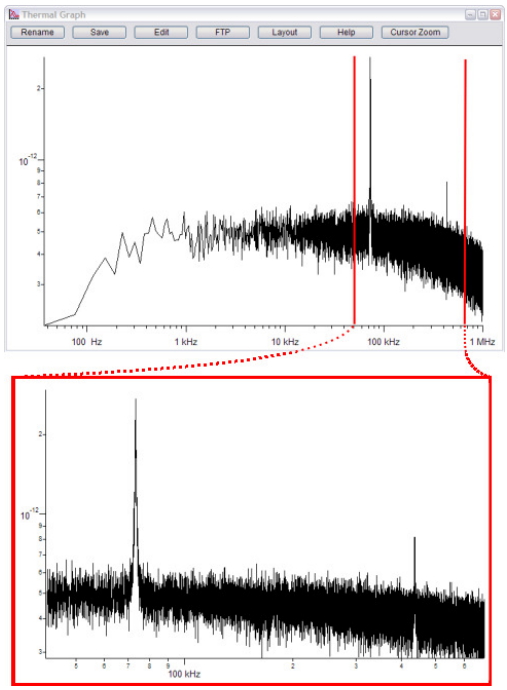
▷ 

Select Mode:

- Select *NanoMechPro tab ▷ Bimodal Dual AC*
- The screen will now re-arrange and 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 290).
- To determine the second Eigenmode, roughly multiply the first Eigenmode frequency by 6.1 - this will be 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 to the right.

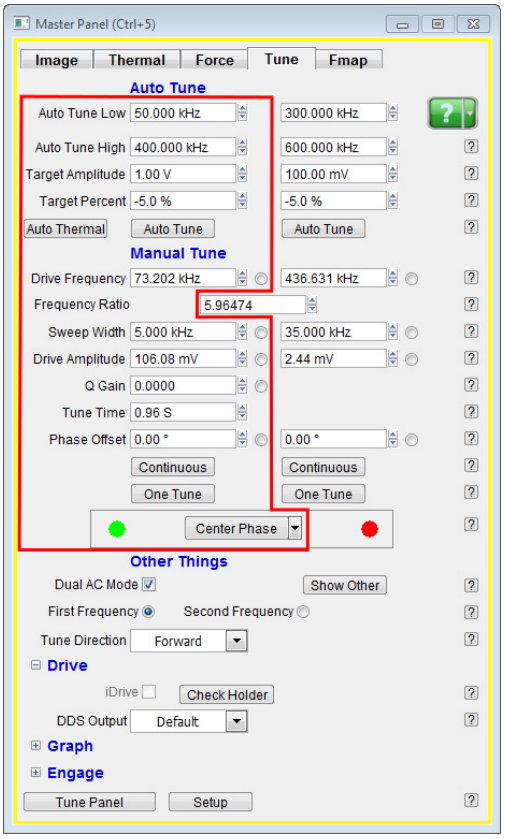


4. Go to the **Tune** tab of the **Master Panel (Ctrl+5)**.

5.

Auto Tuning: First Mode

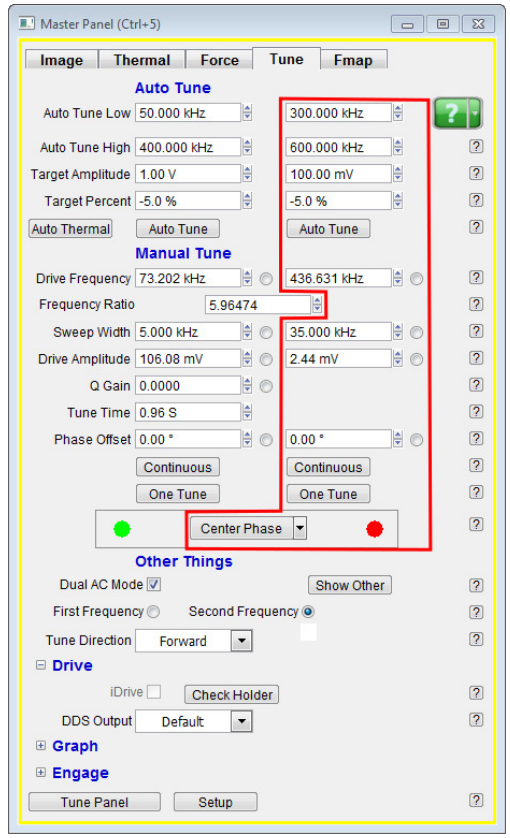
- Confirm that the *Auto Tune Low* and *Auto Tune High* field values cover a range of frequencies the will capture the cantilever resonances earlier determined. The left column are the first Eigenmode tune parameters and the right column are the higher Eigenmode tune parameters.
- Choose a *Target Amplitude* 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 on *Auto Tune*buttun in the left column. This will tune the cantilever as is it normally 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 the will capture the cantilever resonances earlier determined.
- 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 should choose a frequency and update 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 step it down later once imaging commences.

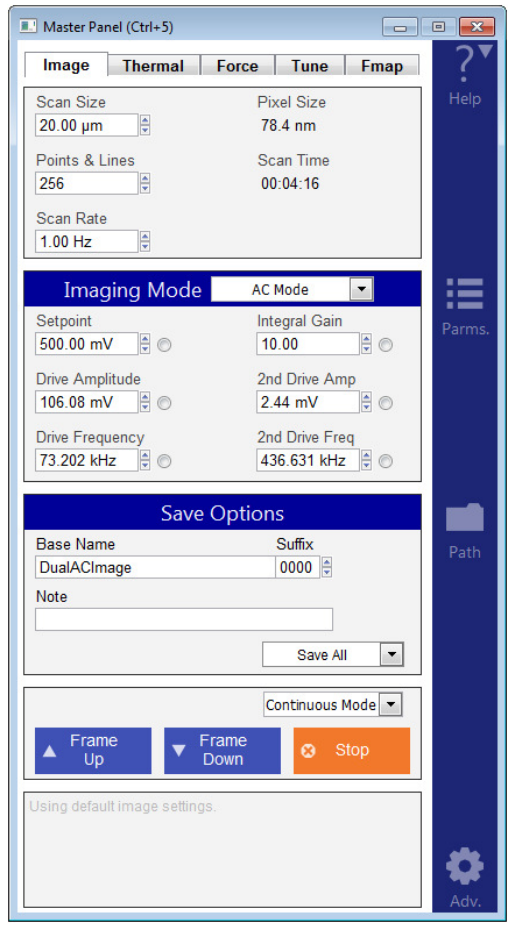


7. Perform a ‘soft engage’ as described in video tutorial for the Soft Engage Method.

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 (i.e., 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* paramteres also with radio button for image tuning.
- Start the scan.



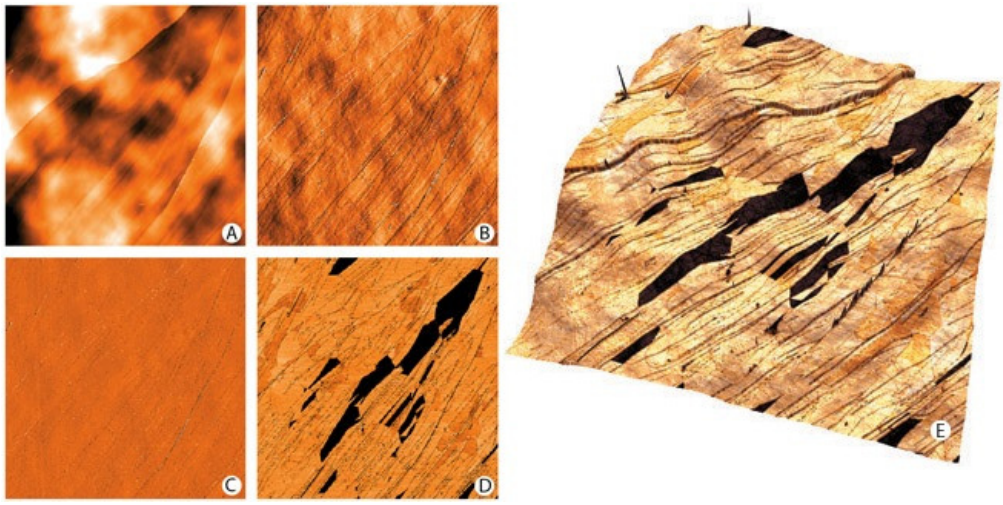


Figure 14.3.: 30µm image of a HOPG surface aquired with an Olympus AC240-R3 Silicon cantilever. (A) Height channel, (B) Amplitude 1 channel, (C) Phase 1 channel, (D) Amplitude 2 channel, (E) Phase 2 channel overlaid on 3D rendered Height channel.

14.4. DualAC™ Imaging in Liquid

DualAC™ in liquid while driving the cantilever acoustically (i.e., 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.

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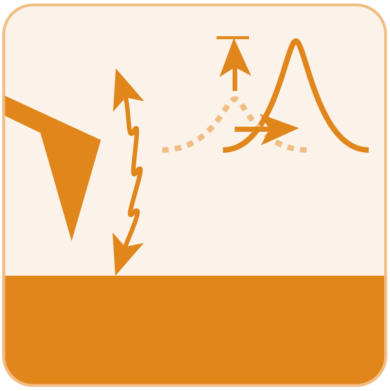
15. AM-FM: Viscoelastic Mapping

CHAPTER REV. 2042, DATED 08/31/2018, 16:24. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



Chapter Contents

15.1. System Requirements for AM-FM

- AM-FM is possible on all MFP-3D AFMs and Cypher AFMs.
- A **High Freqency Cantilever Holder** is highly recommended, but not necessary in all cases.
- If Cypher AFM is equipped with **blueDrive**, this can be used in place of the High Frequency Cantilever Holder 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

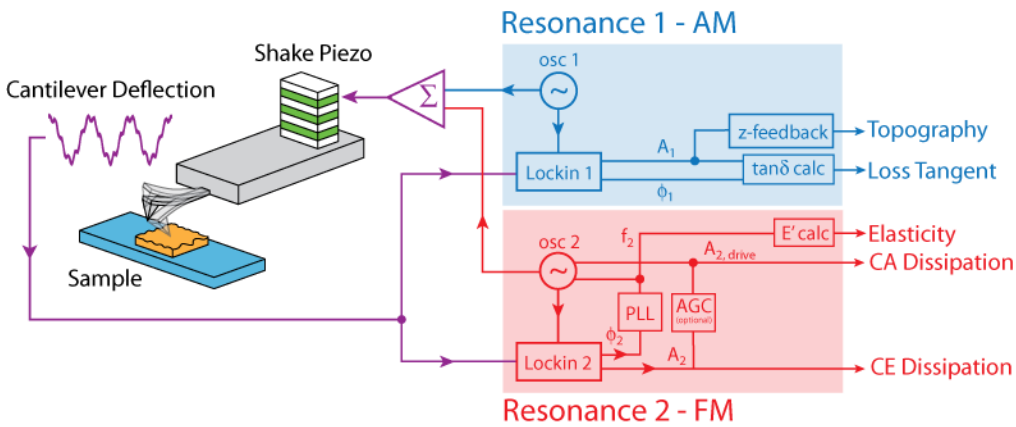


Figure 15.1.: AM-FM Mode. The first mode amplitude is controlled to create a topographic image of the sample (blue). The second mode is used to calculate elasticity and tip-sample stiffness (red).

AM-FM 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 simaltenously driven at two different mechanical resonances, as shown in Figure 15.1 on page 172. When operating in standard AM-FM mode, there are two, or an optional three, feedback loops running simaltaneously. 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 adjustes 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 convential Frequency Modulation (FM) mode, AM-FM is a quantitative technique in wich 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. Setting Up

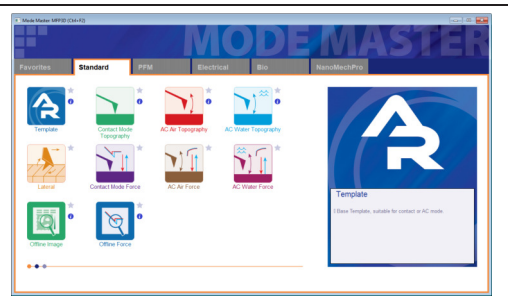
1. For the most recent features use the latest AFM software version 15 or later.

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 in to the High Frequency Cantilever Holder and attach 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.

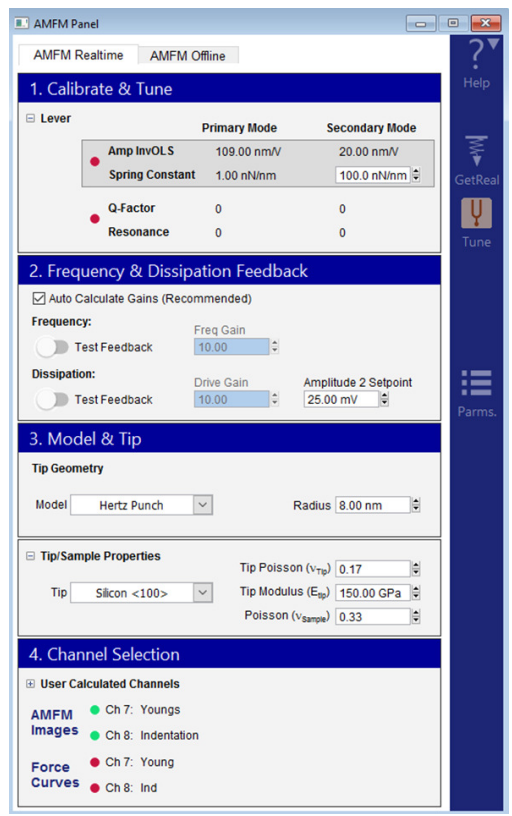
Select Mode:

- Select *NanoMechPro tab ▷ AMFM*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

6.

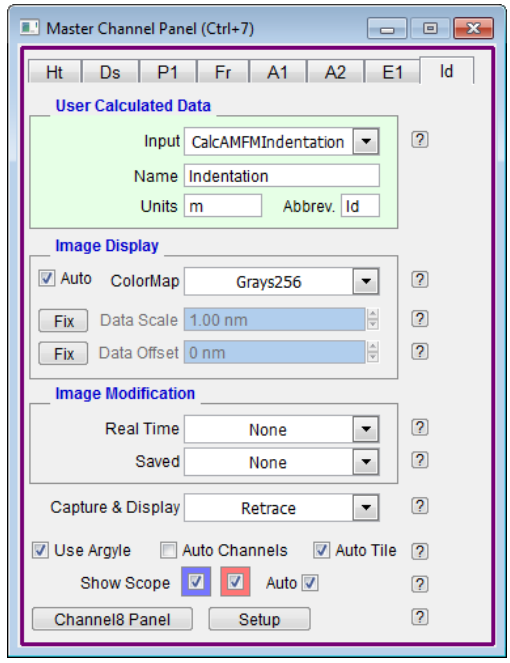
The AMFM Panel

- 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 realtime **tip modeling** parameters.
- The *Offline* tab includes parameters to calculate additional **stiffness/elasticity** images with various **tip models** for AM-FM images after the they have been collected.
.



7. **The Master Channel Panel**

   - The 8 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).



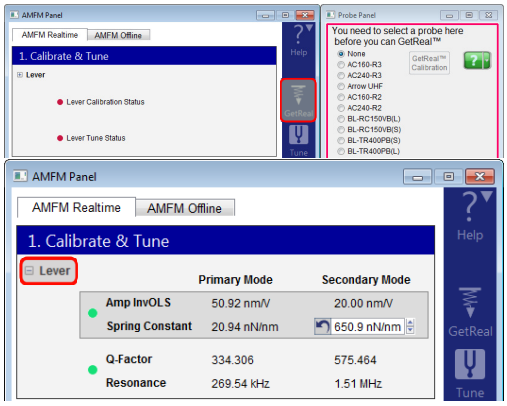
15.4.2. Calibration

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 279or the standard Thermal Method: **??** on page **??**. These instructions will outline GetReal™ cantilever calibration for AM-FM as it is easier, faster, more accurate, and will preserve the probe sharpness.

2.

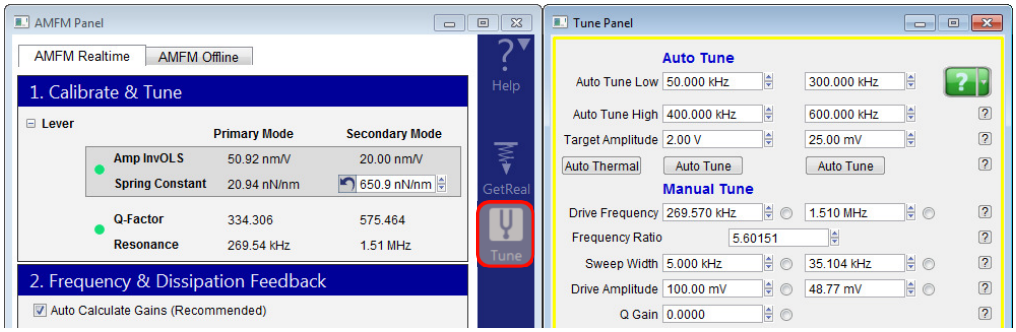
Probe Calibration

- Align your laser on the cantilever and zero your Deflection signal.
- Click the GetReal™ icon on the right column of the **AMFM Panel** and choose your probe from the list.
- Click the button for GetReal™ Calibration 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 both 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 equation 15.1.

15.4.3. Tuning



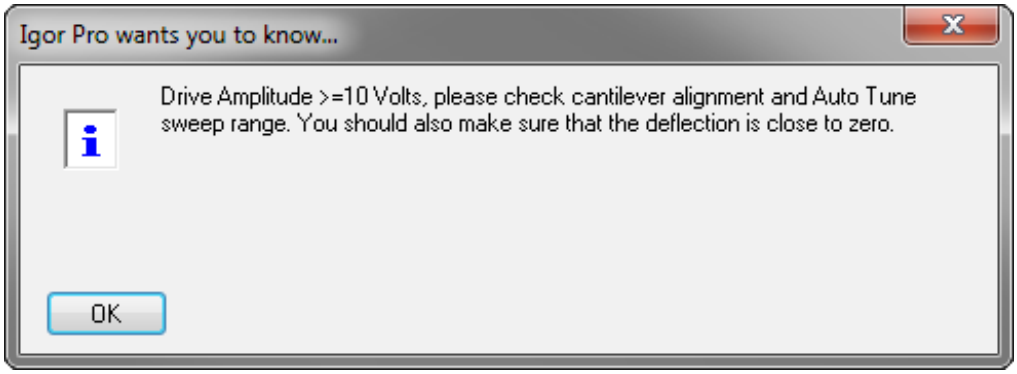
1.

Tuning the Cantilever

- Click the Tune icon on the right column of the **AMFM Panel**.
- Autotune the primary mode by 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 by 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

If you receive a message as seen here after autotuning, you may need to:

- Flip the attenuation switch on your cantilever holder (if you are using an MFP-3D). 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 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

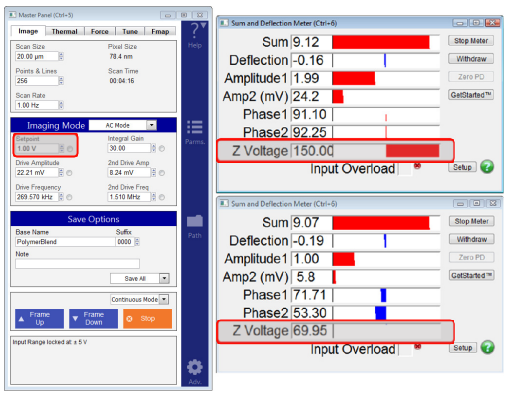
- You want the sensitivity of the optical beam deflection to be as high as possible for the secondary mode. To accomplish this, after you have successfully tuned, you should move the spot as far out to the end of the lever as possible. Observe the Amplitude 2 signal while you do this and pick a laser spot location where you've maximized the Amplitude 2 signal. If you lose a bit of signal in Amplitude 1 in order to maximize Ampltiude 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:

- Pick your imaging *Setpoint* in the **Master Panel**. ~40% of the free amplitude is a good prelimnary 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.

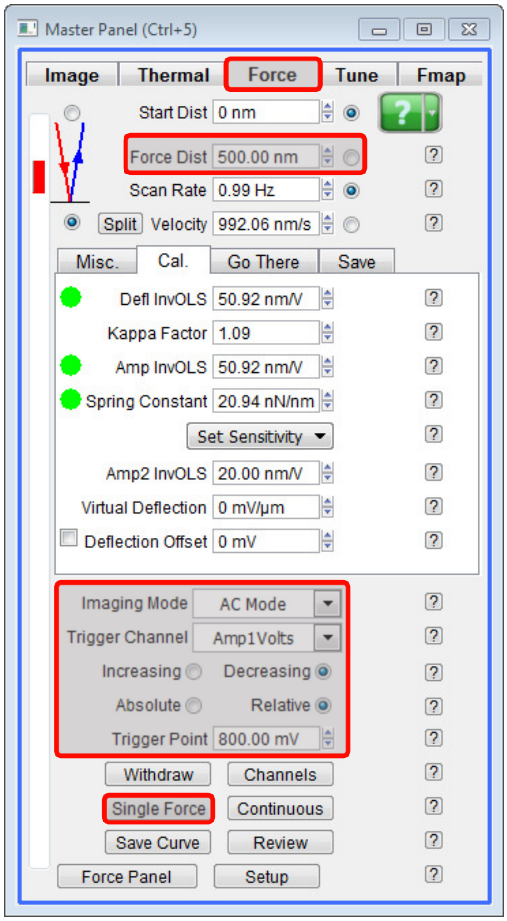


2.

AC Force Curve:

- Do an ~800mV Amplitude 1 Volts triggered AC force curve with a force distance of 500nm.
- Re-tune 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.

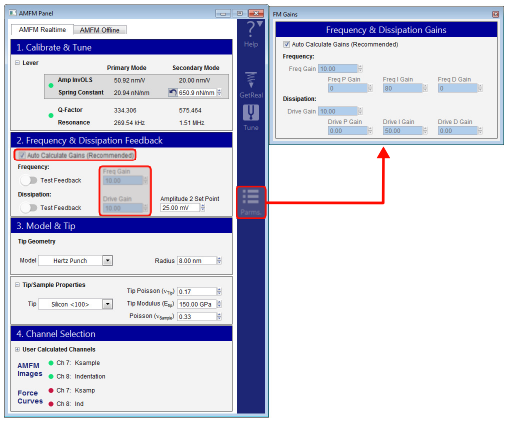
Note This step will help ensure you are exactly on resonance right at the sample surface as this typically will shift a bit from the inital tune as you near the surface.



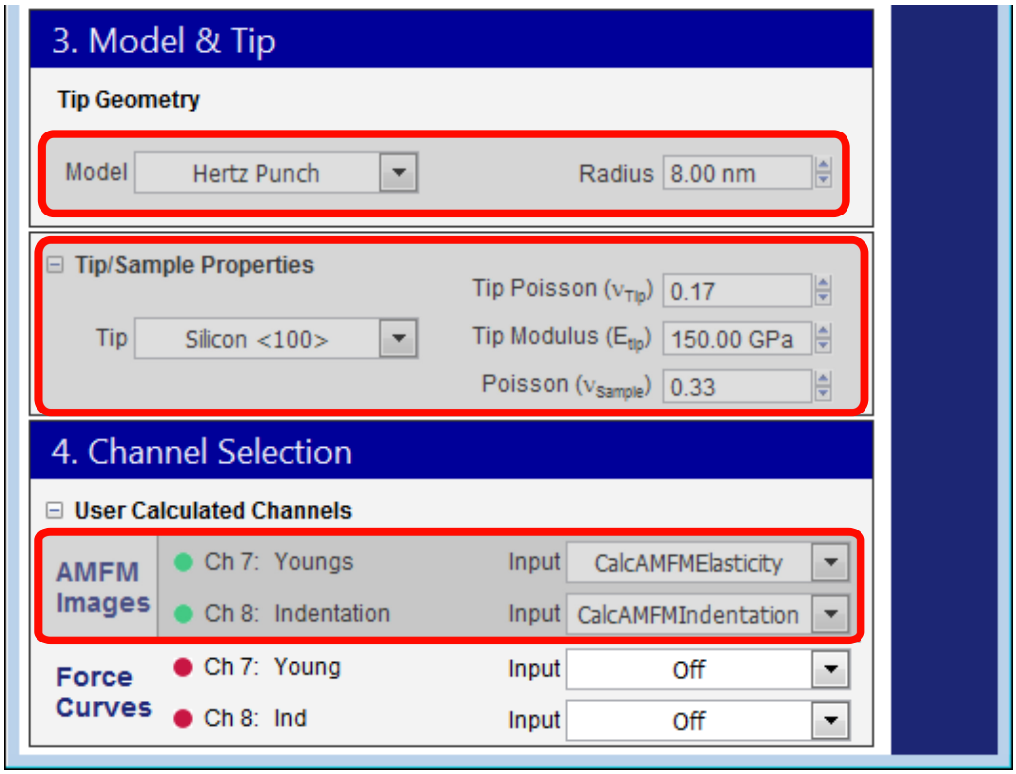
3.

Frequency & Dissipation Feedback:

- While engaged, click the 2 switch icons in the **AMFM Panel** to *Test Feedback* of the Frequency and Dissipation.
- Ensure both Phase 2 and Amp2 appear stable in the Sum and Deflection Meter with *Test Feedback* on.
- If they are not, you may need to manually adjust the feedback gains. Deselect the check box labeled *Auto Calculate Gains*, select the Parms icon on the right column of the **AMFM Panel**, and adjust the P, I, D gains accordingly until stable.



4.



Model & Tip

The last step do before scanning is to:

a) Choose the tip model

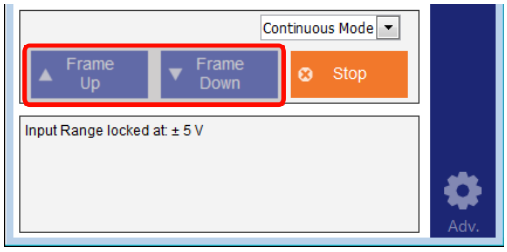
b) Adjust the tip/sample properties

c) Decide which calculated channels you will like to display during the scan.

5.

Begin the Scan

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

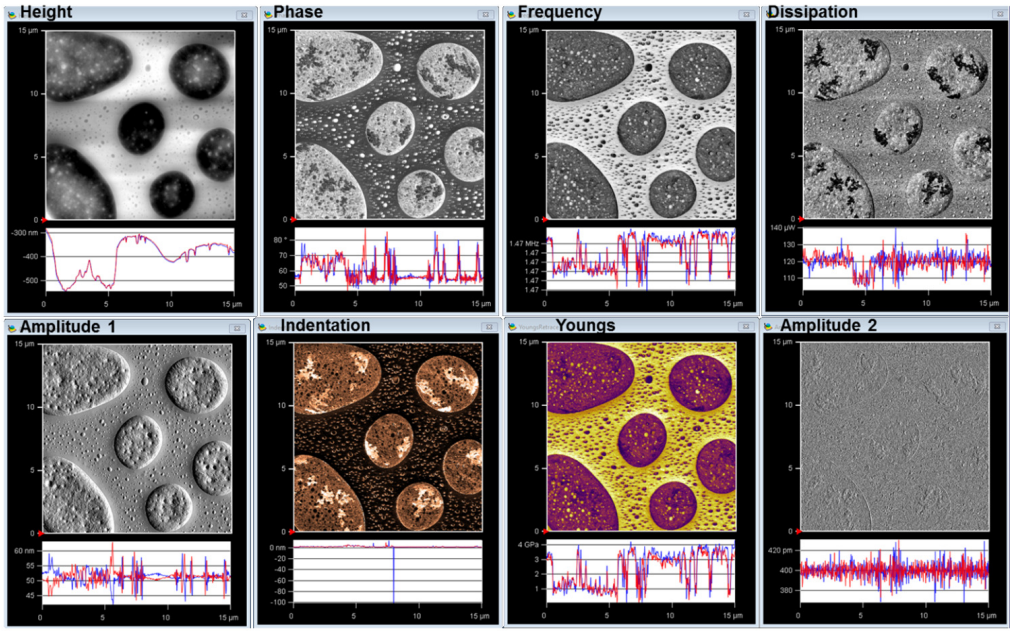


15.4.5. Optimizing the Scan

1.

Repulsive Mode

- It is important to have the cantilever 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* or in the **Master Panel** to maintain 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, it is possible the cantilever tip may have become too blunt or contaminated and should be changed.



Optimizing the Scan

2. 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, just back down the gains a little. This is only causing instabilities 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

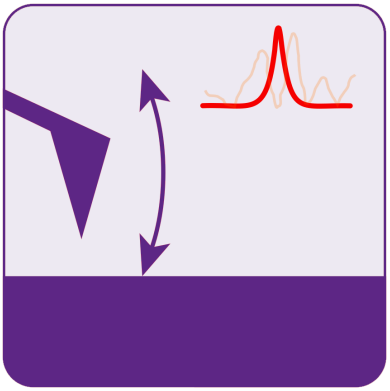
15.5.1. Reference Sample

The AM-FM technique requires a reference sample in order to determine accurate quantitative nanomechanical properties of unknown materials. This is mainly a result of 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 wish to measure modulus information 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. 2038, DATED 08/29/2018, 16:17. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



Chapter Contents

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 184. 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.

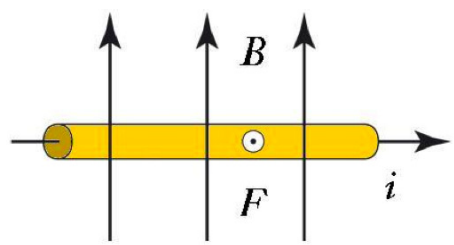


Figure 16.1.: The Lorentz Force F is orthogonal to both current i and the magnetic field B and is pointing out of the page.

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.

[1] Buguin, A./Roure, O. Du/Silberzan, P., Active atomic force microscopy cantilevers for imaging in liquids. Applied Physics Letters, 78 2001 No. 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 No. 1.

[3] Han, Wenhai/Lindsay, S. M./Jing, Tianwei, A magnetically driven oscillating probe microscope for operation in liquids. Applied Physics Letters, 69 1996 No. 26 ⟨URL: http://link.aip.org/link/?APL/69/4111/1⟩.

This means that the tunes are smooth and that other parts of the microscope mechanics are not being excited.

- Because the drive is well defined and is a smooth function of frequency, even in liquid, the phase signal of the cantilever can be used in a manner similar to the phase signal in air. This can lead to some very interesting contrast (Figure 16.2 on page 185, Insulin Fibrils) and even allows advanced techniques such as Q-control.

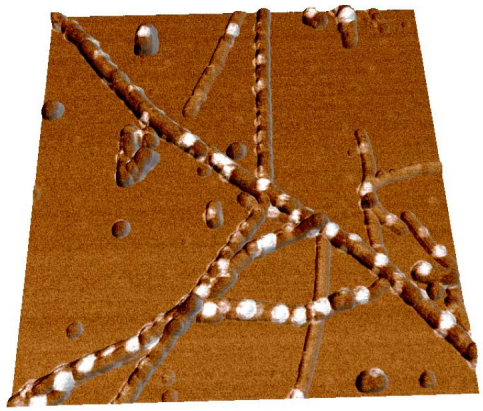


Figure 16.2.: Insulin Fibrils imaged in water. Phase signal overlaid onto topography. The "white" regions in the image are from the phase channel and correspond to higher energy dissipation. 1μm scan.

