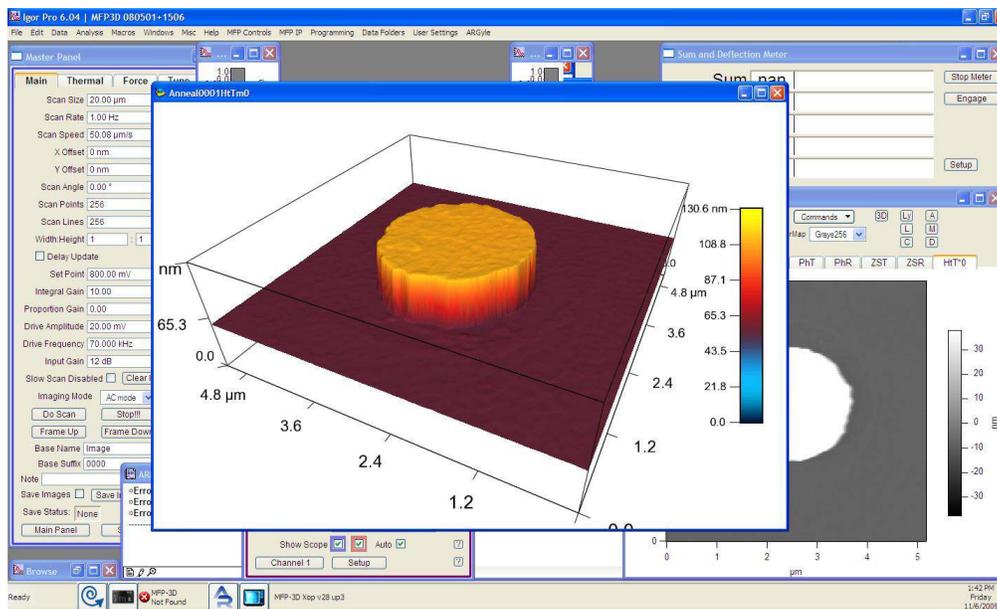


Imaging and Spectroscopy Applications Guide



User Guide

Version 13, Revision: 1578

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Asylum Research
an Oxford Instruments company

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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 *Force Curves and Maps, Spectroscopy, and Lithography*. Step by step instructions for force curves, force volume mapping, lithography, and other Spectroscopies. Quick instructions with a minimum of theory, and when necessary an extra chapter devoted to theory.

Part ?? *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.

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 Happens automatically with software updates, free at <https://support.AsylumResearch.Com>.

Prerequisites

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

**INVISIBLE LASER RADIATION
DO NOT VIEW DIRECTLY WITH
OPTICAL INSTRUMENTS
(MAGNIFIERS)
CLASS 1M LASER PRODUCT
1.0 mW AT 860 nm**

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.

A few words about how to use this part.

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1. Contact Mode Imaging

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1.1. Preparation

1.1.1. Cantilever Selection

See ??for a guide on contact mode 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 AFM, please follow ?, . If you own a Cypher AFM, please follow ?, . This will familiarize you with the basics of loading the cantilever, sample, and such. Once everything is adjusted and the cantilever is “ready to strike” above the sample surface, you can switch over to the sections that follow 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.

LFM

Some of the steps will include instructions for LFM (Lateral Force Microscopy) discussed in detail in Section 1.4 on page 12. Please ignore these steps if you are doing standard Contact Mode imaging.

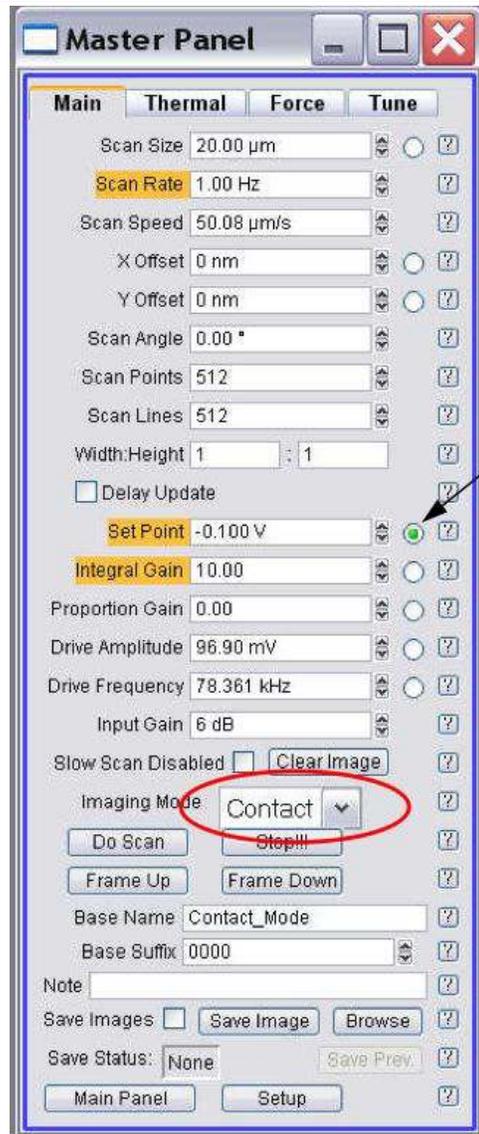
Set up Master Panel:

- In the Main tab of the Master Panel, select *Contact* from the *Imaging Mode* pull-down menu.
 - Setpoint: -0.1 V.
 - Integral gain (I):
 - MFP-3D 8 - 10
 - MFP-3D extended head 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. The figure to the right gives typical values for these fields.

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

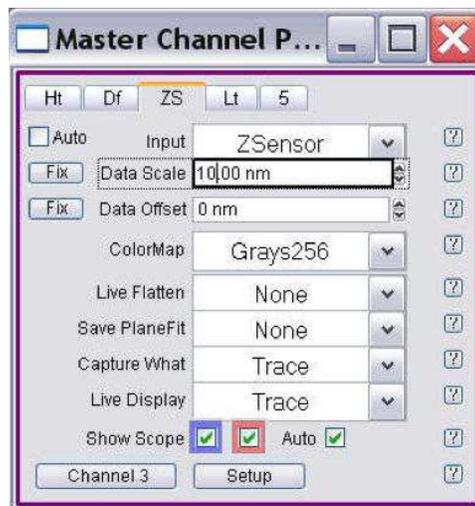
Lateral Force Microscopy Only:

- Scan angle should be at 90° or 270° for lateral force microscopy (LFM). This may be obvious, but some other commercial scopes scan perpendicular to the lever at 0°.



- 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*.

2. **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.



Lateral Force Microscopy Only:

- If doing Lateral Force Microscopy, choose *Lateral* as an additional 4th channel.

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. Better for measuring accurate distances, typically >200 nm.

Lateral: Used to monitor cantilever torsion (twisting) when scanning at 90° by comparing the amount of signal in the left and right halves of the 4 quadrant photodiode.

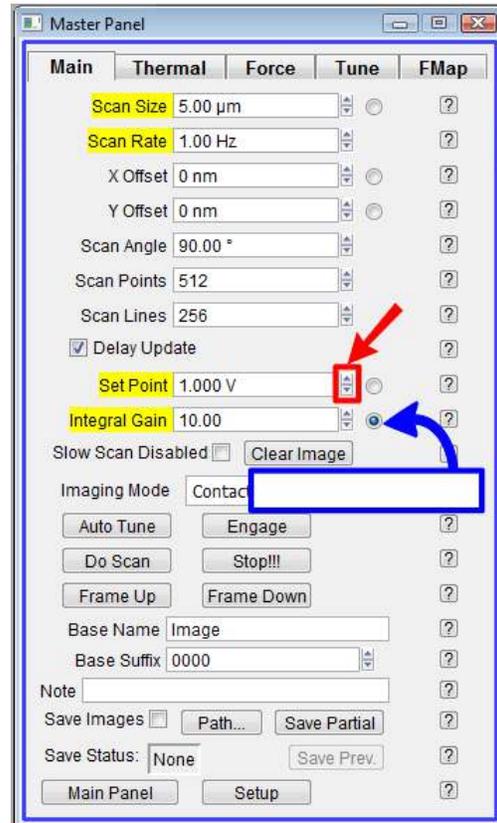
It is useful, though not essential, to activate the Z sensor channel when imaging, especially when sample features are larger than a few hundred nanometers; the LVDT sensors are more linear than the piezos, and thus give a more precise Z measurement.

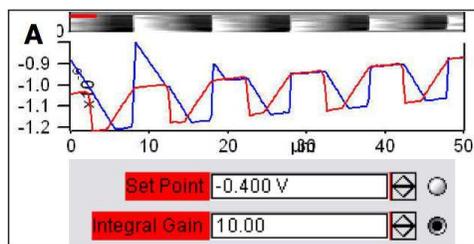
Mode Master: Most of what was described in this section is automatically by the Mode Master when *Contact Mode* is selected from the standard modes.

1.3. Optimize Imaging Parameters

1. Click the 'Do Scan' button on the Main 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:

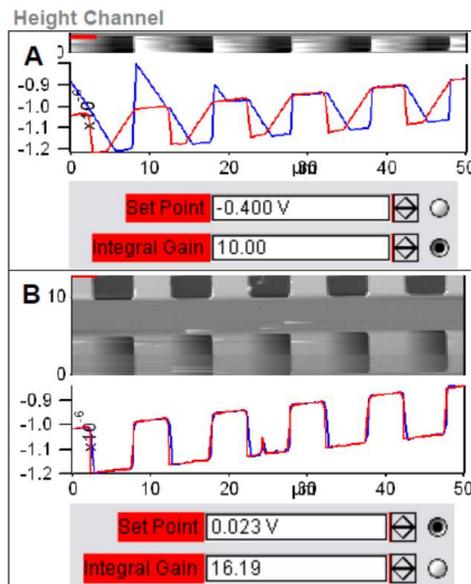
- Image quality can be monitored by image resolution and by the amount of noise in the line traces (located below images).
- Start the learning process on a sample with a known topography, like an Asylum Research Calibration Grating (basically a matrix of square pits). Then you know immediately if the image looks sub-optimal.
- 3. 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**.

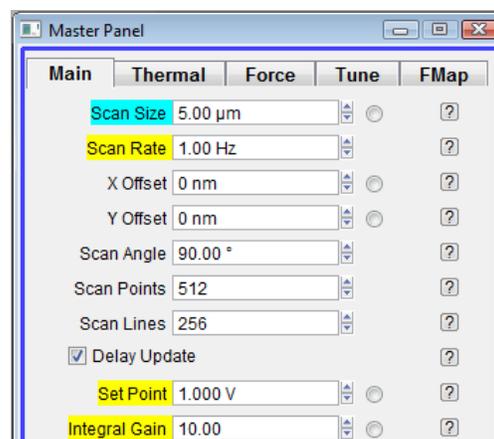
- The Setpoint voltage and Integral gain should first be adjusted to achieve good tracking. The picture to the right shows an example of imaging a calibration grid, with corresponding parameters from the Main tab below.
- 4.
- Panel A) shows the initial tracking of the tip upon engagement with a Setpoint 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 light blue 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).



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 1) 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 blue.

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.

1.4. Lateral Force Microscopy

Lateral Force Microscopy (LFM) can easily be performed on the MFP-3D and Cypher systems. It is a close cousin of contact mode microscopy. One requirement is to run the fast scanning direction orthogonal to the length of the cantilever. Contact mode AFM is normally performed by scanning the tip (or sample) back and forth in the manner of sawing a piece of wood. The lever (saw) moves along its own length. For LFM it moves more like a blind man's cane, perpendicular to its own length.

LFM performs contact mode AFM as described earlier but monitors an extra piece of information: how much the lever twists from the friction experienced by the tip. This signal is measured by the same quadrant photodetector which measures the cantilever deflection, except that different quadrants are subtracted to measure the lateral deflection or twist of the lever.

1.4.1. Zeroing the lateral signal

To best measure the lateral signal (twisting) of the cantilever, the laser spot should be centered on the photodetector. Please consult the manual of your AFM on how to do this. At this writing, the three options are:

MFP-3D AFM with Standard or Extended Z head

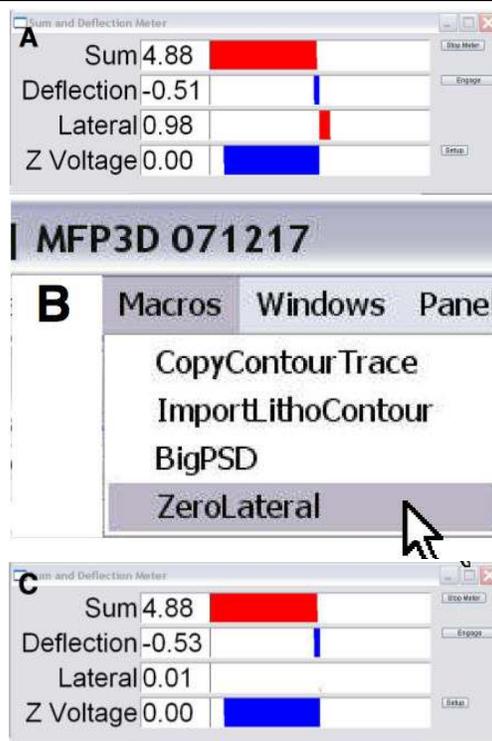
A standard MFP-3D head does not have a mechanical means to zero the lateral deflection on the PhotoDiode, for reasons not to be discussed herein. There is an electronic way to zero the deflection- it is an Igor procedure file (.ipf) called LFM Rocks. Please contact support@AsylumResearch.com for this file.

1. Load the LFM Rocks .ipf- Go to File>Open File>Procedure; load LFM rocks from wherever it is 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 menu bar, as seen in the middle photo.
 - Image C shows the result: electronic zeroing of the lateral signal.



MFP-3D AFM with LFM Head Has special adjustments to mechanically center the laser spot on the detector.

Cypher AFM 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

1.4.2. Example of LFM Imaging

Figure 1.1 shows 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 1.1a shows nearly imperceptible evidence of the two molecular species. Since only their termination differs, they are the nearly the same length. The lateral image in Figure 1.1b, however, shows good contrast, indicating a difference in tip-sample frictional forces between the two species.

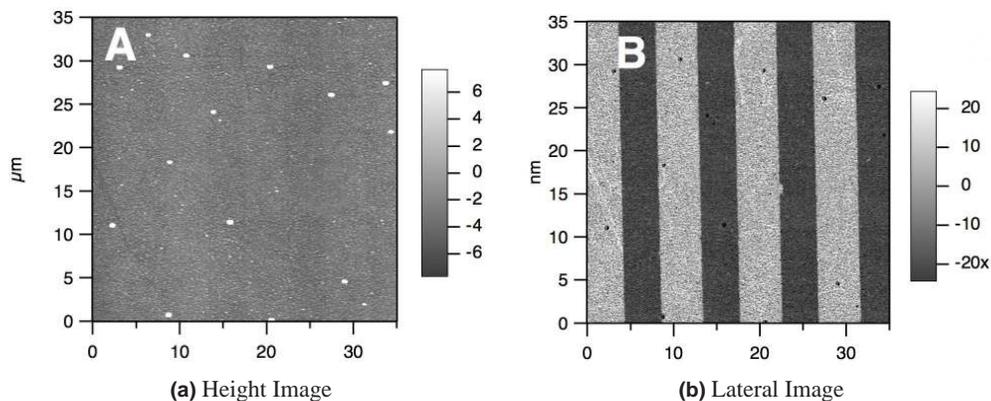


Figure 1.1.: Micro-contact printed alkanethiols on polycrystalline gold. The bright areas of the LFM images are terminated with a carboxylic acid, while the dark areas are a methyl terminus.

1.4.3. ARUP (User Panel) LFM Friction Loop

The Friction loop user panel can be used to calibrate the lateral sensitivity (Lateral InVOLs). This panel can be loaded from the programming > User Panels menu, then double clicking Friction loops, or will be automatically opened when Lateral is selected from Mode Master (Standard imaging profiles).

The friction loop user panel will ramp the Y axis of the scanner to move perpendicular to the axis of the lever, while the tip is engaged on the surface. It will record the lateral signal as a function of Y sensor position. The software will attempt to fit the turn around region to a straight line to obtain the lateral InVOLs value. This can be problematic if the data is not very clear, if there is a jagged turn around, it can come up with some very incorrect 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 trusting it.

2. Contact Mode Imaging: Scientific Background and Theory

CHAPTER REV. 1543, DATED 08/23/2013, 10:10.

USER GUIDE REV. 1578, DATED 08/29/2013, 22:20.

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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 [Chapter 3 on page 18](#)), 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 2.1 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 14 on page 138](#) 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. tip-substrate distance is shown in [Section 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.

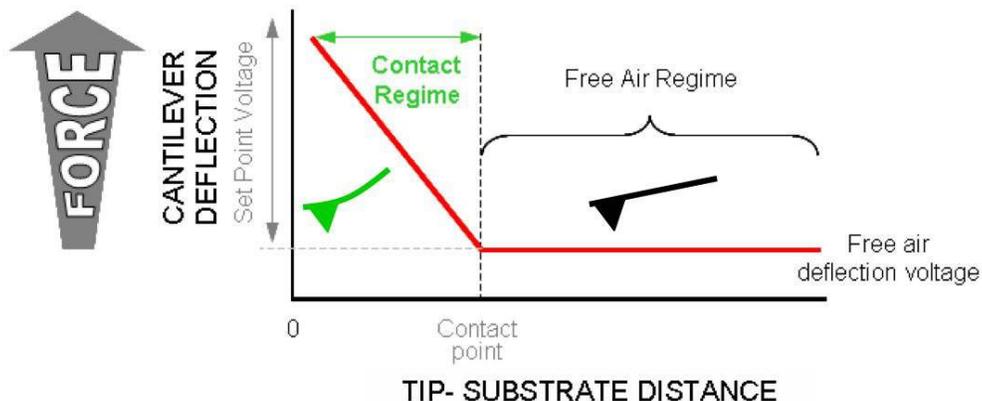


Figure 2.1.: Qualitative depiction of Cantilever Deflection vs. Tip Substrate separation.

The Z feedback loop is what controls the Z actuator. It tries to maintain the specified setpoint voltage (see Figure 2.1 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 full retracted, yet the cantilever is still so bent against the sample the deflection remains higher than the setpoint.

While the Z feedback keeps the deflection constant, the scanner is rastered in X and Y (See ?? on page ??). 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 linear 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 negligible compared to the range itself. If your sample features are around 1 μ m or greater it is good idea to use *Z sensor* rather than *Height*.

If the Z feedback worked perfectly the deflection would never change. In reality the Z feedback is close to maintaining a constant deflection but not perfect. Sudden changes in the sample topography cause the Z feedback to either under or 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.

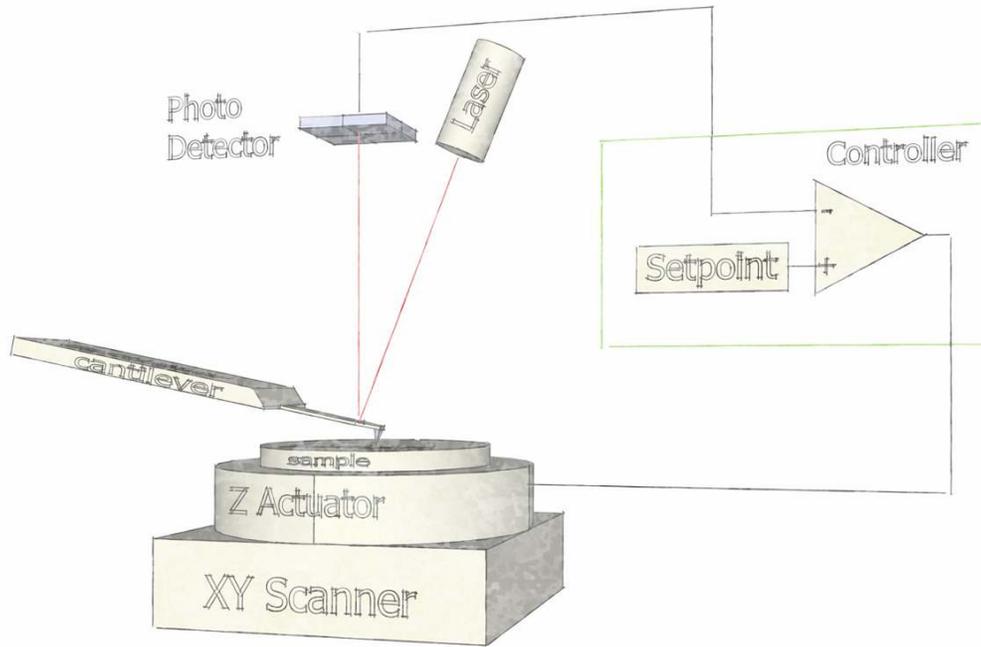


Figure 2.2.: Contact Mode AFM. Z actuator feeds back to keep cantilever deflection at a given setpoint.

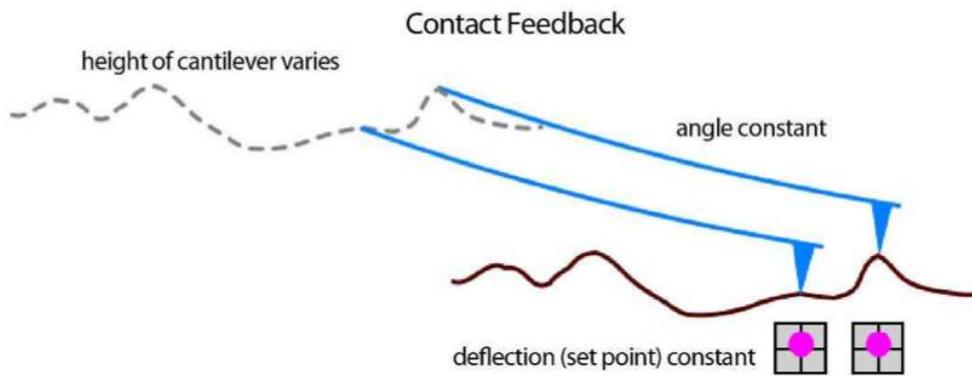


Figure 2.3.: Contact Mode tracking of the surface. Feedback keeps the cantilever deflection constant.

3. AC Mode Imaging in Air

CHAPTER REV. 1578, DATED 08/29/2013, 22:20.

USER GUIDE REV. 1578, DATED 08/29/2013, 22:20.

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3.1. Introduction

This section assumes you are already capable of starting the imaging process. For the MFP-3D AFM. If this is new to you then:

- For the MFP-3D follow this tutorial:¹
- For the Cypher AFM, follow this tutorial:²

The following sections assume the cantilever is “ready to strike” 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 cover that as necessary.