16.2. iDrive Requirements

Attention	iDrive imaging requires a specialized iDrive cantilever holder and specialized cantilevers for operation.

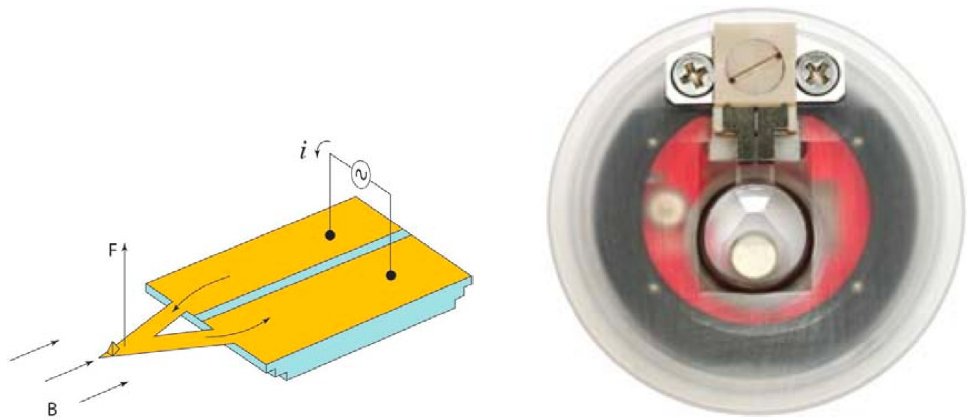
16.2.1. Cantilevers

A generalized schematic of a iDrive cantilever is shown in 16.3a. This hows 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 186) 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

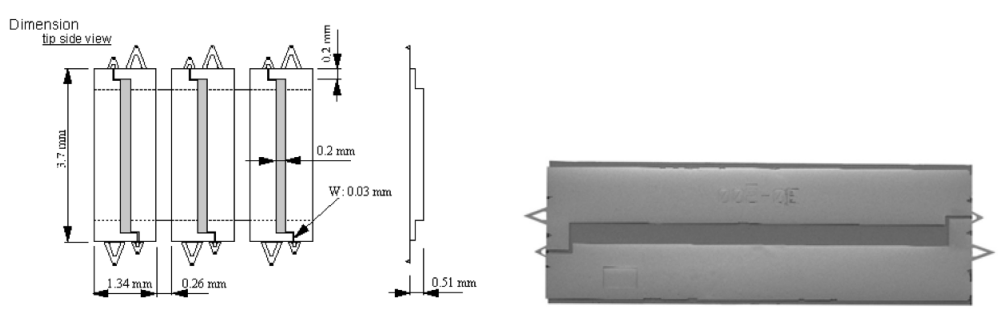
The specialized cantilevers also require a specialized cantilever holder to apply the bias to one side of the chip. This is a modified standard holder with an included NbFeB magnet in the glass insert, to provide that magnetic field (Figure 16.3b on page 186). The fully enclosed magnet is completely 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 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 (i.e. no peak appears in the cantilever tune).

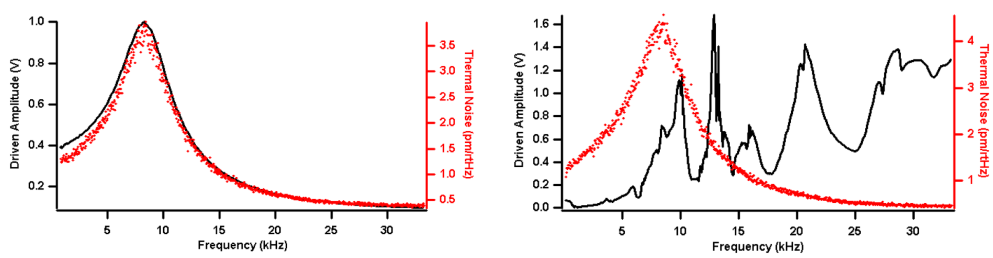
The instructions to measure the resistance across the cantilever chip are listed below. You will need a multimeter.

1. Select "resistance" on your multimeter and, if necessary, the low resistance range.
2. Place the probes on each side of the cantilever chip (i.e. 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, then 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 enviroment. 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 187, where both methods are compared to the Brownian "thermal" tune.

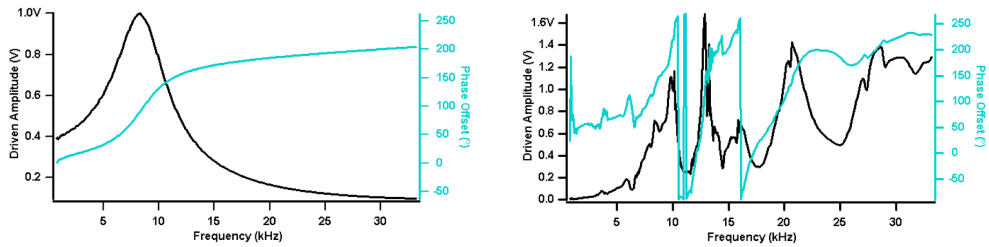


(a) iDrive **(b)** Acoustic

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 188.

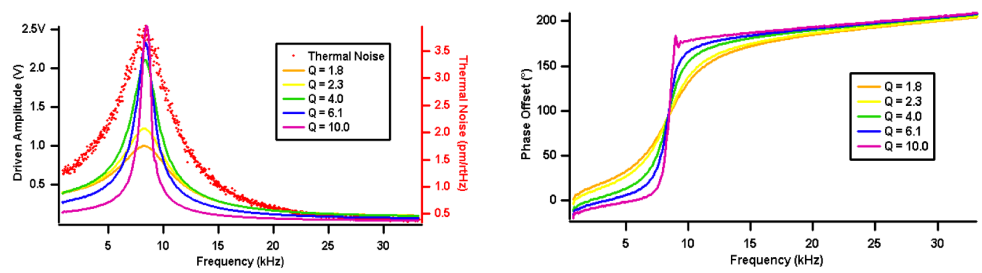


(a) iDrive **(b)** Acoustic

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; 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 188).



(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 189, 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.

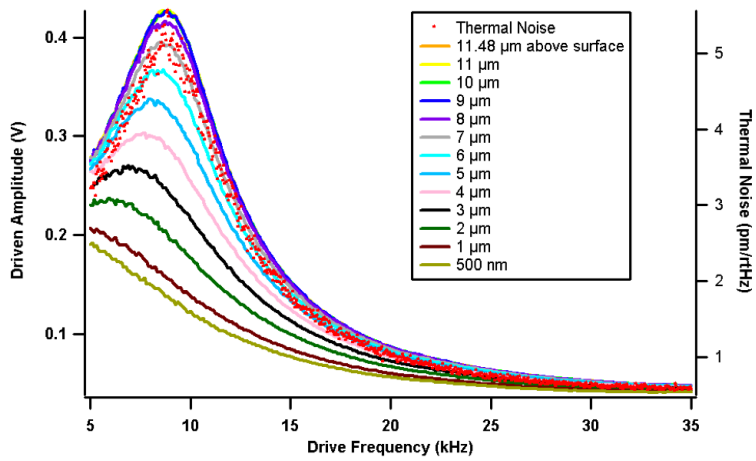
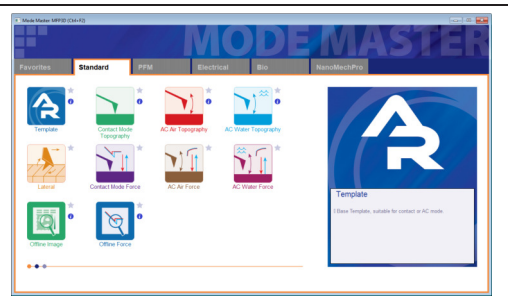


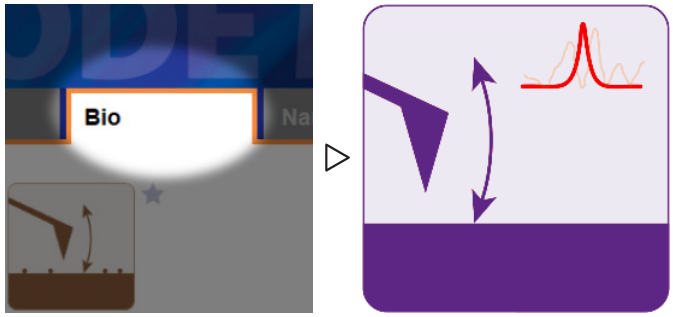
Figure 16.8.: Cantilever amplitudes at different Z positions using iDrive AC mode

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 No. 3-4 ⟨URL: http://dx.doi.org/10.1016/j.ultramic.2003.12.014⟩.

2. 

Select Mode:

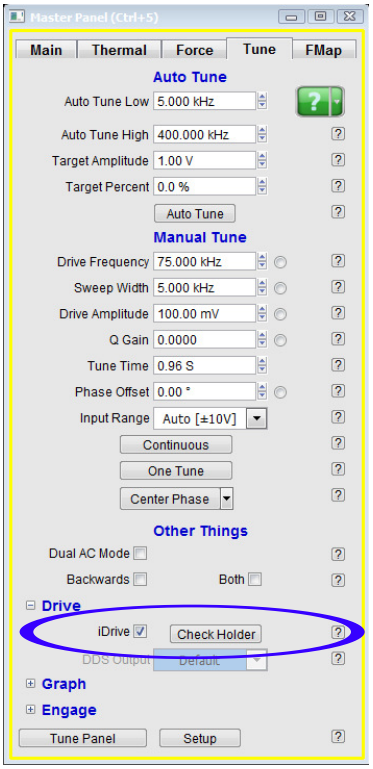
- Select *Bio tab* ▷ *iDrive*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

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

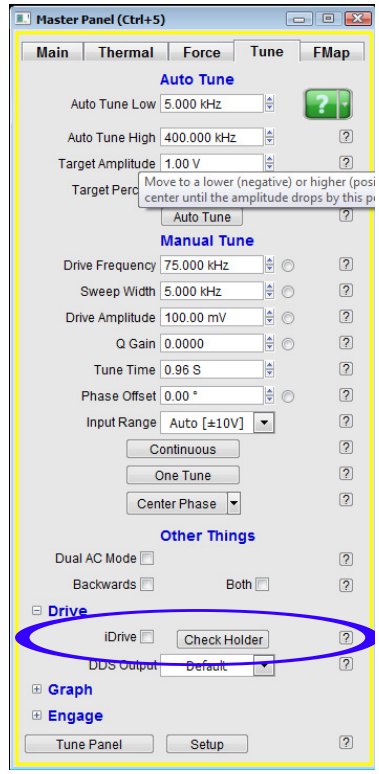
There is no special sample preparation required for iDrive. Standard glass slides with mica, gold or any other substrate can be used. First, install an iDrive compatible cantilever (Model AR-iDrive-N01) into the cantilever holder pocket, as usual, and tighten the PEEK screw until the cantilever chip is secure (do not over tighten).

Similar to operating in piezo-driven AC mode, you will need to:

1. Tune the cantilever, much as described in **??** on page **??**. The exception is that you need to make sure that the iDrive checkbox is checked as shown below 16.9a.
2. Select the appropriate amplitude setpoint value and approach towards the surface (Main Panel)



(a) standard cantilever holders



(b) iDrive cantilever holders but iDrive not selected

Figure 16.9.: Tune Panel configurations.

16.5.1. Tuning 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, then the iDrive box will be shaded (Figure 16.9a on page 191). To enable the iDrive box, attach the iDrive cantilever holder to the MFP-3D head and click on the *Check Holder* button (Figure 16.9b on page 191).

When iDrive is selected, the *Auto Tune Low* parameter will decrease 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 then click on 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 Window 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 window equals the amplitude setpoint and the computer will beep (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 188, it is recommended that you re-tune the cantilever once the tip is on the surface. To do this, click on the *Stop* button, go to the Tune Panel and click on the *Auto Tune* button. Alternatively, the use can choose to enter a *Target Percent* of negative (-) 5-10% which will automatically select a resonant frequency that is 5-10% off and to the left of the resonant peak. This offset will compensate 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.

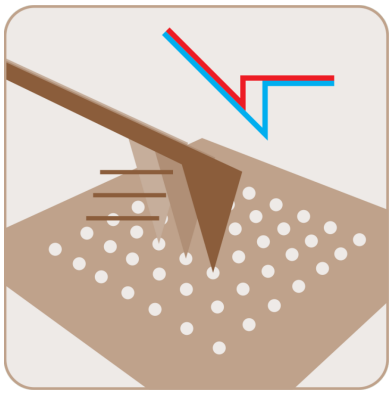
For these particular cantilevers the optimal amplitude setpoint is 200mV or less. Decrease the drive amplitude and setpoint to achieve this final setpoint value. For example, if the tip is tracking the surface well with an amplitude setpoint of 325mV then decrease first the drive amplitude then the amplitude 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.

16.6. iDrive Cantilever Holder Maintenance

As with the standard cantilever holder it is very important to rinse the surface of the holder in order to clean any salt buffer that may contaminate future samples or corrode the metal clamp. Gently rinse with pure water and/or ethanol, and then blow dry with compressed gas. Try to avoid wetting the back of the holder where the circuit board is located. To thoroughly clean the cantilever holder, it can be taken apart and the top (KelF or PEEK portion of the holder) can be sonicated and/or autoclaved.

17. Fast Force Mapping

CHAPTER REV. 2007, DATED 03/27/2018, 20:30. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.



Chapter Contents

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 liscence to be activated.
- AFM software version 15 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 users to acquire data (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, the user 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 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 eletrical bias is applied to the sample and a conductive tip is used, ORCA measurements (See: 9) can be performed at the same time.

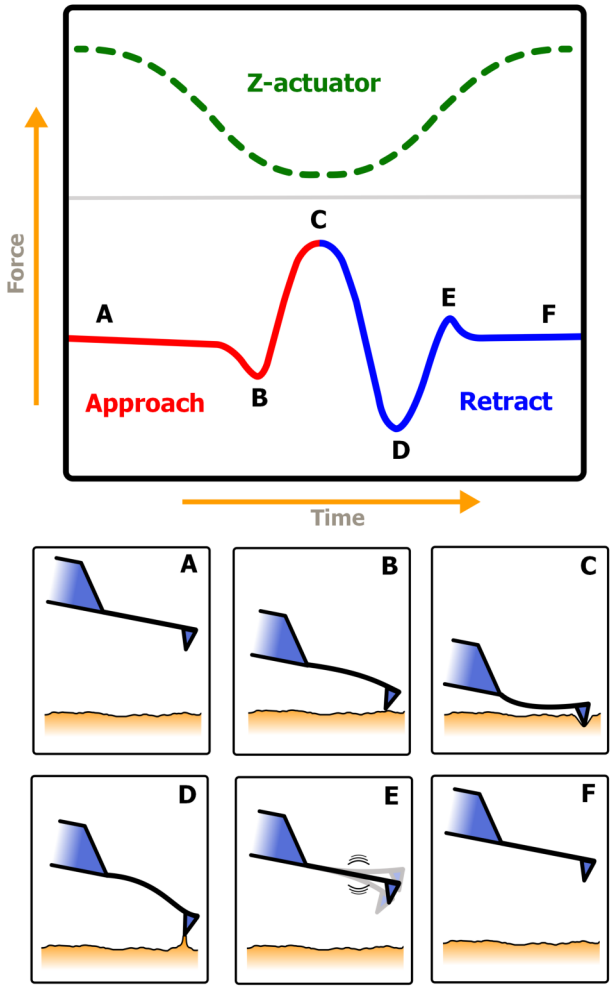


Figure 17.1.: Diagram of general Fast Force Map technique. As the Z-actuator changes position sinusoidally versus time, the cantilever force does as well. A, B, C, D, E, and F show discrete points of interest along the curve during the cantilever approach and retraction.

17.3. Fast Force Mapping in Contact Mode

Fast Force Mapping (FFM) is naitvely a contact mode-based technique, therfore the user needs to choose a probe with a spring constant matching the stiffness of the sample.

For this demostration and example we will use:

- A polystyrene/polypropylene thin film sample
- AC200TS Olympus probe with ~150 kHz resonance and ~10 N/m spring constant

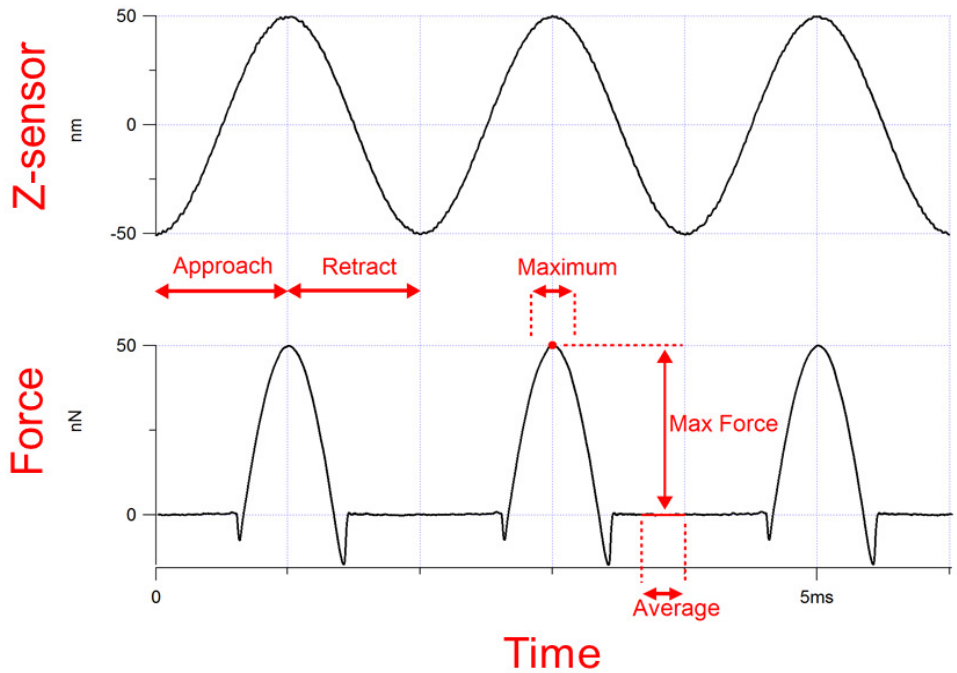
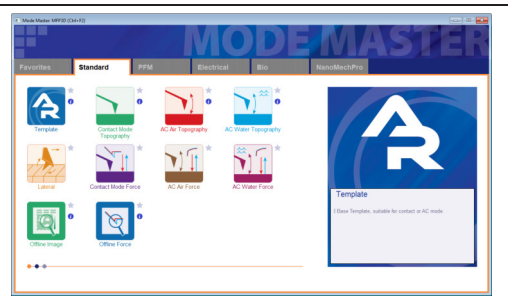


Figure 17.2.: Plot of cantilever oscillation measured by the z-senor (excited by driving voltage) (top) and the resulting force (bottom) when the tip is pushing on a hard surface. The 'Max Force' arrow indicates how the setpoint force is calculated for each force curve.

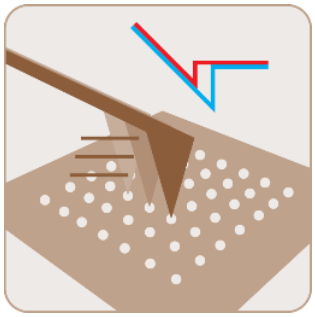
17.3.1. FFM Automated Setup: GetStarted™

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:

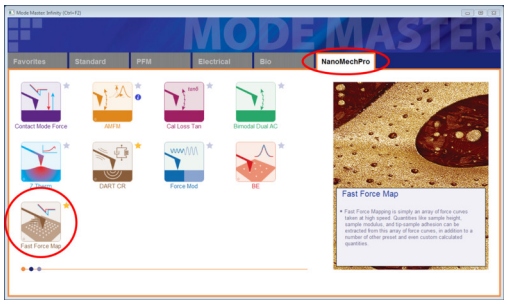
- Select *NanoMechPro tab* ▷ *Fast Force Map*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

Note Loading Fast Force Mode from the Mode Master will automatically start in the **GetStarted™** configuration.

3.

Automatic Imaging Setup: GetStarted™

- Follow the instructions to set up your experiment
- You will be asked to set initial imaging parameters (look for descriptions below)
  - scan size
  - scan points & lines
  - approximate sample roughness
  - Z-rate
  - setpoint
  - force distance
  - sample stiffness
- Some of these parameters are adjusted automatically depending on user input



Specific Parameters for FFM - GetStarted™

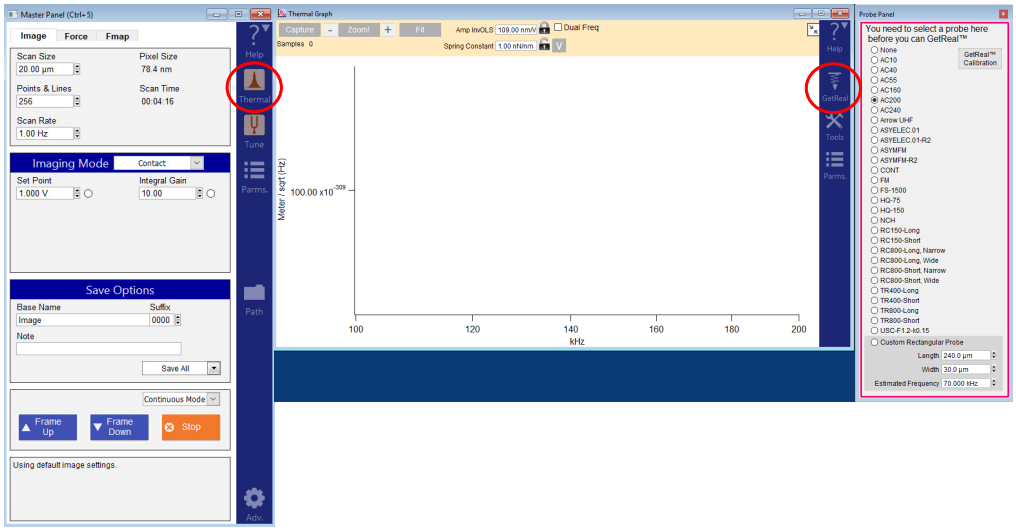
- Scan points & Lines
  - These two parameters determine the number of scan points (pixels) in each scan line and the number of lines in the scanned area. In contrast to other techniques, during FFM the number of force curves corresponds exactly to the number of pixels in the image. For comparison, during 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 2 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 the lowest and highest features on the sample
- Z-rate
  - It's the ramp rate at which the force curves will be performed and it is how fast the Z actuator is moving up and down above the surface. This parameter can be increased until the user sees 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.
- Setpoint
  - It 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. 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
  - It is the distance the piezo will travel during the force curve (extend or retract portion). This distance should match (or exceed) the topography variations of the sample.
- Sample Stiffness

4.

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

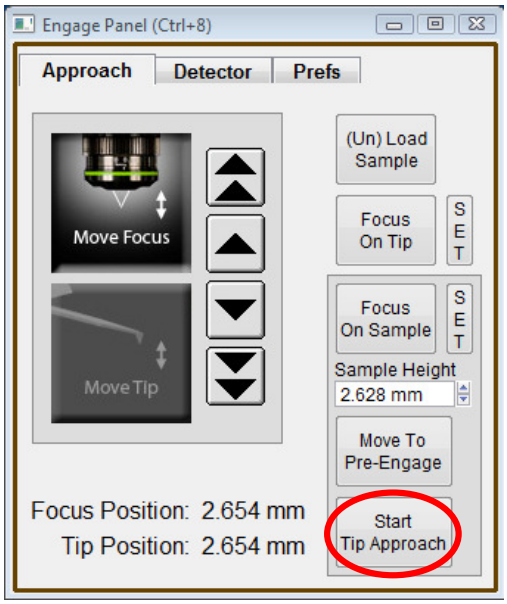
2. 

   Calibrate Probe

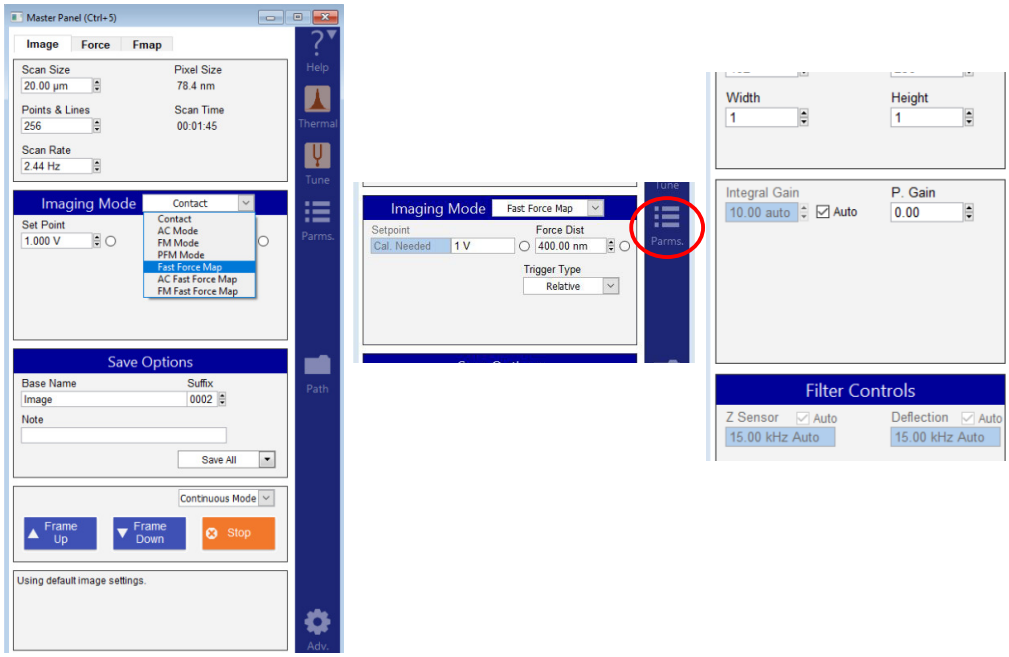
   - Choose the Thermal icon in the Master Panel
     - thermal graph will appear
   - Click on the Get Real icon which in trun will open a Probe Panel
   - Choose the probe that is used for the experiment
   - Click GetReal Calibration
   - Once the calibration is compleated, the Amp InvOLS and Spring Constant values at the top part of the thermal graph will be updated

3. **Approach the sample surface**

- In Master Panel
  - Imaging Mode: Contatct
  - Set Point: 0.2 V (for this particular probe and sample)
- In the Engage Panel
  - Set the tip position while focused on the tip
  - Set the sample position while focused on the sample
  - Click on Start Tip Approach



4.



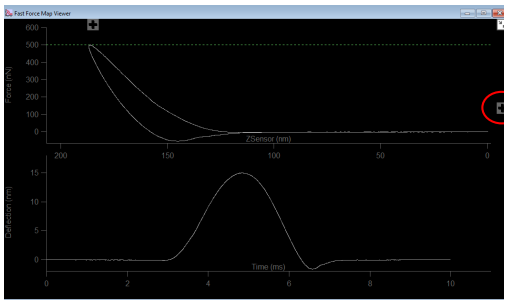
Switch to FFM mode and start imaging

- In Master Panel, switch from Contact Mode to Fast Force Map
- Master Panel will now display parameters related to FFM
  - Setpoint is now displayed in units of Newtons and in Volts: set it to 10 nN
  - Force Distance: 200 nm
  - Trigger Type: Relative
- Set the other parameters in the Master Panel
  - Scan size: 5 µm
  - scan points & lines: 256
  - Z-rate: 500 Hz
- The Gains are located in the Parms panel
- Click Engage in the Sum and Deflection Panel

5.

Engage

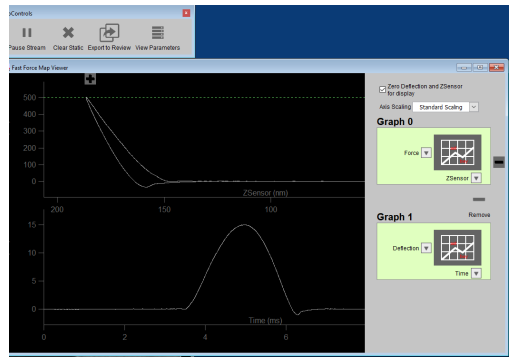
- As the tip engages on the surface, Fast Force Map Viewer will appear
- Display choices are available by clicking on the “plus” sign



6.

Real time Force curves

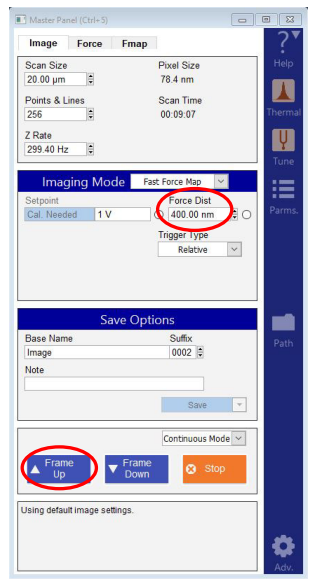
- Fast Force Map Viewer can show up to 2 channels
- Each graph has several options for each axis, in this example:
  - Graph 0 is Force versus ZSensor
  - Graph 1 is Deflection versus time
- The green line on the top graph indicated the setpoint
- Top Controls allow for superposition of several already acquired curves over the realtime curve

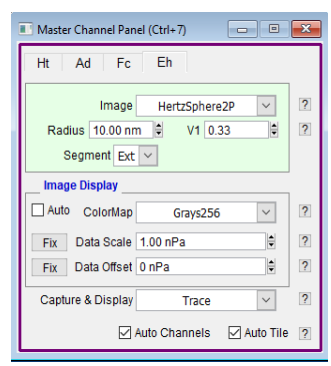


7.

Imaging parameters

- Four Channels can be collected simultanously during FFM imaging
  - Ht - Height
  - Ad - Adhesion
  - Fc - Max Force
  - Eh - Calculated modulus channel. Model (here Hertz) and tip radius must be adjusted prior to imaging
- Adjust the force distance and setpoint
- When the imaging parameters are optimized and the force curves look good in the FFM viewer press Frame UP to start imaging





17.4. Fast Force Mapping in AC mode

The Fast Force Mapping technique can also be operated while simaltaneously oscillating the cantilever at resonance, instead of in contact mode.

The following demostration is for use with a Cypher AFM equipped with blueDrive and is intended for imaging a flat sample in liquid.