3.2. 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

¹?, .
²?, .

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 *Main 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.

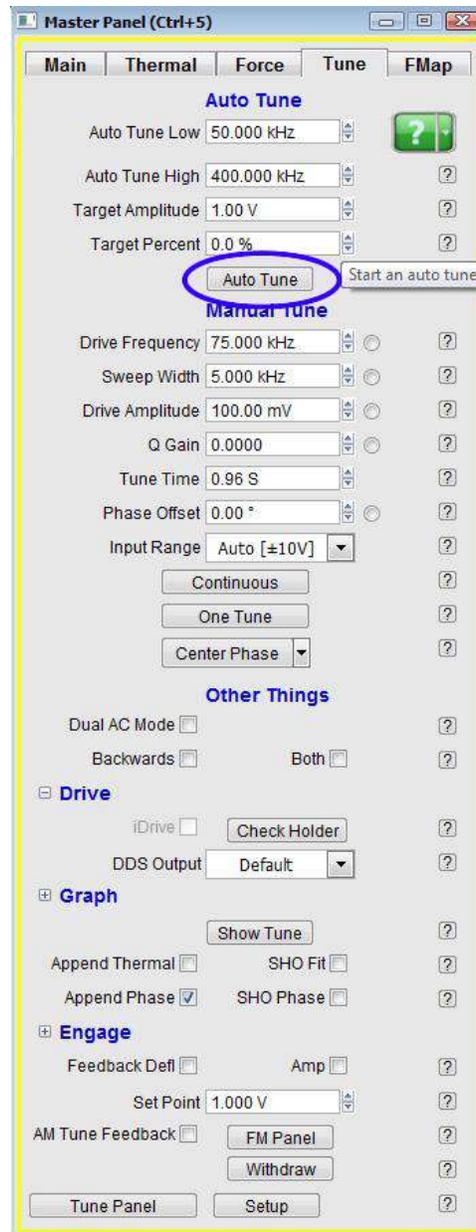


Figure 3.1.: Tune tab

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 4.3 on page 28 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 3.2 on page 20, will appear. The first tune will most likely appear similar to Figure 3.2a on page 20 which then updates to one similar to Figure 3.2b on page 20. 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.

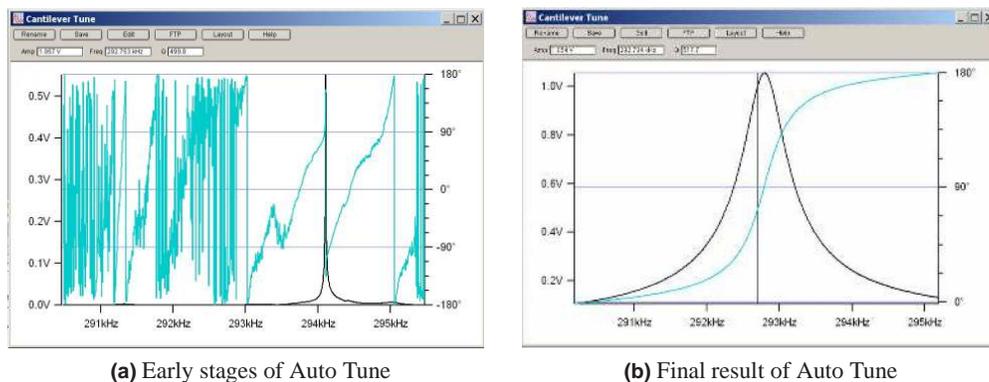


Figure 3.2.: Auto Tune of an Olympus AC160 Si ($f = 300\text{kHz}$; $k = 40\text{N/m}$; nominal values) cantilever in air.

3.2.1. Auto Tune Troubleshooting

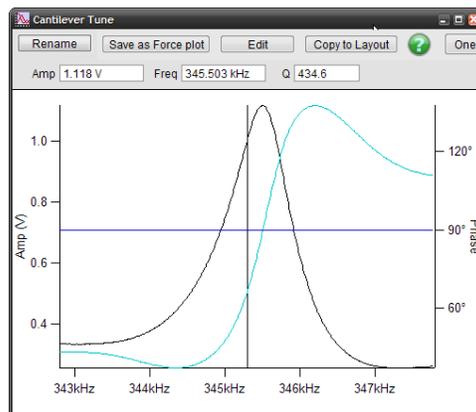
If you see a message about the drive amplitude being too low, see Step 11 on page 123.

If you do not know the resonant frequency of your cantilever, see Chapter 15 on page 148.

3.3. 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.



3.4. Optimizing Imaging Parameters

Please refer to the ARMFP3DUserGuide.pdf : Tutorial:AC Mode Imaging In air, the section on Start imaging. 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 covering 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.

3.5. Attractive and 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, and it is rougher on the tip and sample.

Attractive AC mode is where you track the surface on the small increase in amplitude as the tip is approaching the surface. This is much more difficult to do, can not be done with all levers and samples. But it is much more gentle on the samples, and is sometimes required for soft biological materials.

See also 4.3.2 for a good summary of attractive and repulsive modes.

3.5.1. 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 $< 90^\circ$.

3.5.1.1. 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.

3.5.1.2.

3.5.2. 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 $> 90^\circ$.

3.5.2.1. Chose the Right Probe

Softer longer levers with relatively low resonance frequencies are a good choice. We always have good luck with the [AC240](#). Also see [Asylum Research Probe Store](#).

3.5.2.2. Gently Drive the Probe Above Resonance

Smaller amplitudes and 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.

3.5.2.3. Try Q control with positive Qgain

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

Figure 3.3 on page 23 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 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 .

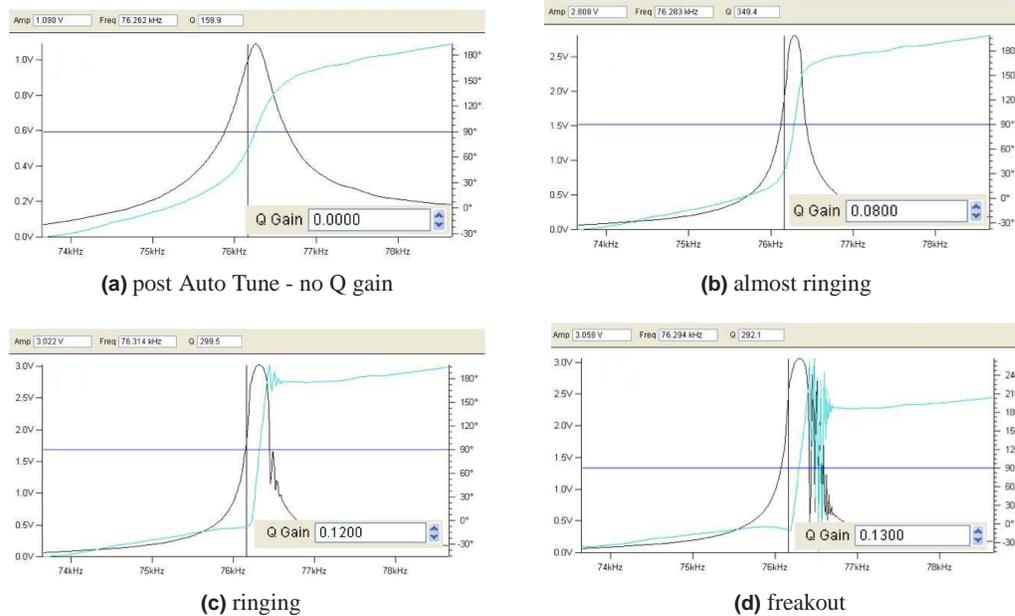


Figure 3.3.

3.5.3. 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 3.4 on page 24 show red regions where phase is much smaller than 90° (3.5.1) and blue where phase is much larger than 90° (attractive behavior). Every 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 get to, you decrease your drive frequency, increase your drive amplitude, maybe decrease your setpoint.

If you are mode hopping and you want to go to attractive mode, you want smaller amplitudes drop 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.

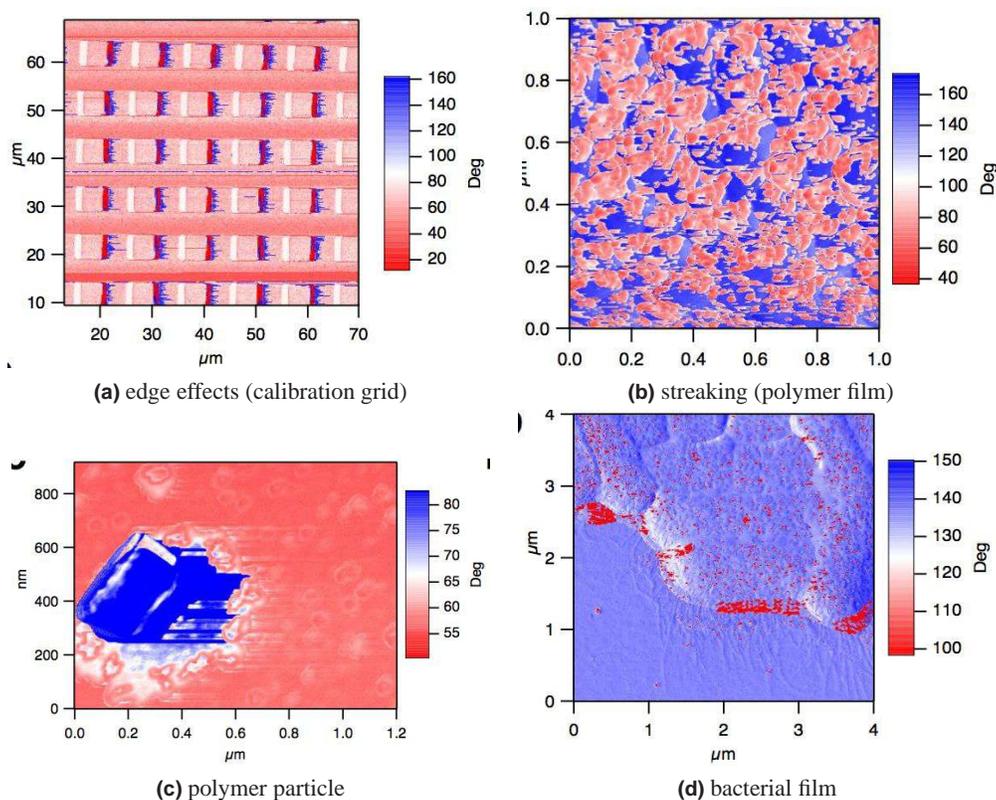


Figure 3.4.: Examples of mode hopping in the Phase Channel

3.5.3.1. Make the Sample less Attractive

If you are still having trouble with mode hopping, 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 3.5). 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. Keith Jones (AR apps scientist) has found that a Pt coated Si cantilever can sometimes work well with sticky samples. We use **Electrilevers** (Olympus AC 240s coated with Pt).

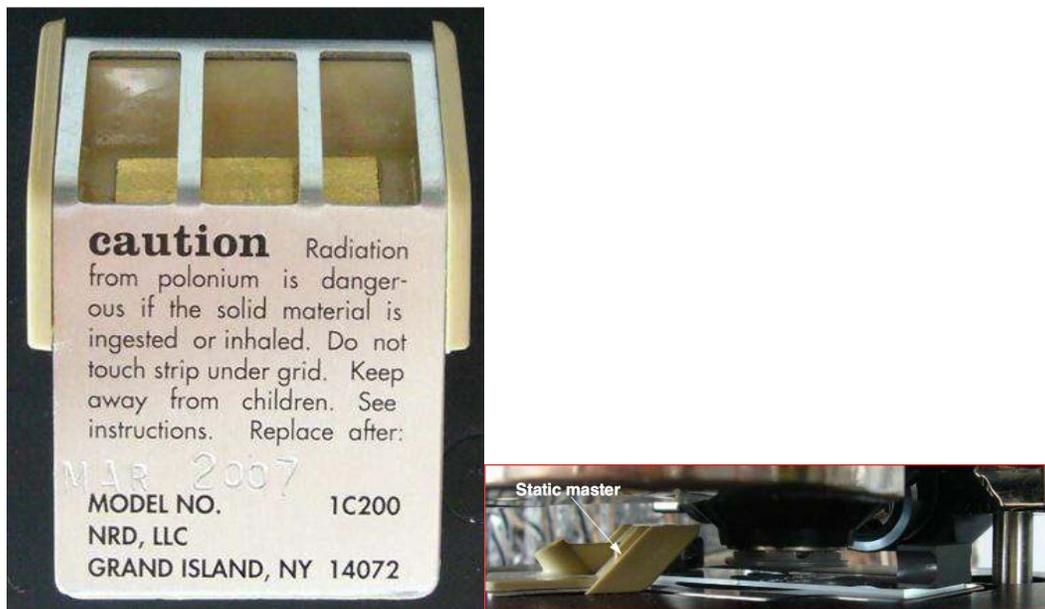


Figure 3.5.: Placing static master near sample to dissipate charge.

4. AC Mode Imaging: Scientific Background and Theory

CHAPTER REV. 1578, DATED 08/29/2013, 22:20.

USER GUIDE REV. 1578, DATED 08/29/2013, 22:20.

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4.1. 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.

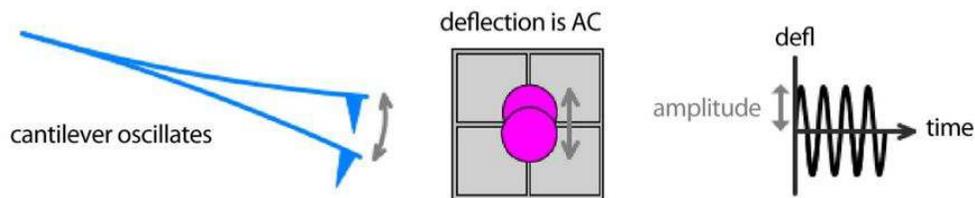


Figure 4.1.: An oscillating cantilever leads to an oscillating signal on the photodetector. Pause to consider the absolute magnitude of these oscillations. Picture a $12\ \mu\text{m}$ tall cone. The absolute motion is twice the amplitude (120nm) or only 1% of the entire conical tip structure. Clearly 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.

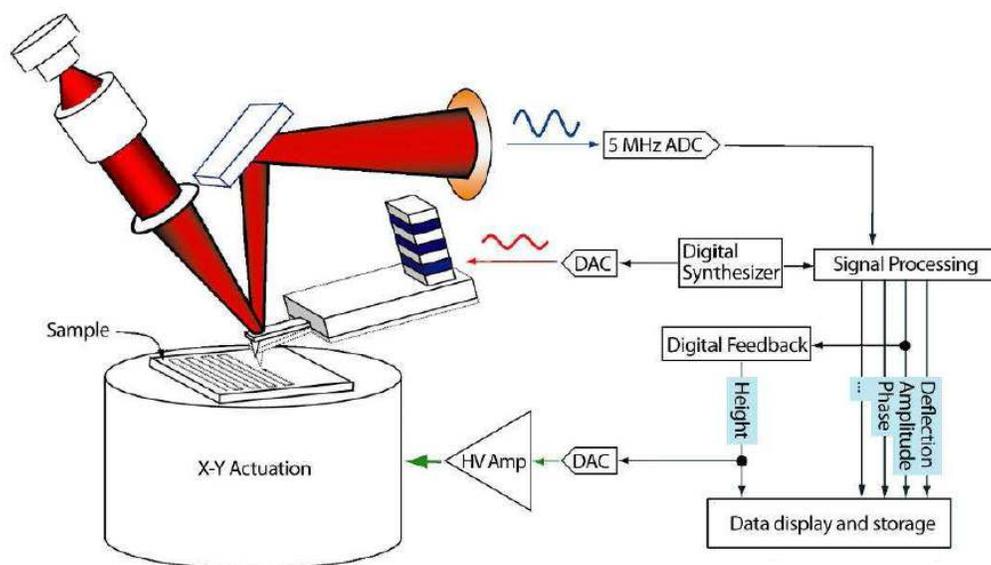


Figure 4.2.: Basic diagram of AC mode imaging.

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 4.3. 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 to 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 4.3 on page 28](#). 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 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 ex-

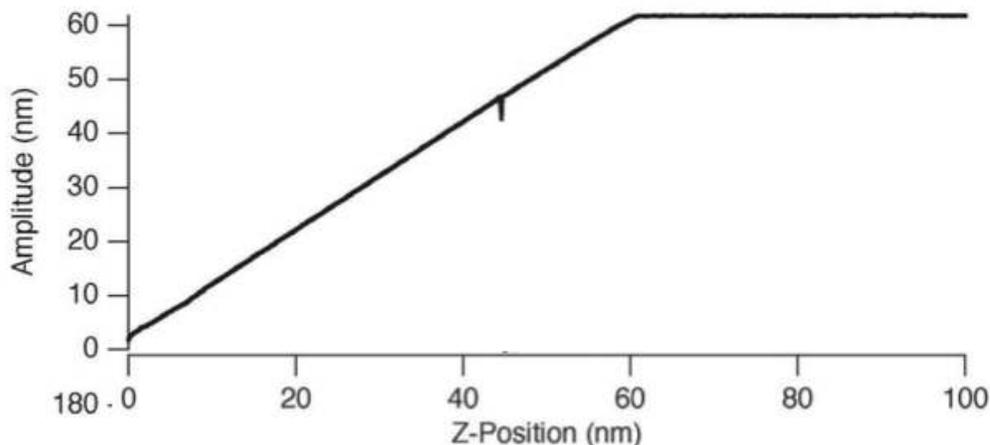


Figure 4.3.: Tip oscillation amplitude as it approaches the surface. The small glitch in amplitude is explained later, see 4.4.

ample, 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.

4.2. 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 explains a lot 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 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 or 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.

4.3. Attractive and Repulsive Behavior

Lets revisit Figure 4.3 on page 28, and this time we’ll show the phase signal as well.

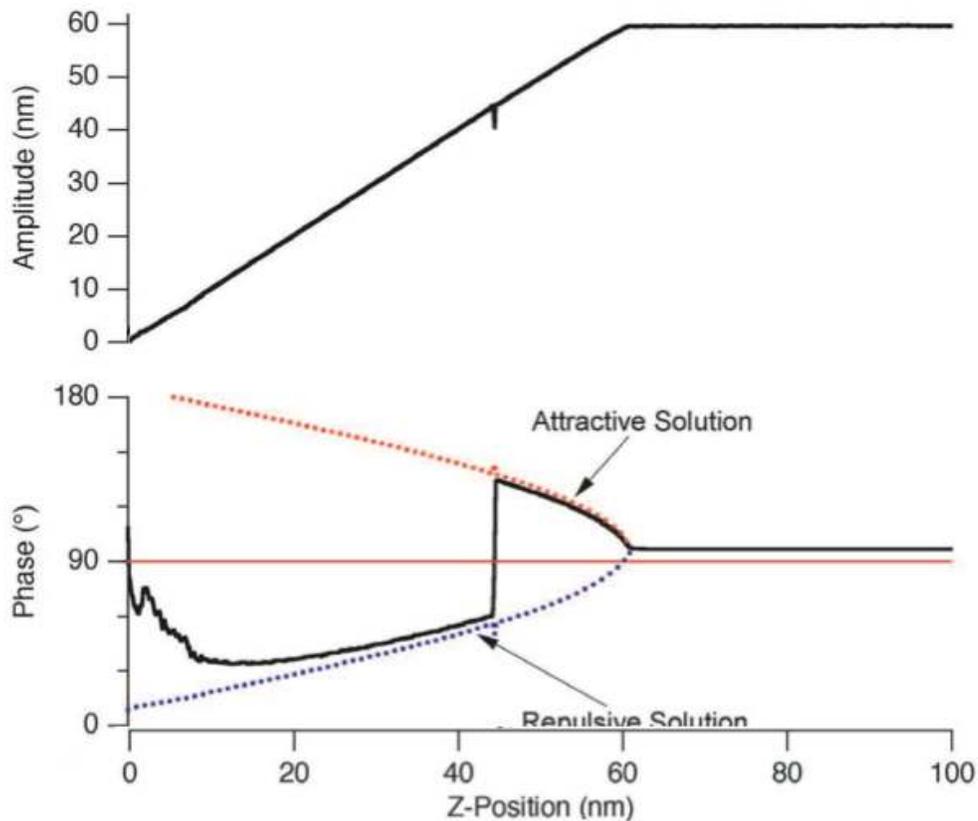


Figure 4.4.: Cantilever amplitude and phase as a function of average tip-sample separation.

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 $> 90^\circ$. 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 frequencies, meaning the drive frequency is below resonance, and you see a phase $< 90^\circ$. This shift in resonance frequency is further illustrated in 4.6.

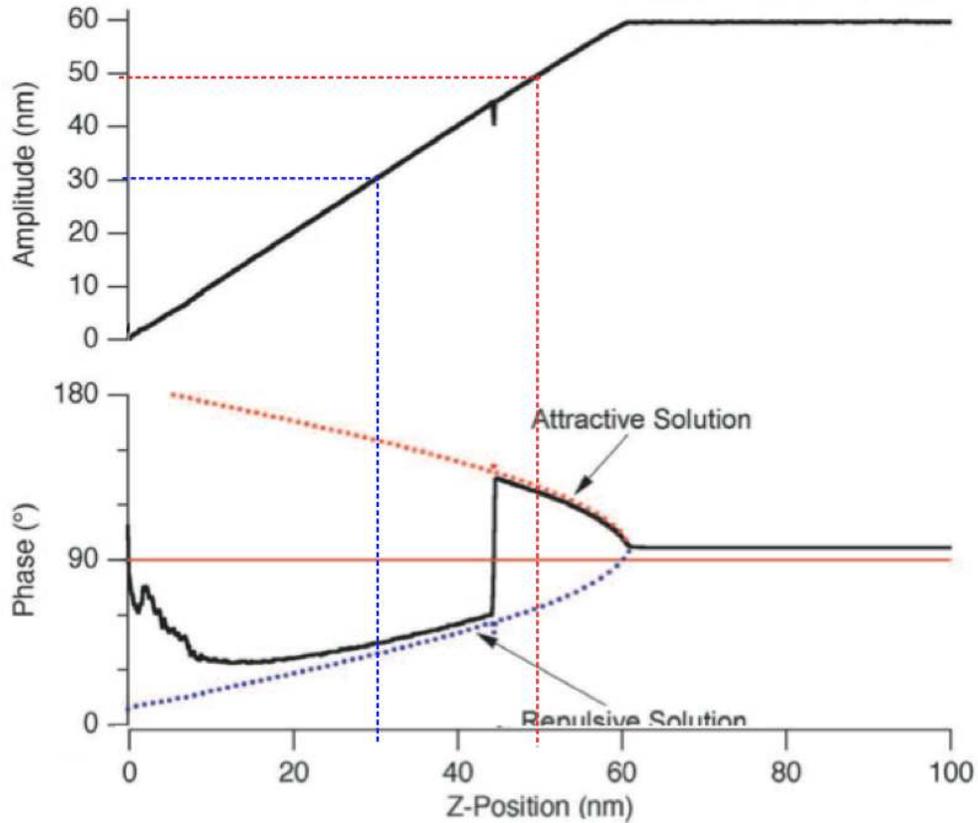


Figure 4.5.: AC mode feedback setpoint of 50nm (red dashed lines) gives attractive mode imaging, a setpoint of 30nm (blue dashed lines) gives repulsive mode imaging.