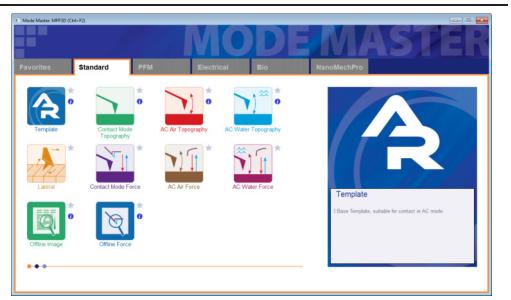
17.4.1. Preparation and Calibration

1. **Prepare**

   - Fresh calcite sample
   - Clean cantilever holder that will be used for imaging (either liquid holder or perfusion holder)
   - Load a FS-1500AuD probe (f = ~1.5 MHz, k = ~6 N/m) in to the cantilever holder.
   - AFM software version 16 or higher and the FFM mode is 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:

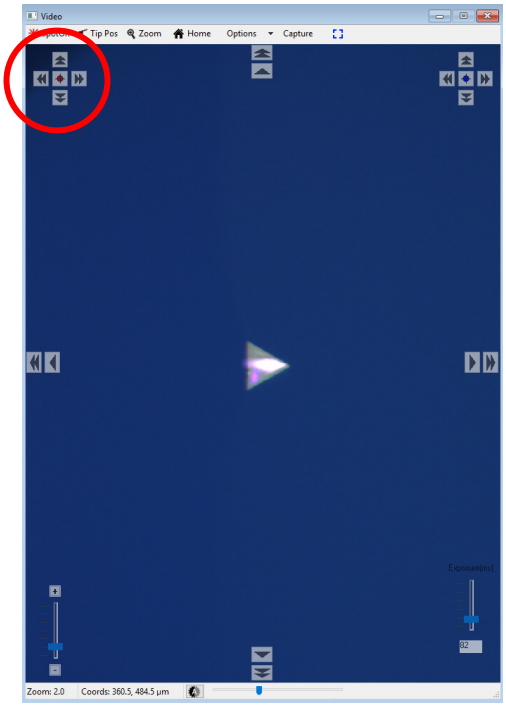
   - Select *Standard tab* ▷ *Template*
   - The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

Note Loading Template from the Mode Master will automatically start in a nonspecific set up of Contact Mode.

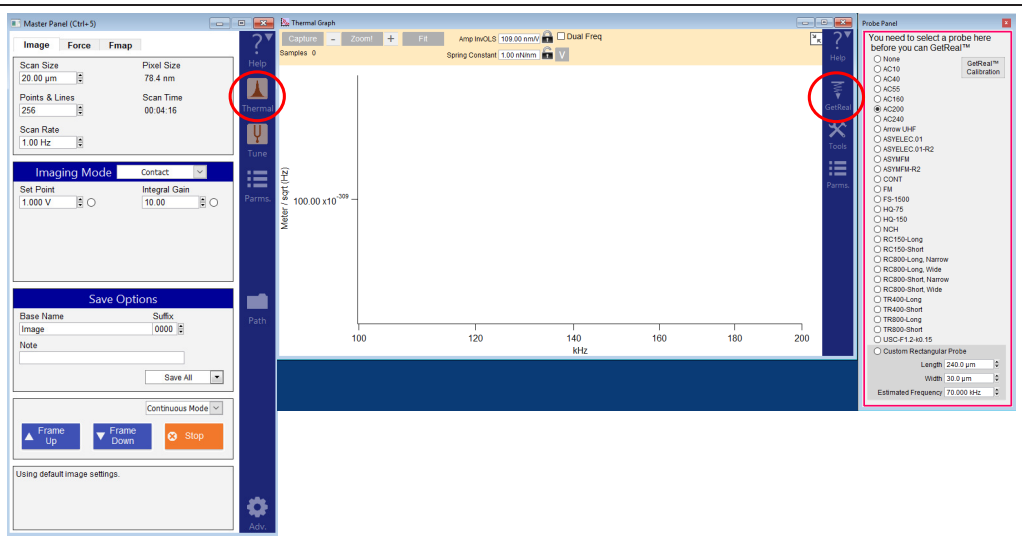
4.

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



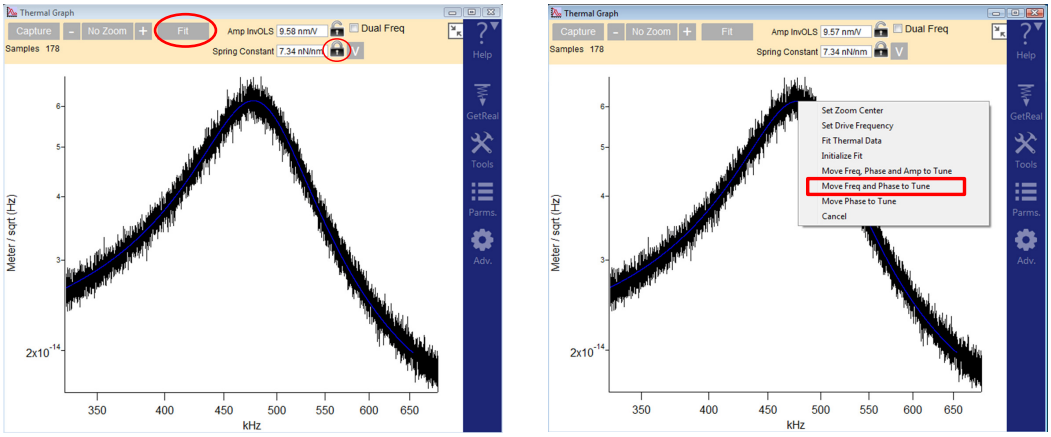
5.



Calibrate Probe (in air)

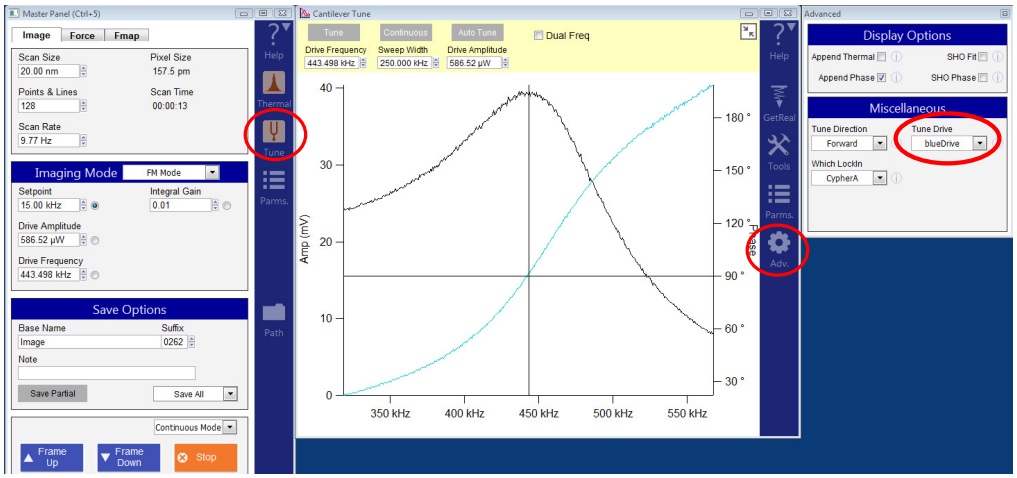
- Choose the Thermal icon in the Master Panel
  - thermal graph will appear
- Click on the Get Real icon which in trun will open a Probe Panel
- Choose the probe that is used for the experiment
- Click GetReal Calibration
- Once the calibration is compleated, the Amp InvOLS and Spring Constant values 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. Re-focus and re-align the laser on the cantilever (now in liquid) - Sum should be ~ 6V
11. 

Capture a thermal of the cantilever in liquid:

- Make sure that the padlock besides Spring Constant is locked
- Re-fit 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. 

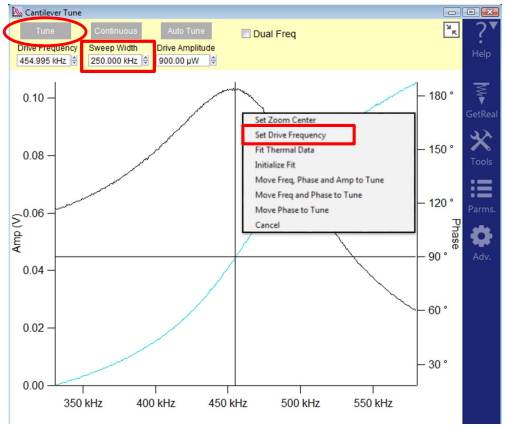
Turn on blueDrive photothermal excitation

- In the Tune graph, click on Adv.
- In the Advanced panel, choose blueDrive form the drop down menu of Tune Drive

13.

Tune panel

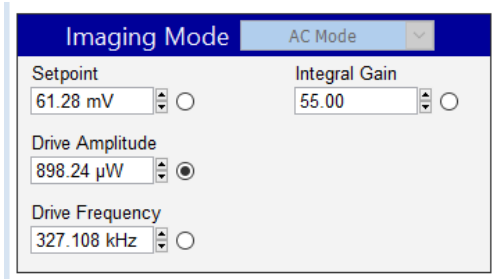
- Set the Sweep width to 250 kHz
- Click one Tune
- When tune is done, right click on the peak and choose set Drive Frequency
- Move the blue laser spot around the base of the cantilever to maximize the amplitude
- Adjust the drive amplitude (in the Master Panel) until the amplitude reaches ~ 80 mV (visible on the graph and in the Sum and Deflection panel)



14.

Master Panel

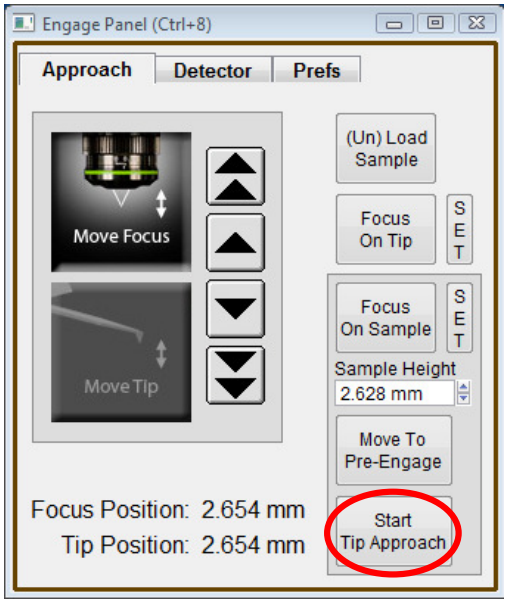
- Set the setpoint to ~ 60 mV
- Set the IG to 55



15.

Engage Panel

- Set the tip position while focused on the tip
- Set the sample position while focused on the sample
- Click on Move to Pre-Engage
- Click on Start Tip Approach



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 besides spring constant on Thermal Graph to re-calculate Amplitude InvOLS

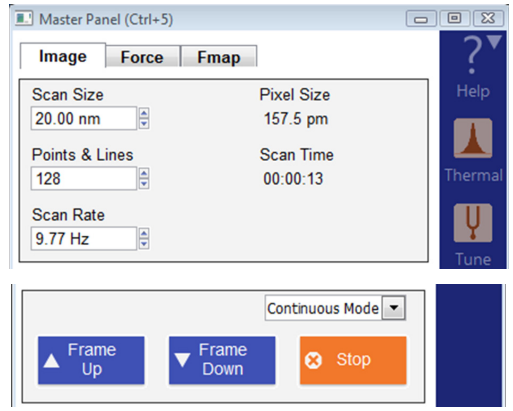
18. Right click on the peak in Thermal Graph and Move Freq and Phase to Tune

19. When the tune is complete,right click on the peak and center phase

17.4.2. Imaging in AC Mode

1. **Imaging parameters - Master panel**

   - Set the scan size to 20 nm
   - Set the scan rate to 10 Hz
   - Start imaging by pressing Scan Down



2. Start imaging

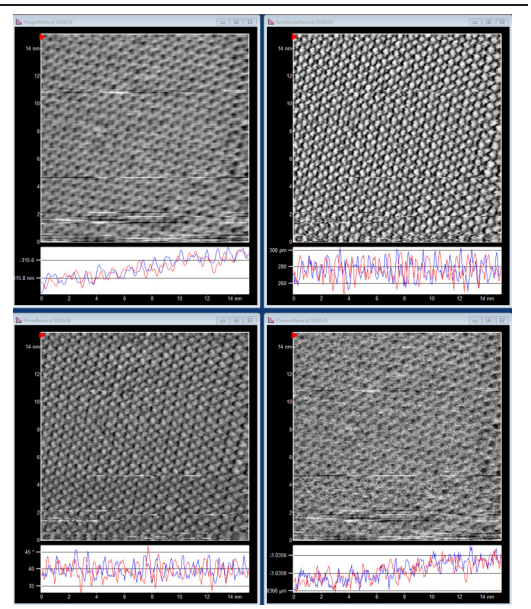
   a) decrease the free amplitude (by decreasing drive amplitude in the Master Panel)

   b) then, decrease the setpoint unil trace and retrace overlap each other

   c) repeat several times to image with the lowest amplitude possible

3. **Imaging**

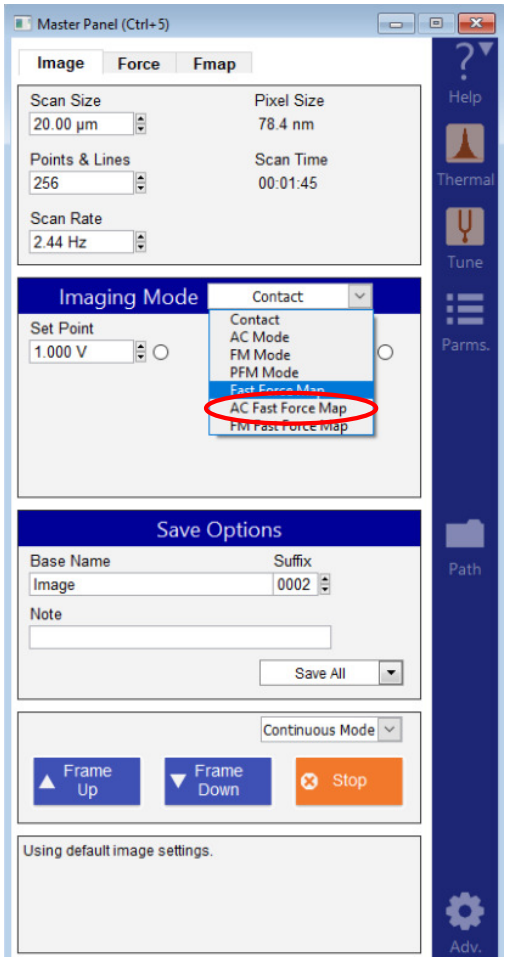
   - Acquire a 20 nm image and re-size the scan to 10 nm
   - Channels that are acquired include
     - Height
     - Amplitude
     - Phase
     - ZSensor
   - Once the imaging looks optimized, stop the scan and switch to AC-FFM mode



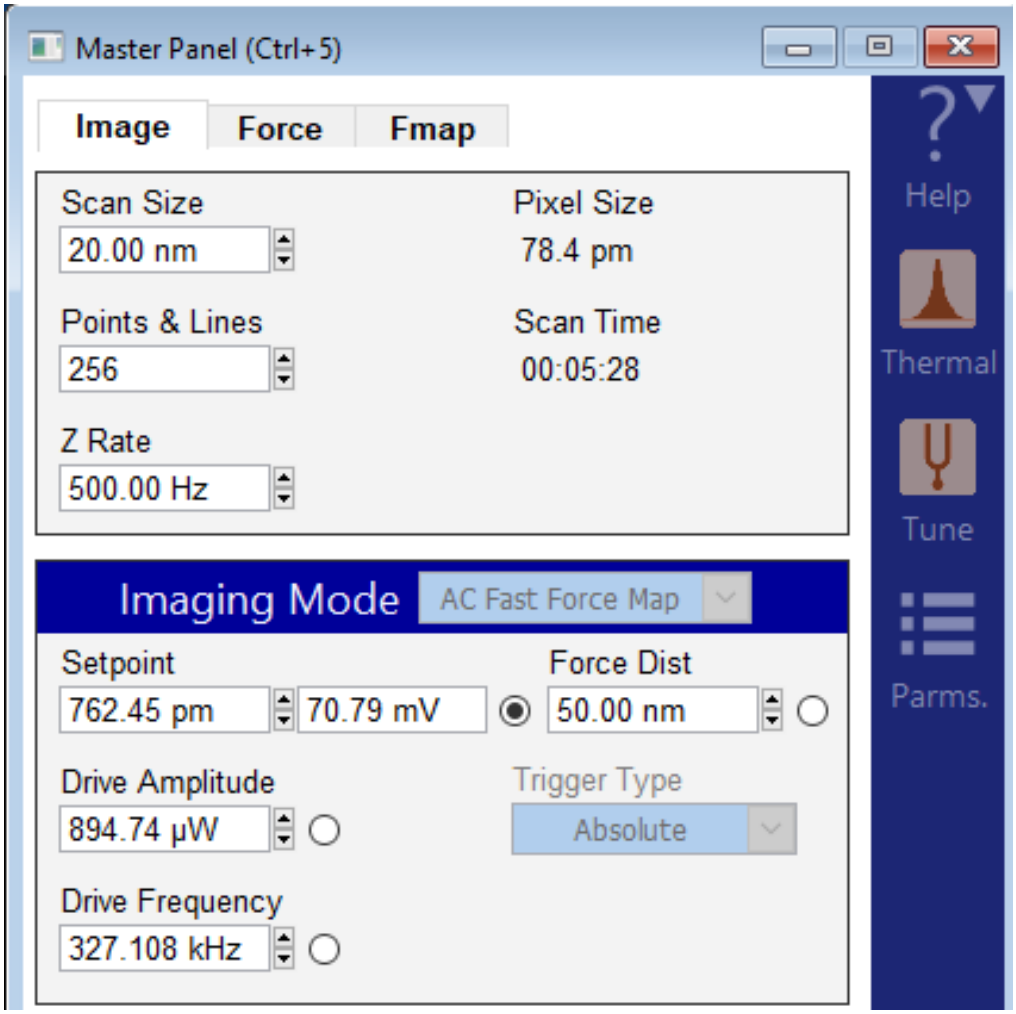
17.4.3. Imaging with AC-FFM

Switching to AC-FFM

1. • switch to AC Fast Force Maps from the drop down menu in Master Panel



2.



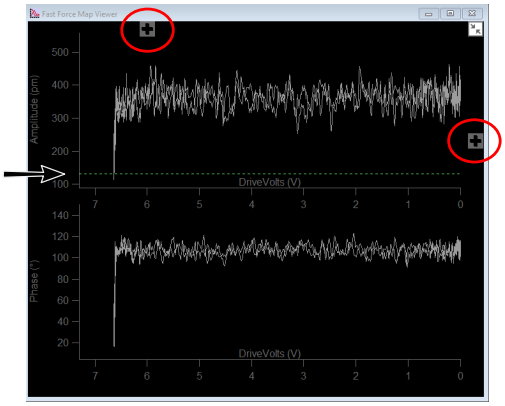
Imaging Parameters

- Master Panel will now display parameters related to AC-FFM mode
  - Setpoint is now displayed in units of meters and in Volts - set it to 70 mV
  - Force Distance - set it to 50 nm
  - Trigger Type - Absolute
- Set the other parameters in the Master Panel
  - Scan size: 20 nm
  - scan points & lines: 256
  - Z-rate: 500 Hz
- The Gains are located in the Parms panel - set to 10
- Click Engage in the Sum and Deflection Panel

3.

Engage

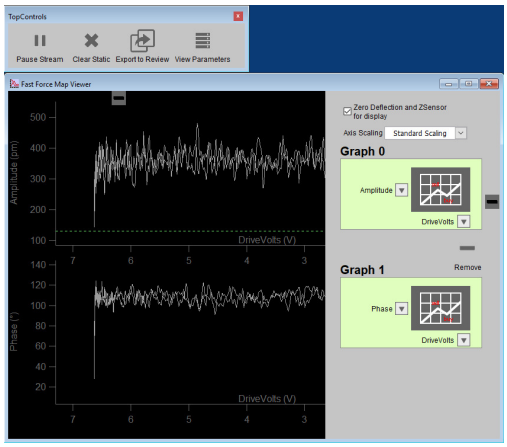
- As the tip engages on the surface, Fast Force Map Viewer will appear
- Display choices are available by clicking on the "plus" sign
- Arrow in the top graph indicates the setpoint value.



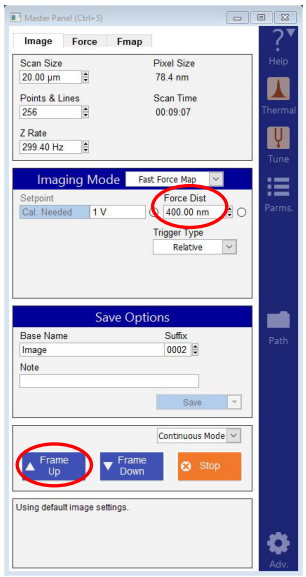
4.

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)
- Top Controls allow for the superposition of several already acquired curves over the realtime curve

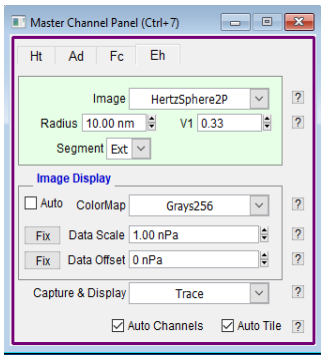


5.

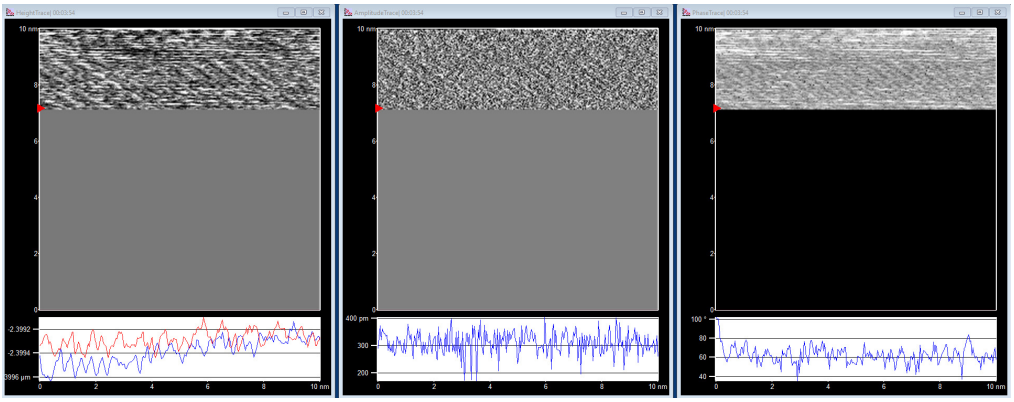


Imaging parameters

- Adjust the force distance and setpoint until the curves in the Viewer look good
- When the imaging parameters are optimized press 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 Fast Force Mapping technique can also be operated while simaltaneously oscillating the cantilever at resonance, instead of in contact mode, with a phase-lock loop to maintain the cantilever drive is on resonance.

The following demostration is for use with a Cypher AFM equipped with blueDrive and is intended for imaging a flat sample in liquid.

The user is guided through the following steps:

1) Calibration of a probe

2) Acquisition of an AC mode image - this step will allow the user 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

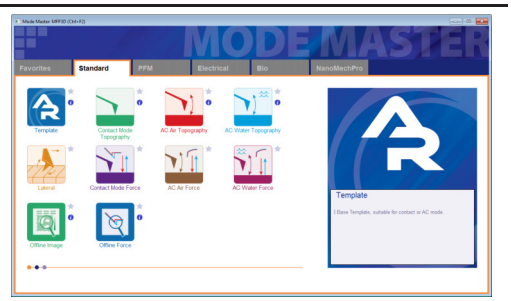
17.5.1. Preparation and Calibration

1. **Prepare**

   - Fresh calcite sample
   - Clean cantilever holder that will be used for imaging (either liquid holder or perfusion holder)
   - Load a FS-1500AuD probe (f = ~1.5 MHz, k = ~6 N/m) in to the cantilever holder.
   - AFM software version 16 or higher and the FFM mode is 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:

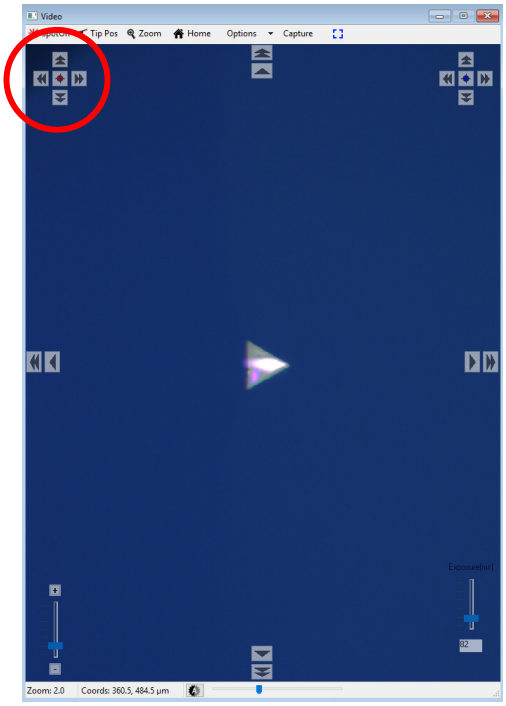
- Select *Standard tab* ▷ *Template*
- The screen will now re-arrange and present all the controls necessary for this type of AFM imaging.

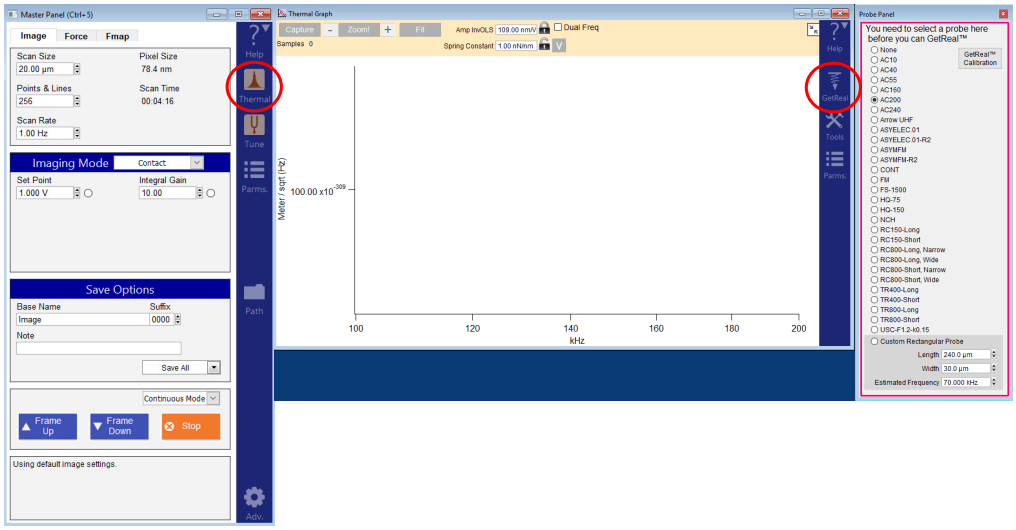
Note Loading Template from the Mode Master will automatically start in a nonspecific set up of Contact Mode.

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 but keep the laser spot at the end of the lever (see figure)



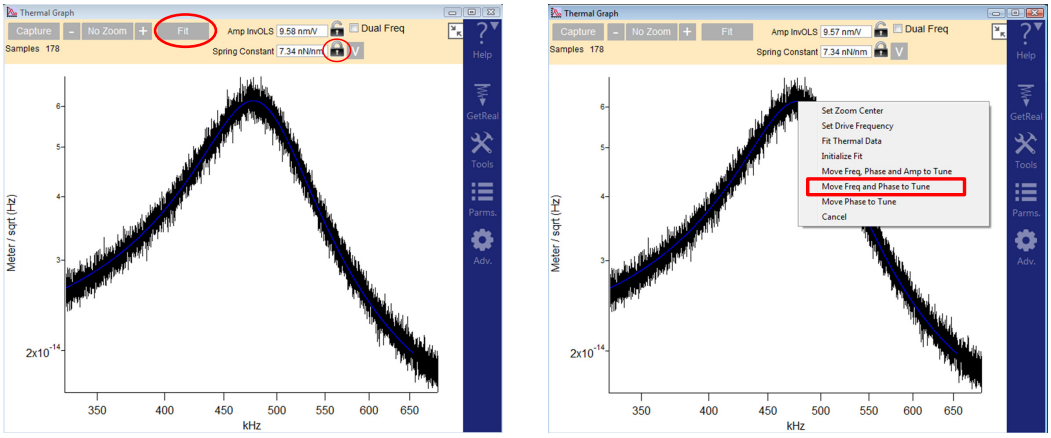
5. 

Calibrate Probe (in air)

- Choose the Thermal icon in the Master Panel
  - thermal graph will appear
- Click on the GetReal icon which in trun will open the Probe Panel
- Choose the probe that is used for the experiment - for this experiment choose Arrow UHF
- Click GetReal Calibration
- Once the calibration is compleated, 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. Re-focus and re-align the laser on the cantilever (now in liquid) - Sum should be ~ 6V

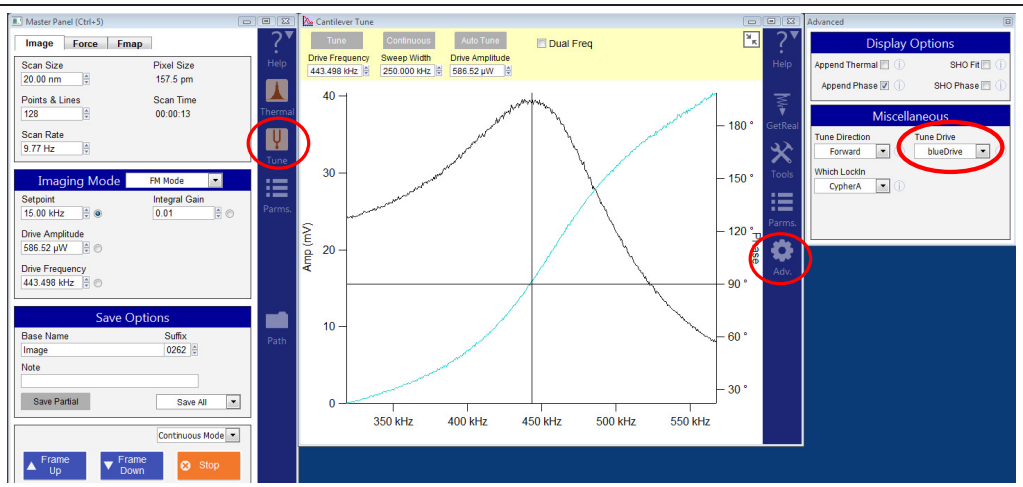
11.



Capture a thermal of the cantilever in liquid:

- Make sure that the padlock besides Spring Constant is locked
- Re-fit the thermal data to obtain the updated (water) InvOLS value
- Transfer the frequency of the thermal peak to the tune panel
  - Right click on the peak of the Thermal Graph and choose Move Freq and Phase to Tune

12.



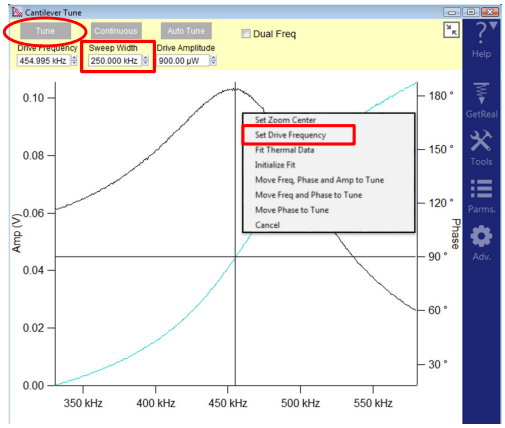
Turn on blueDrive photothermal excitation

- In the Tune graph, click on Adv.
- In the Advanced panel, choose blueDrive form the drop down menu of Tune Drive
- Use the blue arrows in the Video panel (top right side) to move the blue laser spot to the base of the cantilever

13.