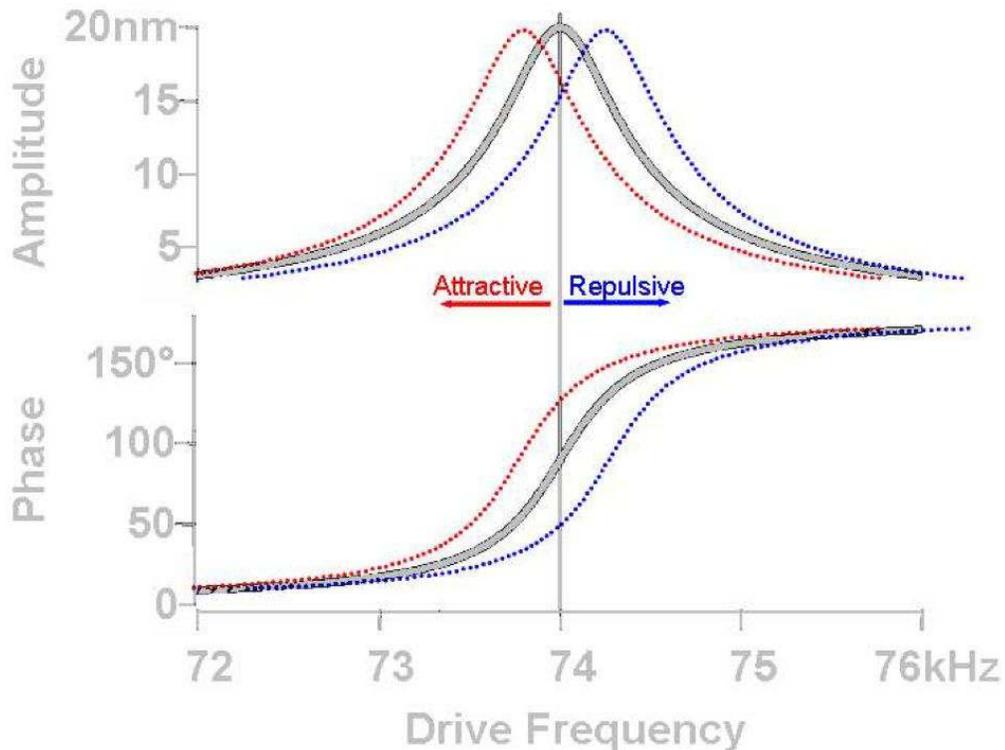


Figure 4.6: A shift in resonance frequency, f_o (at constant amplitude) as the tip interacts with surface results in a change in the phase of the AC signal: Decrease in f_o results in phase increase (attractive mode) while increase in f_o results in phase decrease (repulsive mode). The colors represent the color of the phase on the Sum and Deflection Meter Panel.

Notice from Figure 4.6 on page 30 that if you choose a negative Target Percent amplitude during an Auto Tune (Section 3.2 on page 18), it helps keep the tip in repulsive mode during AC Mode imaging (black vertical line crosses blue dotted line in Section 3.5.1 on page 22). Likewise, positive Target percent amplitudes help the tip stay in attractive mode (black vertical line crosses red dotted line; see Section 3.5.2 on page 22).

For more on this topic, see references^{1,2,3,4}.

4.3.1. Selecting Attractive vs. Repulsive

In practice it can be quite difficult to select attractive mode from repulsive mode imaging. For the specific case of Figure 4.5 on page 30 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.

Take a look at Figure 4.7 on page 32. 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 $> 90^\circ$) for a very small range of setpoints very close to that free amplitude and then stays repulsive (phase $< 90^\circ$) for lower setpoints.

The green, blue, purple, and pink curves are successively for 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 rule of thumb for imaging in purely attractive mode, purely repulsive mode, or something prone to switching between the two.

4.3.2. 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 4.1.: Attractive/Repulsive AC mode imaging rules of thumb

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

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

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

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

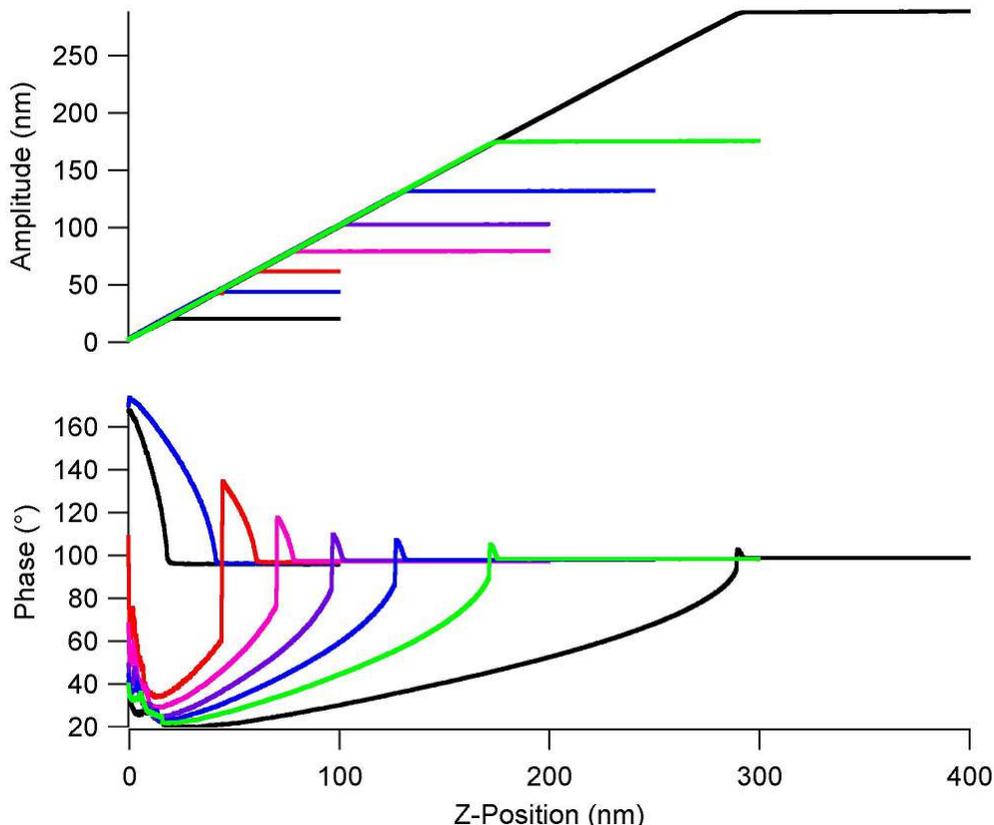


Figure 4.7.: A family of AC force curves for different free amplitudes.

4.3.2.1. 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 4.7 on page 32 show for that cantilever and sample bigger means > 150 nm and smaller means < 50 nm.

4.3.2.2. Cantilever Choice

Stiffer and softer are relative terms, but for the sake of simplicity we will consider the 2 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.

4.3.2.3. 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.

4.3.2.4. 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 relatively 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 trickery in the SPM controller it allows you to increase or decrease the Q. You can read more in 3.5.2.3 .

4.3.2.5. 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 3.5.3.1 on page 24](#) for ways to combat this issue.

4.3.2.6. 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.

5. Single Frequency Piezo Force Microscopy (PFM)

CHAPTER REV. 1514, DATED 08/16/2013, 16:27.

USER GUIDE REV. 1578, DATED 08/29/2013, 22:20.

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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 5.1 on page 35 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.

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 6.

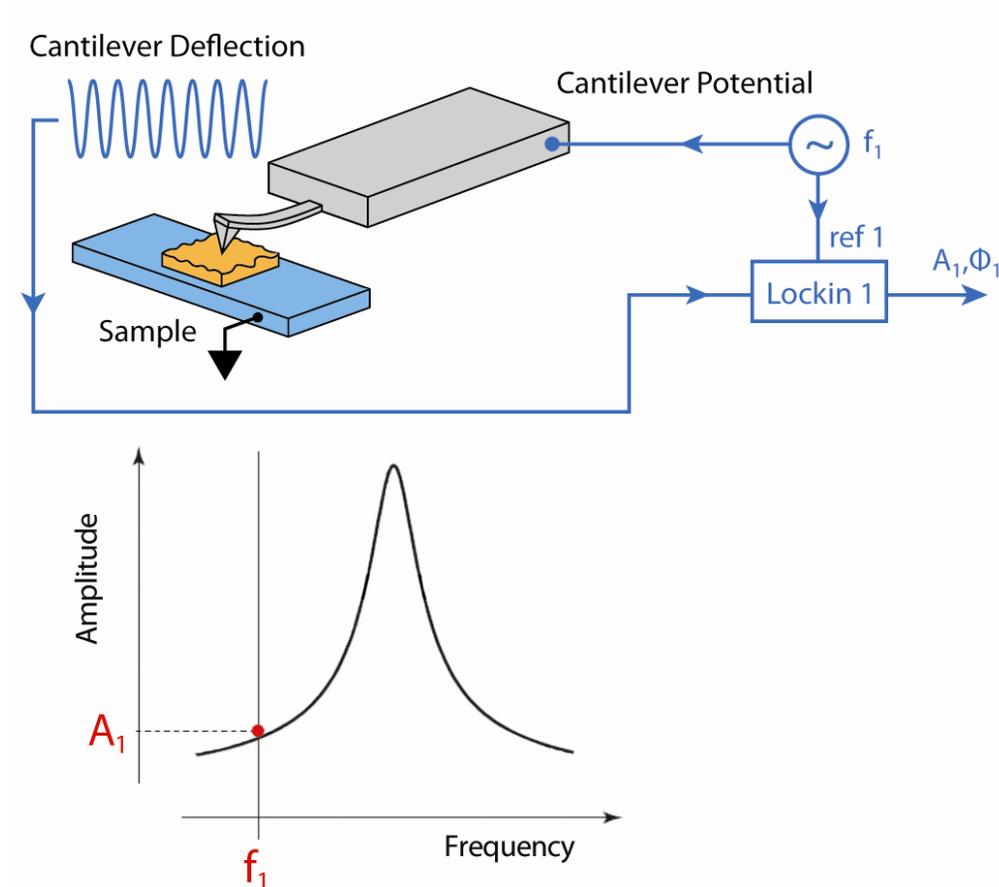


Figure 5.1.: A diagram of Single Frequency PFM

5.1. 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	800-1,200 kHz

Table 5.1.: Typical contact resonance frequency ranges for some common cantilevers

5.2. Single Frequency PFM 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 a high voltage option for your microscope.

5.2.1. 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.

5.2.2. Software Instructions

This assumes you have already approached the tip to about 20 to 50 μm above the surface.

1. To run Single Frequency PFM, open a new AFM template by starting the software from scratch, or by clicking *File* \triangleright *New AFM Template*. The Mode Master window should pop up. If it does not, click [ctrl+F2], or navigate to *User Settings* \triangleright *Mode Master*. Click *PFM*, then click *SingleFreqPFM*.
2. 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.
3. 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).
4. 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.

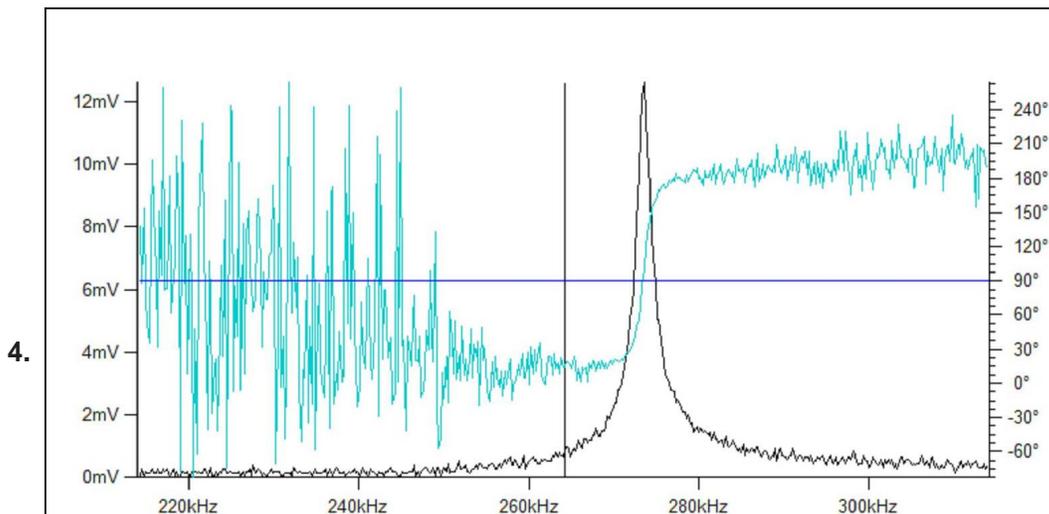
- 5. Position the head above the sample**
- Check that the red wire is seated into the HV connector slot (glowing red on the sample holder. The magnet should suck the end of this wire into the HV connection slot when the head is positioned above the sample.
 - MFP3D only: Take care to ensure that the 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.

Engage the Surface

- 6.
- 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.

5.2.3. Tuning

1. Once you are engaged, it is time to tune. Table 5.1 on page 35 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.

**Center Phase**

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

5.2.4. Imaging

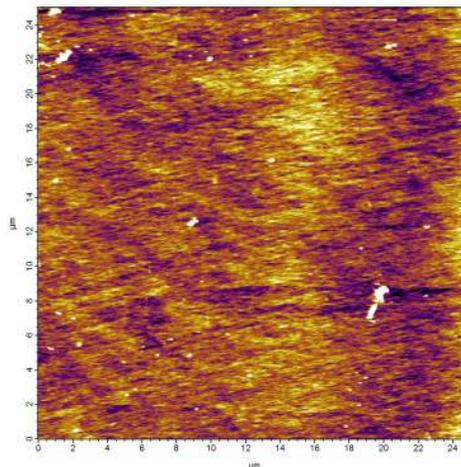
At this point, you're ready to image. Since PPLN the domains are typically many microns across, it can be advantageous to scan a larger size. Also note – you will have more reproducible results if you scan at 90° .

1. Hit *Do Scan* to start imaging.

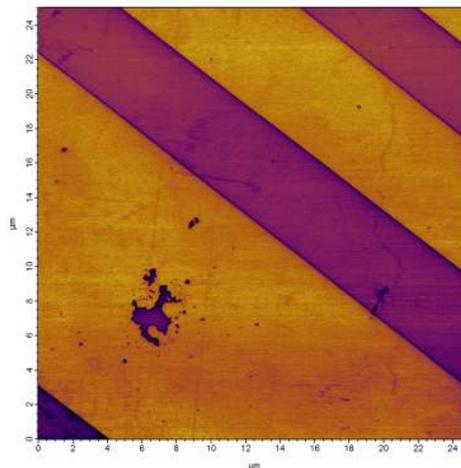
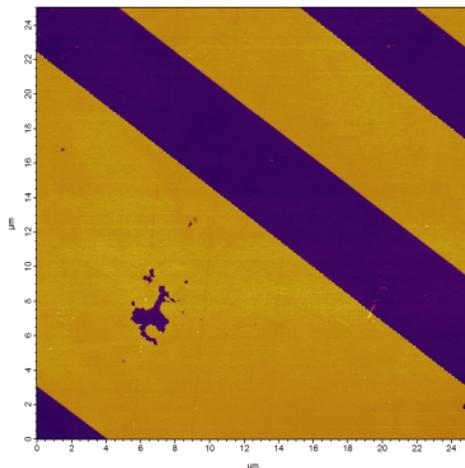
Inspecting data

- If all is well at this point, you should see some 4-10 μm 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.

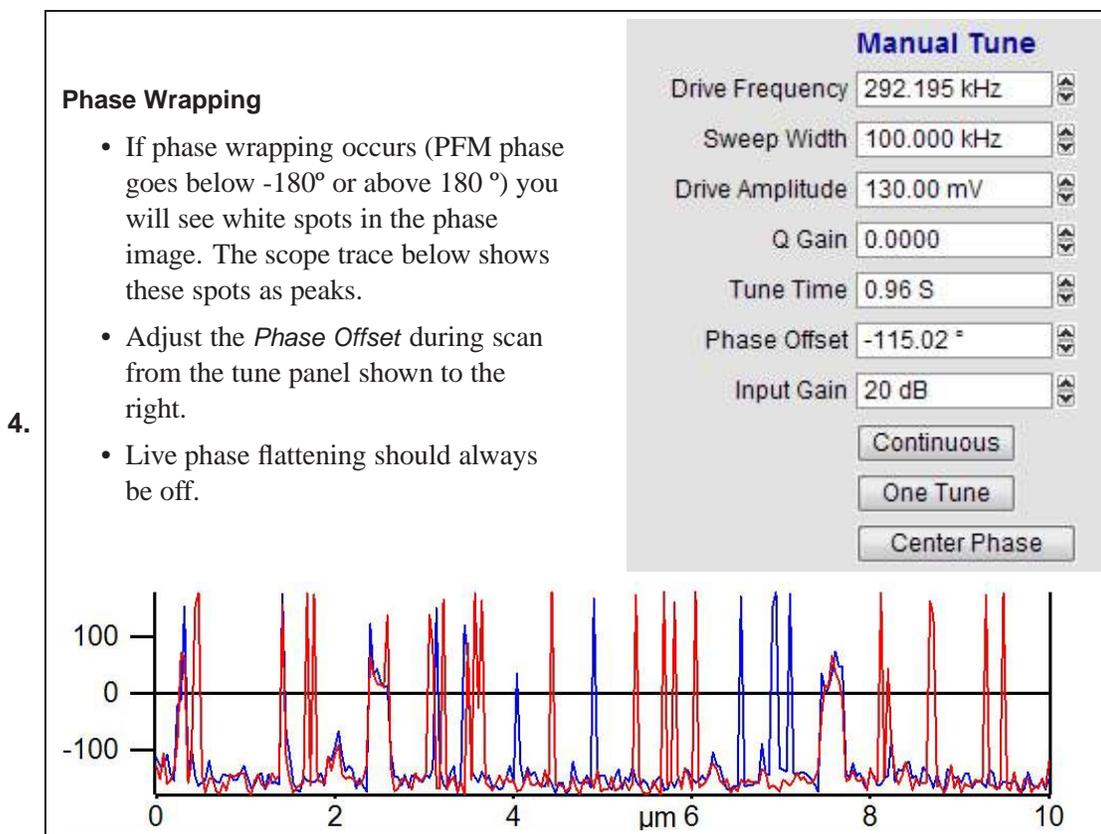
Clockwise, starting from the right, Topography, Amplitude, and Phase.



- 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.



5.3. PFM Lithography with ModeMaster

PFM can 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 6.

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.

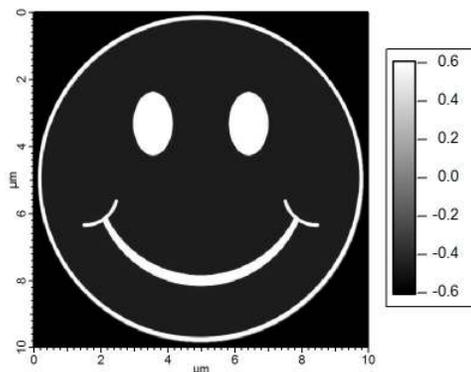
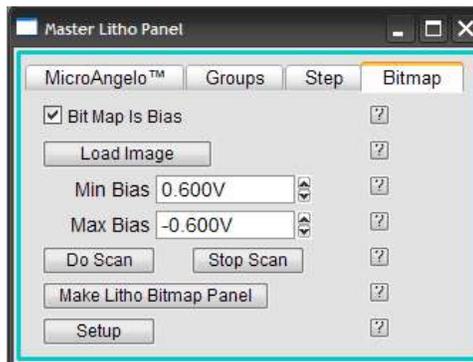
Software Instructions

1. To run LithoPFM, click [ctrl+F2], or navigate to *User Settings* > *Mode Master* Click *PFM*, then click *LithoPFM*.

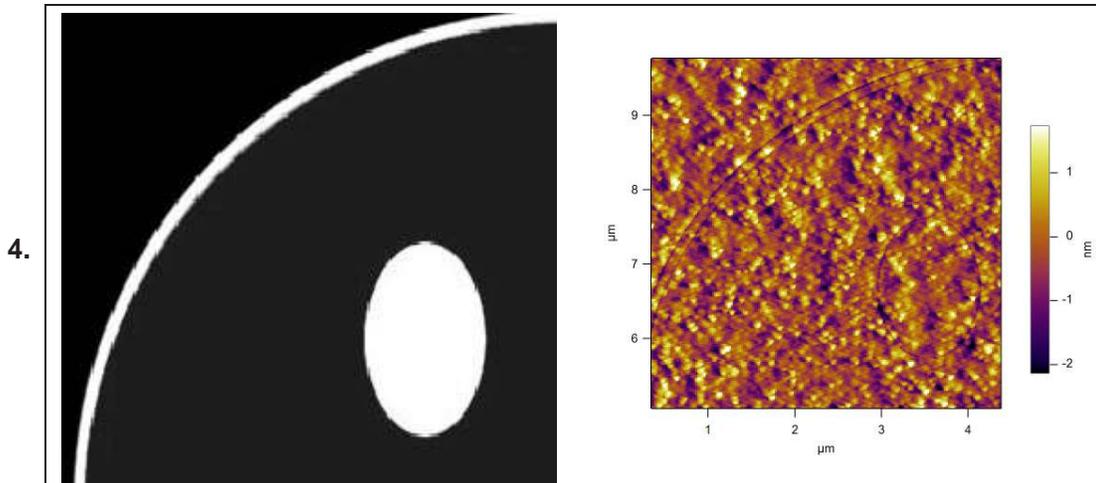
Loading Bitmap Image

- 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>.

2.

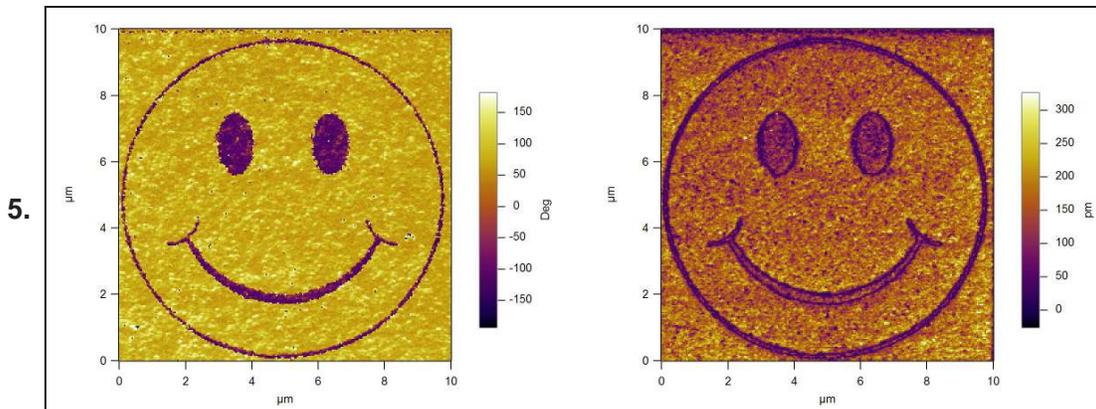


3. Click *Do Scan* to start the bitmap bias lithography.



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



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

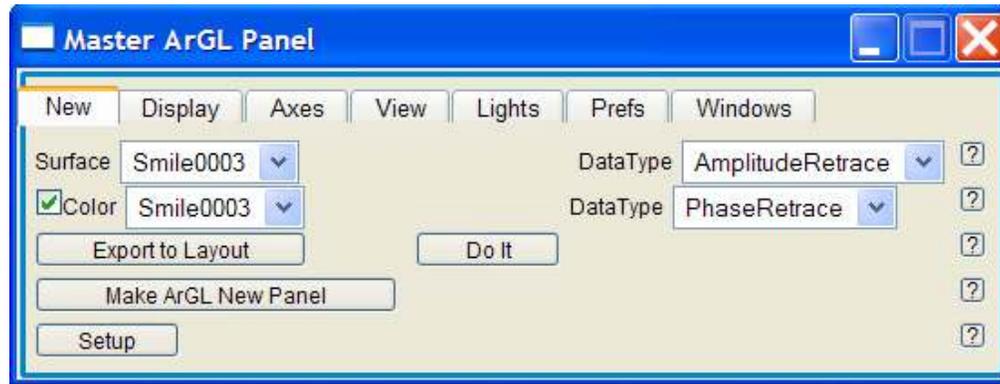
3D Overlay

- 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.

6. See also AR Software Manual:Argyle
3D:Overlaying Data on a Surface.

**5.4. Vector PFM**

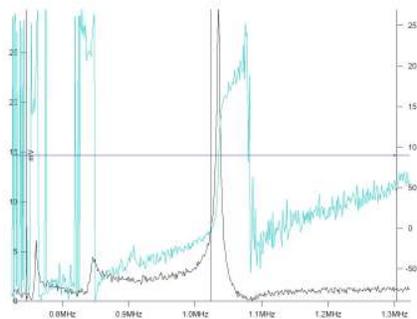
Lateral PFM (Vector PFM) is a two pass technique on an MFP3D and a single pass technique on Cypher. For the MFP3D, it first measures the vertical PFM signal in one pass and then the lateral PFM signal in the second pass. The enhanced LFM head option is required to mechanically center the lateral signal to take advantage of higher gains. The sample used in this example is BiFeO₃/SrRuO₃/SrTiO₃.

1. To run VectorPFM, click [ctrl+F2], or navigate to *User Settings* > *Mode Master*. Click *PFM*, then click *VectorPFM*.

2.

Lateral Tuning MFP3D

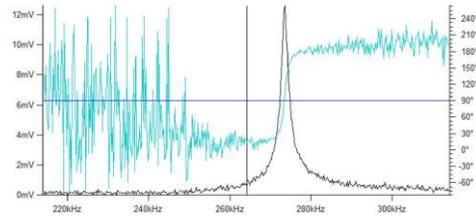
- To tune for the lateral signal, the cross point switch must be manually changed to *InFast* ▸ *Lateral*, click *Write Crosspoint* and select *No Auto Change Crosspoint*
- Engage and tune as normal, the resonance frequency is usually around 1MHz.
- Manually copy the *Drive Frequency*, *Drive Voltage* 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.



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.

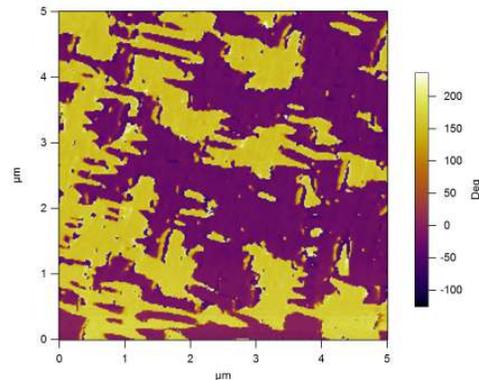
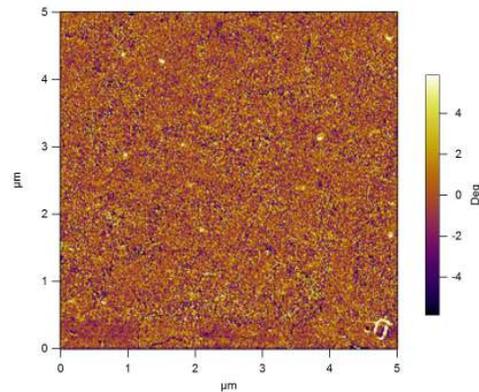
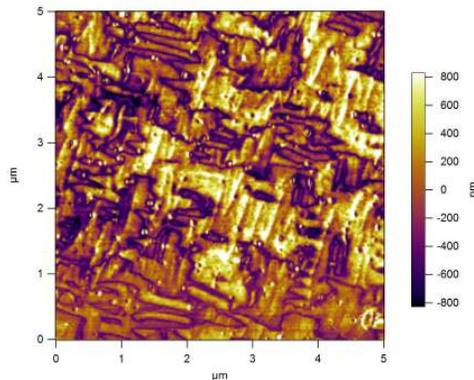


4.

Scan

- Hit *Do Scan* to start imaging.

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



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

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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 Laboratory². This technique is sensitive to ion transport into and out of the lattice in energy storage (battery) materials such as LiCoO₂^{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

¹?,.
²?,.
³?,.
⁴?,.
⁵?,.

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 crosstalk⁶.

In the following pages we will go through:

1. A basic introduction to the Dual AC Resonance Tracking (DART) concept Page (46)
2. Choosing cantilevers and imaging with DART software Page (46)
3. DART Hysteresis loop measurement (aka Switching Spectroscopy PFM) Page (54)

6.1. 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 frequency 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 6.1 on page 47](#) 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.

6.2. 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.

⁶?

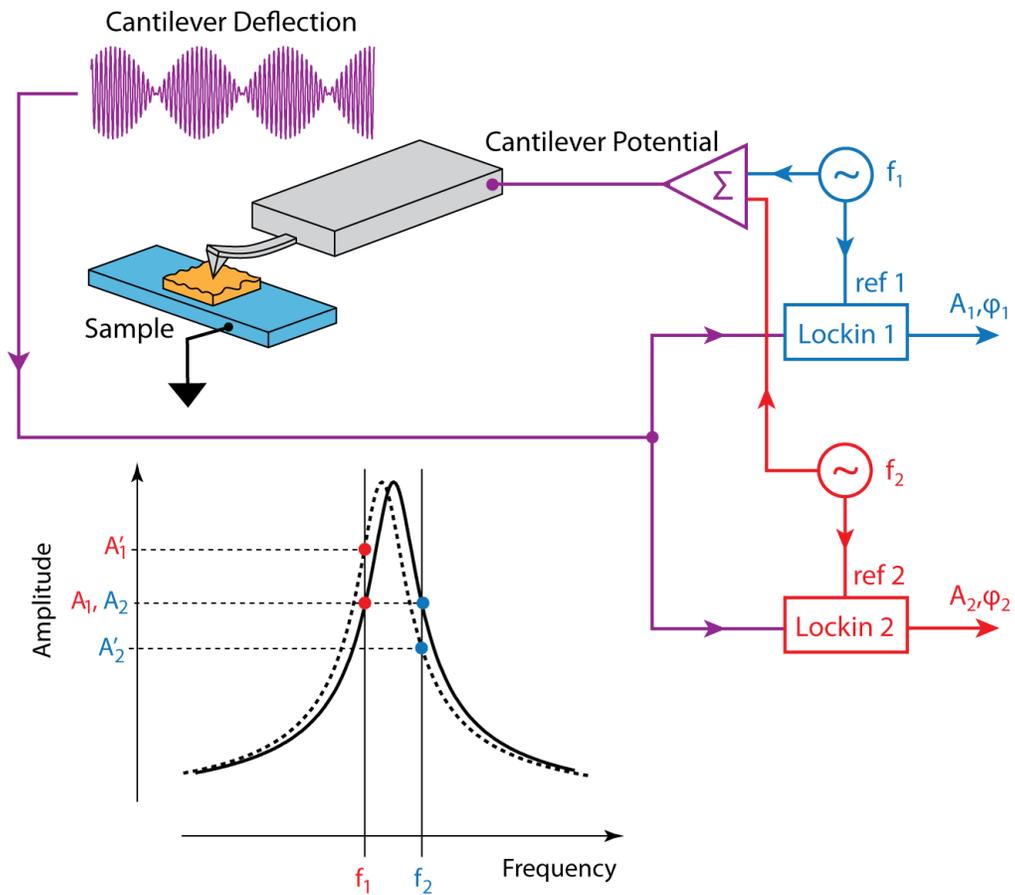


Figure 6.1.: Dart Operation

ModeMaster DART buttons. Selecting the DART option will bring up a template for DART imaging and and point switching spectroscopy (SS-PFM) measurements.



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 6.1.: Typical contact resonance frequency ranges for some common cantilevers

6.3. Dart PFM Example

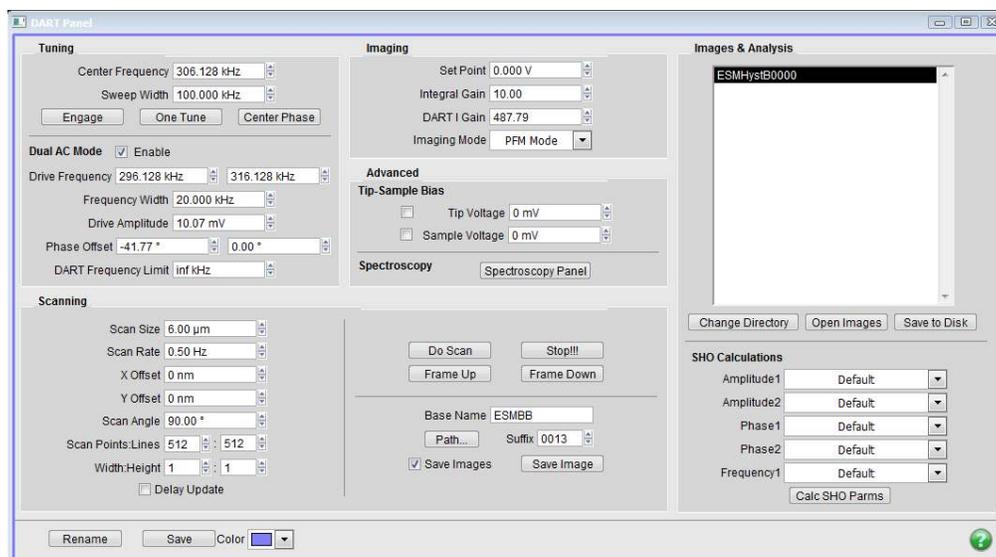


Figure 6.2.: The DART panel.

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>.

DART PFM: Getting Started

1.
 - 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 6.2 on page 46).

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

The Dart Panel:

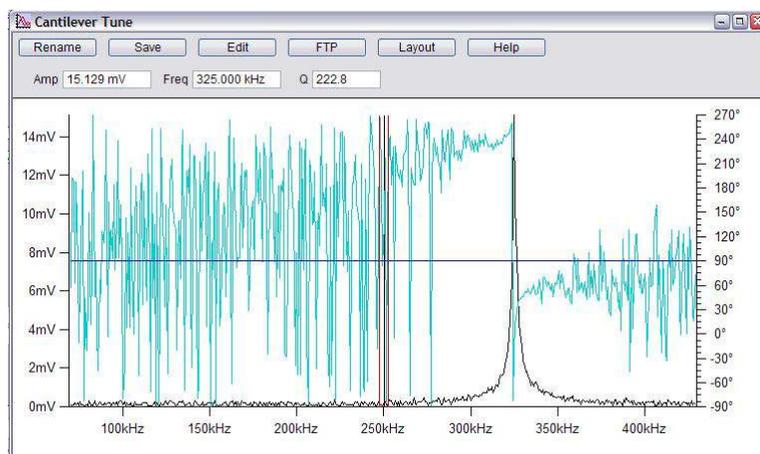
2.
 - The panel in Figure 6.2 on page 48 should appear . If it does not, in the main menu bar go to *Programming* ▷ *User Panels*. You should see a list of available panels, including DART. Select it and the panel below should appear.

PFM Initialization

3.
 - 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 6.1 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 a 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 6.2 on page 48 try a center frequency of 320 kHz and sweep width of 100kHz.
- The frequency width refers to the width of the separation between f_1 and f_2 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.

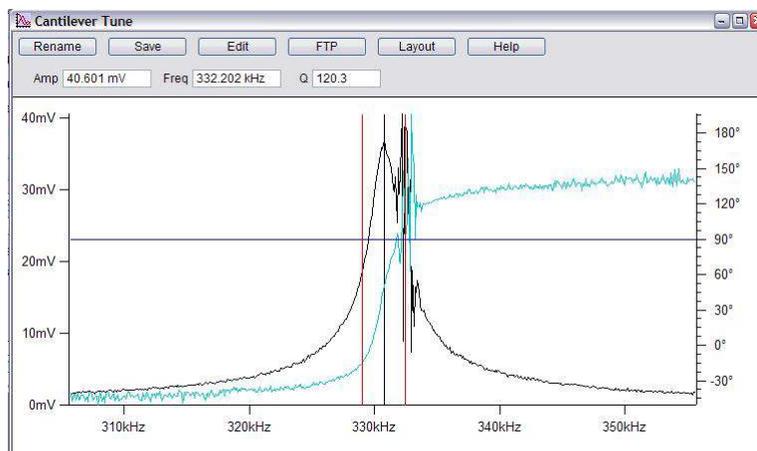
Note If no clear peak is visible, as in the graph above, try widening the sweep width and gently increasing the drive voltage. For an AC240, once the drive goes above ~5 volts you may start to degrade the tip quality. For PPLN and lead zirconium titanate (PZT) samples, 200mV is usually sufficient to 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 lock-in as the first lock-in frequency is swept through the tune range. The ripple in the amplitude and phase channels around the higher drive frequency originate from interference between the first and second lock-ins as the first sweeps through the tune frequencies.

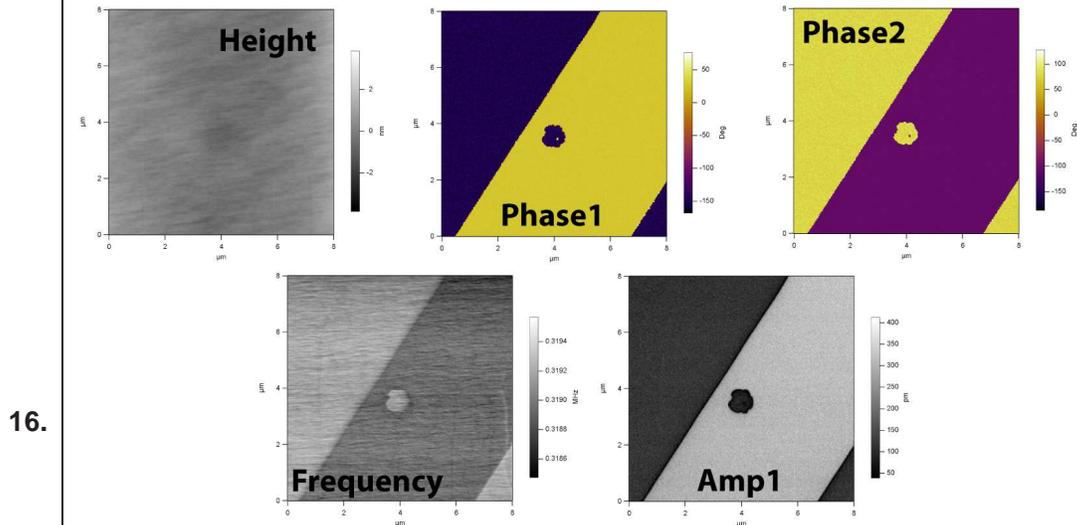
Note It is possible that the position of the resonance peak could shift around during scanning, especially if the surface is rough or the ferroelectric domains are small compared to any thermal drift the system may be experiencing. You may need to “chase” the resonance a bit, especially after you first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

14.

Set Scanning Parameters

- In the Scanning section of the DART panel 6.2 we find it useful to start with a reasonably large scan range (perhaps 20 by 20 μm).
- Set the scan angle of 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



Typical Images, PPLN sample

- If all is well at this point, you should see some 4-10 μm periodic signals on the amplitude, phase and frequency channels corresponding to the poled domains in the PPLN sample.
- Figure above show a typical 6 μm scan over PPLN showing nice contrast over different domains. A small reversed domain is visible in the center of the image where a $\sim 100\text{V}$ 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 less 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.

6.4. Measuring Hysteresis Loops: Switching Spectroscopy PFM (SS-PFM)

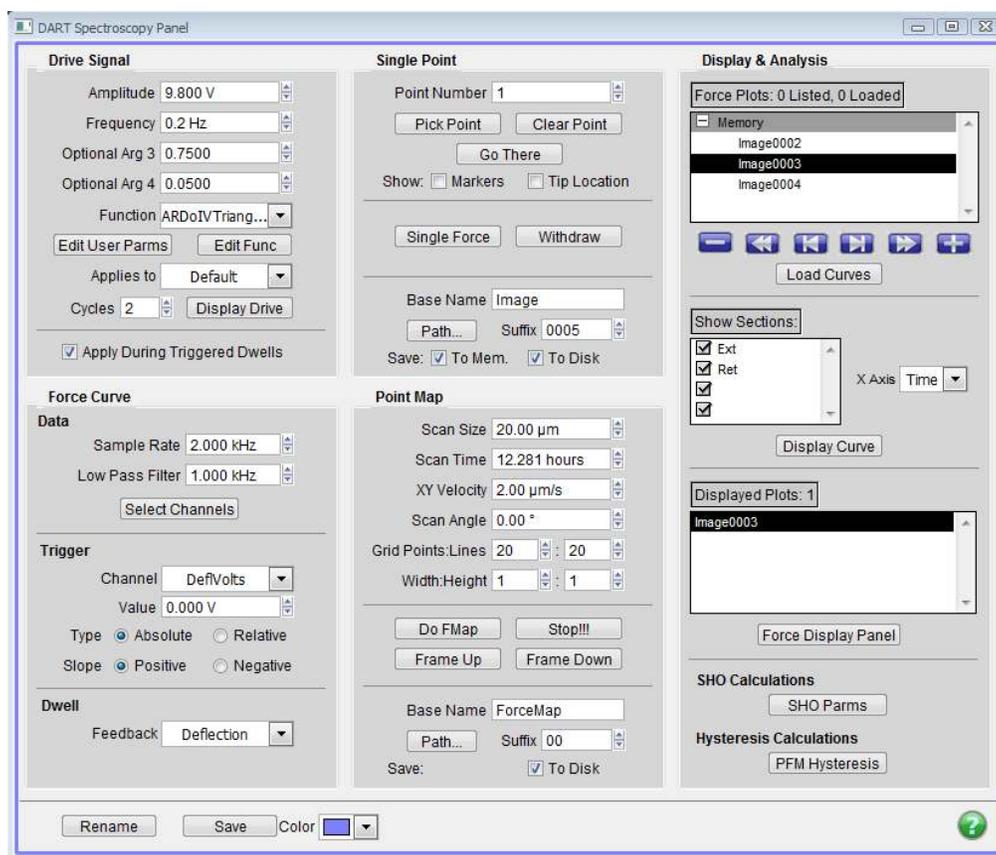


Figure 6.3.: The DART Spectroscopy Panel

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 by⁷. First we'll review some of the terminology related to the triangle step functions which will excite the tip voltage.

⁷?, .

There are four parameters for the triangle step function. The effects of varying them are illustrated in Figure 6.4 on page 55 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 6.5 on page 56.

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.

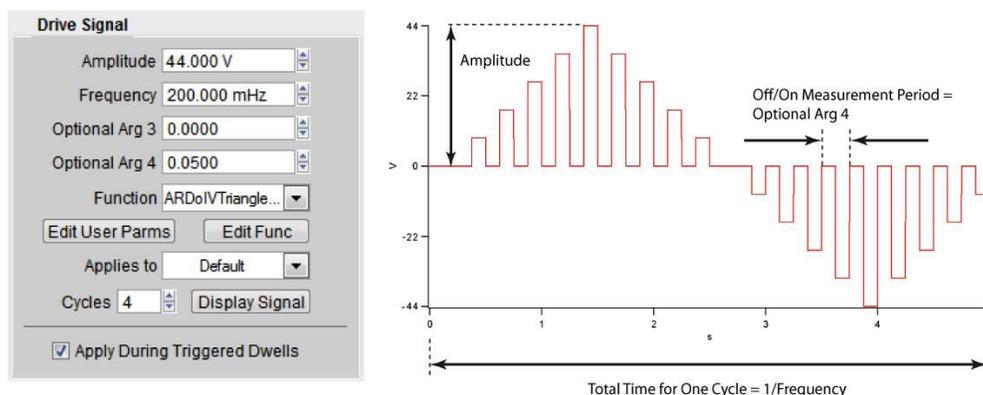


Figure 6.4.: PFM drive waveform parameters

6.4.1. 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 6.3 on page 54), go to the *Drive Signal* group of controls.
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.