Tune panel

- Set the Sweep width to 250 kHz
- Click Tune
- When tune is done, right click on the peak and choose 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 panel)



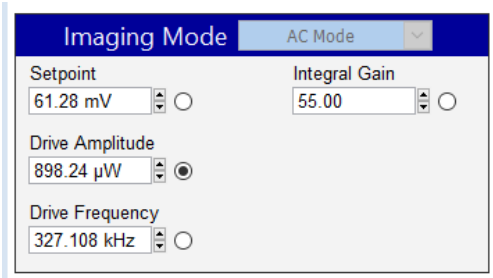
17.5.2. Imaging in AC Mode

Acquire an AC image of the calcite sample to first check probe tip sharpness and to define the area of interest.

1.

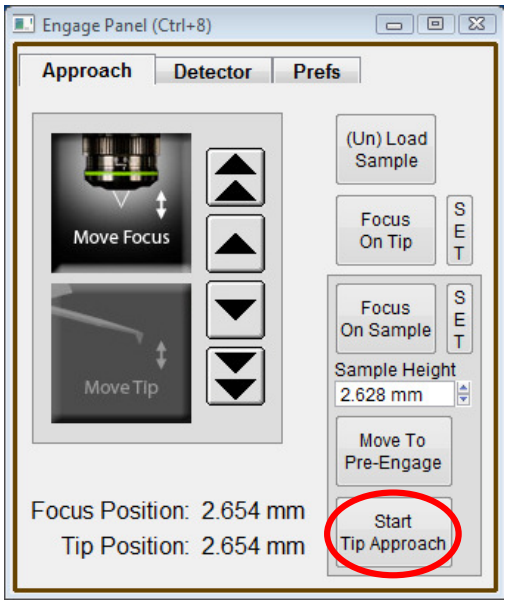
Master Panel

- Set the setpoint to ~ 60 mV
- Set the IG to 55



2. **Video and Engage Panels**

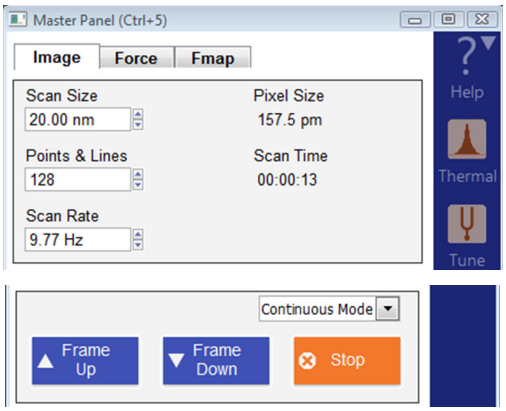
   - Click on SET button besides Focus on tip while focused on the tip
   - Click on SET button besides Focus on sample while focused on the sample
   - Click on Move to Pre-Engage (tip is now 50 μm from surface)
   - Click on Start Tip Approach



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 thermal graph panel, keep the padlock besides spring constant locked and re-calculate Amplitude InvOLS
5. Right click on the peak in Thermal Graph and Move Freq and Phase to Tune
6. When the tune is complete, right click on the peak and center phase

7. **Imaging parameters - Master panel**

   - Set the scan size to 20 nm
   - Set the scan rate to 10 Hz
   - Start imaging by pressing Frame Down



8. Start imaging and optimize imaging parameters

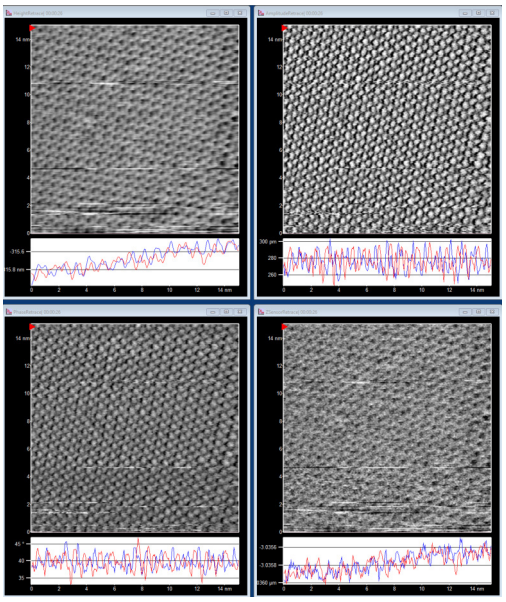
   a) decrease the free amplitude (by decreasing drive amplitude in the Master Panel)

   b) then, decrease the setpoint unil trace and retrace overlap each other

   c) repeat several times to image with the lowest amplitude possible

9. **Imaging in AC mode**

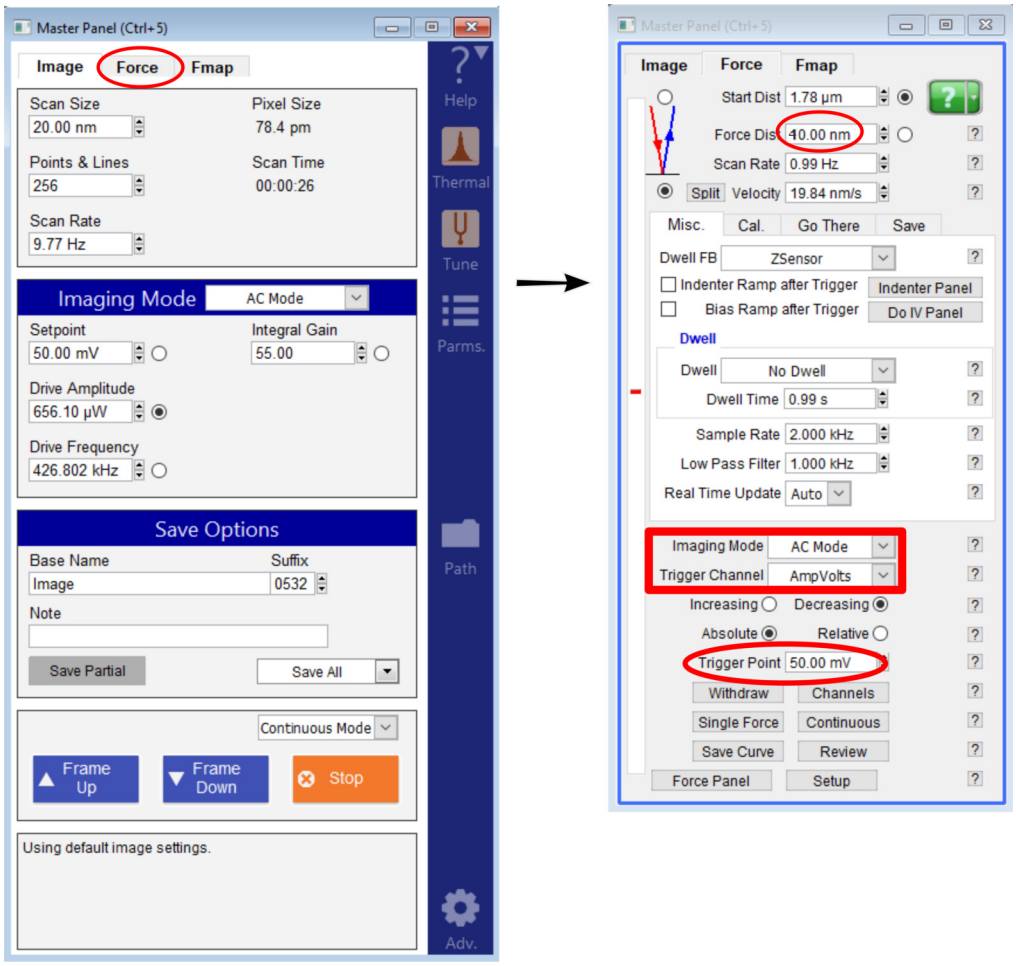
- Acquire a 20 nm image and re-size the scan to 10 nm
- Channels that are acquired include
  - Height
  - Amplitude
  - Phase
  - ZSensor
- Once the imaging looks optimized, stop the scan and switch to the Force tab



17.5.3. AC Force-Distance Curves

The user will acquire several AC force curves to bring the tip within 50 nm of the surface and re-Tune the cantilever.

1.



Force Tab

- In Master Panel, switch from AC mode imaging to AC mode force curves
- Master Panel Force tab will now display parameters related to AC mode force curves
  - Set the setpoint to about 60% of the amplitude (if amplitude is 80 mV set the setpoint to 50 mV)
  - Begin with force distance set to ~ 500 nm. Force distance will be the distance between the tip and sample after the curve has been acquired
  - Set the Trigger Channel to AmpVolts, Decreasing and Absolute

2.

AC Force Curves

- Acquire a single force curve
- Click on 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 on Stop curves
- The z voltage should remain between 70 and 150 Volts

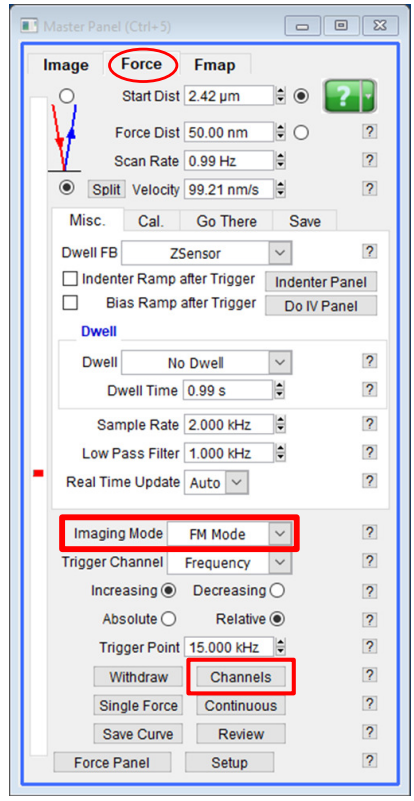
Re-tune 50 nm away form the surface

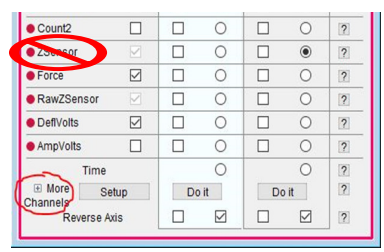
- Click tune on the Tune Graph
- Right click on the peak of the graph and choose Set Drive Frequency
- Right click on the graph again and choose Center Phase
  - Z voltage should still be at the same value as when the force curve was acquired (meaning that the tip is ~ 50 nm away from the sample)

17.5.4. FM Force-Distance Curves

This step will allow the user to calculate FM gains and check if the setpoint is appropriate.

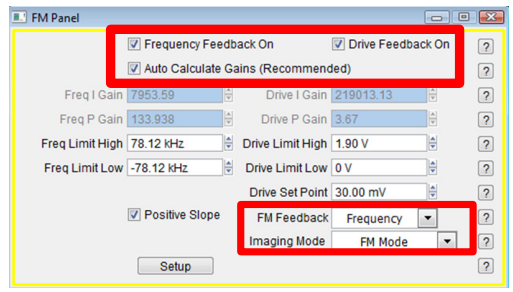
1.
- Choose FM Mode for Imaging mode
- Click on Channels button in the Force tab and select
  - Frequency
  - Dissipation
  - Amplitude
  - Phase
  - Make sure to plot versus DRIVE (not ZSensor) for short curve (tens of nm) as Drive is a quieter signal than ZSensor (which has 50 pm of noise in a1 kHz bandwidth)
- Choose trigger: Frequency
  - Increasing
  - Relative
- Trigger Point: 5 kHz is a good start

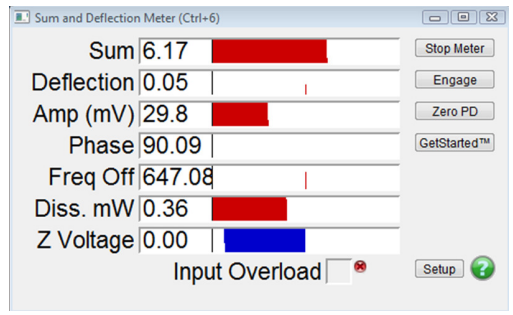


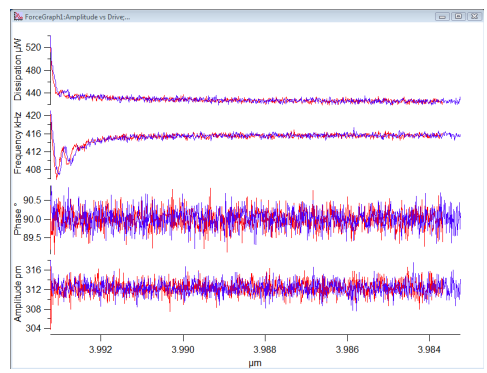


2.

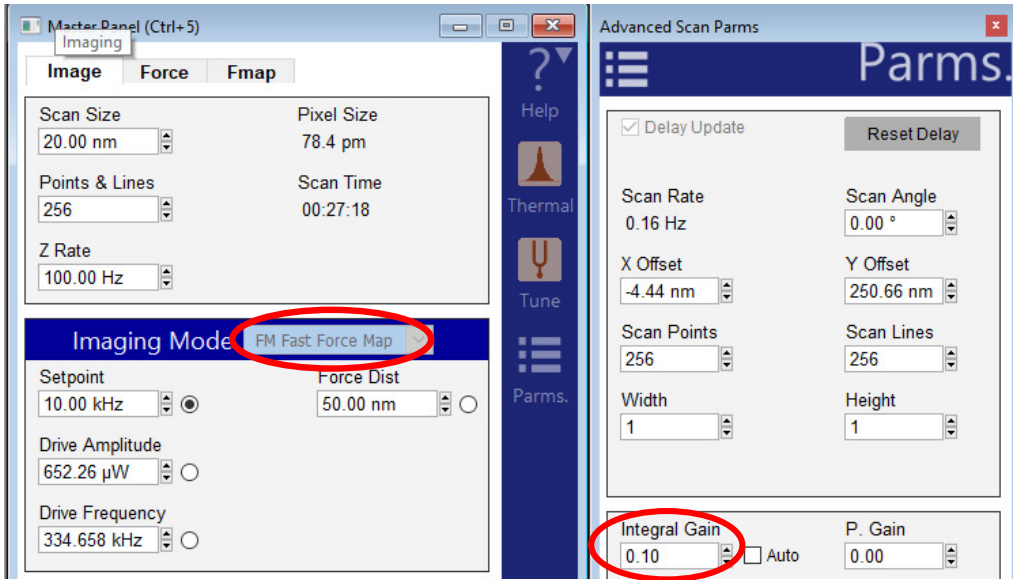
- Locate the FM Panel (AFM Controls –> Other –> FM Panel)
- Un-check and check the Auto Calculate Gains checkbox, the values should update
- Set the Drive Set Point to the required amplitude
  - start with 3 Angstroms: 3A = 0.3nm;
  - Ex: 0.3nm / 10.25nm/V InvOLS = 0.0292V = 29.3mV
- Activate Feedback Loops by checking the boxes besides
  - FM feedback ON
  - Drive Feedback ON
- When the frequency and drive feedbacks are ON, the following values will 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
- Click Single curve to acquire a FM force curve
  - If the FM gains are correct, force curve should appear on the graph







17.5.5. Imaging with FM-FFM



1.

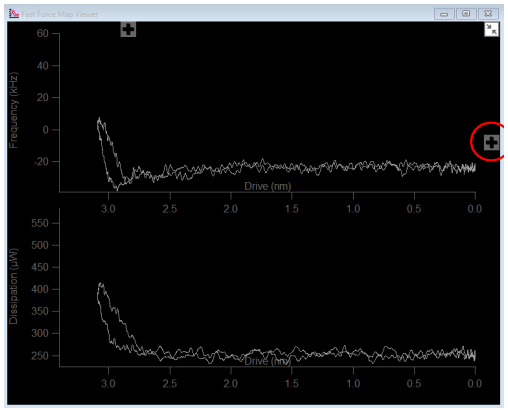
Imaging Parameters

- In Master Panel, switch from FM Mode to FM Fast Force Map
- Master Panel will now display parameters related to FM FFM mode
  - Setpoint is now displayed in units of Hertz (if frequency feedback is chosen, and watts if dissipation feedback is chosen in the FM Panel)
  - Set the force distance to 50 nm
- Set the other parameters in the Master Panel
  - Scan size: 20 nm
  - scan points & lines: 256
  - Z-rate: 100 Hz
  - Z-voltage should still be between 70 and 150 V
- Click Engage in the Sum and Deflection Panel
  - FM FFM settings should now be reflected in the Sum and Deflection panel
- Set the IGain located in the Parms panel to 0.1

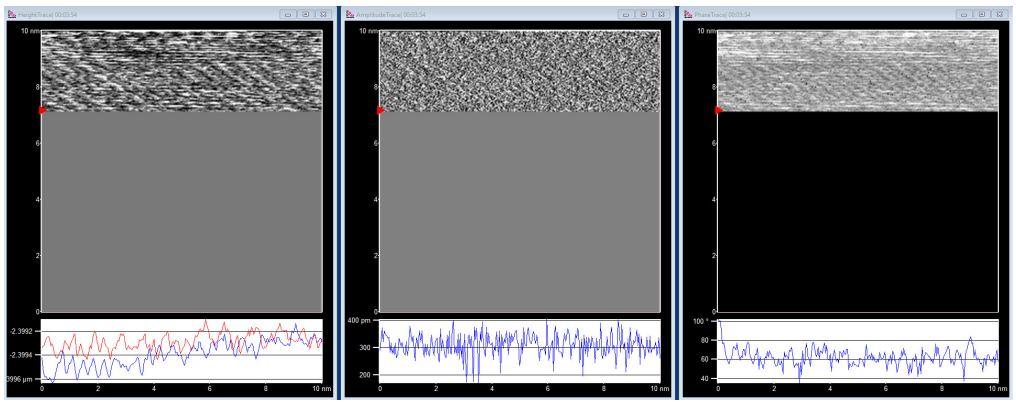
2.

Real time Force curves

- Fast Force Map Viewer can display up to 2 channels (to add a graph, click on the plus sign on the right side of the viewer)
- Each graph has several options for each axis, user 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 is collected in 3 channels during FM FFM imaging
  - 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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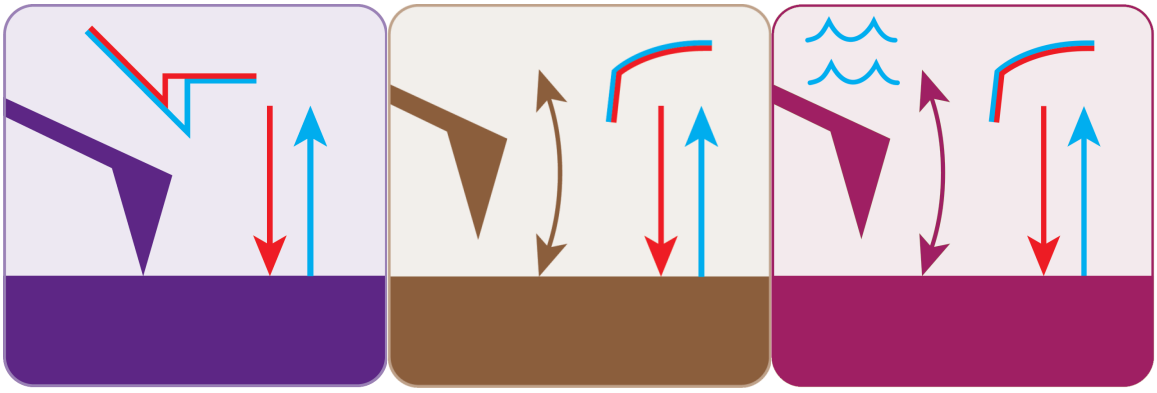


Chapter Contents

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 in order to measure intra-/inter- molecular 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 18.1, 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, the user can also acquire a Force Map which is an array of force curves over a user defined XY scan range.

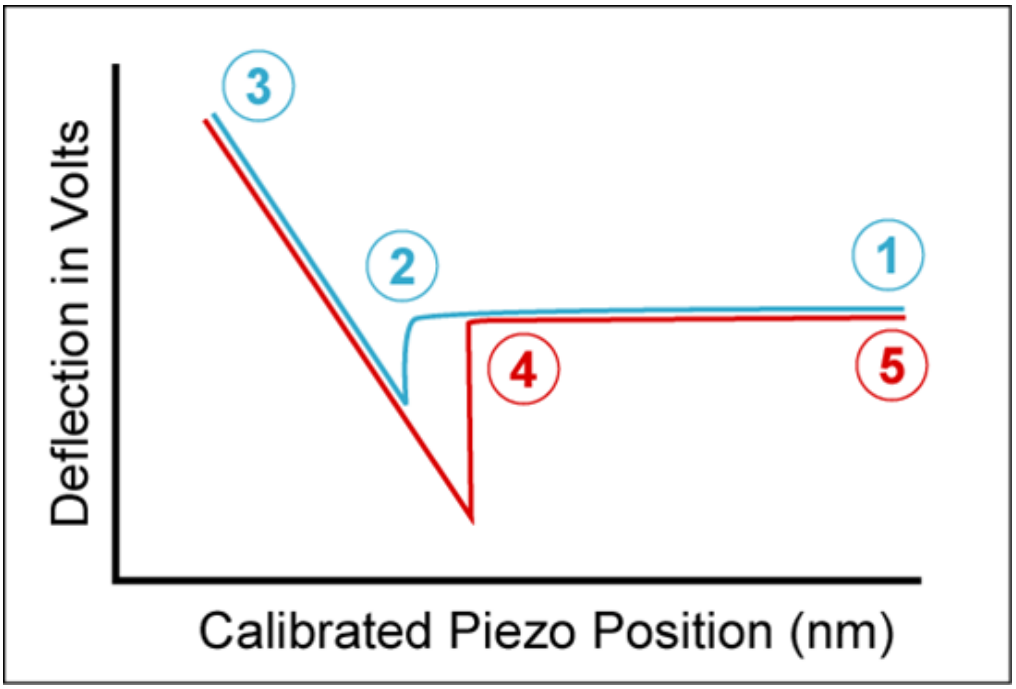


Figure 18.1.: Components of a Force Curve as the cantilever approaches and retracts from the sample surface.

There are two major classifications that most force spectroscopy experiments measure:

- A pulling event in which the tip interacts with the surface, and some adhesion dissociation event between the tip and surface is measured on the retract cycle (18.2).
- A modulus or compliance measurement where the tip pushes into the sample surface. (18.3).

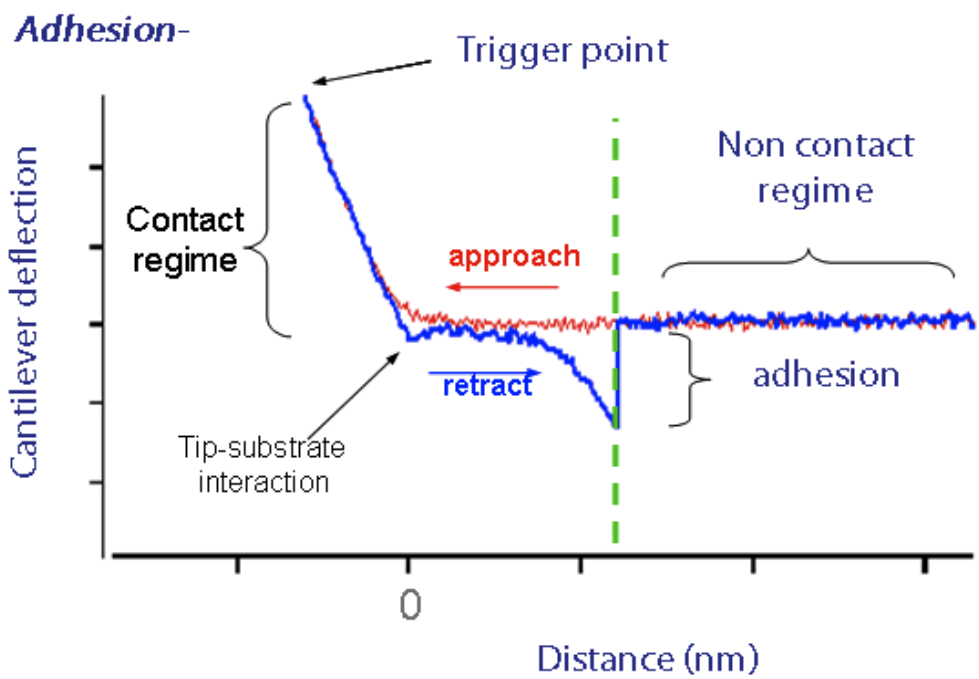


Figure 18.2.: Adhesion force spectroscopy classification. Pulling can yield adhesion forces.

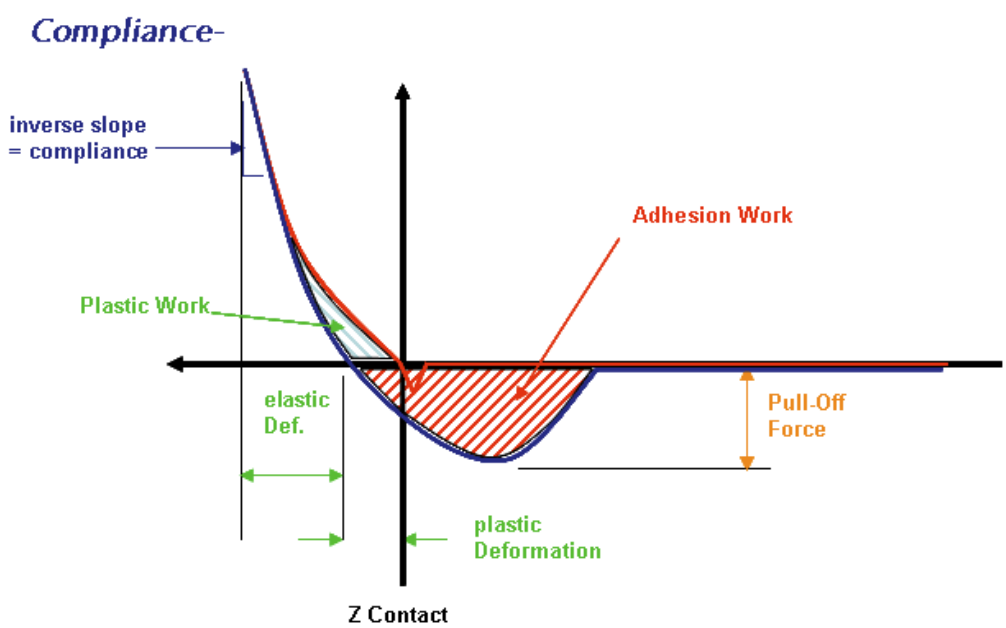


Figure 18.3.: Compliance force spectroscopy classification. Pushing can measure compliance forces.

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, then small forces

may not be recognized; if a lever is too floppy, perhaps not enough force can be applied during compliance measurements. Another way to state this is to say that 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 will appear as no different from the noise in the meausrement. Other considerations are the cantilever material regarding how well it will work in air/ fluid, and the ability to do chemical cross-linking to it.

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 will vary from cantilever; and as a result the applied force will 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 278.

To briefly summarize, the Thermal Method requires two steps:

- First a force curve is acquired to calculate the cantilever sensitivity (InvOLS).
- Second a thermal tune is captured and used to calculate the cantilever spring constant (k), using the calculated cantilever sensitivity.

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:

- First cantilever spring constant (k) is calculated based on the dimensions and frequency of the cantilever.
- Second a thermal tune is captured and used to calculate the cantilever sensitivity (InvOLS). This is the opposite of what is done in the Thermal Method. GetReal 'locks' the k and calculates the InvOLS.

There are some excellent force spectroscopy review papers in the literature; we list here a few:

- 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

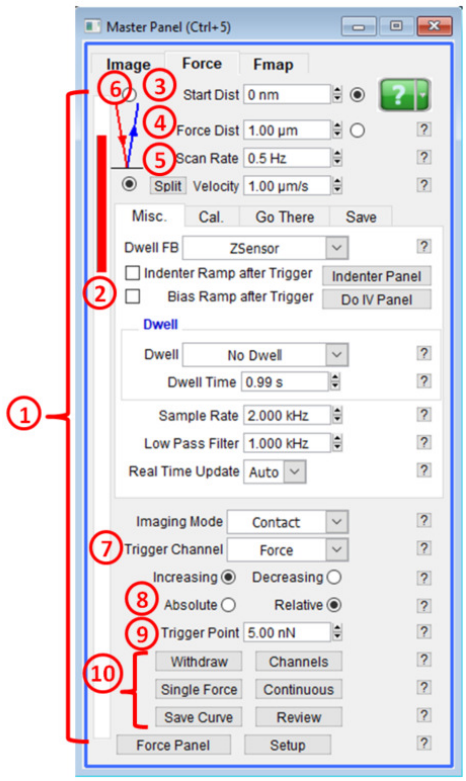


Figure 18.4.: Force tab of the Master Panel.

18.2.1. The Force tab of the Master Panel

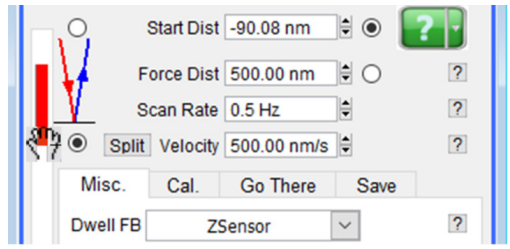
The Force tab of the Master Panel is where most the basic force spectroscopy software control occurs. First we'll describe some of the general controls, and then more controls hidden under the additional tabs on this panel, also known as sub-tabs, as numbered in Figure 18.4 on page 232.

① **White Bar** Along the left of the panel represents 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.

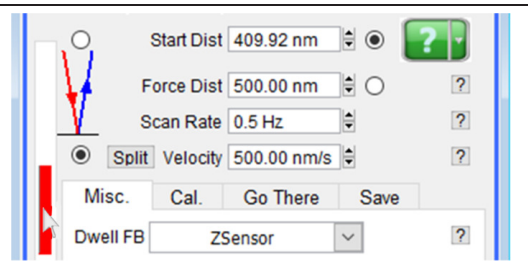
② **Red Bar** Within the white bar represents the 'Force Distance' of the force distance cycle, relative to the entire travel of the Z piezo (see below). Note that values in microns given are in reference to the piezo sensitivity. The captured and displayed force curve will show slightly different values as corrected by the z sensor.

③ **Start Distance** This parameter is the point at which the piezo begins its Z/ force distance cycle (i.e., the starting voltage applied to the piezo is another way to think about it). 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 will automatically set to 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 cursor somewhere in red region
   - Left mouse click until hand icon appears
   - Drag red bar throughout the Z piezo range (white slider bar)



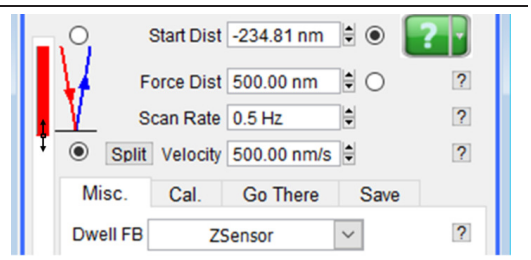
2. **Method 2**
   - Left click mouse in the white area to roughly scroll the red cursor up or down.



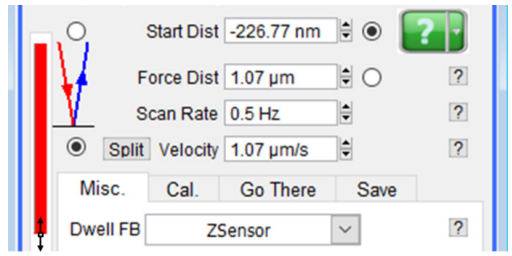
The Hamster wheel (detached or on the front of MFP controller) can also execute this movement in real time when the start distance radio button is activated.