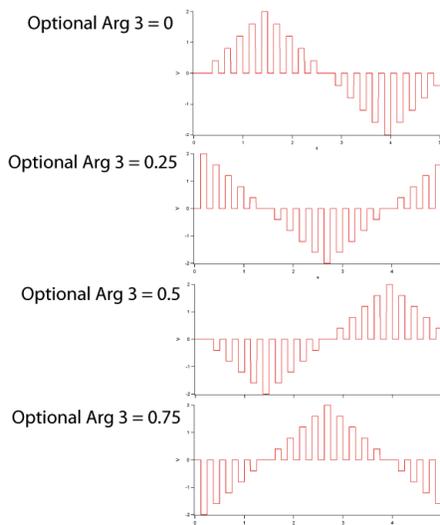


Figure 6.5.: Some examples of the effect of Optional Argument 3

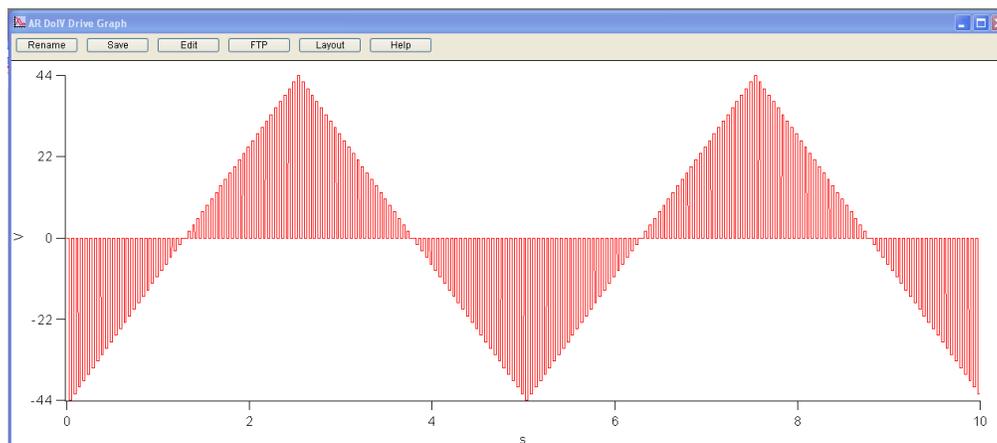


Figure 6.6.: A typical scan waveform for measuring in-field and remnant hysteresis loops for SS-PFM.

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 6.7 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 6.9 below for a nice example of data extracted from the time waves shown in Figures 6.7 and 6.8.

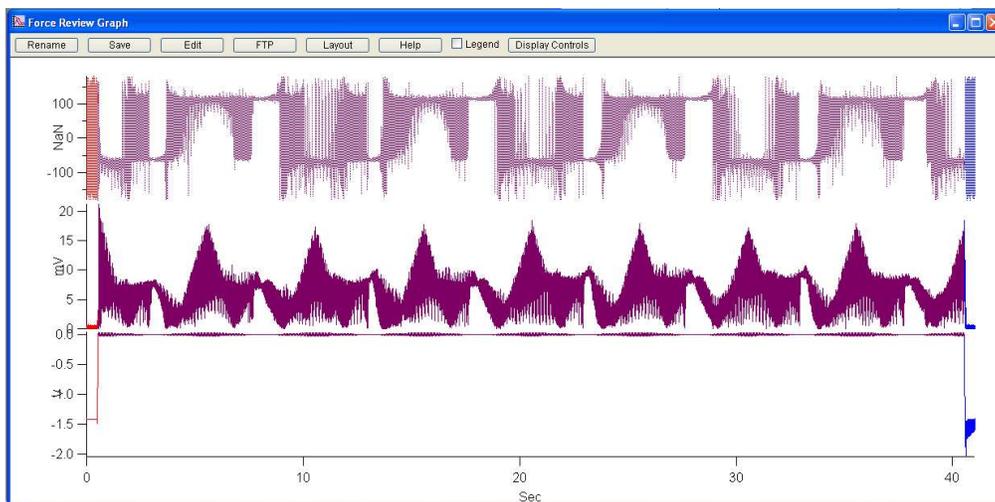


Figure 6.7.: The SS-PFM raw data set. The phase1, amp1 and deflection traces for an 8-cycle, 40 second hysteresis loop. The details of the switching are difficult to discern from the time data and need to be processed to produce hysteresis loops.

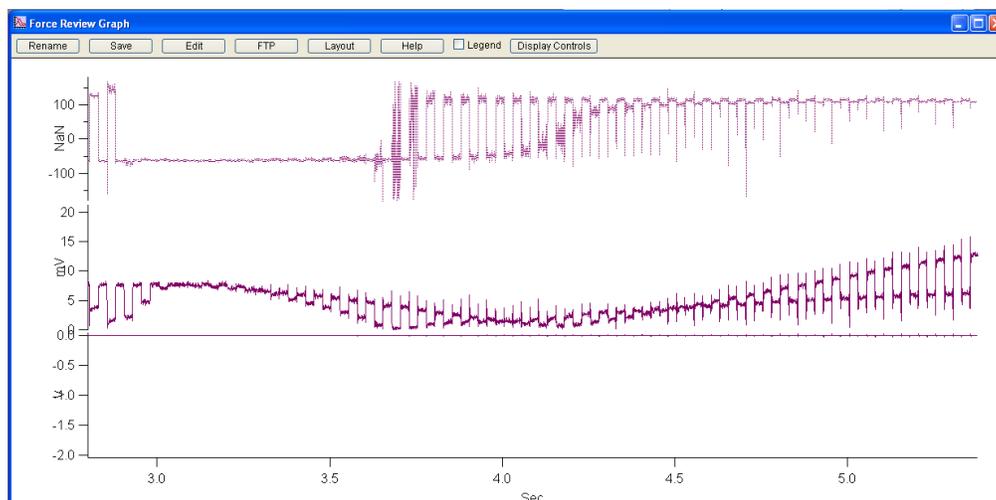


Figure 6.8.: Zoomed in region from Figure RawData. Here, the individual steps from the stepped SS-PFM method of⁸ are evident. Repeat the above steps as many times as you would like, adjusting the drive parameters as you wish. If you notice noise during the higher voltage portions of the curves, you may have your *Freq Igain* set too large. Reduce it until the effect disappears.

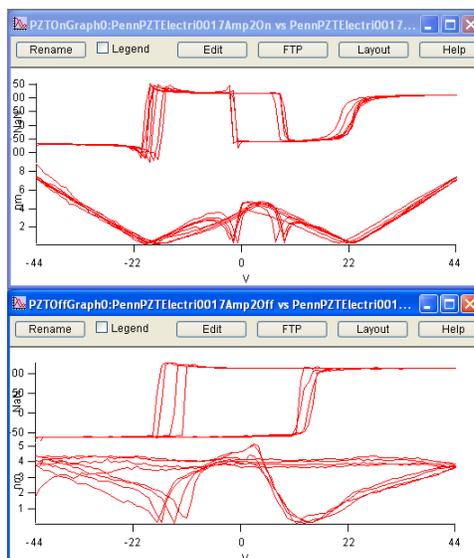


Figure 6.9.: The applied (“On”, top graph) and remnant (“Off”, bottom graph) hysteresis loops obtained from the processed time waves of figure 6.7 and 6.8. The phase loops are on the top of each window and the amplitude “butterfly” loops are on the bottom.

6.4.2. To do an array of SS-PFM 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.

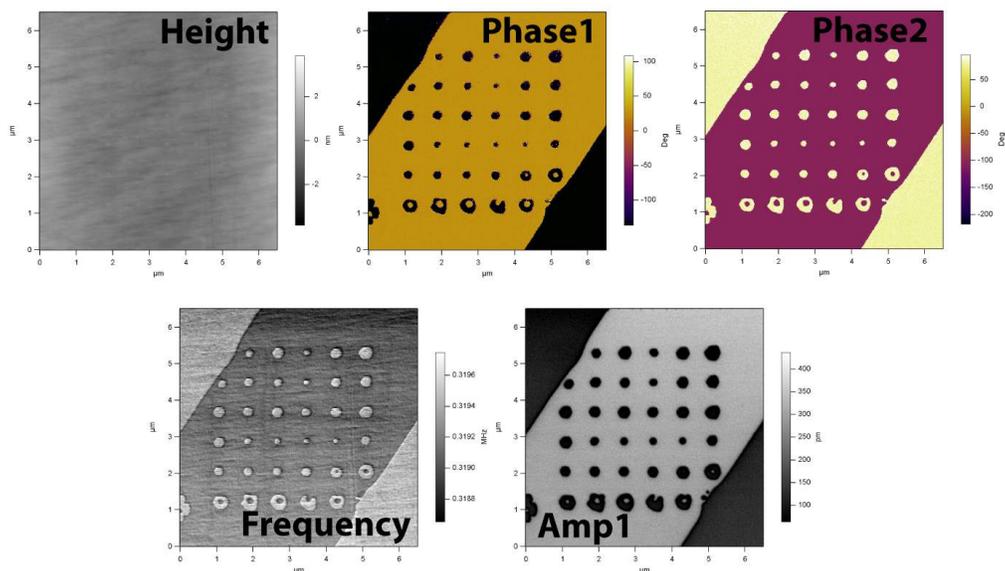


Figure 6.10.: DART PFM image of PPLN after a 6 by 6 array of hysteresis loops have been measured.

6.5. DART Imaging Guidelines and Troubleshooting

6.5.1. 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.

6.5.2. 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 90° to minimize tip shape effects, which can potentially have a large effect on the contact resonant frequency.

6.5.3. 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°.

6.5.4. “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.

6.5.5. 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.

6.5.6. 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.

6.6. MFP3D 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 6.11 on page 61.

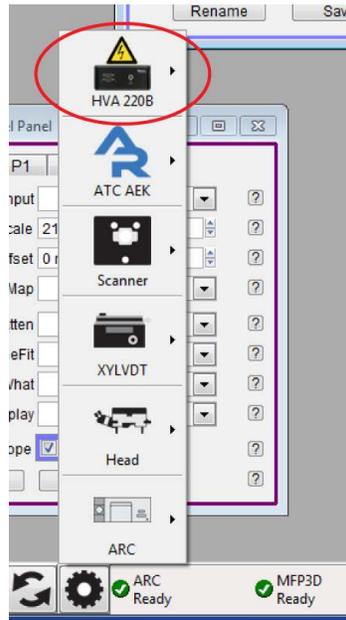


Figure 6.11.: Testing communication with the HVA using the SmartStart Device interface. The HVA icon indicates that the HVA is present and active.

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.



7. Piezo Force Microscopy: Scientific Background and Theory

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by Roger Proksch¹ and Sergei Kalinin²

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7.1. 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 7.1.: PFM amplitude channel overlaid on AFM height (top) and phase image overlaid on height (bottom) of lead zirconium titanate (PZT), 20 μm scan.



Figure 7.2.: 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.

7.2. 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 phenomena³. 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) devices⁴. The ability of ferroelectric materials to switch polarization orientation – and maintain polarization state in a zero electric field – has led to emergence of concepts of non-volatile ferroelectric memories and data storage devices⁵. 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, of the broad arrays of piezoelectric, biological and molecular-based electromechanical systems.

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⁵ ? . .

Progress along this path requires the ability to image and quantify electromechanical functionalities on the nanometer and molecular scale (Figure 7.1 on page 63 and Figure 7.2 on page 63). 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 materials⁶.

7.3. Principles of PFM

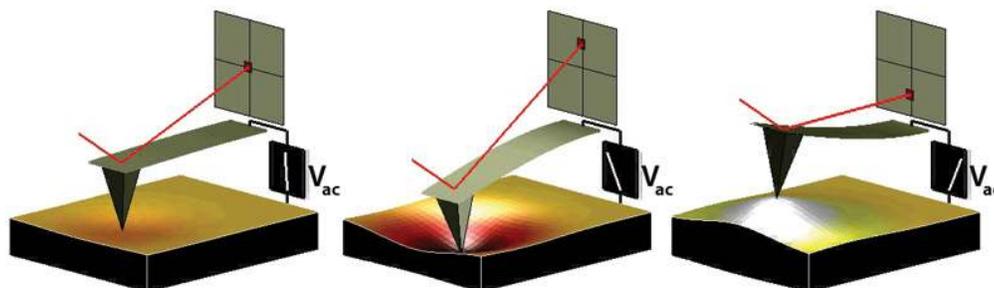


Figure 7.3.: Depiction of PFM operation. The sample deforms in response to the applied voltage, which in turn, causes the cantilever to deflect, which can then be measured and interpreted in terms of the piezoelectric properties of the sample. Image courtesy S. Jesse, ORNL.

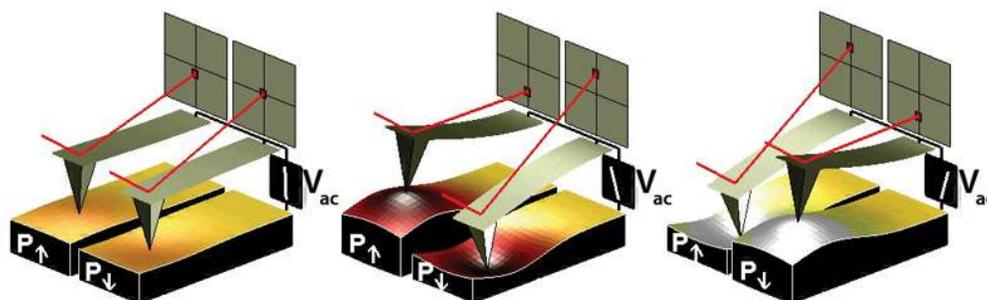


Figure 7.4.: Sign dependence of the sample strain. When the domains have a vertical polarization that is pointed downwards and a positive voltage is applied to the tip, the sample will locally expand. If the polarization is pointed up, the sample will locally contract. The phase of the measured response is thus proportional to the direction of the domain polarization. Figure courtesy of S. Jesse, ORNL.

7.3.1. 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 expands or contracts as shown in Figure 7.3 on page 64

⁶?, .

When the tip is in contact with the surface and the local piezoelectric response is detected as the first harmonic component of the tip deflection, the phase φ , of the electromechanical response of the surface yields information on the polarization direction below the tip. For c- domains (polarization vector oriented normal to the surface and pointing downward), the application of a positive tip bias results in the expansion of the sample, and surface oscillations are in phase with the tip voltage, $\varphi = 0$. For c+ domains, the response is opposite and $\varphi = 180^\circ$. More details are given in Section 7.3.2 (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 PFM⁸. 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) .

7.3.2. 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} component¹¹, since it couples directly into the vertical motion of the cantilever. The voltage applied to the tip is

$$V_{tip} = V_{dc} + V_{ac} \cos(\omega t) \quad (7.1)$$

resulting in piezoelectric strain in the material that causes cantilever displacement

$$z = z_{dc} + A(\omega, V_{ac}, V_{dc}) \cos(\omega t + \varphi) \quad (7.2)$$

due to piezoelectric effect¹². When the voltage is driven at a frequency well below that of the contact resonance of the cantilever, this expression becomes

$$z = d_{33}V_{dc} + d_{33}V_{ac} \cos(\omega t + \varphi) \quad (7.3)$$

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⁸ ? . .
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¹⁰ ? . .
¹¹ ? . .
¹² ? . .

Table 7.1.: 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
LiNbO_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 BiFeO_3	FeRAM	3-10	1-10	10-20
Ultrathin Films				
1-5nm BiFeO_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 7.6 $A = d_{33} \text{Vac} Q$ 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 $A > 30\text{pm}$. The ultimate limit is $A = \text{thermal noise}$.

^{††} 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.

where we have implicitly assumed d_{33} depends on the polarization state of the material. From this last equation and from Figure 7.3 on page 64, 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 7.3 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.

As mentioned above, the direction of sample polarization determines the sign of the response. Figure 7.4 on page 64 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

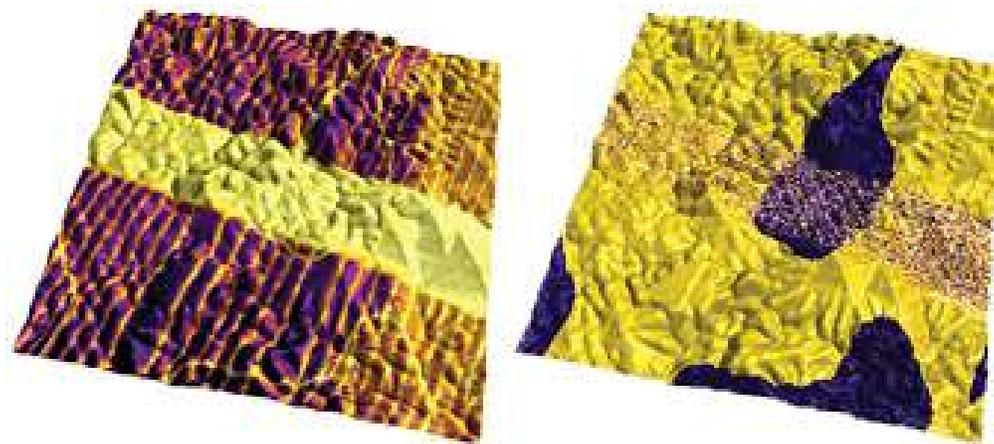


Figure 7.5.: Vertical PFM amplitude overlaid on AFM topography (left) and PFM phase overlaid on AFM topography (right) images of lead titanate film, 5 μm scan. Images courtesy of A. Gruverman and D. Wu, UNL. Sample courtesy H. Funakubo.

of the polarization orientation of the sample when an oscillating voltage is applied to the sample.

The relationship in Equation 7.1 and the values for d_{33} in Table 7.1 on page 66 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 $<100\text{kHz}$. 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 $>300\text{kHz}$.

7.3.3. PFM Imaging Modes

The three typical PFM imaging modes and piezoelectric lithography are briefly described below.

7.3.3.1. Vertical PFM

In vertical PFM imaging, out-of-plane polarization is measured by recording the tip-deflection signal at the frequency of modulation. Figure 7.5 on page 67 shows an example image of vertical PFM for a lead titanate film. Antiparallel domains with out-of-plane polarization can be seen in the PFM phase image, while in-plane domains are seen in the PFM amplitude image as yellow stripes due to the weak vertical piezoresponse signal

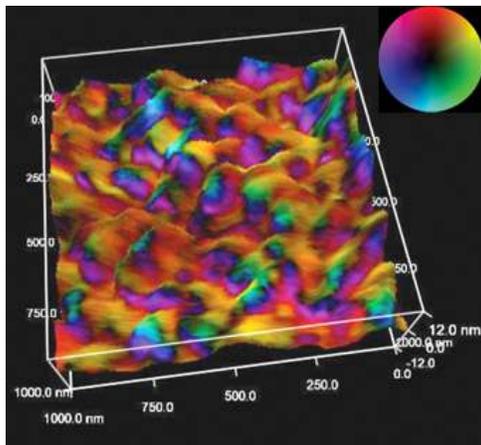


Figure 7.6.: BST film with vector PFM overlaid on AFM topography, 1 μ m scan. Image courtesy of C. Weiss and P. Alpay, Univ. of Conn., and O. Leaffer, J. Spanier, and S. Nonnenmann, Drexel University. Color wheel indicates PFM vector orientation.

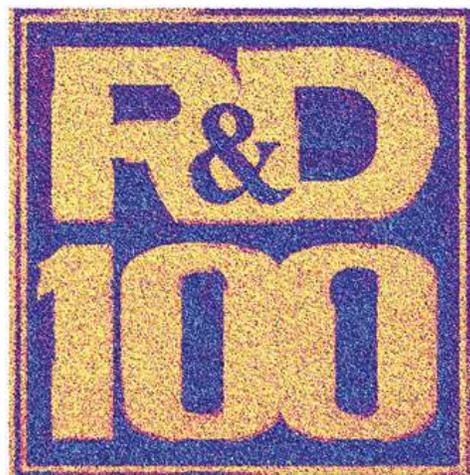


Figure 7.7.: R&D 100 logo written on a sol-gel PZT thin film by PFM lithography. PFM phase is overlaid on top of the rendered topography, 25 μ m scan. Oak Ridge and Asylum Research were awarded an R&D100 award for Band Excitation in 2008.

7.3.3.2. 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 PbTiO₃ thin films¹⁶.

7.3.3.3. 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 7.6 on page 68 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

¹³, .
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¹⁵, .
¹⁶, .

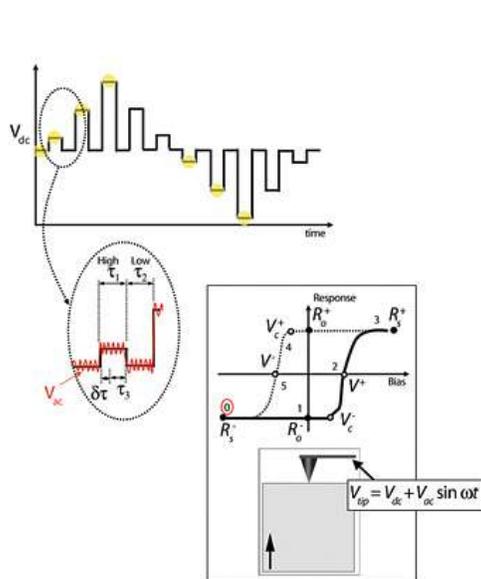


Figure 7.8.: Switching spectroscopy PFM diagram (see text for discussion). Reused with permission from Jesse, Baddorf, and Kalinin, Applied Physics Letters, 88, 062908 (2006). Copyright 2006, American Institute of Physics.

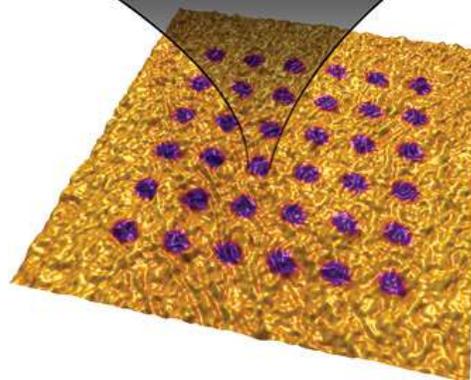
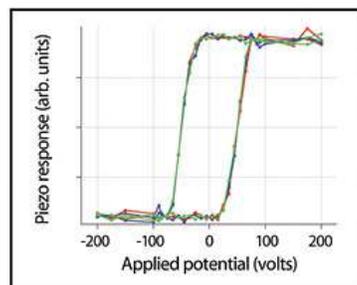


Figure 7.9.: Rendered topography of a LiNbO₃ sample with the PFM signal overlaid on top, 4 μ m scan.