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 edge of red bar.
   - Hold left mouse button down until double arrow appears



2. **Step 2**
   - Drag cursor to desired force distance value.
   - Notice the change in the 'Force Dist 'value in the box to the right.



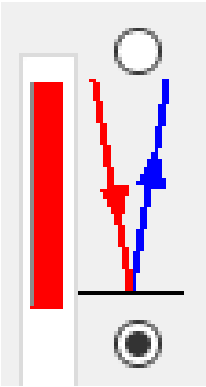
⑤ **Scan Rate/Velocity** This is the estimated PIEZO velocity (NOT the tip velocity); changing the velocity, or scan rate, affects the other.

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). See Section 18.2.8 on page 246 to select velocities that are not subject to hydrodynamic effects.

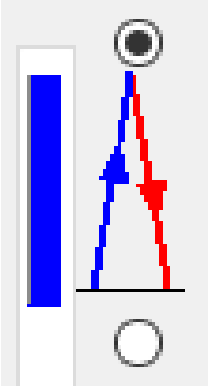
⑥ **Forward/Reverse pulls** These radio labels change whether the tip starts on or above the surface (Red mode/ Blue modes) :

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.

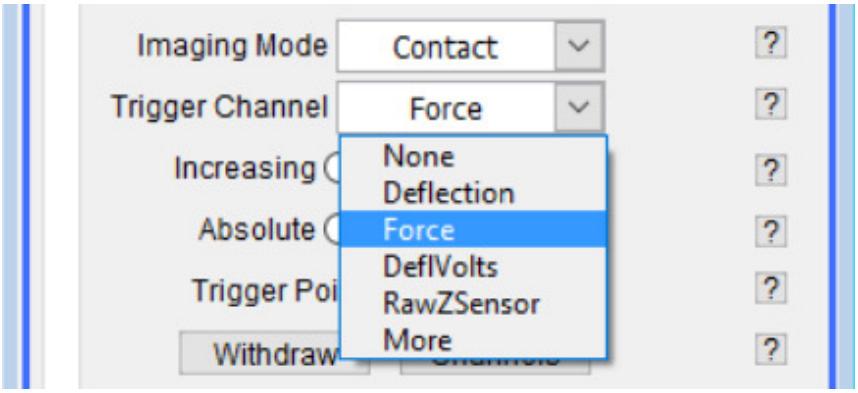
Reverse pull (**Blue mode**) Tip starts cycle at surface and travels (retracts) to free air (non-contact), then returns to surface; This mode is good for 'Fishing' , possibly Force Clamp (**??** on page **??**). See Figure 18.5b on page 235.



(a) Forward pull



(b) Reverse Pull



(c) Selecting a Trigger Channel

Figure 18.5.: Examples of different Trigger types.

⑦ **Trigger Channel** This pull-down menu allows the user to define what units to measure for 'triggering'- essentially a user defined Force that the tip applies before switching direction (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 the user has a higher risk of grinding the tip into the surface.

Deflection Monitors the deflection (nm) on the photo-detector. The piezo will only move the cantilever if the InvOLS (and ideally the spring constant) has been calibrated (See Chapter 20 on page 278).

Force Monitor the force applied to the sample. This value is only accurate if both the InvOLS and spring constant have been calculated (See Chapter 20 on page 278).

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 only use this 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 If you select this option the drop down menu will expand to show other 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 since 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 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 Photo-detector 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; 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.

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 amount of continuous curves can be limited using the 'Limit Cont. to' parameter.

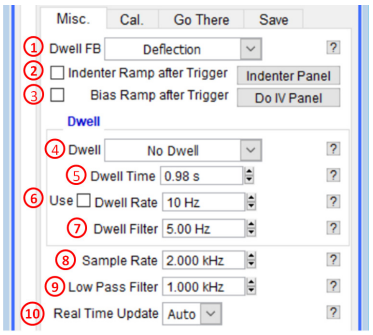
1. 'Limit Cont. to' is an advanced parameter that is hidden with the default display. Click on the 'Setup' button. Click on the checkbox.
2. Click the 'Looks Good' button. This parameter should now appear.
3. Enter the amount of curves needed to collect continuously in the 'Limit Cont. to' setvar.
4. Click the 'Continuous' button to acquire your force curves.

Save Curve If the curve looks like it is something you want saved, and you do not have all curves being saved in the Save sub tab, click this button. By default, all force curves are automatically saved to Memory and Disk.

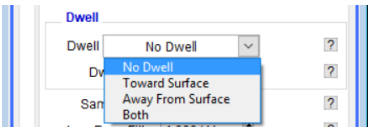
Review Loads force curves for review/ analysis by bringing up the Master Force Panel.

18.2.2. The Misc. Sub Tab

This sub-tab controls dwell periods / sampling and sampling / bandwidth options. The MFP-3D software allows the user 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 perhaps the tip (functionalized or not) needs 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 Pulldown Menu

Figure 18.6.: The Misc. Tab allows the user 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 drop down menu allows the user to set the Dwell FB (Feed Back) loop input.

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 a bit experimental and requires a well calibrated InvOLS.

② **indenter Ramp After Trigger** This will call whatever function has been developed in the Indenter Panel. The Indentor button brings up the indenter panel. Section 18.3.2 on page 251

③ **Bias Ramp After Trigger** This will execute an I-V curve using whatever settings are applied to the DoIV. See 9.7.

④ **Dwell Pull-down menu** Activate Dwell function here as shown in Figure 18.6b on page 237.

None No Dwell period is applied.

Toward Surface This is a Dwell period applied when the trigger point is reached. Bandwidth options during this segment can be chosen.

Away from Surface This occurs for reverse pull (see blue mode); a dwell is applied after the tip pulls away from the surface and before it approaches it again.

Both This applies a dwell when the tip retracts from surface, then again as the tip approaches the surface.

⑤ **Dwell Time** This 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.

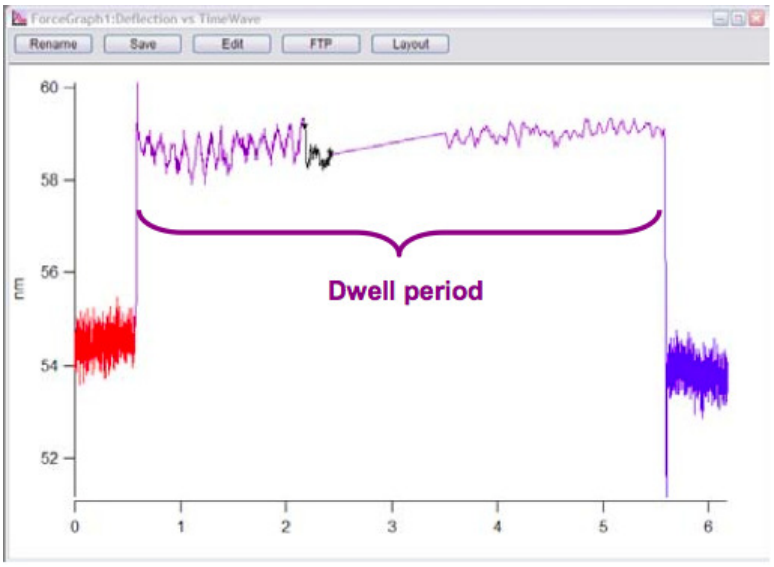


Figure 18.7.: Example of a temporal force distance curve (plotted as cantilever deflection vs. time, with a multi second Dwell towards surface.

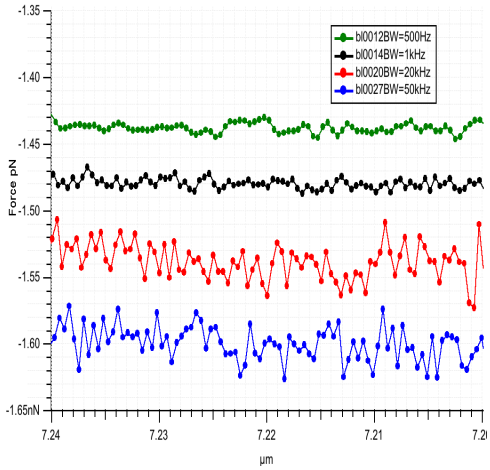
⑥ **Dwell Rate** This is the rate at which data is collected during a dwell- i.e., maybe the experiment calls for more or less points to be collected during this time. It is an advanced parameter that is hidden with the default display. Click on the 'Setup' button then click the checkbox to the right. Click the 'Looks Good' button. This 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** This is a low pass filter that can clip from 1 Hz to 1kHz; often not a default parameter. Must be activated in the setup menu of the panel similar to the Dwell Rate.

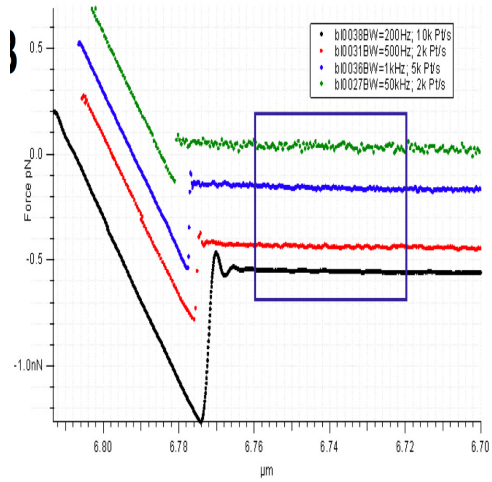
18.2.2.2. Sampling and Bandwidth Options

The band width (BW) options for the curve collection. Note, to view the BW options (in most version of Asylum Research software), they must be activated to be shown using the 'Setup' button. Setup can also be activated by right clicking anywhere on the panel.

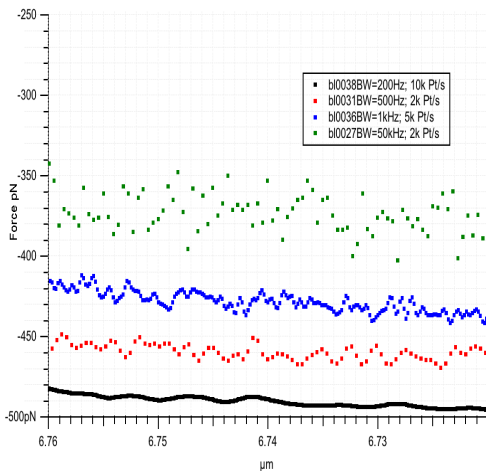
⑧ **Sample Rate** This is essentially how many points per second that are collected during the force curve acquisition (See Figure 18.8a on page 239 for example).



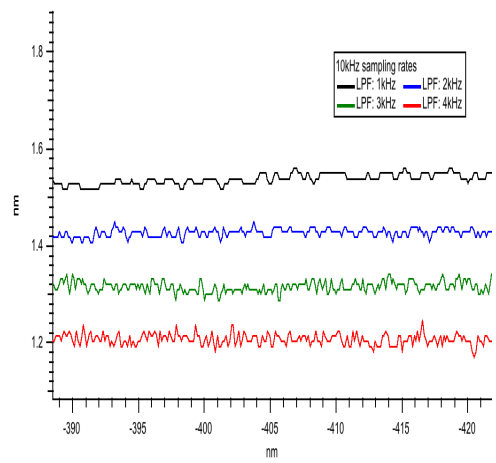
(a) Various data streams from a free air portion of a force curve collected at 2kHz (i.e., 2,000 points/sec) with different low pass filter settings. Notice higher filter settings are more noisy.



(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 were deflection resolution with distance is important.

⑩ **Real Time Update** This drop down menu allows the user to choose how the Force graph gets updated:

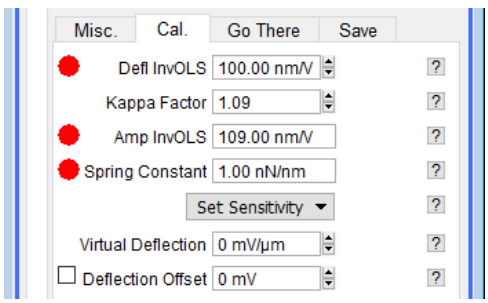
Never Force plots updates *after* the acquisition is complete.

Auto Will update with some time constraints within the acquisition parameters.

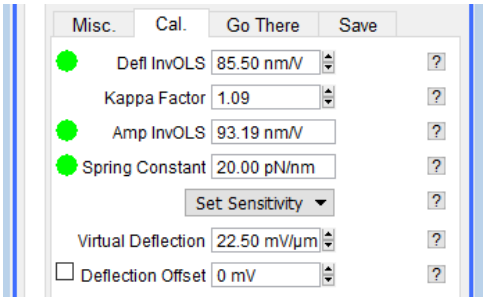
Always Force plot is updated as it is being collected; this will be evident by a black line that appears within the data stream in the force curve. See Figure 18.7 on page 238 for an example of this.

18.2.3. The Cal. Sub-Tab

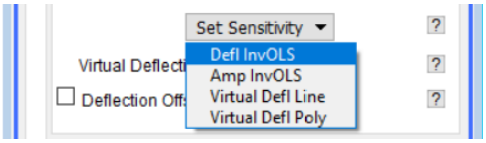
This sub-tab is where the calibrations are executed and displayed for spring constant determination (see Chapter 20 on page 278 for a complete description of that protocol).



(a) The 'Cal' Sub Tab of an uncalibrated cantilever.



(b) Set Sensitivity Pull-down Options.



(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. Ge-

tReal (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 will change 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. 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 will be displayed here. See Chapter 20 on page 278 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).

18.2.3.1. Set Sensitivity

Set Sensitivity pull-down menu allows the user to select what will get calibrated. (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 horozontal 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 abberant 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 278.

18.2.4. The GoThere Sub Tab

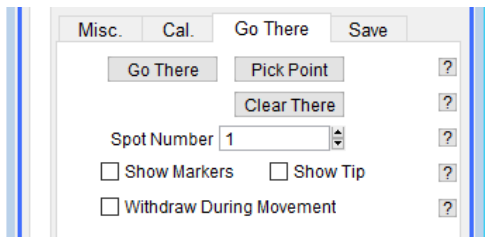


Figure 18.10.: The Go There Sub Tab for Point and Click force curve acquisition.

The Go There Sub Tab of Force tab in the Master panel allows the user 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, the user can 'pick points' on the image in order 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 pull down 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 swithcing 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' ☑ checkbox. By default the 'Show Tip' ☑ checkbox will also get 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. The user can grab and drag the ⊕ cursor to new location to mark additional points for either force or IV curves.

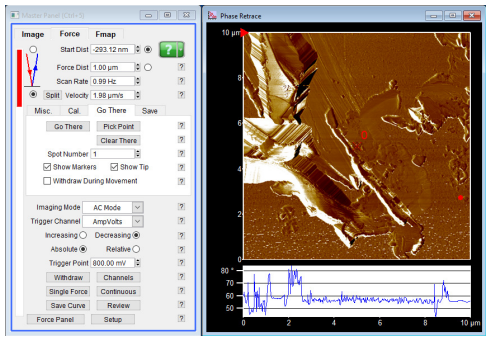


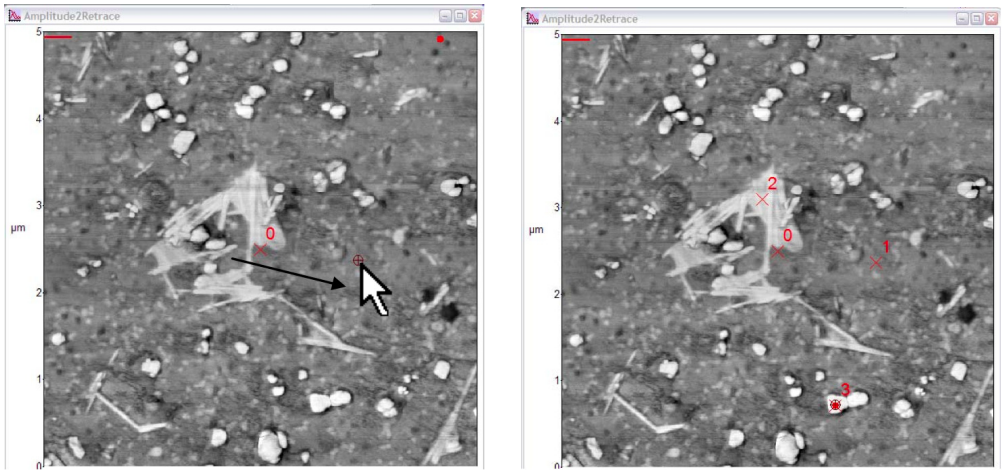
Figure 18.11.: The Go There subtab for Point and Click force curve acquisition.

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' - Position 1. This locks the point at that spot, meanwhile the Spot number index is advanced by one in anticipation of the next point location being chosen. Notice the user defined location/ spot is marked on the image with an 'X' and also labeled with the spot number (Figure 18.12b on page 244).
7. Also, notice the Spot Number base suffix will increase by one, expecting another point to be picked.
8. Continue to grab and drag the ⊕ cursor until you have selected all of your points (Figure 18.12a on page 244).
9. When ready to acquire the force curves, enter the Spot Number for the position on the surface you want to probe.
10. 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.
11. 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 163). 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 positionof 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).

Recall 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.

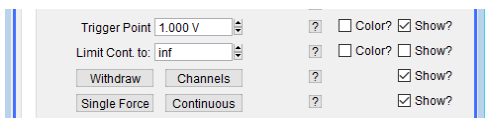


Figure 18.13.: Continous force limit count of the Force panel.

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 get 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.

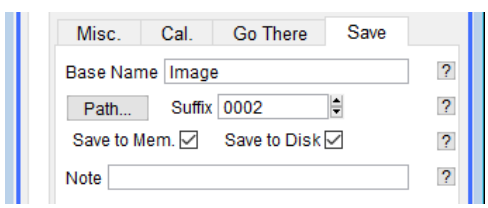


Figure 18.14.: The Save Sub Tab.

As curves are acquired, the base suffix will increase by 1.

18.2.6. Force Channel Panel

This panel assigns the data channels displayed and saved in Force Plots.

Figure 18.15.: Force Channel Panel

The Green / Blue / Red circular dots to the left of the channels describe the settings of those channels as follows:

- Red = Channel is not on (no activation checkmarks)
- Blue = Channel is ONLY displayed (i.e., YDisp checked for a given Channel, but Save not checked)
- Green = Channel is saved (does not care if channel is displayed or not).
- The 'Channel' column gives all the possible data channels- note that depending on the experiment, some 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' pull-down allows up to 5 channels to be plotted simultaneously. Depending on the number of graphs selected in the pull-down menu, that many X&Y columns will be displayed.
- Each Graph column (1,2,. . . 5) allows 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.

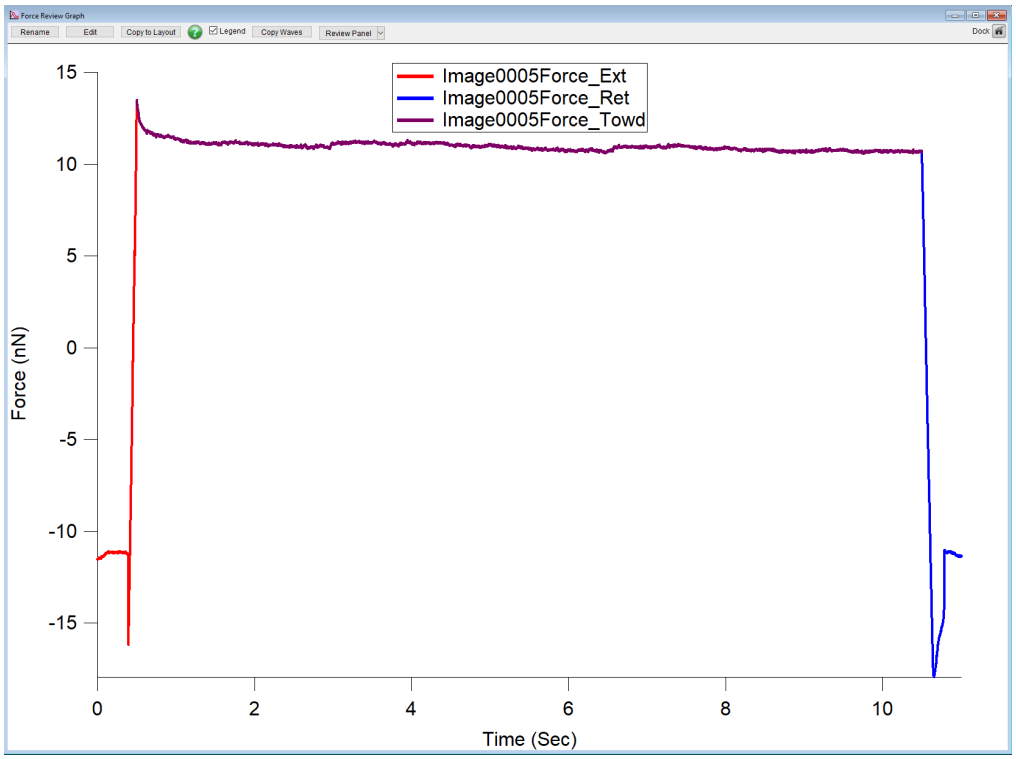
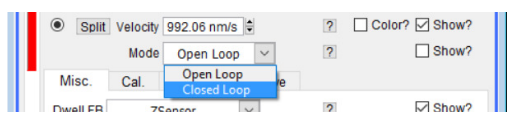


Figure 18.16.: Deflection vs. Time Plot

1. To turn on go to the setup menu to activate it.

2. **Select Closed Loop**

   - From the pull-down 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 that tip several hundred microns up if possible, and the peak should be sharper (For an example, see 20.3.5).

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. Another way to think of this is 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 particularly common in labs that have 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 to these 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.

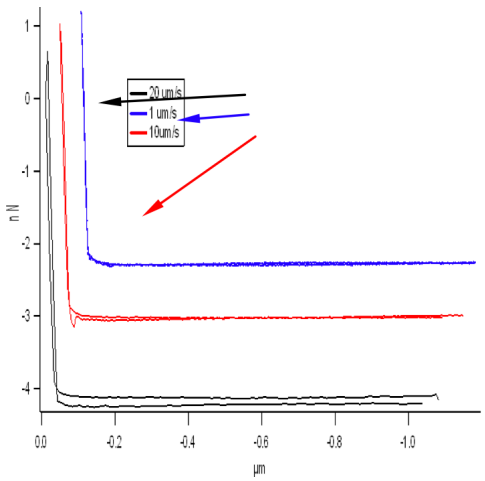
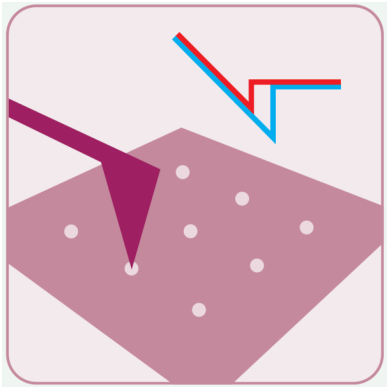
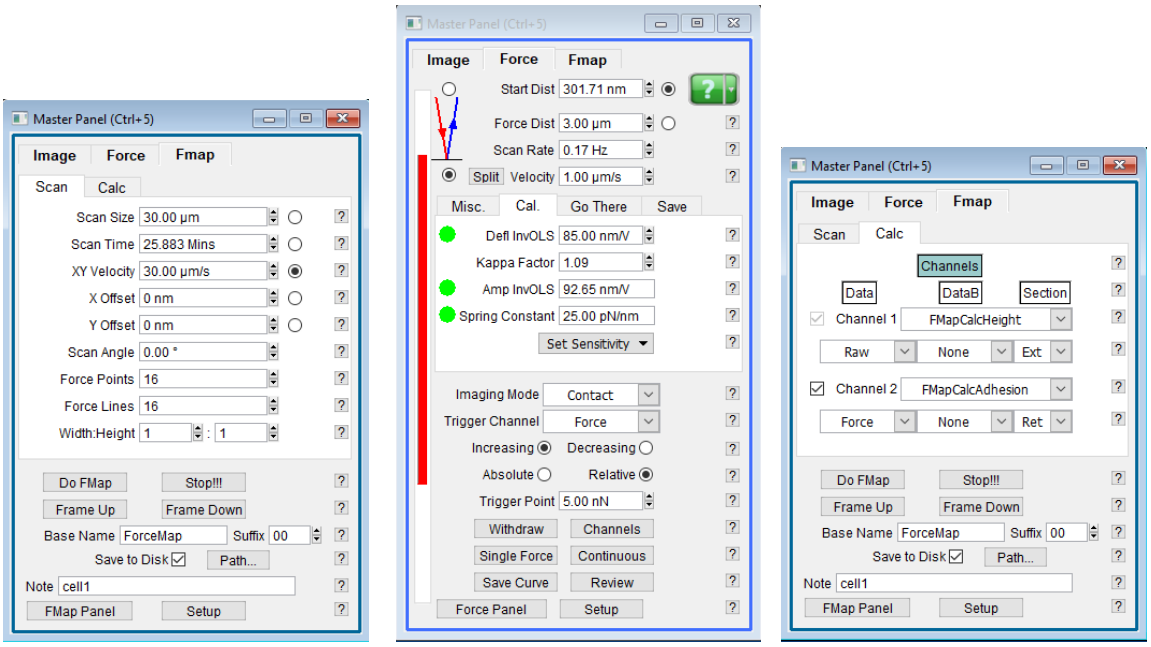


Figure 18.17.: Hydrodynamic effects can occur in fluid with fast force curve acquisition cycles

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 Sub Tab Showing Parameters

(b) Force Tab Showing Triggering Parameters

(c) Calc. SubTab Defines Data Channels per Respective Axis

Figure 18.18.: Force Map 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.

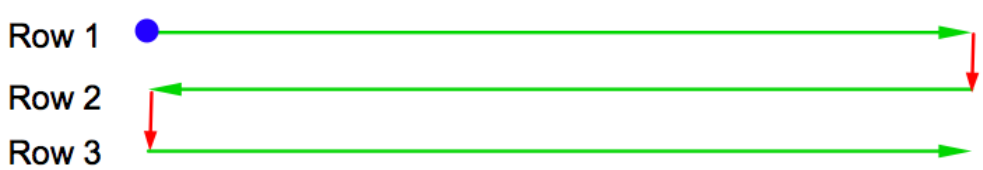


Figure 18.19.: Curve Collection

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.

- For the MatLab users out there, exporting is an important consideration for post processing due to the way the files are exported.

The FMap Panel has two Sub Tabs that include similar controls found for imaging in the main tab 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).

- Data Column displays / captures the X axis data.

- Data B Column displays / captures the Y axis data.
- Section Column gives option 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 will collect force maps continuously in the same scan range region until told to stop.

18.3.1. Procedure for Force Map Acquisition

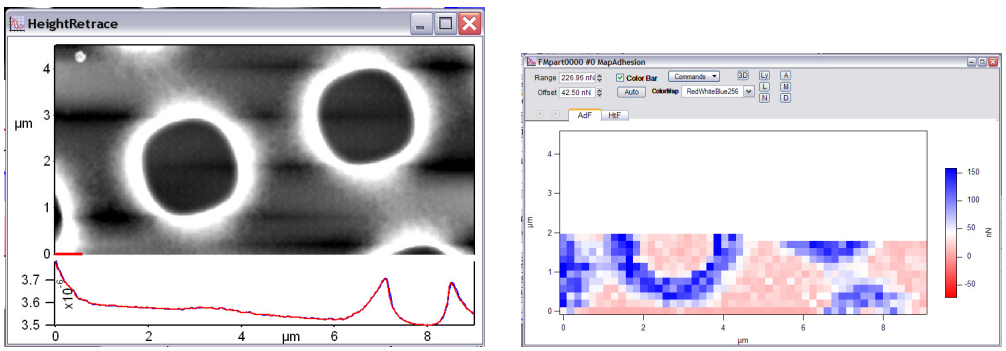
Since Force Maps may take minutes to hours to acquire depending on the sample and parameters necessary for quantitative measurements it is a good idea to acquire several individual force curves, ideally at different locations, and analyze them using either the Analyze or Elastic Tabs in the master Force Panel.

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 will already have that area dialed in from the 'Scan Size' field of 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 then 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. The user 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, typical won't give you enough for statistics. So the user will need to optimize Scan Size, Force Points and Force Lines for each sample and measurement.
   a) The 'Scan Line Time' setvar will tell how long it will take to acquire the Force Map based on the parameters defined (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 will use these parameters for the Force Map. The main parameters to adjust include:
   a) Force Distance: 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 importatnt to select a velocity that won't artifically increase modulus.

c) Trigger Channel/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; the user will have to wait until after the entire Force Map has been acquired before using the Elastic Tab. Something that looks like an MFP-3D 'Display Window' appears during acquisition, with tabs that correlate to what Channels that are 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.
- The tip material is important because it must be 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:

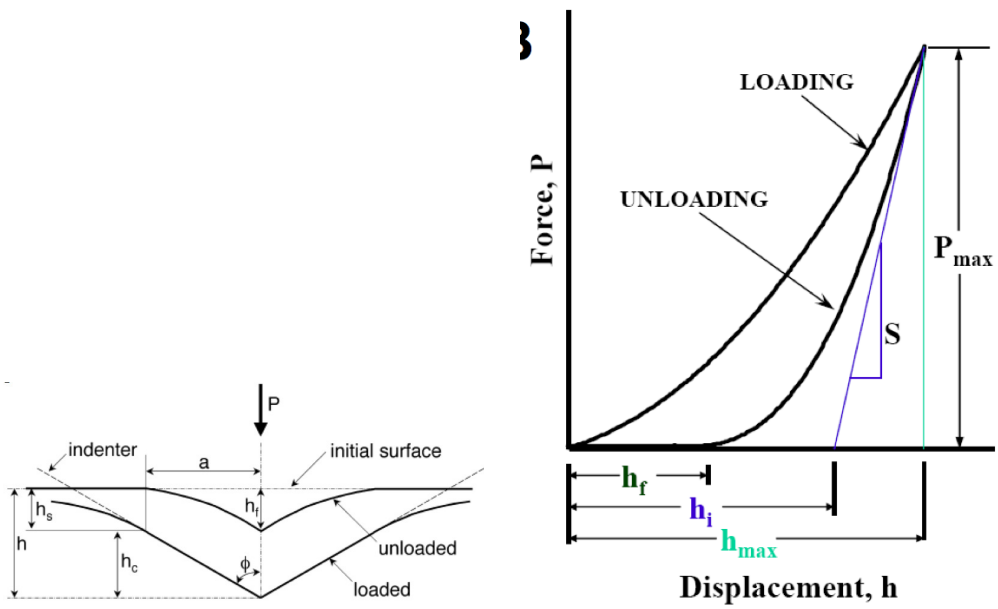
P is Load; typically in the force dimension (N).

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 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 **??** on page **??**. Panel A shows a fully elastic system; there is no hysteresis between the load and unload components, indicating 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 the 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 must 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, it is assumed that the following steps have occured.

1. Load tip, align SLD on cantilever, 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.

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

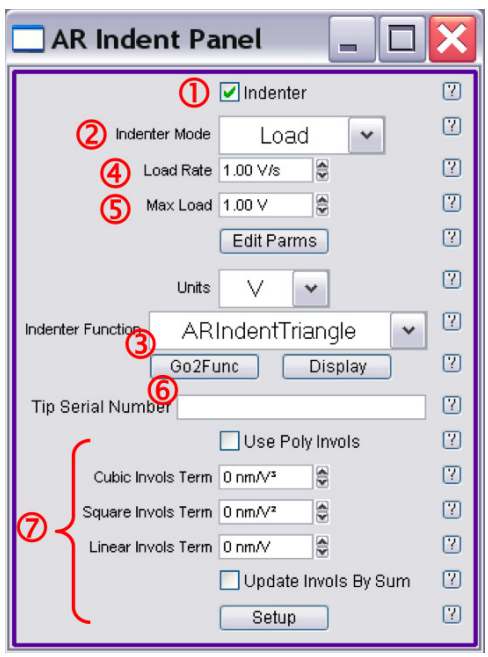


Figure 18.22.: AR Indent Panel. Numbers correspond with steps below.

1. When the indenter check box is activated, this panel uses Closed loop Z sensor to apply the load (after the initial force trigger used to find the surface).
2. The indenter mode pull-down menu gives 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' pulldown lets the user choose between Volts, distance (m) or Force (N).
4. Loading Rate can now be chosen in a comfortable dimension.
5. Maximum load can also be selected.
6. Indenter function pulldown 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. The pull-down menu options are:

   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 customer 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 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.

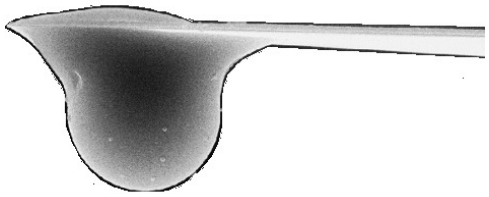


Figure 18.23.: Colloid Lever Side View

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 $\propto \partial(\text{depth})^{\text{m}}$, 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

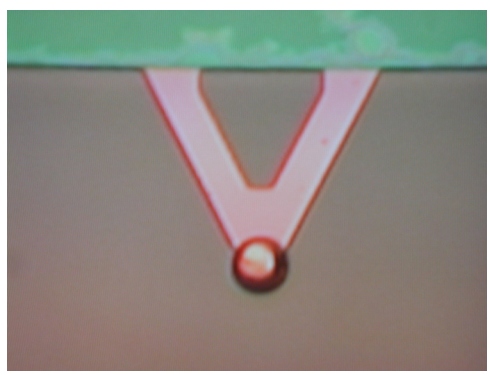


Figure 18.24.: Colloid Lever Top View

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 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:

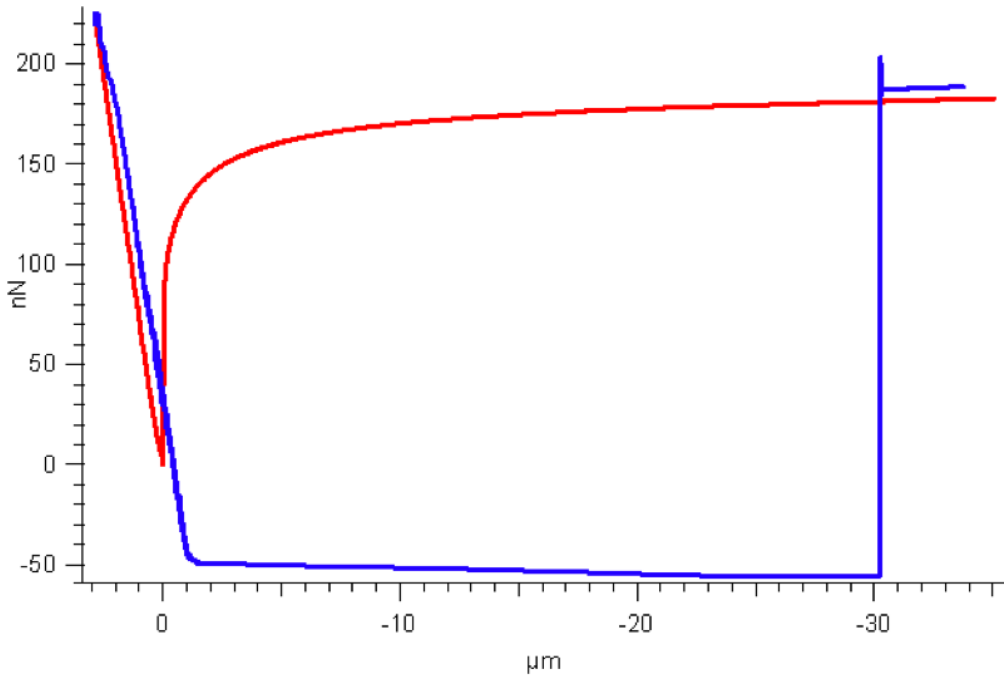
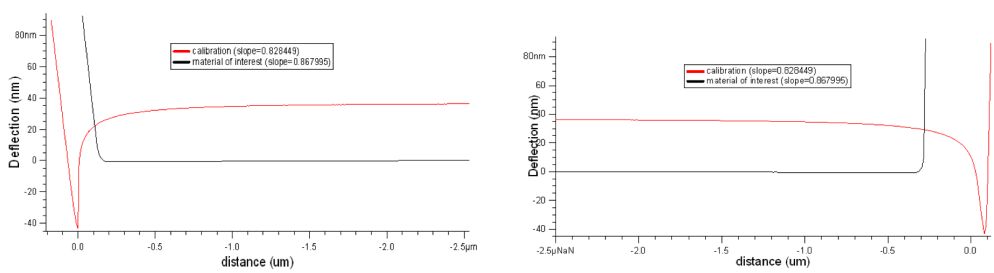


Figure 18.25.: Large surface forces can act on a sphere affixed to cantilever (in air; k ?)

- 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 Panel A), it is not really 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.



(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 (do not 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 **??** on page **??**).

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.

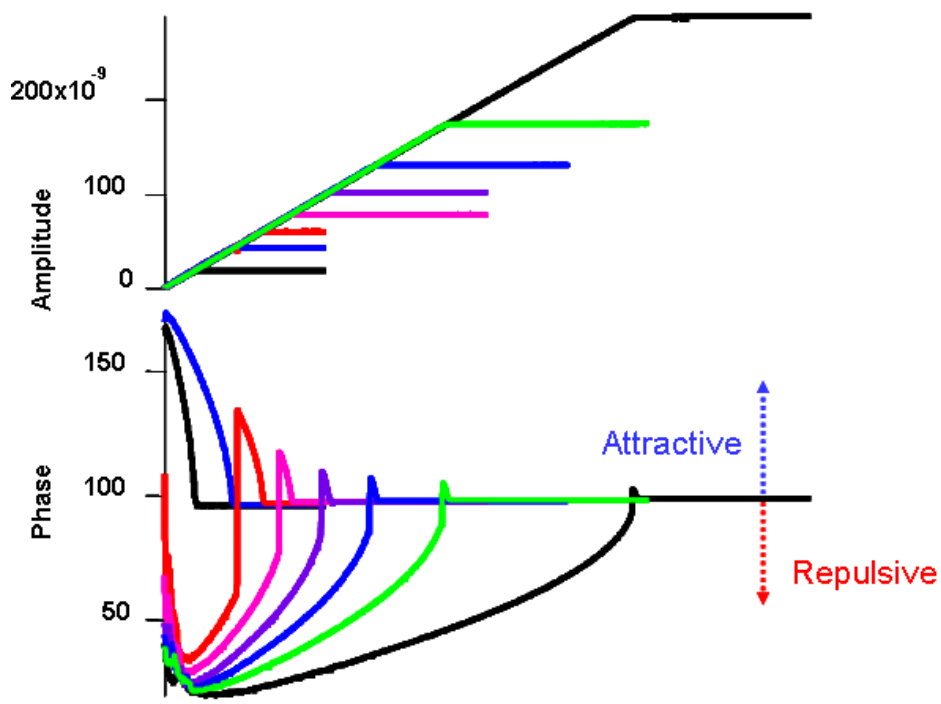


Figure 18.27.: AC force spectroscopy.

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 & 4 (black and blue, respectively) are similar during the approach (extension) curves, there is an initial attraction to the surface, then repulsion; at some point, the tip becomes closer to the surface, it flipped back to attractive mode.
- Point 3 (red) never goes into the repulsive regime.

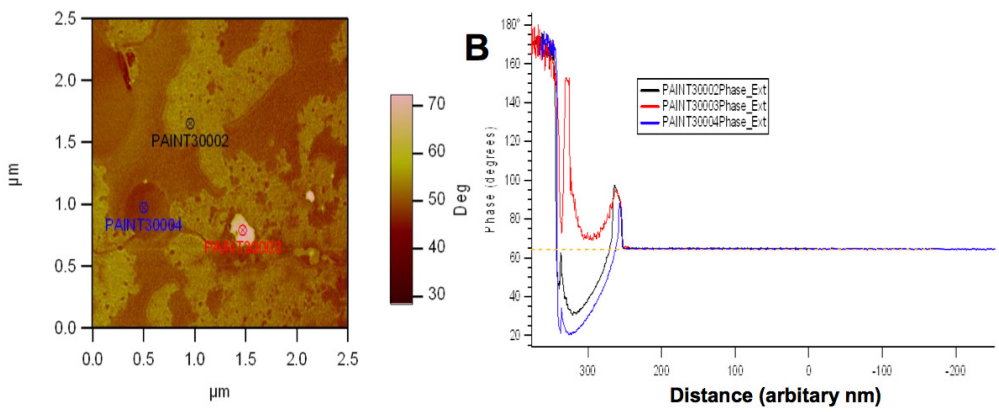


Figure 18.28.: AC point and click Force curves on heterogeneous sample: A) Phase image of paint sample with location of user defined force curves; B) individual AC force curve Phase vs. distance (Z)- orange dash line indicates Free Air Phase values of ~64 degrees. (yes, that is the di color table on the 3D)

18.3.5. The MFP-3D Function Editor

The MFP-3D software has a 'Function Editor', which is very useful for applying custom made waveforms for force spectroscopy, indentation, and various electrical techniques.

To open, click the Function Editor option in the indenter panel. A panel like the one shown in below will appear.

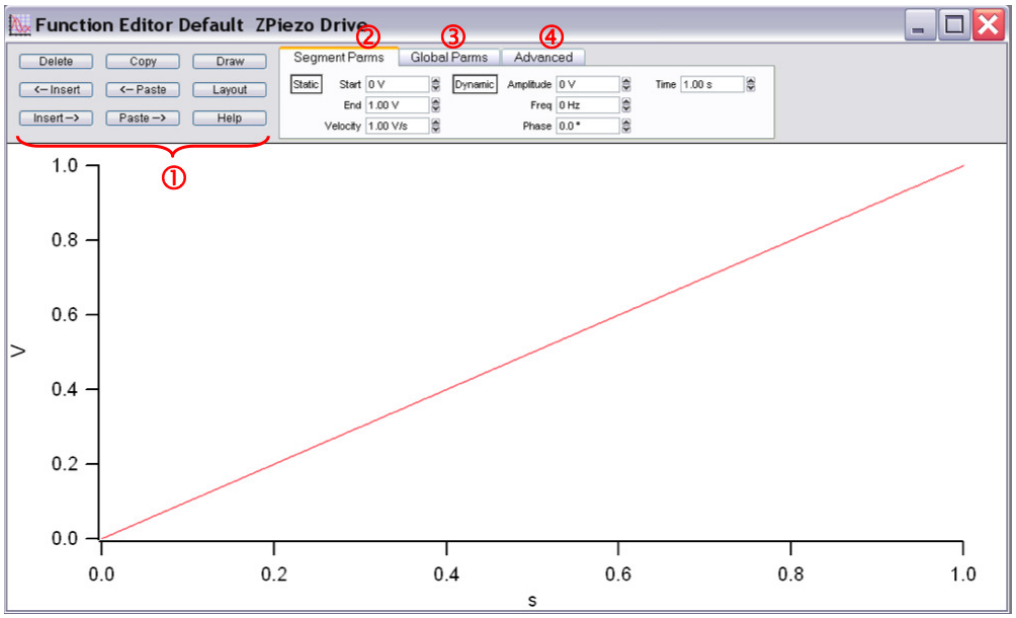


Figure 18.29.: The AR function editor allows custom wave forms to be applied to the Z piezos, or tip sample biases, for advanced force spectroscopy experiments.

1. The buttons to the right of the panel allow the segments to be copied then pasted to the left or right of the copied segment.

   Insert Will insert 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 Paste a copied segment; will have the same parameters as copied segment.

   Copy Copy a segment. Segments must in activated (red) with mouse click to copy them

   Draw Allows 'free hand' line segments that are strung together.

   Layout This dumps image of function to an Igor Layout (which are awesome!).

   Whichever segment is displayed as red is the forward most segment to be manipulated via the buttons or setvars.

2. Segment Parms Subtab:

   Static This is for DC voltages applied to the piezo (stack); the 'Start' and 'End' Piezo voltages, and velocity can be user defined.

   Dynamic This is for AC voltages applied to the piezo (shake); the amplitude, Frequency and Phase setvars can be adjusted accordingly. This is useful for oscillating the tip for viscoelastic materials.

3. Global Parms Subtab:

   This scales and offsets the signals in the function generator.

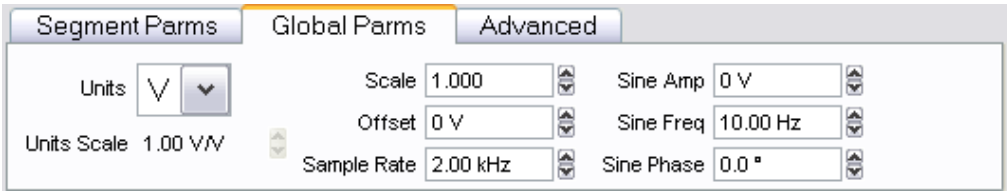


Figure 18.30.: Global Parms Subtab

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 Will offset the signal on the Y axis.

Sample Rate What frequency (# of pts/sec) the data is collected at.

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.

Advanced:

This tab allows the user to save functions for future use, and to define what the function will drive.

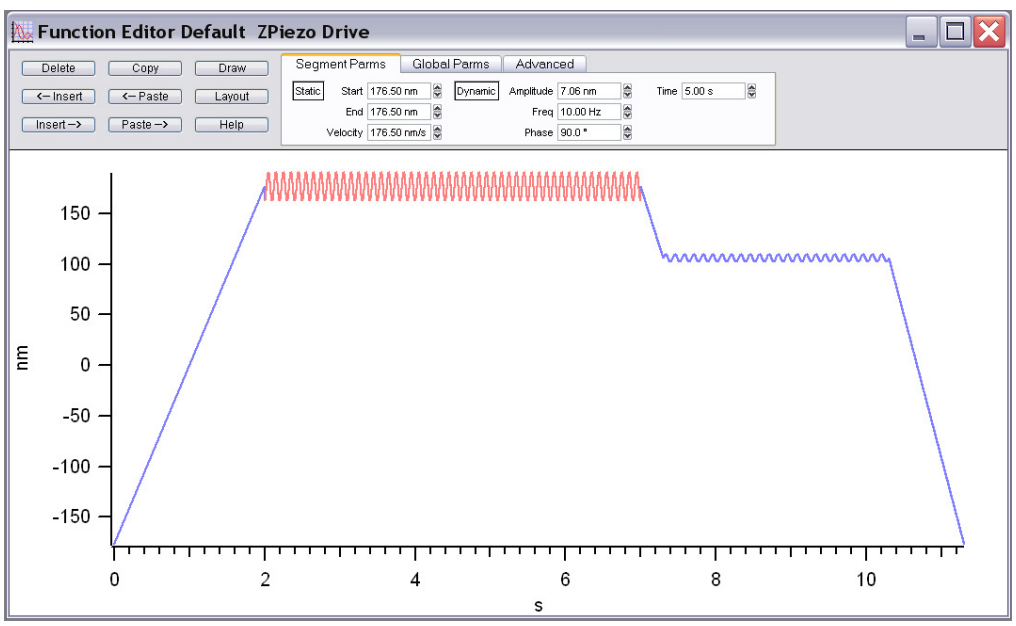
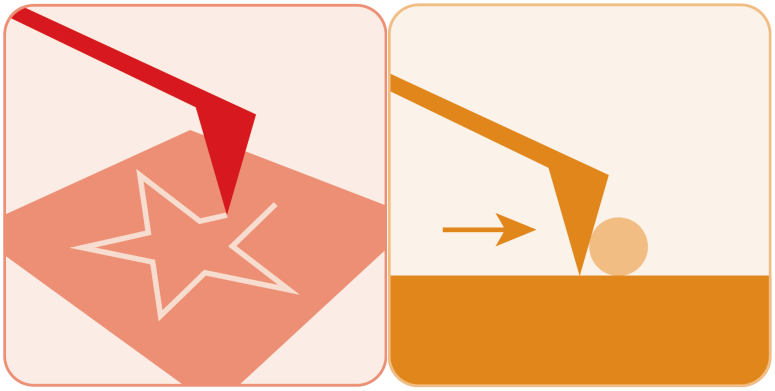


Figure 18.31.: An Example Custom Waveform

Figure 18.31 on page 261 shows a simple function that was generated in the 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°, then return to a negative deflection.

19. MicroAngelo™ Lithography & Manipulation



CHAPTER REV. 2027, DATED 07/13/2018, 16:14. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

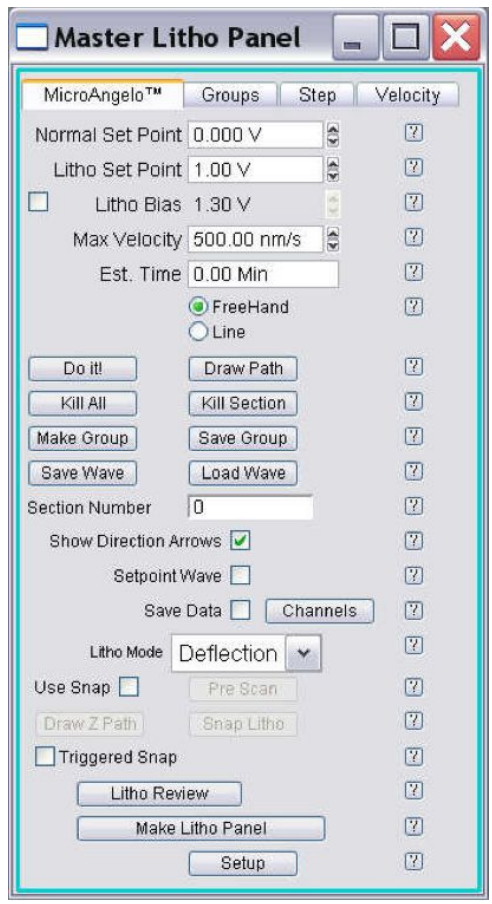
Chapter Contents

The closed loop X-Y-Z positioning of the MFP-3D and Cypher systems makes it 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 non-destructively (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, set point voltage (or tip bias) ranges defined for the execution, and the scripting performed.

These instructions and example assume the user is using an MFP-3D AFM and is set up with desired tip and sample; and is engaged on the surface.

19.1. Introduction to Software Panel

1. In the Main Menu bar of the ARSPM software, open the lithography panel by choosing *MFP Controls ▷ Litho Panel*. The panel consists of four tabs including MicroAngelo, Groups, Step & Velocity.



The MicroAngelo tab allows the user 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; other features of this window will be described later in this procedure text.

The Groups tab allows the user 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.

The Step tab allows the user to make arrays of points by defining number of points, spacing between points, dwell times (& ranges), and tip voltages (& ranges, if applicable).

The Velocity tab varies the tip velocity along the path based on the pixel color on an imported image.

The help menus are excellent for the MicroAngelo software. To access them, just click on the question marks to the right of every parameter or button in the software panels.

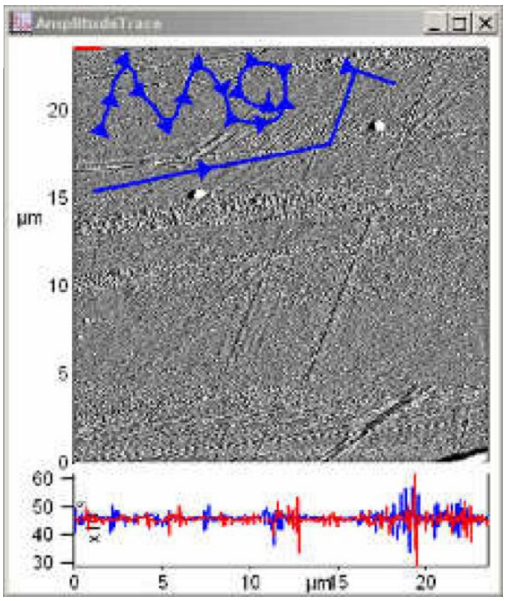
2. Image the substrate to determine an area suitable for lithography or the manipulation. Depending on your substrate material or application, it may also be a good idea to have the spring constant determined prior to the manipulation, so the user is aware of the amount of force applied at a given Set Point voltage.

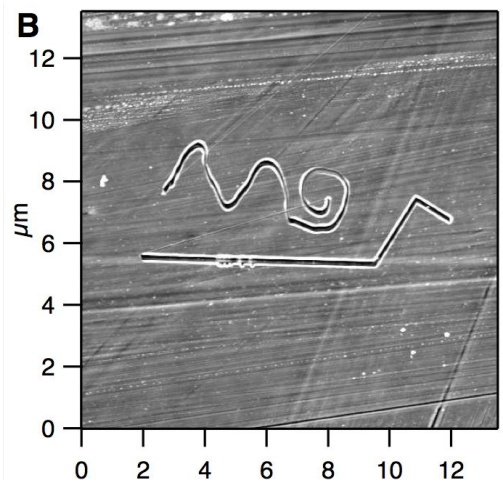
19.2. Drawing lines

19.2.1. Basic Operation

MicroAngelo allows the user to draw linear or free hand lines representing tip paths on an image window. Multiple lines can be grouped together, offset, rotated and rescaled if necessary.

1. In the MicroAngelo tab, activate the FreeHand or Line radio button depending on what kind of line you want to draw.
2. Click on the 'Draw Path' button. Notice the button now reads 'Stop Draw', the free hand and line selections become faint because they can not be switched between each other, and a cross hair cursor will be present when the mouse is on one of the image channel windows.
3. Draw a path on the image window with mouse/cursor. If drawing more than one path (with the line tool or free hand tool), left click mouse button at each segment; double click mouse button when finished drawing line segment.
4. Paths can be grouped together: If you want to save the pattern for future use, Click 'Make Group', then 'Save Group', name it in the dialogue that shows up. Notice this name will now appear in the list in the Groups tab.
5. If you want to switch between the FreeHand & Line tools, click 'Stop Draw', switch to the other; then click 'Draw Path' again. Notice the drawn line turns from red to blue once clicking 'Stop Draw'.
6. For scratching based lithography or manipulations, switch the imaging mode to Contact in that Main tab imaging mode pull down menu of the Master panel, or in the 'Litho Mode' pull down in the Litho 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/ 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 now reads 'Stop Litho'.
10. The tip will be withdrawn when completed with the lithography/ manipulation execution.
11. The 'Show Directions Arrow' checkbox will show the direction the path will traverse, and will become apparent on the image. To adjust the number of arrows on the path pattern, double click on 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.
12. 



The figures above show a simple set of lines drawn with MicroAngelo: one was by free hand, one by line. The two paths were grouped by clicking the 'Make Group', then 'Save Group' buttons- a dialogue came up asking what the group is to be named. The Free Air deflection (from S&D 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.

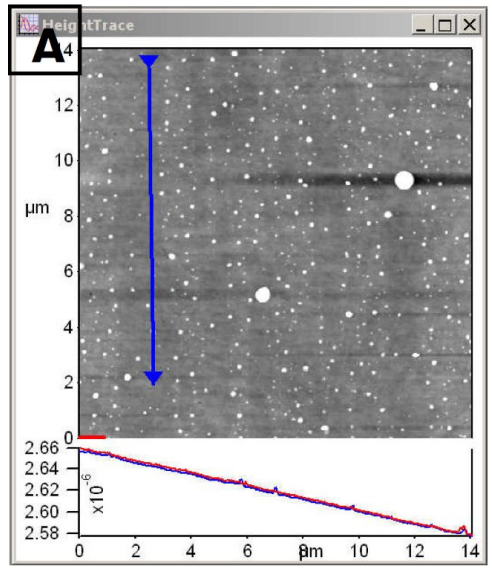
- Similarly, this same approach can be applied for sample manipulation via the tip.
- If you rescale the image/ scan area to view results, you will probably have to kill the section by clicking 'Kill All' button.

19.2.2. Using the Setpoint Wave Checkbox

You may use the Setpoint Wave checkbox in the MicroAngelo tab to systematically vary the Setpoint.

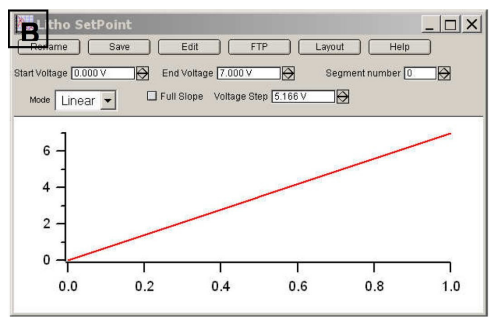
1. When Setpoint Wave is checked, a new dialogue is presented that allows Set Point voltage ranges to be applied along an individual line/path, in either a linear or staircase waveform. Note that is not able to distinguish between objects in a group.

2. After imaging the surface in AC mode, the path is drawn by clicking the 'Draw Path' button, and then the'Stop Draw' button.



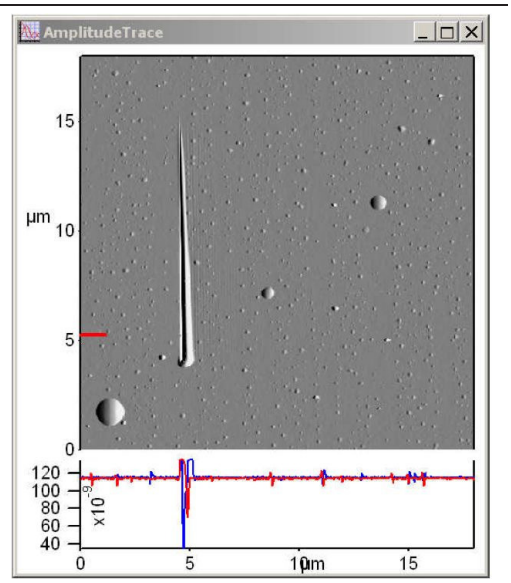
3. Check the 'Set Point Wave' checkbox , which brings up the Litho SetPoint window. The control parameters at the top of this window allow the user to define a start and ending Set Point voltage, and whether that ramp is stepped linearly, or as a step function.

- In this example, 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.



4. Click the 'Do It!' button to execute the MicroAngelo event.

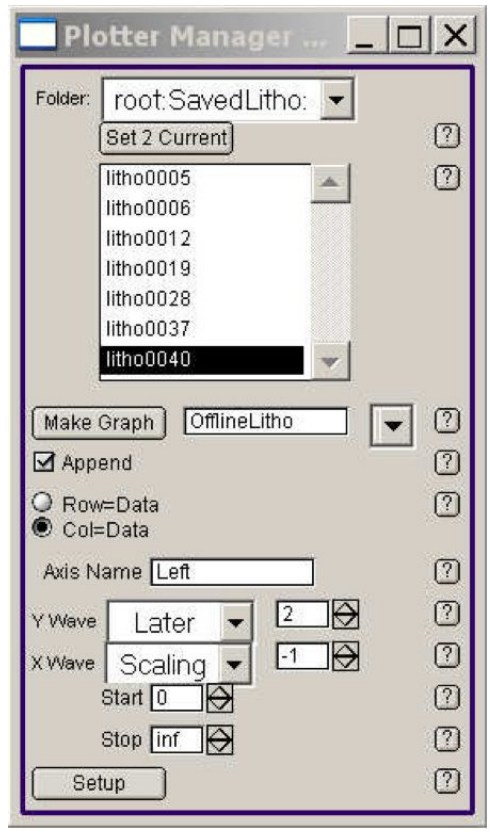
- The figure shows the results of this ramping Set Point (applied force). As expected, with increasing Set Point voltage/ force, the tip plows further into the polycarbonate surface as it traverses the defined path, seen as a feature increasing in width.
- The path was defined such the tip would experience increasing torsion as the pattern was traversed, to demonstrate what is described in the next section.



19.2.3. Save Data checkbox

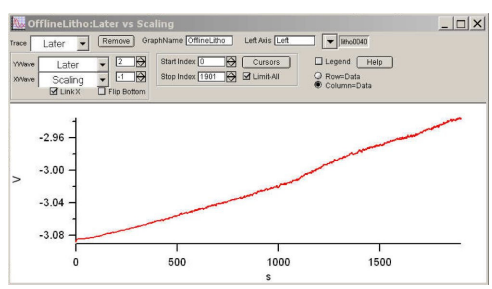
This checkbox allows the deflection and lateral signals during the event to be viewed.

1.
- To view the deflection, lateral and time signals during the manipulation event, click the 'Save Data' checkbox BEFORE the event (before clicking the 'Do It!' Button).
- After the event, click the 'Litho Review' button, which will bring up a panel similar to the one seen in the example. This panel shows a list of the litho events that were performed 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 pull down menus.



2.
- Click 'Make Graph' button to view the data; a plot similar to the one seen in the figure.
- In this case, the lateral data is shown, representing the torsion on the cantilever during the lithographic event shown in Figure 10.4 (above).

Note that 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.

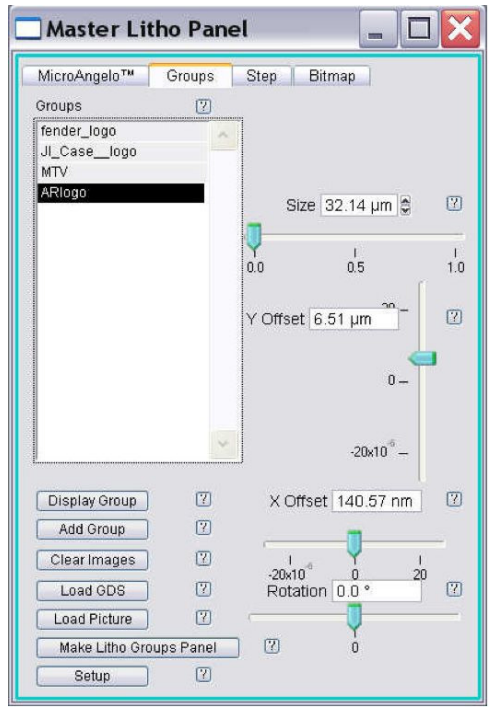


19.2.4. Importing Images as Patterns

Importing an image to use as a group in MicroAngelo^{tm} is a very straightforward task. Many image files (tiffs, jpeg, Bitmap, etc.), as well as GDS (graphics design system) CAD drawing files can be imported.

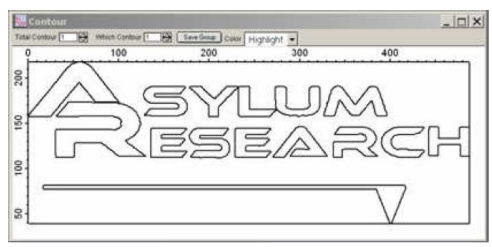
1.