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.

7.3.3.4. 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 7.7 on page 68 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.

7.3.3.5. 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 ac-

cess 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 ($\sim 10^3 - 10^5 \text{ kT}$) 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 switching¹⁷. 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.

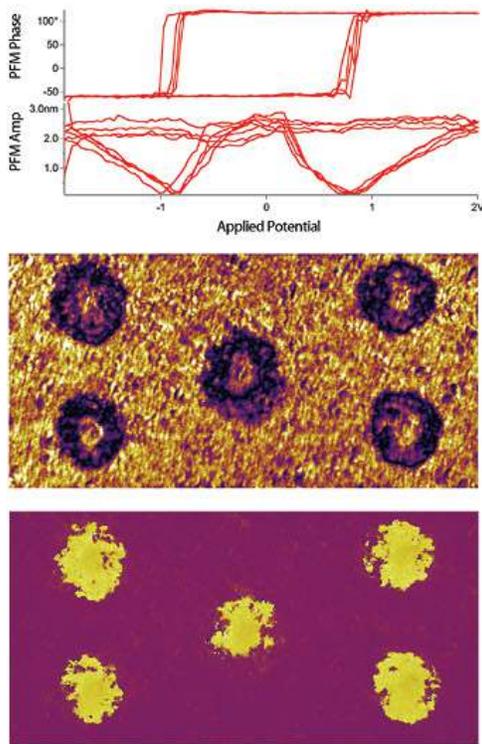


Figure 7.10.: Sol gel PZT sample where local hysteresis loops were measured and displayed (representative phase and amplitude loops shown at top). After the switching spectroscopy measurements, the area was imaged, the DART amplitude (middle) and phase (bottom) are shown, $3.5 \mu\text{m}$ scan.

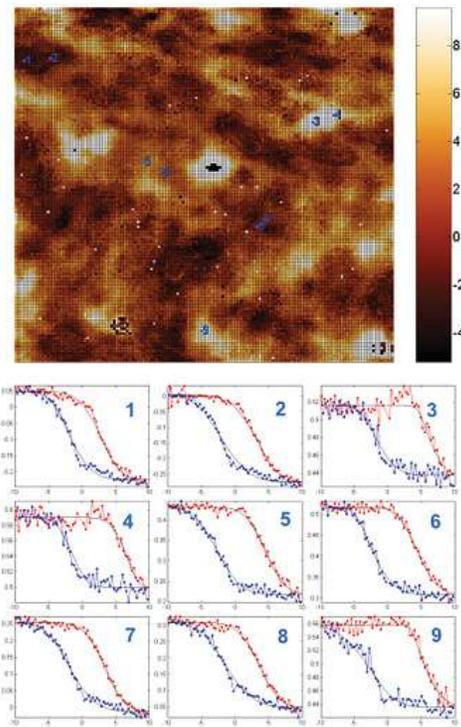


Figure 7.11.: SS-PFM and hysteresis loops of capacitor structures. Data courtesy K. Seal and S.V. Kalinin, ORNL. Sample courtesy P. Bintacchit and S. Troler-McKinstry, Penn State Univ.

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

¹⁷, .

ferroelectrics, thus resolving the structural origins of the Landauer paradox¹⁸. 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 7.8 on page 69 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 7.9 on page 69 shows an example image of a LiNbO₃ 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 7.10 on page 70 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 7.11 on page 70 shows SSM-PFM of capacitor structures and Figure 7.12 on page 71 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.

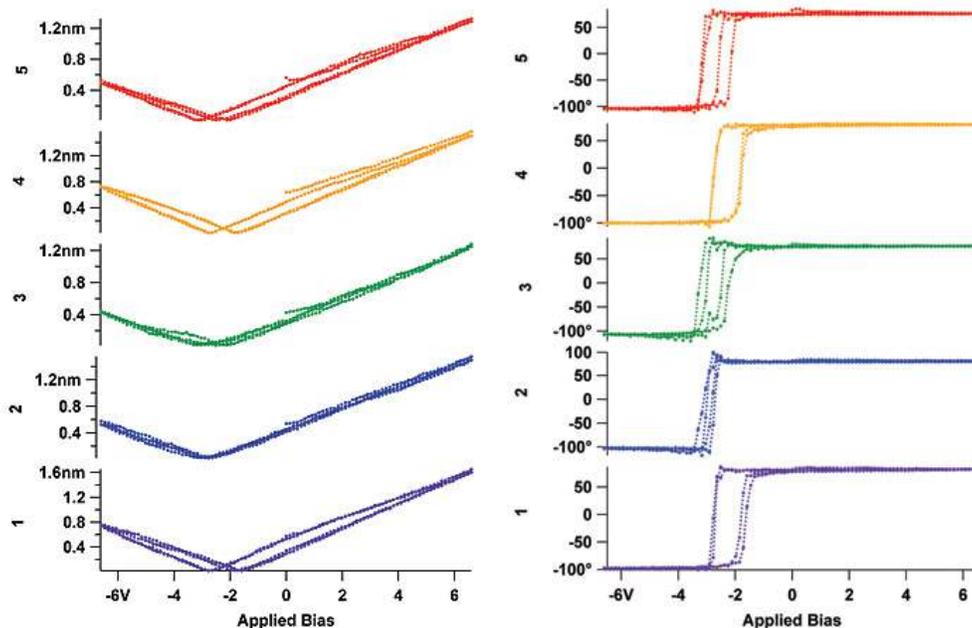


Figure 7.12.: Amplitude (left) and phase (right) hysteresis loops measured at five different locations on a PZN-PTi thin film.

¹⁸ . . .

7.4. 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.

7.4.1. 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 LiNbO₃) allowed direct imaging and spectroscopy of ferroelectric materials sufficient for applications corresponding to a detection limit of 50pm at ~100 kHz. Measurements of lower sensitivity materials require the use of higher voltages or the use of contact resonance.

7.4.2. 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 reactions¹⁹.

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 Sader²⁰ in the vicinity of a resonance for small damping ($Q > 10$), the amplitude and phase frequency response can be described using the harmonic oscillator model²¹ as

$$A(\omega) = \frac{A_{\max} \omega_0^2 / Q}{\sqrt{(\omega_0^2 - \omega^2)^2 + (\omega_0 \omega / Q)^2}} \quad (7.4)$$

$$\tan \varphi(\omega) = \frac{\omega_0 \omega}{Q(\omega_0^2 - \omega^2)} \quad (7.5)$$

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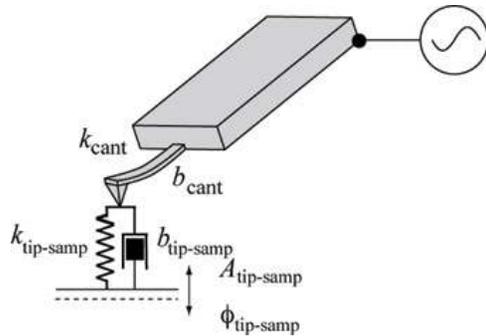


Figure 7.13.: In PFM, the cantilever voltage is modulated, usually at some fixed frequency. This causes the sample to distort at some amplitude and phase. Mediated by the contact mechanics, this drives the tip which, in turn, is monitored by the AFM sensor.

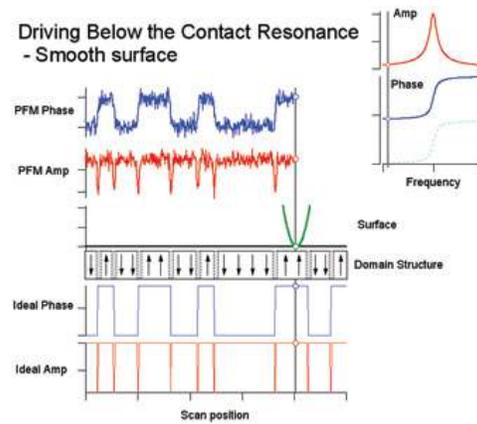


Figure 7.14.: This figure shows the ideal and measured PFM response of an idealized tip (green) scanning over a smooth surface (black line below the “tip”). The domain structure of the ferroelectric sample is shown below the surface where the arrows correspond to the sample polarization direction. The 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.

where, A_{\max} is the amplitude at the resonance ω_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

$$A = d_{33}V_{ac}Q \quad (7.6)$$

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 7.15.

Figure 7.13 on page 73 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 ($A_{\text{tip-samp}}$, $\phi_{\text{tip-samp}}$). 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

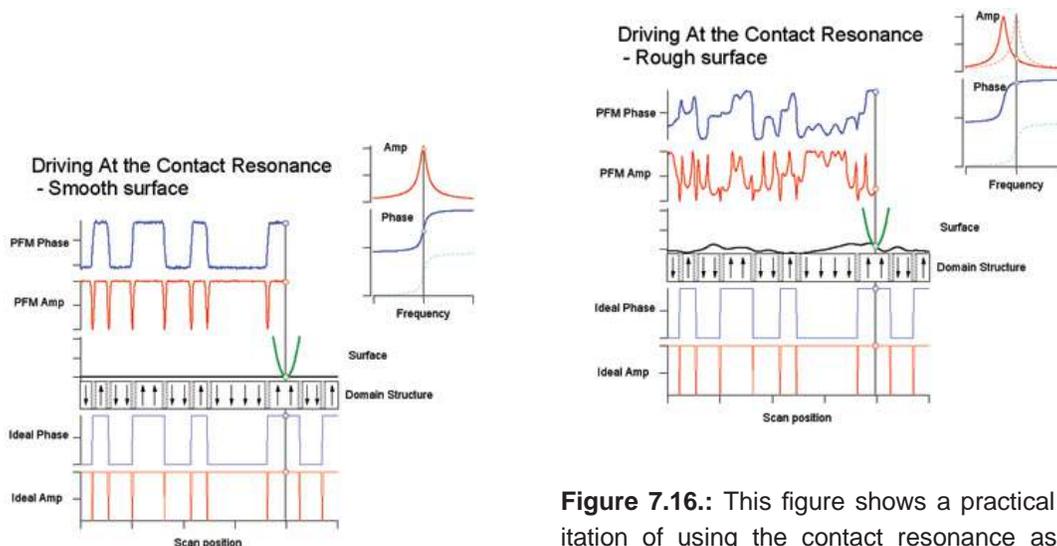


Figure 7.15.: This figure shows the same situation as described in Figure 7.14 on page 73, except that here we are using resonance enhancement to boost the small PFM signal. The inset frequency tune in the upper right corner shows the drive frequency. In this case, since the Q -factor of the resonance is 100, the SNR of the measured PFM amplitude (red, thick curve) and phase (blue, thick curve) has dramatically improved.

Figure 7.16.: 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.

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, affects the resonance frequency.

To understand how resonance is affected in PFM, we first describe an “ideal” situation as illustrated in Figure 7.14 on page 73. 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 simula-

tion 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.

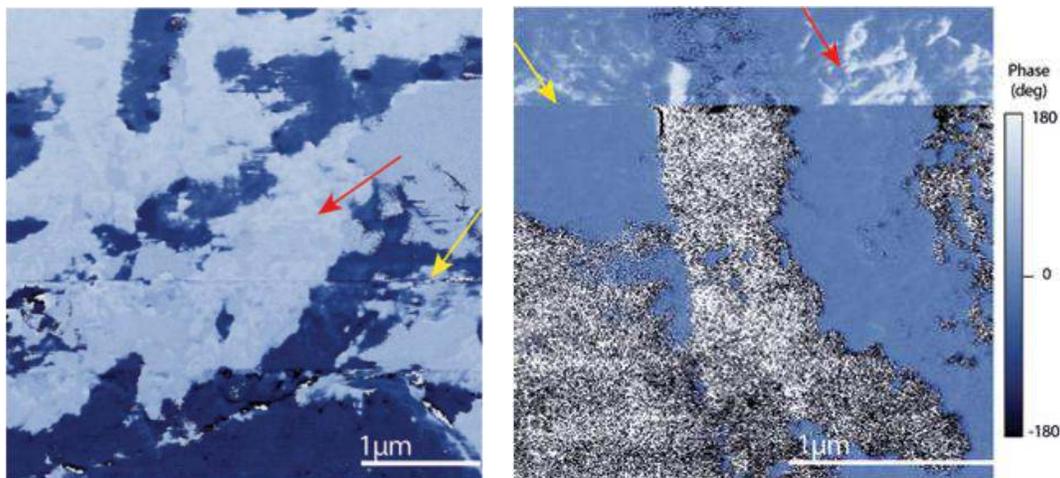


Figure 7.17.: PFM phase channel on a polished PZT sample. The cantilever was driven near the contact resonance to enhance the SNR. There is significant crosstalk between the sample topography and the PFM signal. Red arrows indicate “roughness” where the contact stiffness changes the measured phase. The yellow arrows indicate a sudden tip change causing a change in the contact resonance, and consequently causing significant 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 7.19 on page 76, Figure 7.17 on page 75, and Figure 7.18 on page 76 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 consequence, interpretation of the domain structure becomes much more difficult and in many cases, impossible. Figure 7.16 on page 74 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

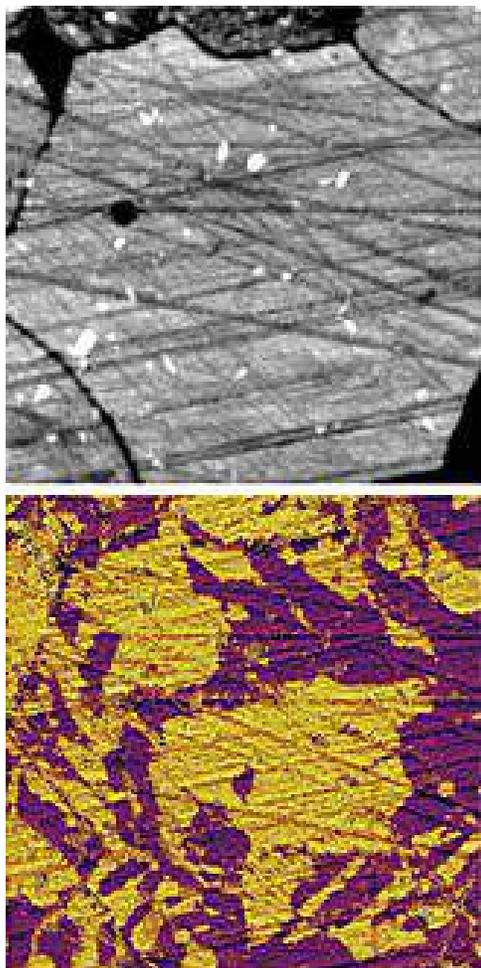


Figure 7.18.: PZT showing crosstalk, 14 μm scan.

kHz. This causes large, discontinuous changes in the measured phase. [Figure 7.17 on page 75](#) and [Figure 7.18 on page 76](#) 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 7.19 on page 76](#). 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.

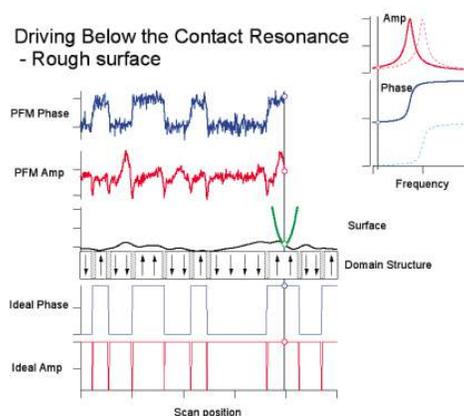


Figure 7.19.: Driving below contact resonance with conventional PFM. Here, the cantilever is driven well below the contact resonant frequency. The effects of surface roughness are minimized, though still visible in the measured PFM amplitude. However, this reduction in crosstalk comes at the high price 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.

7.5. Solutions to Limits of Conventional PFM

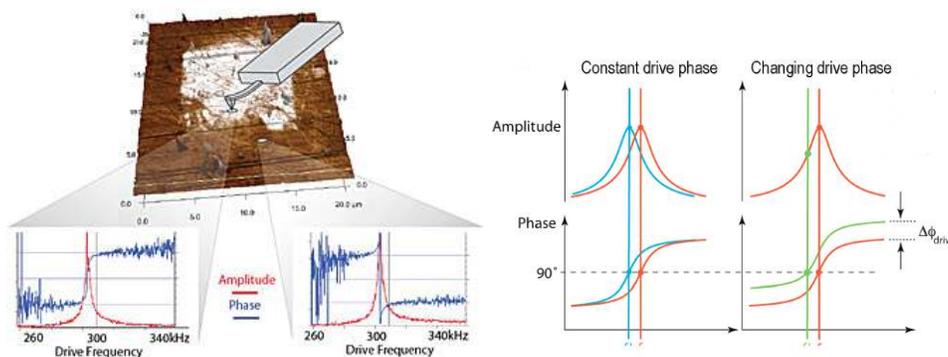


Figure 7.20.: For domains with an antiparallel (180°) orientation, conventional PLLs drive the PFM frequency away from resonance. (Top) Amplitude, red, and phase, blue, cantilever response over antiparallel domains. In the measurement, phase is offset by 180° over anti-parallel domains (see curves on 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 7.17 on page 75 and Figure 7.18 on page 76.

Figure 7.20 reprinted with permission²².

7.5.1. 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 7.6).

7.5.2. Using Contact Resonance as a PFM Amplifier

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

As noted in 7.4, 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

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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 7.20 on page 77 is an example where, for antiparallel domains, a conventional PLL will actually drive a PFM away from resonance.

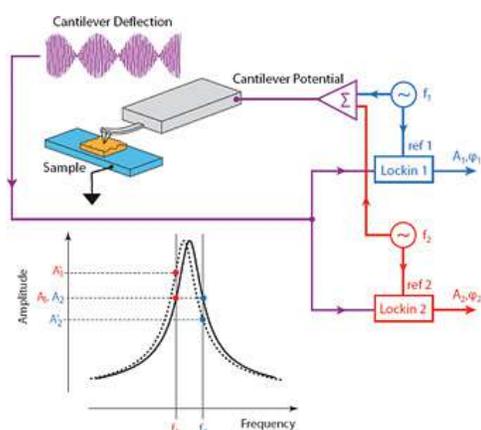


Figure 7.21.: Schematic diagram of DART, showing a drive phase independent feedback signal.

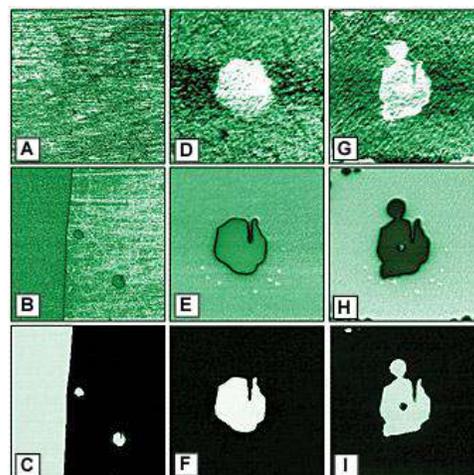


Figure 7.22.: (A), (D), (G) Resonance frequency, (B), (E), (H) piezoresponse amplitude and (C), (F), (I) piezoresponse phase images of antiparallel domains in lithium niobate. Shown are images of the (A)–(C) native domain structure, (D)–(F) an intrinsic domain and (G)–(I) domains switched by $\pm 176V$ (locations marked in (E)). The images are obtained at $\omega f = 4\text{kHz}$ and $V_{ac} = 66V$. The frequency images have been flattened to account for minute changes of contact radius from line to line.

Figures 7.21 and 7.22 reprinted with permission²⁵.

7.5.3. 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 7.21 on page 78 shows how DART works. The potential of the conductive cantilever is the sum of two oscillating voltages with

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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 changes²⁶.

DART-PFM studies of polarization switching are illustrated in Figure 7.22 on page 78, where the resonant frequency (A), amplitude (B) and phase (C) images of a lithium niobate surface are shown Figure 7.22A. 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 7.22 (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 domain²⁷.

Additional DART images of ferroelectric materials are shown in Figure 7.23 on page 80 and Figure 7.24 on page 80. Figure 7.23 on page 80 shows a series of images of PFM on multiferroic BiFeO₃ nanofibers. Figure 7.24 on page 80 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.

7.5.4. 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 Laboratory²⁸ 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 7.4 and 7.5)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 7.25 reprinted with permission³⁰.

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

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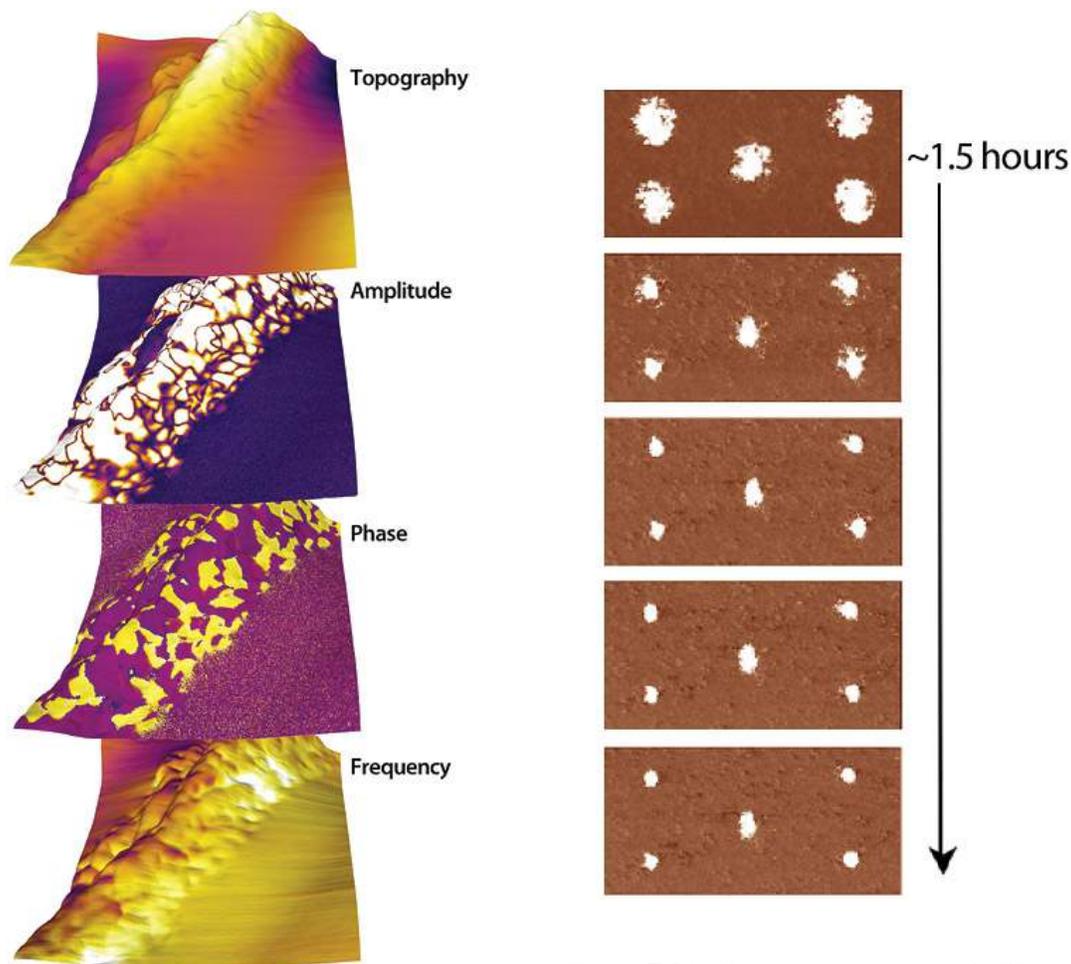


Figure 7.23.: PFM of multiferroic BiFeO_3 nanofibers, 1 μm scan. Collaboration with Shuhong Xie, Xiangtan University, China and JiangYu Li, University of Washington.