- Open the Groups tab.
- Click on the 'Load Image' button; a browse dialogue comes up letting you select the stored file.



2.

- The image loads and Igor immediately makes a 'Contour' of the image. This figure 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.



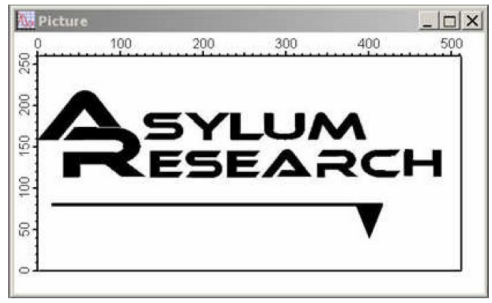
3.

- Notice at the top of the Contour window, there are some 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'. Contour effects are more pronounced in imported images that are grayscale or have color. It's best to try it on your own.

'Total contour' has something to do with edge effects- its similar to a threshold in the mask function in the modify panel.

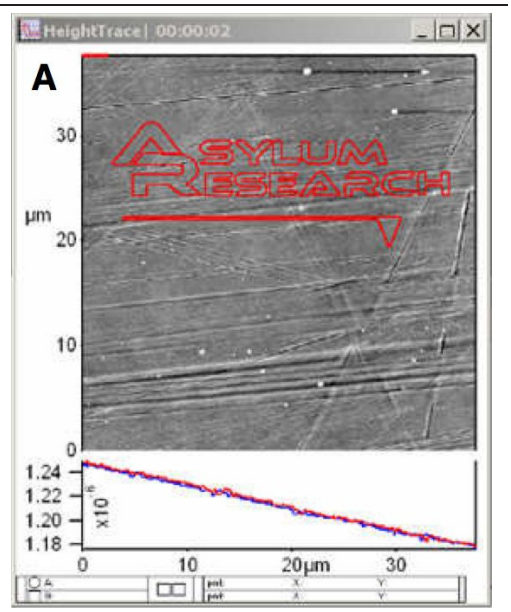
'Which contour' chooses which contours will be included in the group ultimately used as the path.

Color is a pull down menu that will color the different contours.

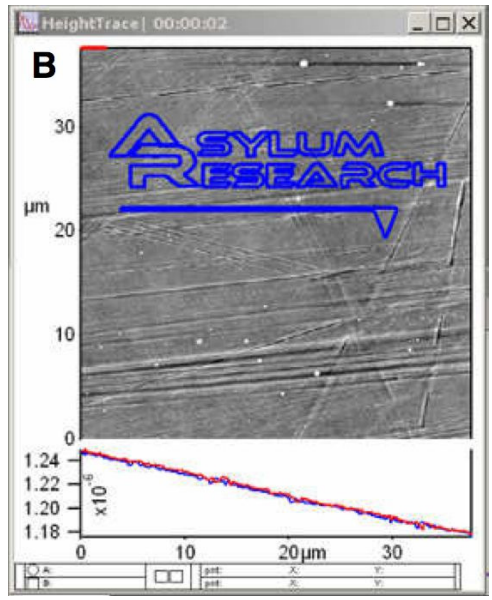


4.

- Click the 'Save Group' button to add this group to the list. (In this example it is called 'ARlogo')
- Highlight the group of your choice.
- Click the 'Display Group' button- this will display the group (colored red) in the image window (see example).

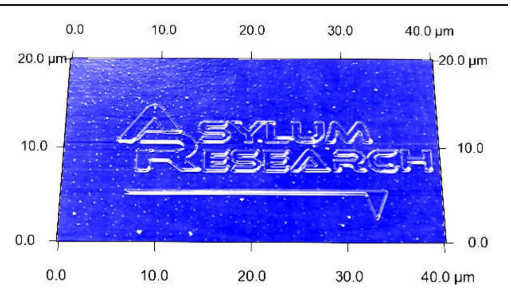


5.
- Position the group where you want it in the image area using the X & Y offset slider controls, along with rotation and size slider controls.
- Click the 'Add Group' button, which will make the group pattern blue in the image window.



6. Go back to the MicroAngelo^{tm} tab.
7. Define the 'Normal' & 'Litho Setpoint' voltages and tip velocity.
   - Choose a 'Normal Set Point' voltage value that doesn't damage the surface between paths.
   - Choose a 'Litho set point' 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 from the pull-down menu (if that is the desired mode). 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 will show the direction the path will traverse, and will become apparent on the image. To adjust the number of arrows on the path pattern, double click on 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 that represents 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 will be 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 this figure.

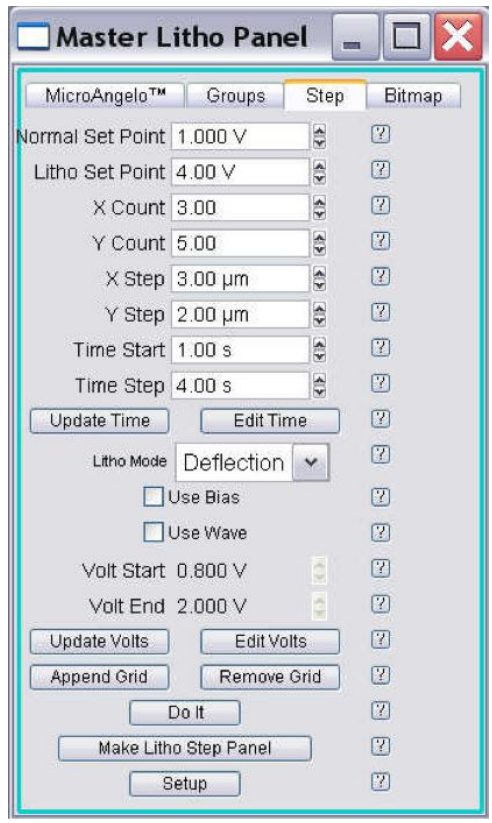


19.2.5. Creating Array Patterns: The Step Tab

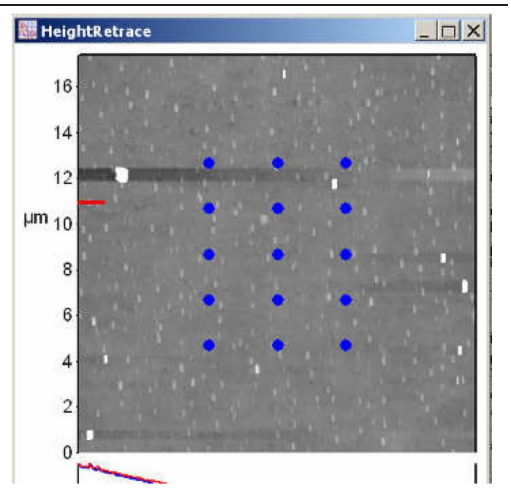
Arrays can be created using the Steps tab. This tab can have the tip dwell at each subsequent array point for a user defined time or apply a different force. Both functions can be systematically increased, or custom values can be manually entered.

19.2.5.1. Creating simple arrays

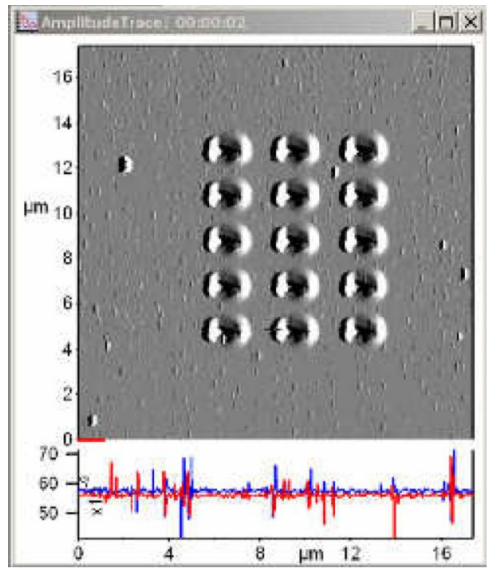
1.
- Choose number of points desired in the array with 'X Count' and 'Y Count' setvar inputs.
- Choose the distance desired between points in the array, in both X & Y dimensions using 'X Step' & 'Y Step' setvar inputs.
- The 'Time Start' setvar value is the amount of time the tip will be in contact with the surface (dwell) at each point in the array. If it is desired to have the tip have the same dwell at each point, enter 0 s in the 'Time Step' value input.



2.
- Click 'Do It' button to fabricate the array pattern. Notice the S&D meter deflection values reaching the defined Set Point during the indents.
- Point 1 in the array start with the lower left, move to the right, then moves to left array in next row.



3.
- The array will be centered in image window; the XY offsets in the group tab doesn't move the array to a user defined area in the image (like it does when using a pattern).
- In the example shown 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.

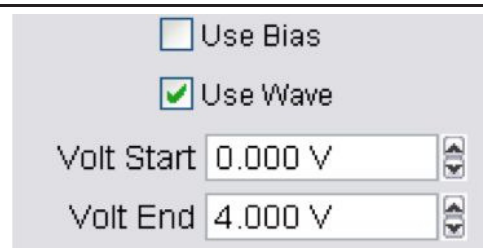


Varying Tip Dwell Time at each Array Point The amount of time the tip stays in contact with the surface at each array point can be independently or systematically varied using the Steps tab.

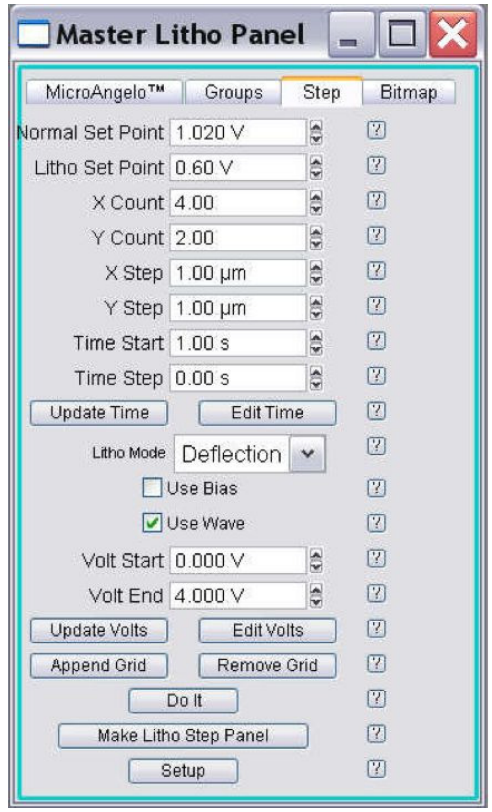
- To systematically vary the dwell time at each subsequent point in the array-
  1. Define a 'Start Time' value which represents the amount of time the tip dwells at point 1.
  2. Define a 'Time Step' value: this is the amount of additional time spent at each subsequent point. For example, if you want the tip to spend 3s longer at each subsequent point, enter 3s in 'Time Step'; (Start Time being 1s). Point 1 (lower left of the array) would spend 1s; point two would spend 4s; point three, 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. This will bring up a spreadsheet that allows the user to 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 checkbox above the 'Volt Start' setvar input (this is actually called 'Use Voltage Wave). This will disregard the 'Litho Setpoint' input value in the MicroAngelo tab.

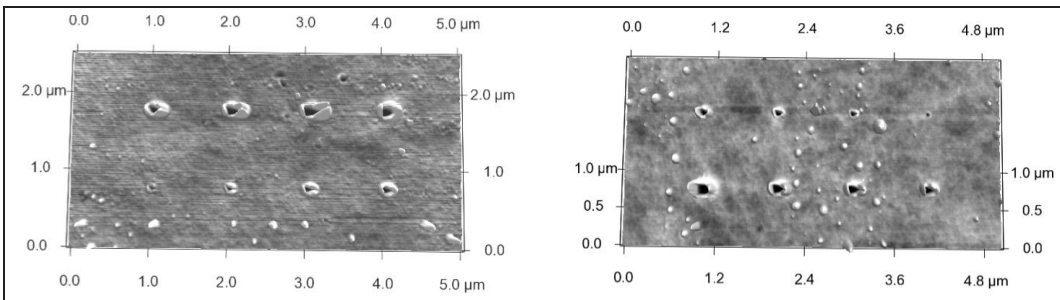


2. To systematically vary the applied force at each subsequent point in the array-
   - Define a starting Set Point voltage using 'Volt Start' setvar input.
   - Define an end Set Point voltage using 'Volt End' setvar input.
   - Click 'Update Volts' button to activate this change.



3. The individual Set Point setvar 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 be monitored in the S&D meter.

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 (Tine Start = 1s; Time Step = 0s).

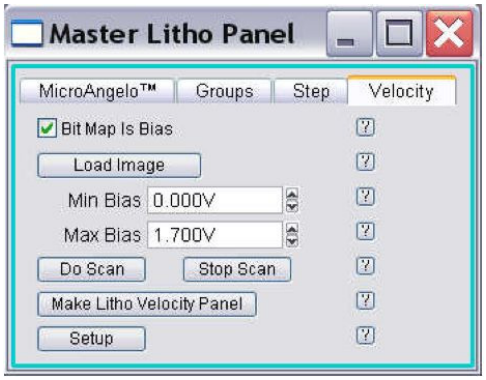
To independently vary the applied force at each subsequent point in the array-

When a more custom Set Point variation is required- in this case, click the 'Edit Volts' button. This will bring up a spreadsheet that you can go into and manually change the Set Point at each respective point.

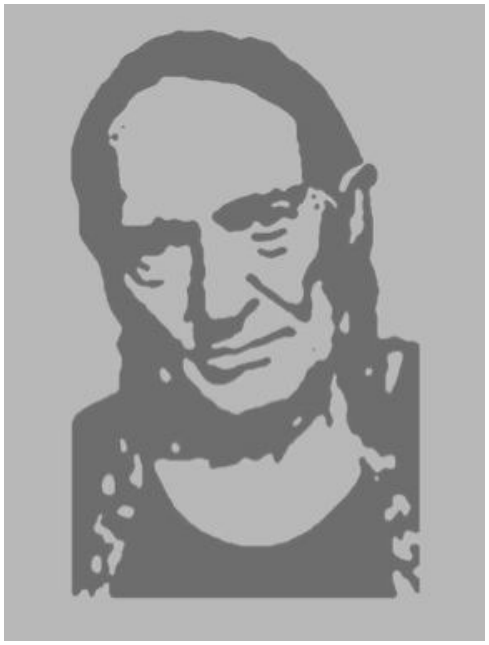
19.2.6. The Velocity Tab

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' while the bright grays are patterned at the 'Max Velocity'. 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. 2027, DATED 07/13/2018, 16:14. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

Chapter Contents

20.1. Introduction

Before you begin taking force curves on your sample, you will need to first 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. All of these methods, we will discuss 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; this list is always growing as well. Asylum has implemented aspects of this method in an automated calibration technique called 20.2™, which we will 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 a member yet, 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™ will calibrate 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 *(internet connection required)*.

20.2.2. How to Guide

In the **Thermal** tab, click on the 'GetReal Probe Panel' button and select the lever you have loaded from the list. This loads a number of constants that describe that specific cantilever geometry. Then click the 'GetReal Calibration' button and the software will begin 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 get the InvOLS, see Section 21.3 on page 294 for more details on this.

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 280.
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 283.
3. Withdraw tip & 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 290.

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 288.

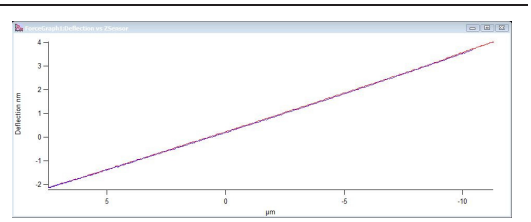
NOTE: If at all possible, it is often best to do the spring constant calibration in air as it is easier to fit the higher Q response in air.

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, resulting in 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 for, 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 depends on how the light is aligned on the cantilever as it travels through the Z-piezo range. By performing this step first, it will give you 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.

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 the start dist to -inf, or drag the red bar to the top
4. Set the force dist to inf, or expand the red bar all the way to the bottom
5. **Do a force plot**



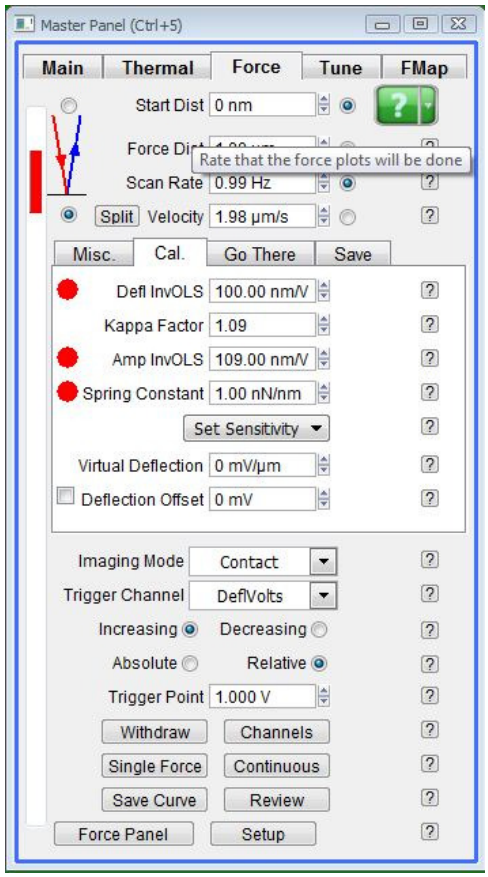
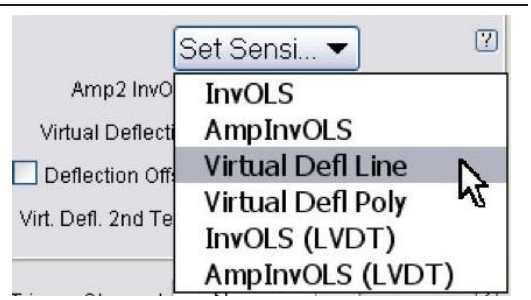
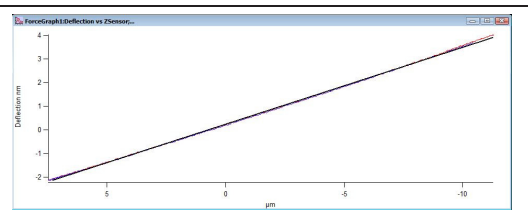


Figure 20.1.: *Master Panel ▷ Force Tab ▷ Calibration Tab.*

6. Right click on the graph and select Virtual deflection
or
or from the cal sub tab, Set sensitivity control, select virtual deflection



7. **Check the fit**

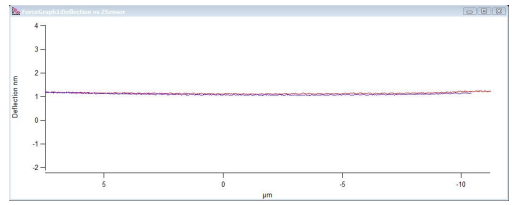


8. You can fit a sub region of the force curve by using the Igor cursors (shown with Ctrl + i). But you should avoid doing second order poly virtual deflection on sub regions of the curve, they can greatly alter any data later collected outside of the region fit.

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 non-linear fashion.

10. **Review** The virtual deflection has now been corrected so the approach now looks level (as it should since it is not experiencing any deflection during the approach) in subsequent force curves.



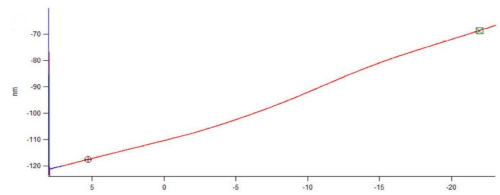
20.3.1.1. Tips and Tricks

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 cuve may not appear linear at all.

- In this case you should select 'Virtual Defl Poly' in Step 6 on page 281 to account for the curvature of the approach curve.

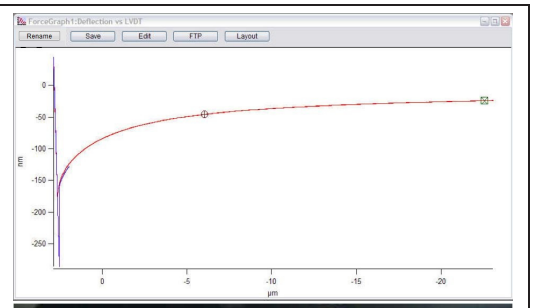
Note! is it critical you do a full range force plot to calibrate the poly invols, as described above. Extrapolating a polynomial past the fit region can be very inacurate.



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 measurment 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 photodector combination as a whole. In the previous section we eliminated the possible instrumental errors of this measurement which might otherwise contribute to cascading inaccuracies of the Spring Constant measurement.

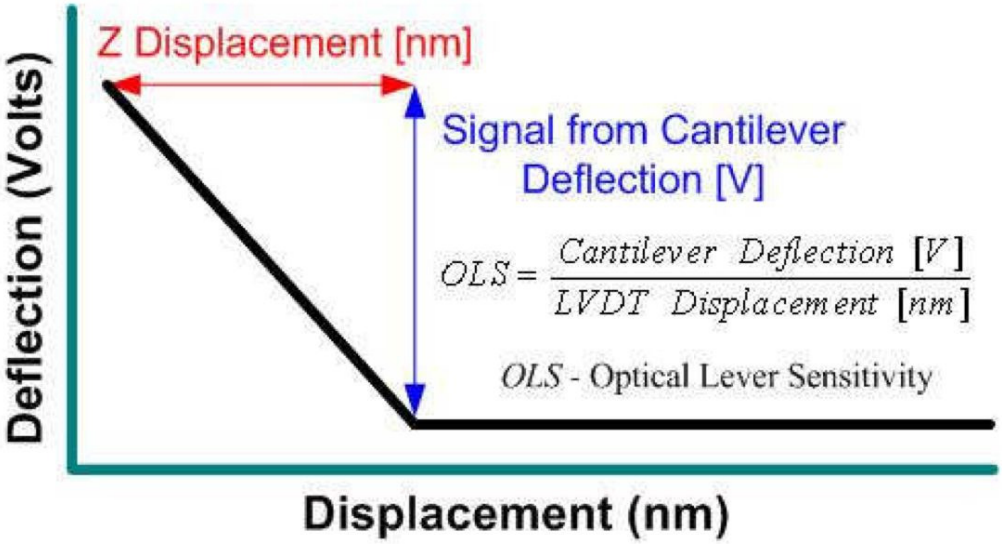


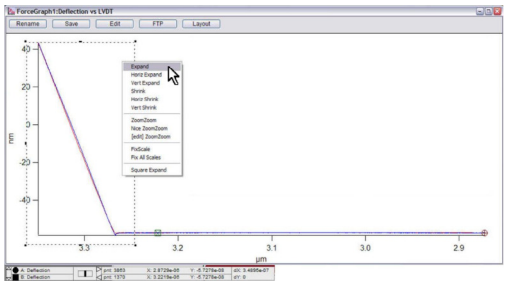
Figure 20.2.: Inverse Optical Lever Sensitivity measured from a force curve on a hard surface.

Figure 20.2 on page 283 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 deflection or DeflV.
- An absolute trigger of 1V is a good place to start for most tips and samples.
- Click the *Single Force* (Ctrl +3) to do a force plot.
- Optionally click and drag a box around the sloped region of the curve and right click to expand on it for better a better view.

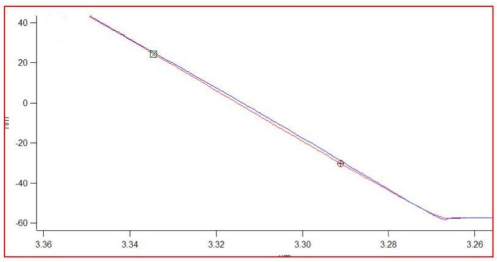


Note the non-contact region of the curve should be horizontal if the virtual deflection correction is working properly.

2.

Find Invols

- Right click on the contact region at a reasonably large force, and select Calculate Invols.
- It will put up a fit to the deflection vs Z Sensor and calculate Invols for you.



3.

[Optional] Fine Tune Cursor Positions

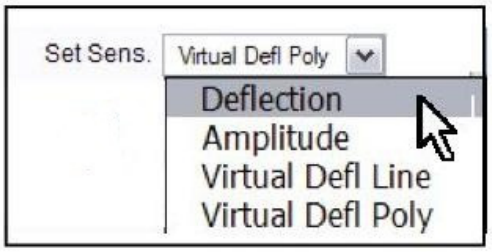
- You can move the A (circle) and B (Square) cursors after the fit to change the fit region.
- You can drag them each individually

OR

- Deactivate one of the cursors on the curve by going into the lower margin of that Igor window and clicking on the open circle/square.
- When it's deactivated, it will turn black allowing you to finely position the other cursors with the arrow keys.

4. **Perform Line Fit**

   - On the *Master Panel* ▷ *Force Tab* ▷ *Cal sub tab*
   - 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 281), 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. Lets try 100 curves.

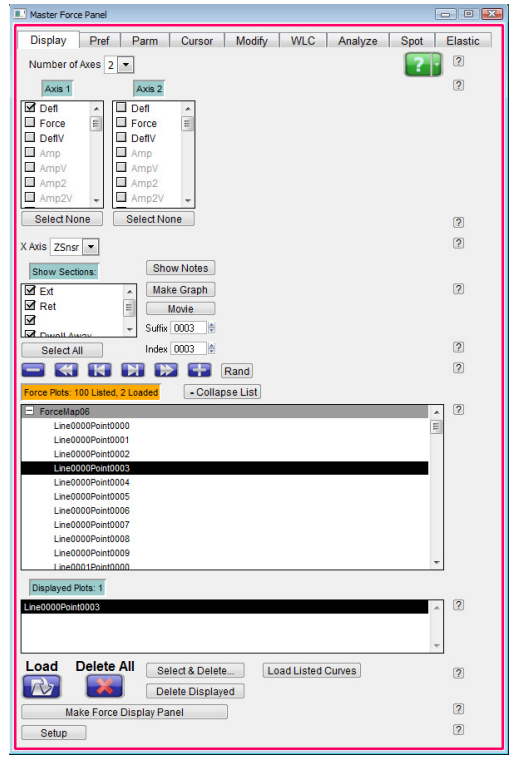
   Note This will allow a set number of force curves to be sequentially acquired at the same XY location, without clicking the 'Single Force' button repeatedly.

3. Click the 'Continuous' button to acquire the force curves. This may take a few minutes, since each curve requires a second or two.

4. **Activate** 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.

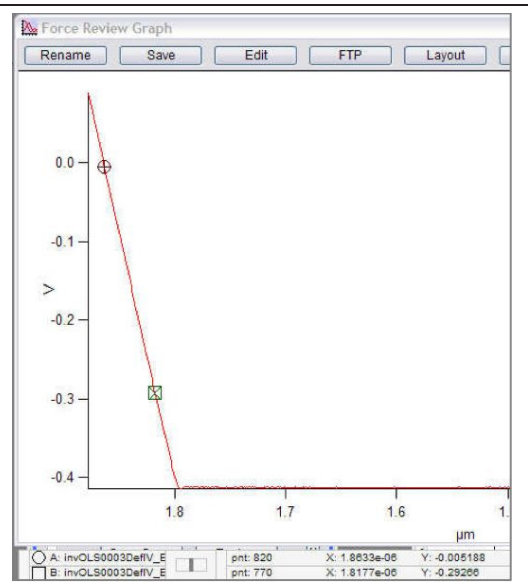
Note Take note of the suffixes of these curves because they will have to be designated in the Analysis tab.



5. **Plot** the Force Curves

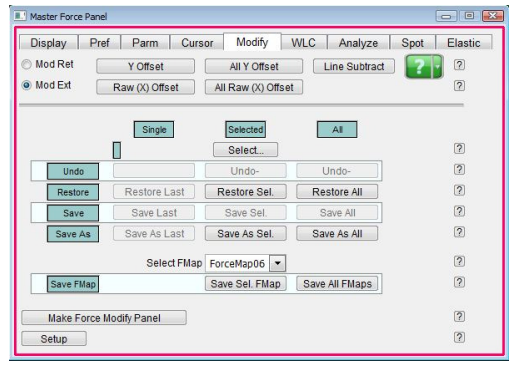
- Plot the Force curves as Deflection volts versus ZSnsr.
- With the graph window selected, click Ctrl + i to show the info area on the bottom of the graph.
- Drag the cursors from the info area onto the sloped part of the graph.

Note Take note of the Y axis values, these will also be used in the Analysis tab to designate the *deflection range* during the averaging.



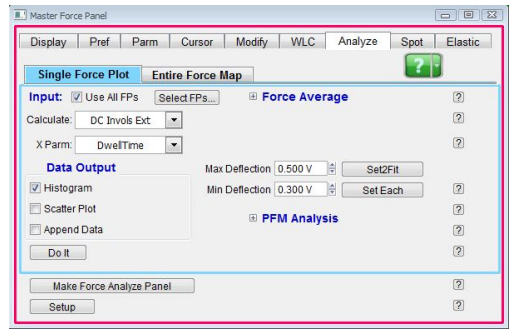
6. **Offset** the Data

- Go to Modify tab of the force review.
- Select Ext in the upper left and then All Y offsets
- This will offset the data to deal with 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.
- Under the 'Calculate' pull-down, select 'DC InvOLS'.

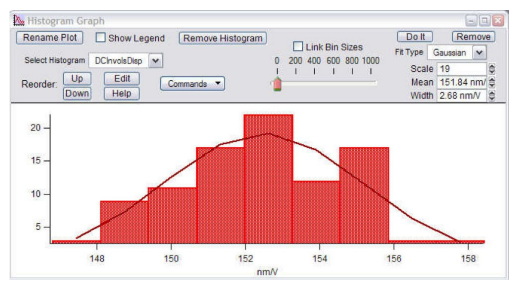


8. [Optional] Click on the Select FPs... button to select which force plots you want to analyze.
9. Under the 'Max Deflection' control, enter the value of the higher cursor.
10. Under the 'Min Deflection' control, enter the value of the lower cursor.

11. **Create** the Histogram

- Check the 'Histogram' Check box.
- Click the 'Do It' button, a histogram should be generated that will show a distribution of InvOLS values.
- In the figure a Gaussian fit was applied, although other fit types are available in the 'Fit Type' pull-down.

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, which is needed to fit for the spring constant. Thermals are outlined in the next section21. 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.