Figure 7.24.: 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.

a single pixel in conventional single-frequency SPM. BE allows quantitative mapping of local energy dissipation in materials on the nanoscale³¹. Figure 7.26 on page 82 shows an example image of an amyloid fibril (bovine insulin) on mica imaged in water using the BE-PFM technique. The image size 250nm x 250nm.

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

- 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.

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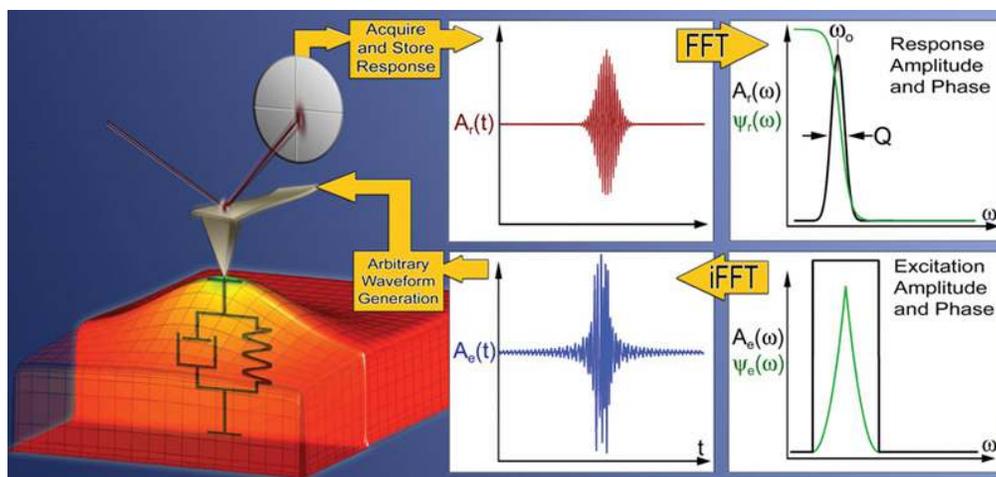


Figure 7.25.: Operational principle of the BE method in SPM. The excitation signal is digitally synthesized to have a predefined amplitude and phase in the given frequency window. The cantilever response is detected and Fourier transformed at each pixel in an image. The ratio of the fast Fourier transform (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.

- Imaging modes and hardware are fully integrated.

7.6. Emerging Applications for PFM

7.6.1. 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 $\sim 2-8$ MHz and potentially higher, opening a pathway for high frequency studies of polarization dynamics. Figure 7.27 on page 83 illustrates the different information that is revealed by imaging a ceramic PZT material at various frequencies.

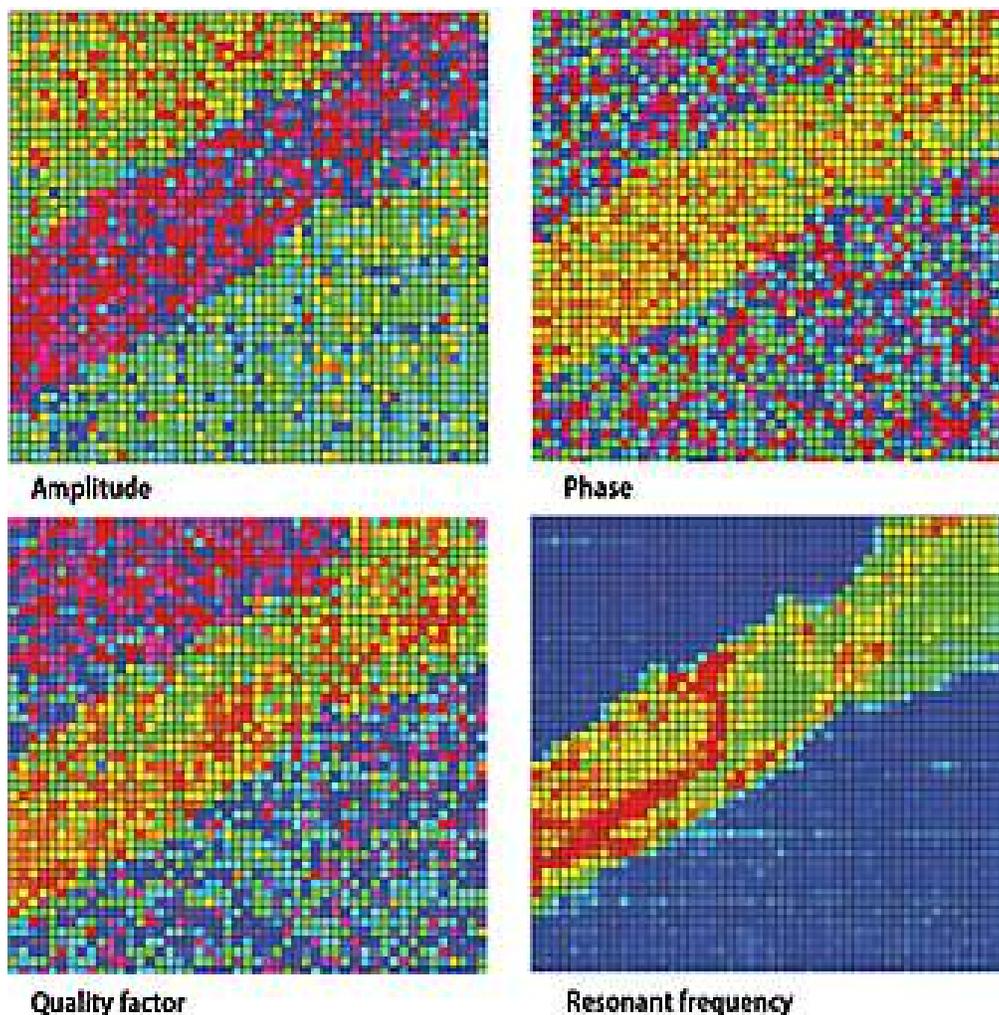


Figure 7.26.: Amyloid fibril (bovine insulin) on mica imaged in water using BE-PFM technique, 250nm x 250nm. Image courtesy of G. L. Thompson, V. V. Reukov, A. A. Vertegel, M. P. Nikiforov, Clemson University, Dept. Bioengineering, and S. Jesse, S. V. Kalinin, Oak Ridge National Lab.

7.6.2. 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 second³².

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 7.28 on page 83 and Figure 7.29 on page 84.

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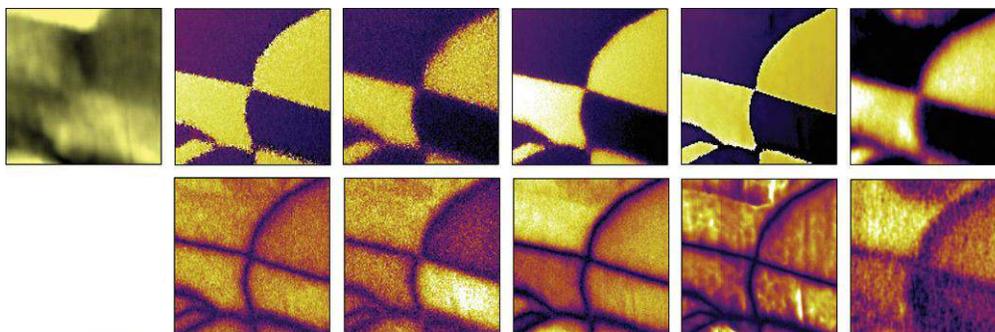


Figure 7.27.: High Frequency PFM using Asylum’s fast photodiode on a ceramic PZT sample at different frequencies (phase left, amplitude right) – below first resonance (top row) and at cantilever resonances (all others) using a MikroMasch NSC 35B cantilever. $1\mu\text{m}$ scans. Image courtesy of K. Seal, S. Kalinin, S. Jesse, and B. Rodriguez, Center for Nanophase Materials Science, ORNL.

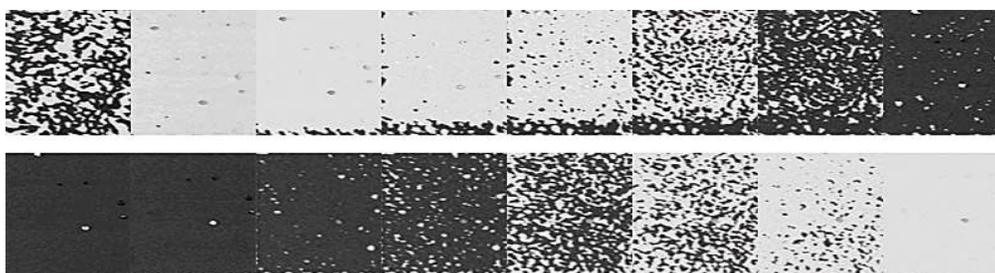


Figure 7.28.: This image sequence (left to right, top to bottom) is excerpted from a movie of 244 consecutive High Speed PFM images ($4\mu\text{m}$ scans) depicting in situ ferroelectric memory switching. For the first half of the movie, the tip is biased with a positive DC offset throughout the measurements. 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.

The more general High Speed Scanning Property Mapping (HSSPM) allows rapid measurements of mechanical compliance, electric fields, magnetic fields, friction, etc, with similar benefits for novel dynamic measurements of surfaces³³.

7.6.3. 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

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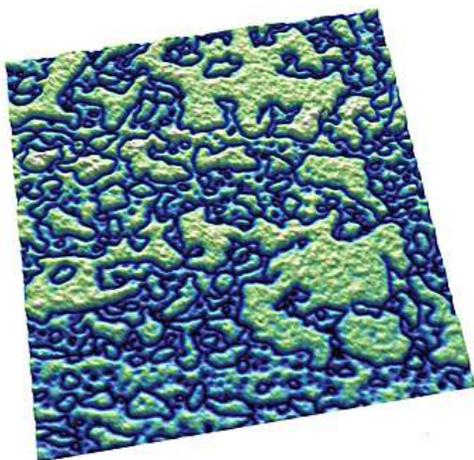


Figure 7.29.: (001) domains in a PZT thin film, 3.8 μm scan. Image courtesy N. Polomoff and B. D. Huey, University of Connecticut Institute of Materials Science. Sample courtesy R. Ramesh, UC Berkeley.

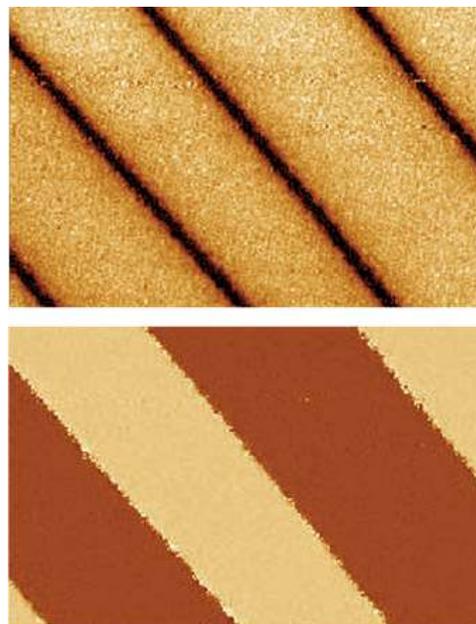


Figure 7.30.: PPLN amplitude (top) and phase image (bottom) acquired with the MFP NanoIndenter, 50 μm scan.

sample surface. This motion is not well characterized and in most cases is not even measured.

The ability to probe forces and directly image the piezo response of a sample with the Asylum Research MFP NanoIndenter is an emerging application area³⁴. 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, measured 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 7.30 on page 84 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 7.31 on page 85. 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,

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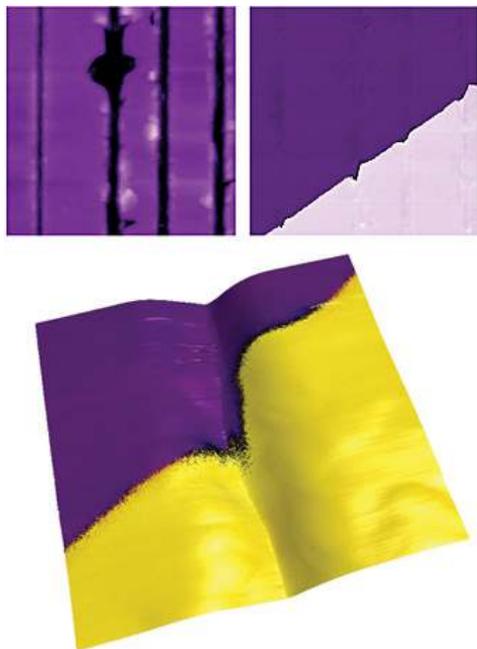


Figure 7.31.: Surface topography of PPLN after it has been purposefully scratched with different loading forces using the NanoIndenter, 10µm scan (top images). 1µm scan (bottom).

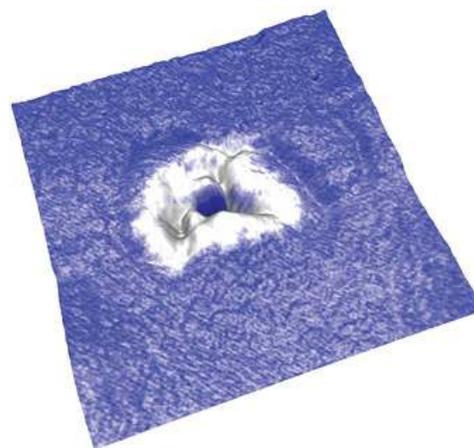


Figure 7.32.: Zoom of the top surface of a red blood cell. The surface shape was rendered to show the topography while the phase channel is overlaid on top to show piezo response. A small sub-micron region on top (white) of the cell exhibited a much different piezo response than the surrounding cell surface. 2µm scan. Image courtesy of B. Rodriguez and S. Kalinin, ORNL.

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 distortion in the domain structure.

7.6.4. 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

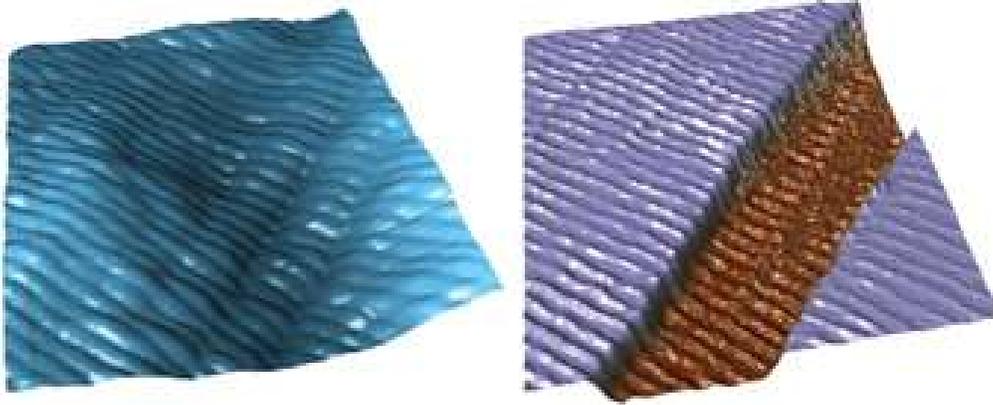


Figure 7.33.: Topographic (top) and PFM phase (bottom) images of collagen fibers, 1.4 μ m scan. Image courtesy D. Wu and A. Gruverman, UNL. Sample courtesy G. Fantner.

polymers, bacteriorhodopsin, and connective tissue^{35,36}. 7.33 shows 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 7.32 on page 85³⁷. This image shows a zoom of a red blood cell with the PFM phase channel painted on top to show piezo response.

7.7. Additional Reading

7.7.1. 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.

- ?,³⁸
- ?,³⁹
- ?,⁴⁰
- ?,⁴¹
- ?,⁴²
- ?,⁴³
- ?,⁴⁴

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- ?, 45
- ?, 46
- ?, 47
- ?, 48
- ?, 49
- ?, 50
- ?, 51
- ?, 52

7.7.2. Comprehensive Material

These references provide key papers and comprehensive reviews on PFM:

- ?, 53
- ?, 54
- ?, 55

7.8. 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.

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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.

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.

8. Conductive AFM (ORCA)

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8.1. Introduction

Conductive AFM (ORCA on an Asylum Instrument) can be considered anything that looks at 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 in principle all do the same thing; they measure one of the variables in Ohm's equation $V=IR$, where V is the voltage applied between the tip and the sample, I is the current passing through the sample and the tip, and R is the resistance of the entire circuit.

8.1.1. Terminology

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

Subcategories:

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

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

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

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

8.1.1.1. 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 $n \times n$ points. Spectroscopy normally entails taking AC mode images of a surface, then changing over to contact mode and collecting the spectra where desired.

8.2. Probes

There are several probes available for this technique. Ideal spring constant range is between 0.5 and 5 N/m. If you get much softer it becomes difficult to overcome the contact resistance between the tip and the sample, if you get much stiffer then your wear can quickly become too large.

- Metal coated Si probes. Pt, PtIr, Au, CoCr, Tungsten Carbide, and Ir coated probes such as the **electri lever**, see also <http://www.asylumresearch.com/ProbeStore/APPLICATION?Entry=JustLooking#EFM,%20KPFM> 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 lose its ability to conduct.

8.3. 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 modification** 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. Some materials can be modified by biases to become either conducting or insulating with a current or a bias. 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.
- **Sample preparation**, other problems that can lead to poor reproducibility involve the circuit, rather than the tip or sample. This is more common when comparing results from sample to sample, but can also occasionally occur in multiple experiments on the same sample. This includes things like poor silver paste used to mount the sample, or poor contact being made with one of the connections in the circuit. This could be where a clip contacts a pad on the sample, or a break in the silver paint once it has dried. It is a good idea to double check this even if you think the circuit is fine.
- **Silicon Oxide**, this one really belongs in the previous item, but it is so common, it should be stressed. On Si samples, you need to scratch away the SiO₂ from the surface, then as quickly as possible apply conductive paint to the surface (Ted pella <http://www.tedpella.com/> leitsilber is very good) to insure you don't have a metal-oxide-semiconductor (MOS) contact in your circuit.

8.4. The Hardware

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

- ?,
- ?,

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.

8.5. Current imaging

A schematic of CAFM is shown in 8.1.

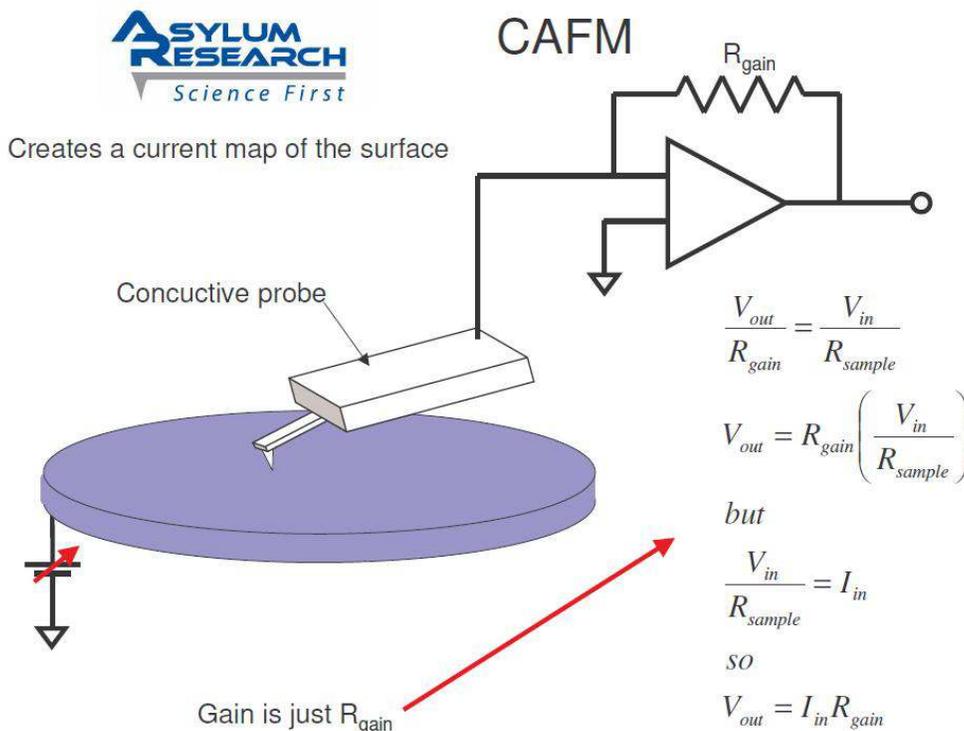


Figure 8.1.: Schematic of CAFM with relevant equations.

Current is passed through the tip and into a transimpedance amplifier, converting the current to a voltage.

Set the system to operate in Contact mode. Align the SLD, adjust the PD, then engage on the surface as normal for contact mode.

Select current as your channel type under the channel panel. For the dual gain orca you will have current 1 and current 2.

The ORCA bias can be found on the DoIV panel, and is called Bias Voltage.