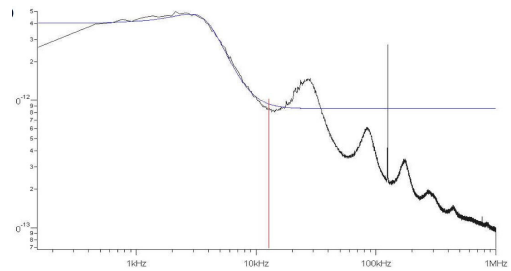
20.3.5. Tips and Tricks

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 by:

- Taking 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 of the tip a turn or two of 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

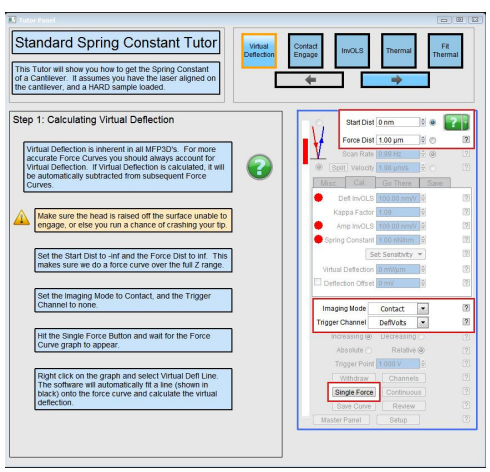


Figure 20.3.: Spring Constant Tutor

The Spring Constant Tutor allows all three steps of the spring constant (k) determination steps Section 20.3 on page 280 to be performed in a single software panel; particularly useful among 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 substeps 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. 2022, DATED 07/09/2018, 16:52. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

Chapter Contents

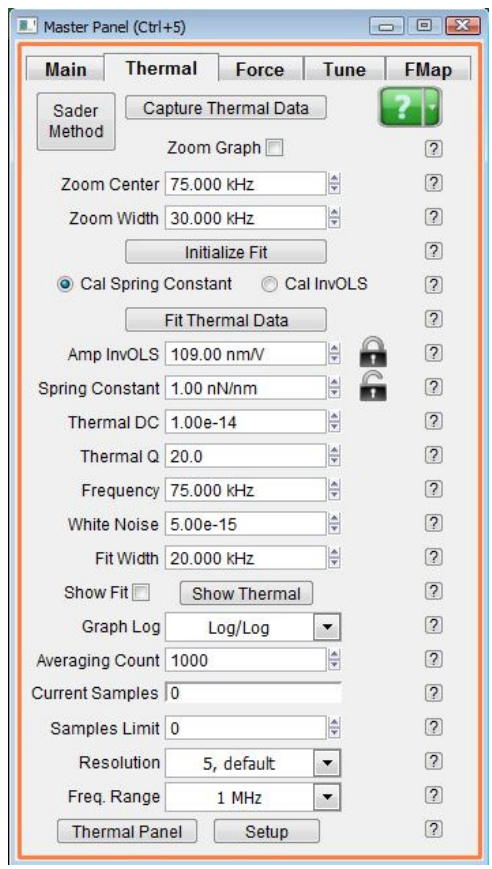
Capturing a thermal spectrum consists of pulling back data at a high rate and doing a FFT to determine the spectral components. The MFP3D can run thermals up to 2 MHz and Cypher AFMs up to 32 MHz. These figures are based on sampling rates and anti aliasing filters for the respective instruments.

Thermal Tunes are mostly used for a few specific tasks:

- Determining the cantilever Spring Constant OR InvOLS.
- To determine 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).

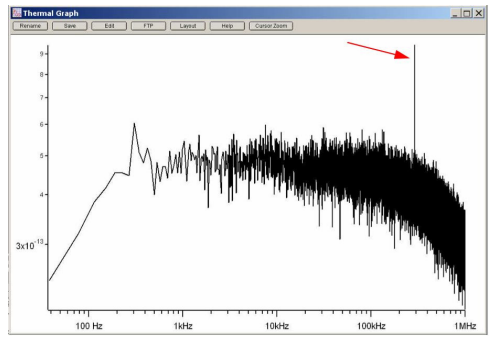
21.1. Capturing the Thermal

1. The Thermal tab is located in the Master Panel (Ctrl + 5).

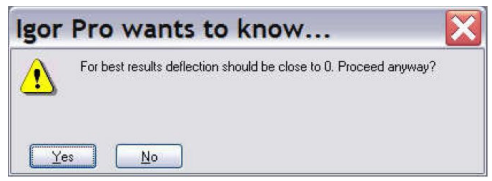


2. Assuming a probe is properly loaded, SLD aligned and the photodiode (PD) zeroed, follow the procedure below to acquire a thermal tune of the cantilever

3. Click the “Do Thermal” button (Short cut: Ctrl+2); a power spectrum plot will appear, continuously averaging spectrums in real time. In this example, 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. Doing thermals in liquid is much more difficult to identify peaks, you should start out looking at thermals in air.



4. If the deflection is not zeroed, a dialogue will come up. On Cypher there will be an option to zero the PD then. On an MFP3D you can either click No and zero the PD, or if precision is not your top priority, click Yes and continue. The reason to zero the PD is to have a more linear response in their center.



5. The user 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 3).

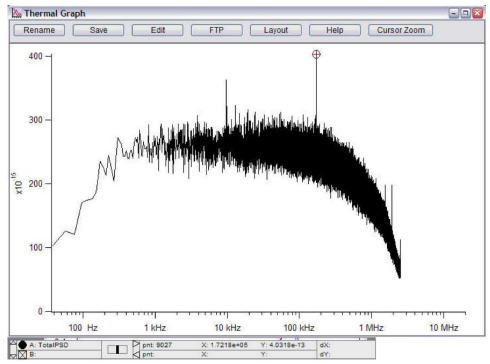
6. Click the Stop button to terminate the collection of power spectrums (ShortCut: Ctrl+2).

Resolution of the acquisition can be changed in the resolution pull down menu of the Thermal tab. Larger numbers go faster (less resolution); while smaller values acquire slower, but give less noise. The way the data is plotted (i.e., linear vs. logarithmic) can also be changed in the Graph Log pull down 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.

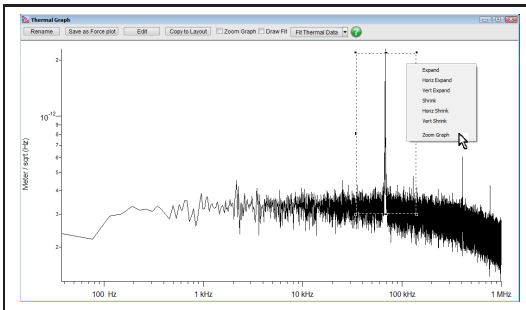
Be careful when selecting the 2.5 MHz Range on an MFP3D, as you can see all the way to Nyquist, the anti alias filters may not have removed all of the aliasing: signals between 2 and 2.5 MHz may actually be from 2.5 to 3 MHz.

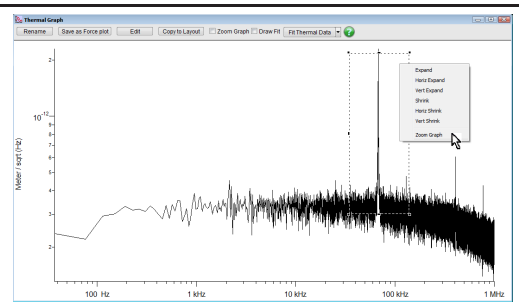


Save Thermal tune plots by

- Clicking the Save as Force plot button at the top of the thermal plot; saves as data as a force plot.
  - This is a little strange, in that the PSD is saved as deflection (what is really m/sqrt(Hz) is labeled as m).
  - and that the you need to change the X axis of the force plot to frequency to display it correctly in the force review.
- 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 283 for more details.
2. 



   Select peak and fit thermal:

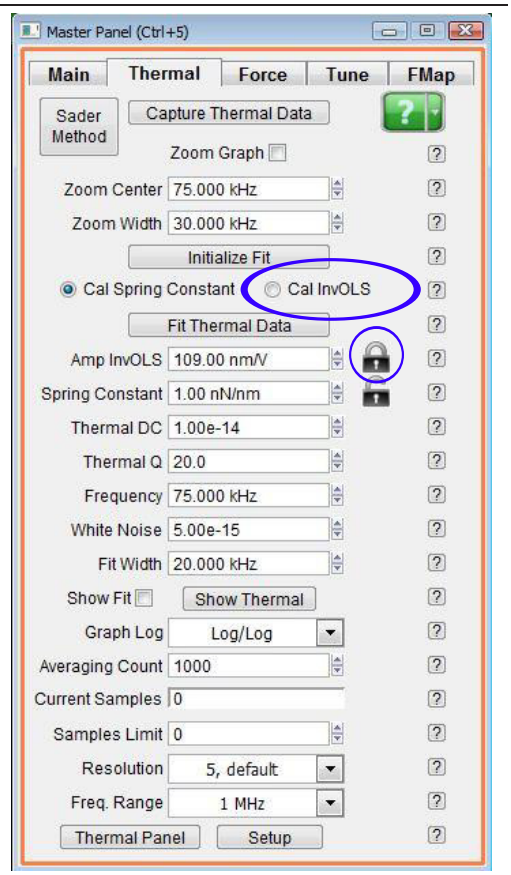
   - Zoom into the area of the peak by left mouse dragging around the peak, then right click to see the menu.
   - Choose Zoom Graph (at the bottom); the result would look similar to the graph on the right.
   - Click on fit thermal, the blue curve will appear.
3. The default setup is that AmpInvols is 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 some clean hard surface. The spring constant should not change. Invols will depend on numerous factors, temperature, spot position, liquid, etc, so if you know the spring constant, you can collect thermal data, and find what Invols value would give you the same spring constant.

General outline:

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 on 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

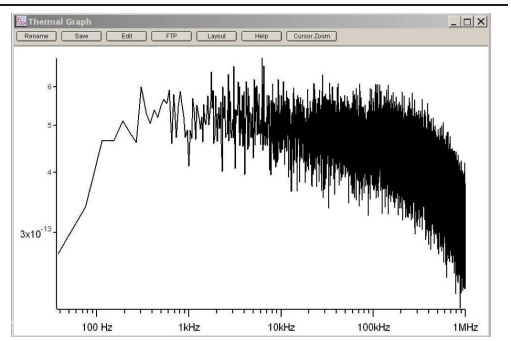
- Thermals work very well for determining the resonant frequencies (and ultimately drive frequencies) of cantilevers in fluid.

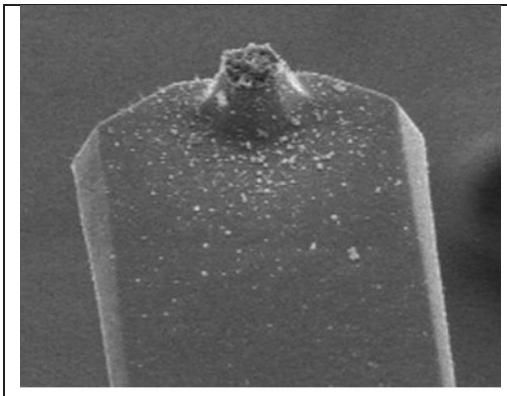
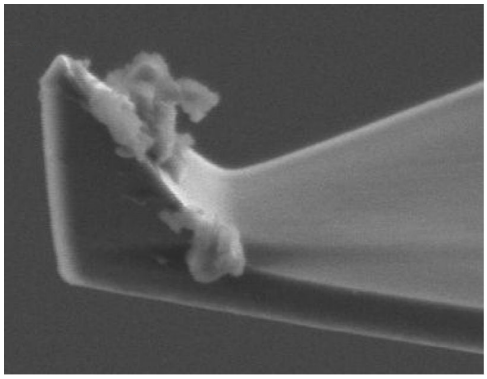
- After you have fit the thermal peak, you can right click on it and select Move Freq, Phase and amp to tune. This will set the drive amplitude, drive frequency and Phase offset for the cantilever drive based on the thermal fit.
  * You can do this operation from the right click menu, or from the right hand side of the Fit Thermal Data button the thermal, or the right hand side of the Center Phase button 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 figure to the right shows 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.



2. 


The figures 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. 2035, DATED 08/15/2018, 14:50. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

Chapter Contents

Even though this user guide focuses on specific SPM applications techniques, it is still essential and, hopefully, of interest to understand the working concepts behind the **Scanning Probe Microscope** (SPM) in order 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 will cover very generic ideas on SPM. Each following chapter in this part of the user guide will elaborate 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: create a map of the earth by flying a plane across its surface. With a radar altimeter the pilot gets 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 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 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 descend the plane when the altimeter reads above the setpoint. This is a very basic example of something called 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 become:

Tip motion Given the relatively small distances over which the tip moves (usually microns down to angstroms) the preferred mechanical actuator is made of piezo electric materials, which 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 the 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. So, 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 postioners.

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. Section discusses the subject further and tells you how to use the AR SPM software to do STM imaging.

22.3. The Atomic Force Microscope (AFM).

Where does the name come from? It's a force microscope since it measures force between tip and sample (opposed to tunneling current with an STM). It is not required that the sample is conducting; one of many reasons why AFMs are much more commonplace today than dedicated STMs. An AFM is atomic since it is capable of resolving atoms. The list of basic requirements becomes:

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 allows 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 micromachined 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 piezo electric 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 **??** for further discussion and how to do 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 on this in **??**. There are at least a dozen commonly used imaging modes beside Contact and AC. These all get their own chapter in this part of the user guide. Each chapter will start off with a little theory and will then describe how to it is put into practice in the AR SPM software. In some cases special accessories are required, for which you will 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, spring constants. (See the Probe Storefor an assortment.) Figure 22.1 on page 303 gives a typical example of a Silicon cantilever. The three main components are:

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 will tweezers and clamp it down in the AFM.

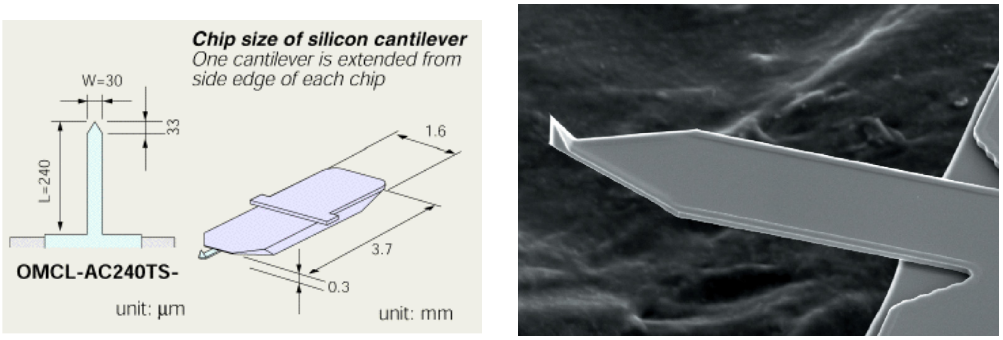
Lever or Cantilever The diving board protruding from the end of the chip.

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'll do our best to call the entire object the cantilever, and the sub-parts the chip, lever, and tip. Context will usually make it clear if the word cantilever refers to the whole object or the lever itself.

22.4.2. About the Lever Deflection Detector

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 303.



(a) AC240 cantilever and chip dimensions **(b)** AC160 SEM micrograph

Figure 22.1.: Cantilever Examples from Olympus

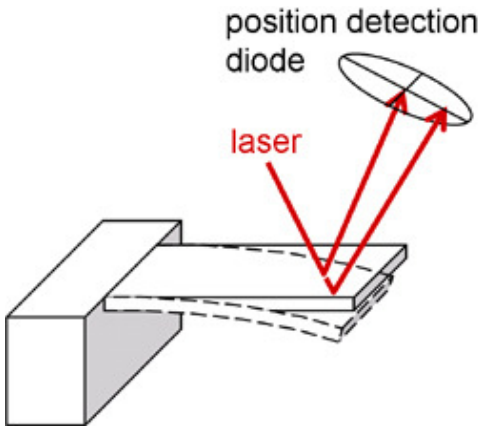


Figure 22.2.: Cantilever optical detection. The optical lever.

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 nice 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 will tell 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 possible to detect cantilever deflection and also cantilever torsion. The latter is useful for something called Lateral Force Microscopy or LFM (See **??**.)

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 intereference effects. We'll typically refer to this optical lever light source as "laser". The word light source could be used, but can be confusing since 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

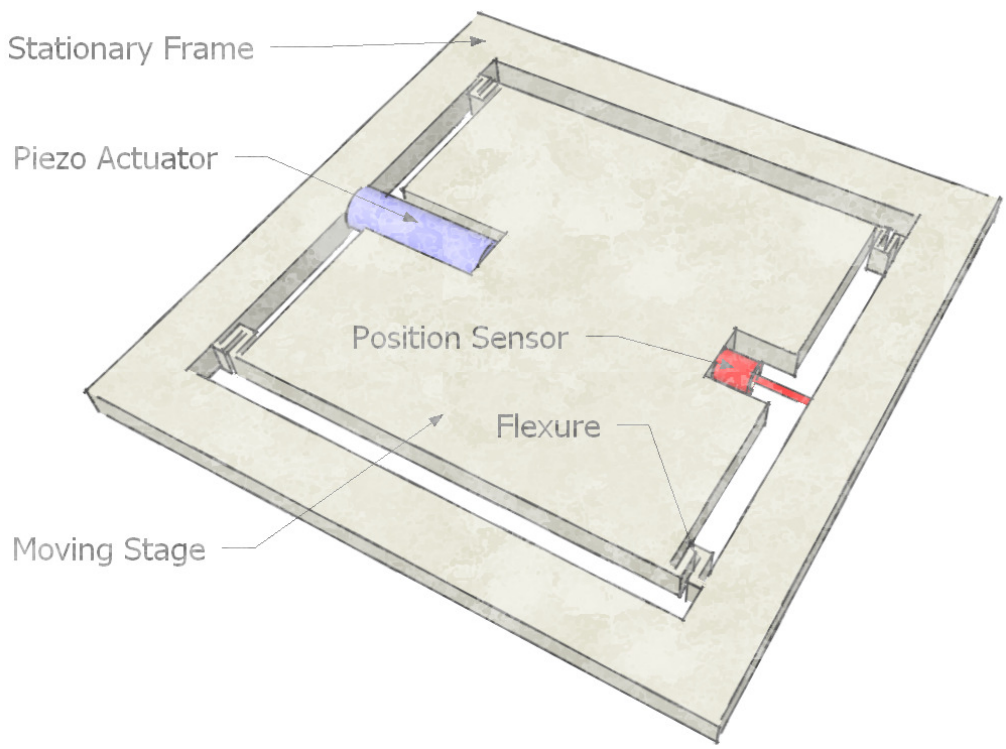


Figure 22.3.: Diagram of a flexure based nano positioning stage with position sensor. The frame, flexures, and stage are all cut from a single piece of metal.

Motion in SPMs is typically accomplished by pieces of piezoelectric material which expand when voltage is applied. In Asylum Research AFMs these so called **piezo electric actuators** (or simply **piezos**) are used to push between a frame and a metal element which 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 304 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.

22.4.4. A Basic AFM Diagram

Let put the bits of the last two sections together in a rudimentary diagram of an AFM. 22.4 shows a sample mounted on a Z positioning stage with is in turn mounted on an XY positioning stage. Position sensors inside the positioning stages are not shown.

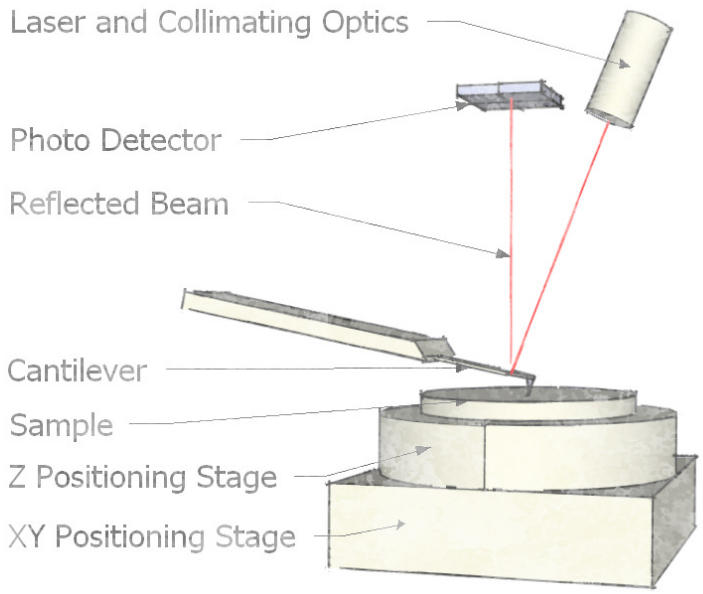


Figure 22.4.: A basic sample scanning AFM (such as the Asylum Research Cypher AFM). A rigid frame to which the cantilever, detector, laser, and XY stage are attached is not shown.

A collimated laser beam is focused on the cantilever and the reflected beam is directed toward the split photodetector.

Not shown are:

- is the rigid mechanical frame which holds the cantilever, laser, detector, and XY positioning stage fixed together.
- The mechanism which 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 which 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.

22.5. AFM Electronics

As you may have guessed, the voltages for driving the piezo actuators, and 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 be too often 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 305 will be re-drawn with the necessary electronic connection and circuit elements required for the imaging technique.

23. Controlling the XY Scanning Motion

CHAPTER REV. 2027, DATED 07/13/2018, 16:14. USER GUIDE REV. 2053, DATED 09/28/2018, 15:06.

Chapter Contents

Only read this chater if you are particularlly interested in the minute details of how the SPM software specifically controls the XY-axes during scanning of samples.

Chapter 22 on page 299 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 aforementioned anaology 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 will 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. You should 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 it's 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 309 shows the voltage applied to fast scan piezo as a function of time. This is a typical 1Hz scan, where the X positon 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 swatooth with 512 peaks.

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.

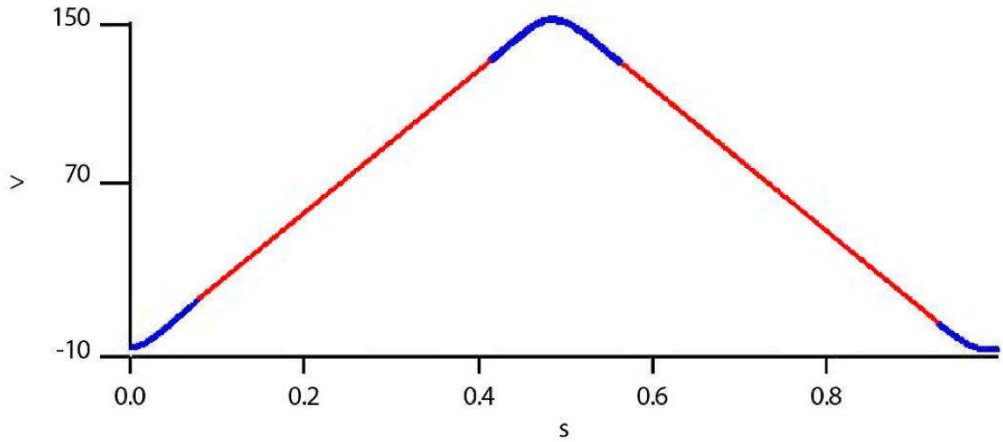


Figure 23.1.: Fast Axis X Piezo Drive (at 0°scan angle). Y-axis of graph is piezo voltage; X-axis is time. The rounded caps (blue regions) are called the turn-around, where the fast axis changes direction. Data from the turn-around is not typically saved, though it is possible to access it for special applications.

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, since 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, (where the fast scan is changing linearly), the slow axis is held constant. The striaight "flight paths" of the tip are parallel and the turnarounds are nicely rounded to minimize transient vibrations caused by scanner turnaround.

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 311) 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 311 and Figure 23.5 on page 312.

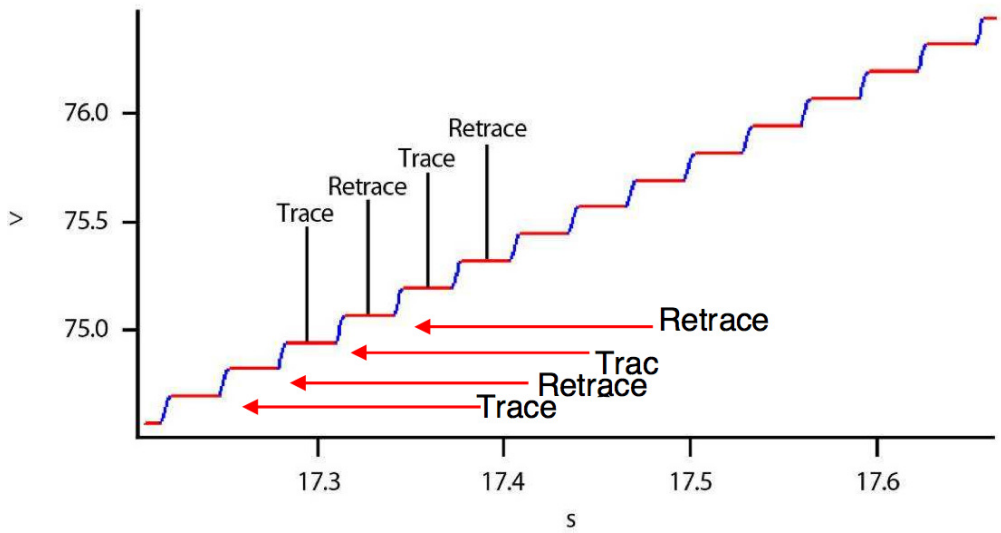


Figure 23.2.: Slow Axis of Ortho Scan. In image, vertical axis is Y piezo voltage (scan angle 0°); horizontal axis is time. Axis limits are zoomed in to s how the same region as in preceding Figure (Slow Axis of Raster Scan). The blue sections denote turn-around regions. Approximately 7 scan lines are shown here, each comprising two steps (one for Trace and one for Retrace).

In the early days, when AFMs were controlled by analog electronics, raster scanning was typical as 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 312). 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 312, 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 much raster scanning continues to this day. All Asylum Reserach AFMs have closed loop scanners and digital controllers and uses ortho scanning almost exclusively. This mode eliminates the pixel position problems discussed (above) in raster scanning.

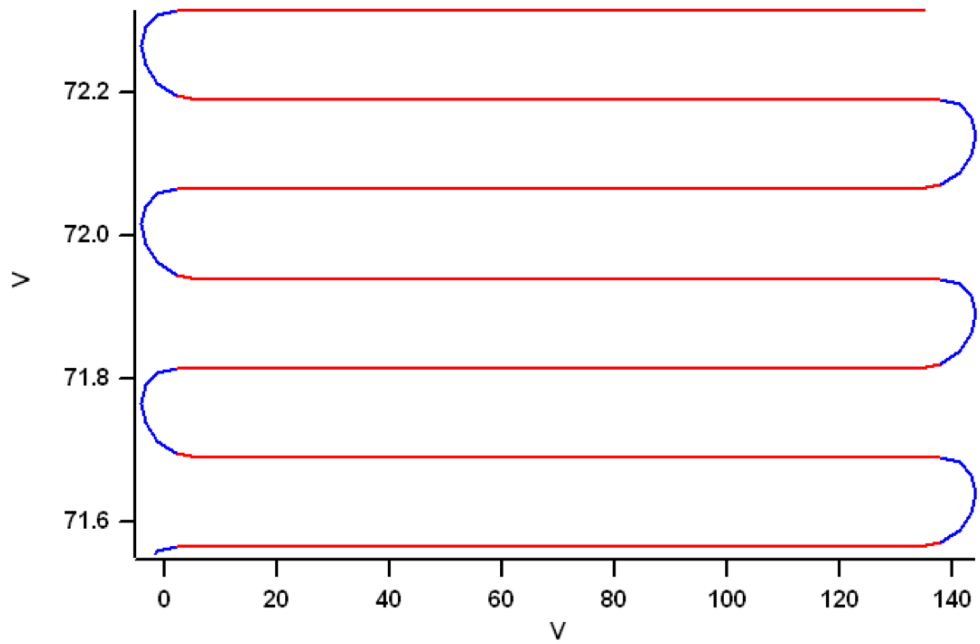


Figure 23.3.: Motion of the Tip Across Surface in Ortho Imaging. The X-axis is the fast axis drive, while the Y-axis is the slow axis drive. Three scan lines are shown. The blue sections denote turn-around regions.

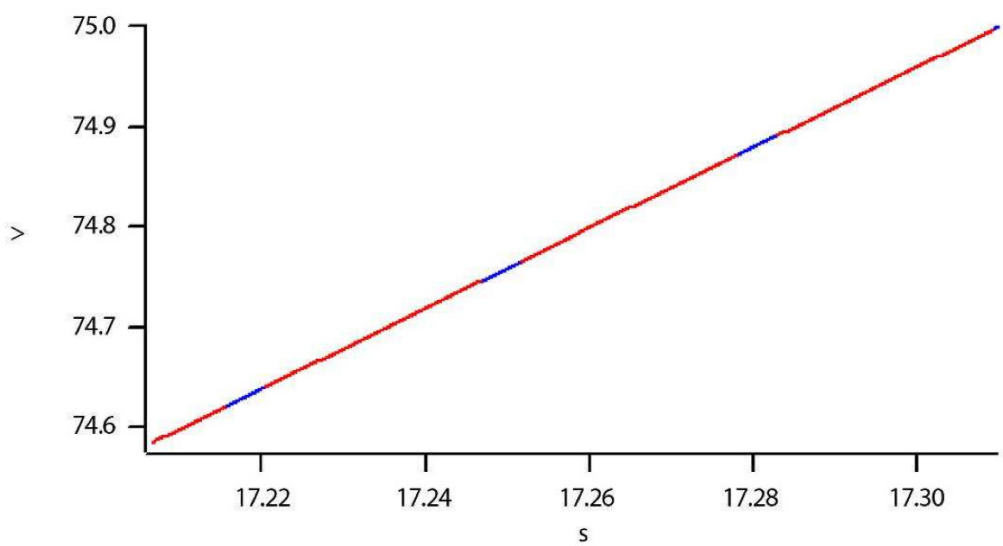


Figure 23.4.: Slow Axis of Raster Scan. Y-axis is Y piezo voltage (scan angle 0°); X- axis is time in image. Axis limits are zoomed in to show the same region as in Figure. The blue sections denote turn-around regions.

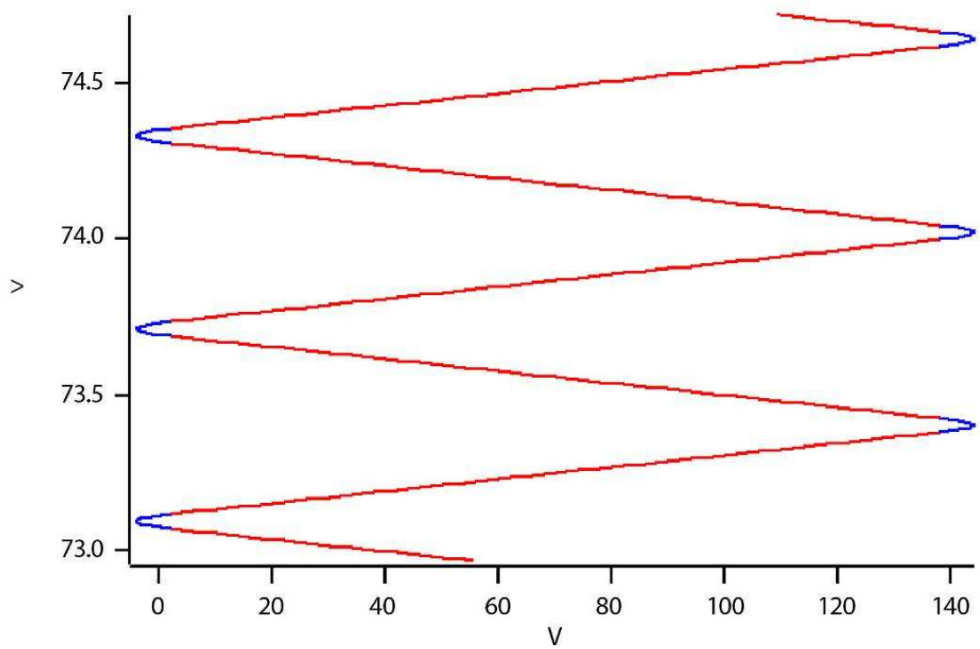


Figure 23.5.: X-Y Motion of the Tip across surface in raster imaging. The X-axis is the fast axis drive, while the Y-axis is the slow axis drive. Less than 3 scan lines are shown to better illustrate the concept. The blue sections denote turn-around regions.

Part V

Bibliography, Glossary, and Index

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