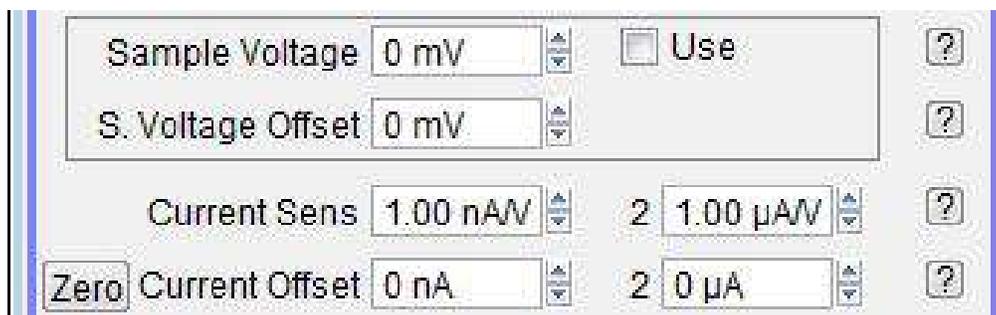


Figure 8.2.: These controls, found on the DoIV panel, control the bias to the sample while the sample is imaging.

Once you are imaging in contact begin raising the ORCA voltage until you see a current response from your sample.

You should start with a light force, but may find that you have to slowly raise the force to 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 AC240 based cantilevers, or other 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.

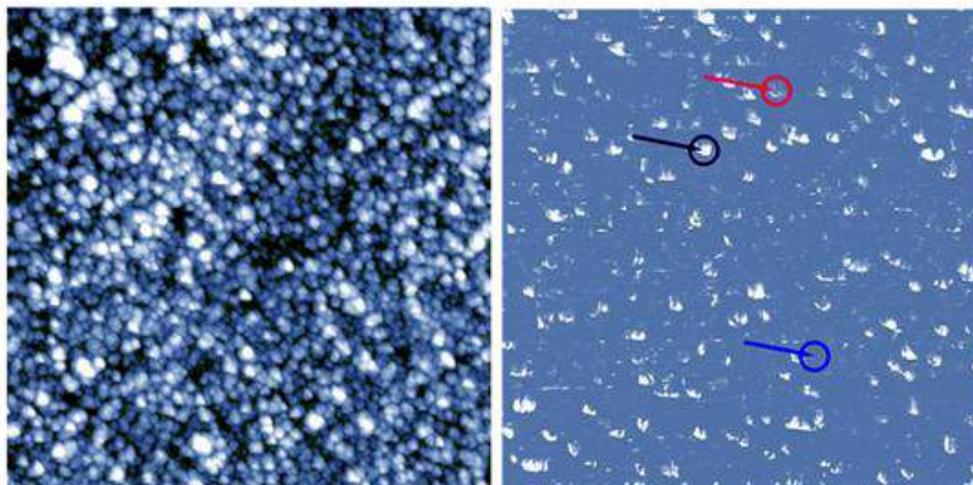


Figure 8.3.: This image is fairly typical of a thin film CAFM image. Note that tip shape artifacts are visible on the current image, but not on the topographic image. As the metal coating on the probe tip wears, or the tip is contaminated, these artifacts will be more prominent. Rocky Mountain nanotech now makes a solid Pt probe that does not suffer from this problem.

8.6. Current-Voltage (I-V) Spectroscopy

IV curves are done using the DoIV Panel which is opened from the menu *AFM controls* \triangleright *Do IV Panel*. This contains all of the controls that a user needs to position the tip over features, place the tip on the surface, and do IV curves with any of our existing functions, or any custom user functions that a user may wish to write.

A description of all of the individual listings on the Do IV Panel can be found in the help file embedded in the software by clicking on any question mark to the right of the control in question.

Before loading your sample, you may want to 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 higher for the CAFM measurement. This is because the number of conductive paths are much more numerous for the larger area probe than for a 20nm probe. Of course, this is not always the case, particularly for hydrophilic or metallic samples. Once you know the resistance of the current loop, you can estimate the bias you will need by using Ohm's law, $\text{Voltage} = \text{Current} \times \text{Resistance}$.

Before attempting to do IV curves on a sample, it is recommended that you first run I-V curves on a test resistor.

Running Current-Voltage curves generally involves first acquiring an image of your sample surface. For delicate samples that would be damaged or modified by contact mode, AC mode is generally used.

I-V curves are 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 most convenient way to design your I-V curve is to display the function on a graph before you execute it. To do this, click the 'display' button under 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. Adjust the amplitude and frequency of your drive wave, and observe the results in the display window. These will allow you to adjust the high and low voltage, and how fast the voltage will change. The amplitude gives you the maximum voltage above and below zero. One volt will produce a ramp that will start at zero, rise to one volt, then drop to negative one volt, then return to zero. A one Hertz frequency will complete one full cycle in one second.

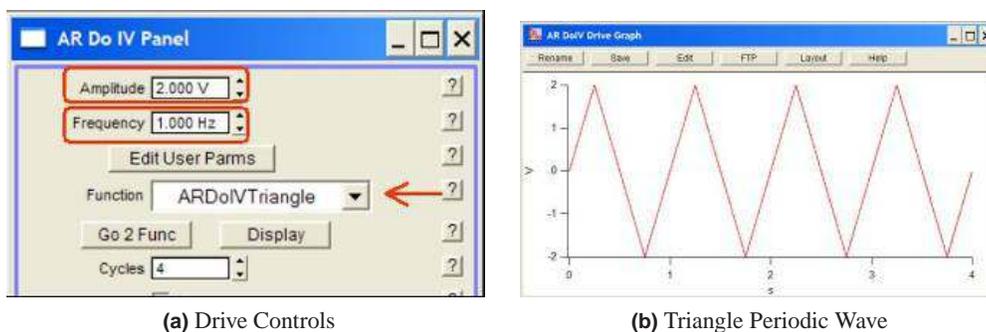


Figure 8.4.: Periodic functions can be selected as drive waves under the function drop down menu. There are two hidden arguments that can be displayed by clicking 'setup' at the bottom of the panel and checking the box to the right of arg3 and arg4. Descriptions of these are embedded in the help and can be accessed by clicking on the question mark to the right of the entry on the panel.

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 Optional Arg 3 and Optional Arg 4. These can be shown by clicking on the 'setup' button at the bottom of the DoIV panel. The Arg stands for argument and is different for different functions. Generally, Arg 3 is phase, and Arg 4 is offset. Phase is a ratio, where 1 is a 360° phase shift, 0.5 is a 180°, etc. The offset is in units of volts. Typing in -1 will produce an offset of -1V.

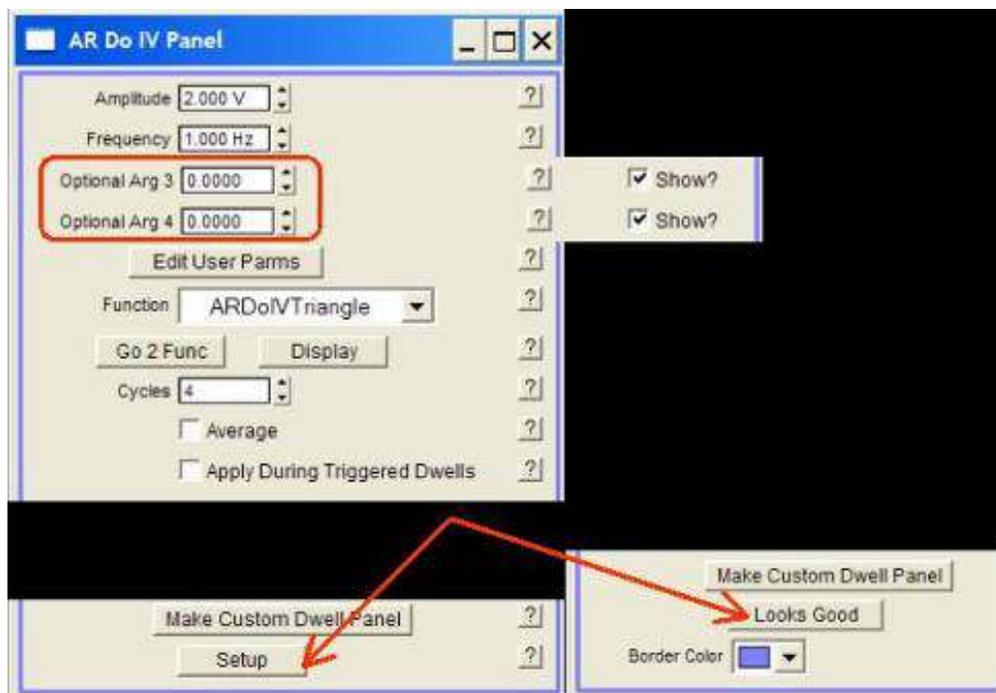


Figure 8.5.: [Setup on the DoIV panel] You can select the fields you would like to display using the setup button at the bottom of the DoIV panel.

Checking the 'average' box will average each cycle of the periodic function to produce a single I-V curve. Use caution with this function, as often the current will change drastically over multiple I-V cycles. These changes will be lost in the averaging. On stable curves, however, averaging is a good way to improve signal to noise.

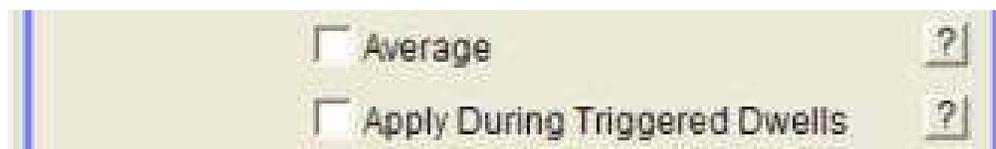


Figure 8.6.: [Triggered Dwell] You can attach an IV to the end of a force curve, or a force volume map by checking the 'applied during triggered dwells' box. Averaging can improve signal to noise, but caution should be used, as conductivity can change over time, thus skewing the average.

Checking the 'Apply during triggered Dwells' box will tie the IV curves to the force curve and force mapping functions. If this is checked, whenever the system does a force curve, it will automatically execute an IV curve during a dwell on the surface. This will allow force mapping to be applied in order to produce a current spectroscopy map.

For I-V curves at frequencies faster than 20 Hz, using a triangular or square drive wave is not recommended, as the construction of these waves would require DACs with much higher frequencies than 100kHz. To take IV curves above 20Hz, use a sine wave as the DoIV Function . Also, for high frequencies, you will need to increase the number of points in the wave. Do this by increasing the DoIV Points per Sec on the ARDoIV Panel. You can tell if you have increased this to a high

enough value by looking at the graph of the drive wave. Increase the number of points until the drive wave looks good on the display graph (see DoIV Drive Display).



Figure 8.7.: [Filtering] You can set the digital filter to as low as needed. below 50Hz artifacts may appear. Be sure your filter is set to at least twice the frequency of your drive wave. note that complex wave forms (non sinusoidal) require much higher bandwidths to drive and acquire.

To adjust the filter on the analog to digital converter that will collect the current signal from the orca holder, simply change the field labeled 'low pass filter' on the AR 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 100kHz ADCs is used.

It is also possible to write custom functions with the AR Do IV panel. Custom waves can use the user parm waves that are brought up by clicking on the 'edit user parms' button.

Now you are ready to pick points on the surface at which to perform I-V curves. To this, check the Show Markers on the AR Do IV Panel. This will allow you to see the spots you have selected for I-V curves. The buttons for this are conveniently grouped together on the Do IV panel:

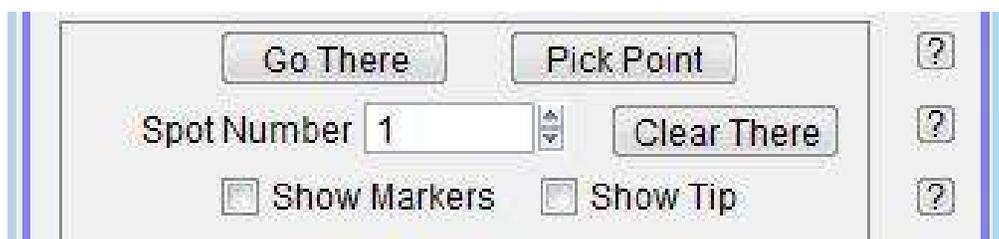


Figure 8.8.: [Go there] Use the pick a point, that's it, and go there buttons to move around the surface and collect IV curves. Use 'numbered markers' to graphically depict the selected locations on the AFM image.

The Pick Point button is the same for ORCA I-V curves as it is for force curves. First select the image you would like to use as your reference for the IV curve. If you are using an AC mode image to pick points on the sample, use the real time height image.

Note, there is an offset between the location of objects as detected by AC mode and when you are operating in contact mode from the difference in the bending of the cantilever. This can be large enough to miss smaller objects.

If you are using a current image, select the real time current image. Click the Pick Point to bring up a cursor that you will place on the point of interest. Once you have selected a point, the button should now read 'that's it'. You may then continue this for other points. Each time a point is

selected, the spot number will automatically increase by an increment, and a new spot may be selected.

Once you have the spots selected at which you want I-V curves, select the spot number you want your first I-V curve on 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 do your force curves.

8.6.1. 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.

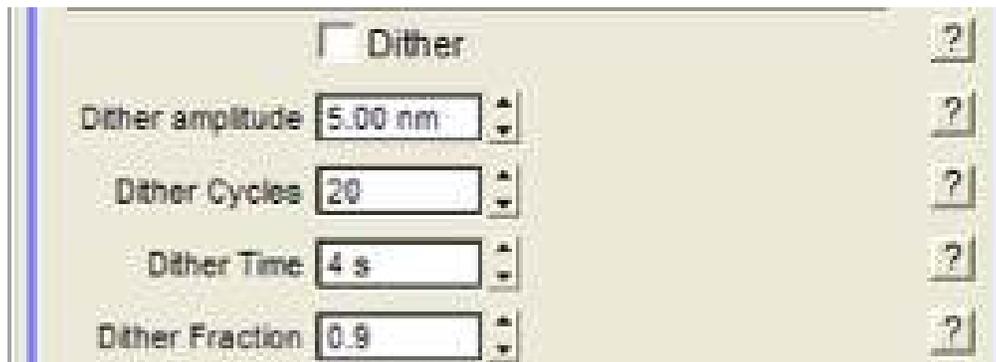


Figure 8.9.: [Dither] The dither function will create a spiral pattern the probe will trace out. This is intended to remove debris or to score the surface in order to get through oxides, thin films, or contamination on the sample surface.

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.

8.6.2. 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 masterful little piece of code 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.

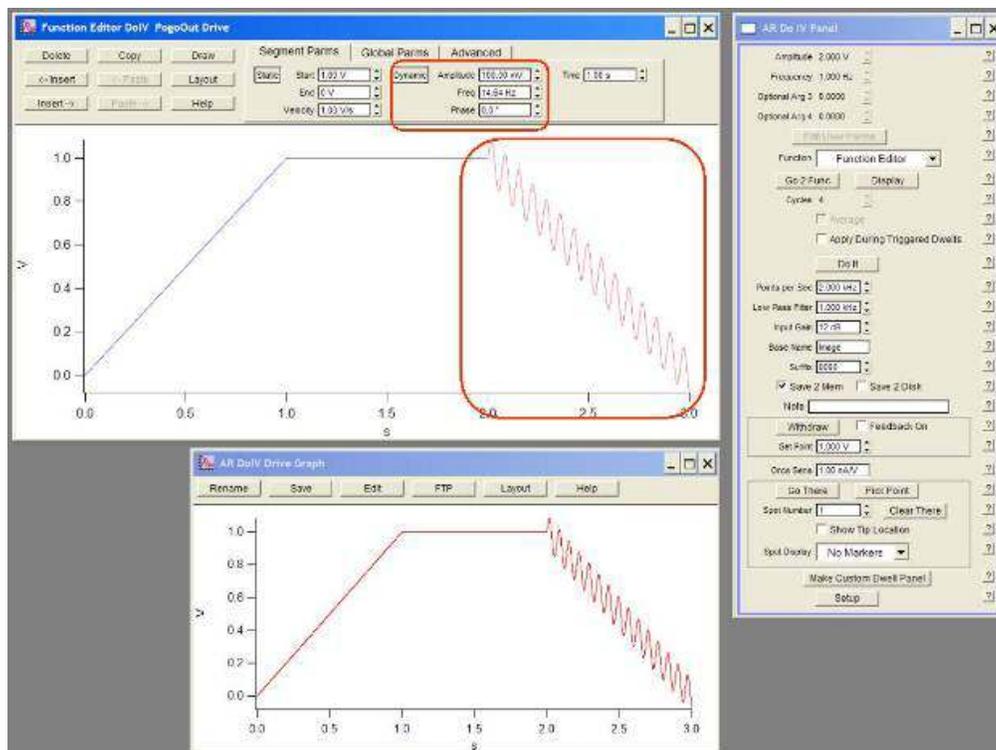


Figure 8.10.: [The Function Editor] The function editor can be used to make a variety of drive waves for current measurements. Insert new segments using the Insert button. Select different segments by clicking them. Each can be modified independently.

Under Global Parm's, 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.

Advanced allows you to save an entire drive wave to be used later.

8.6.3. Viewing I-V curves

I-V curves are treated like force curves. The actual data is treated as a dwell on a force curve. To display the curve, open the Force Display Panel . If you have just taken some I-V curves, then the

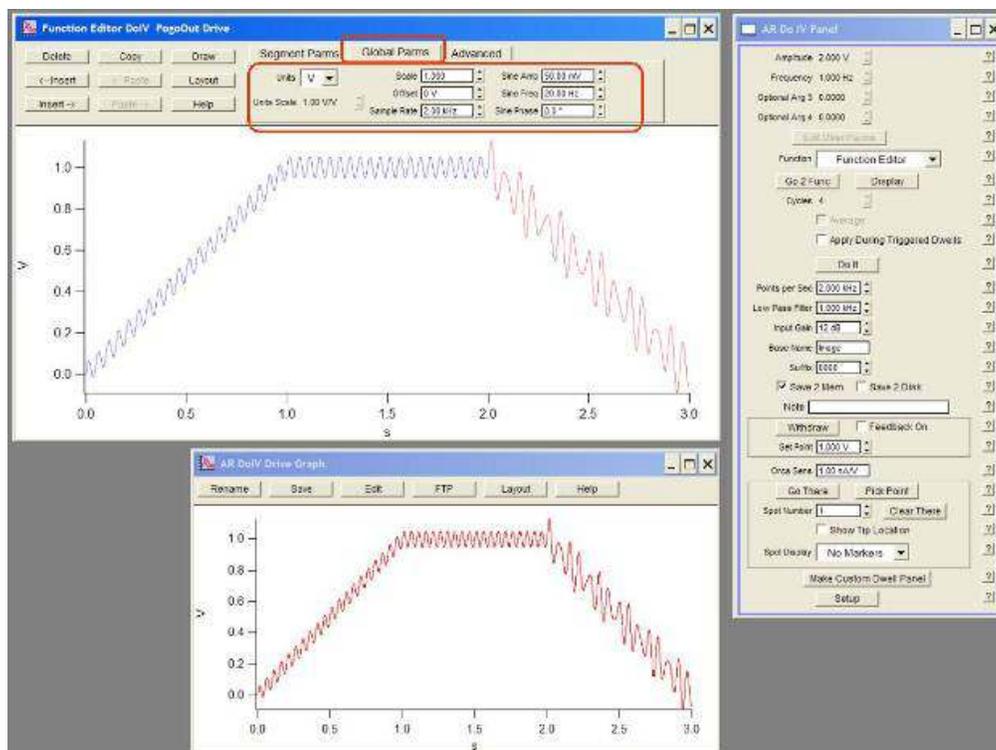


Figure 8.11.: [The Function Editor 2] Use the Global tab to modify all the individual segments at one time. Of particular use in this section is the Scale field. This allows you to scale up or down the entire wave function you have created. So if you had a custom wave from 0-1V with a dwell, scaling it by 5 would make it go from 0-5V.

force review panel should be looking at the right directory with the curves already loaded. If you are attempting to look at old data, then click the button at the bottom labeled 'load curves'. Then navigate to the folder that contains your IV curves.

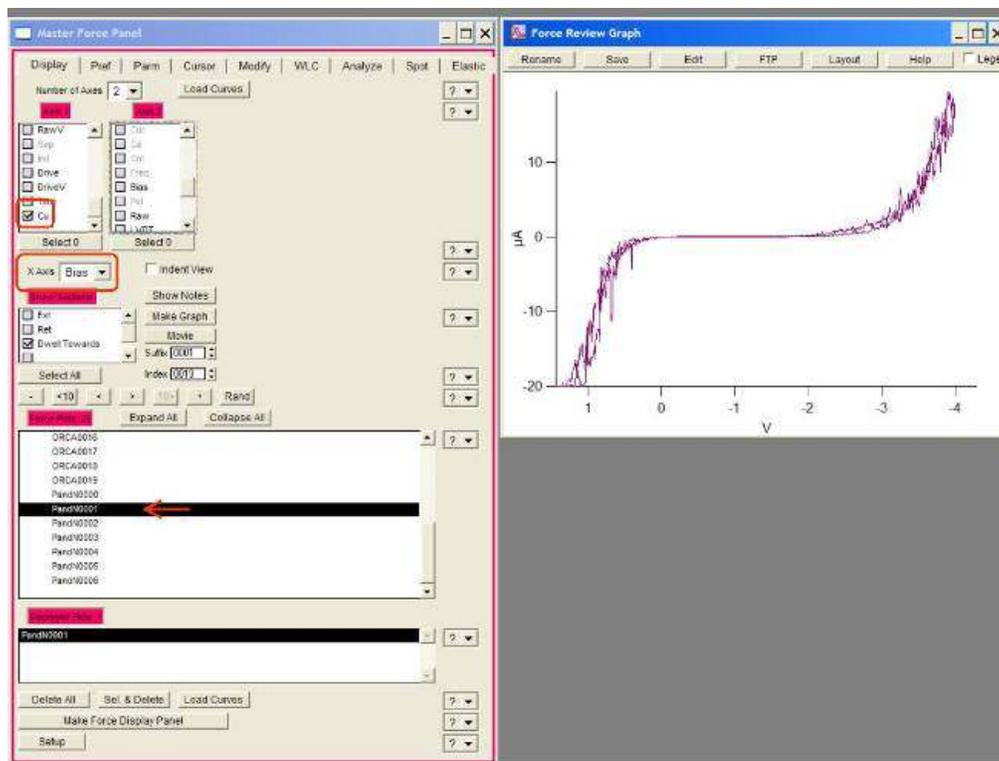


Figure 8.12.: [Current vs. Bias] To display IV curves, open the force review panel, view current (and current2 if you have a Dual Gain ORCA), Then set the x axis as bias, then select the dwell towards under the sections window. Click 'make graph' if the graph does not appear.

Next click on one of the curves to display it.

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).

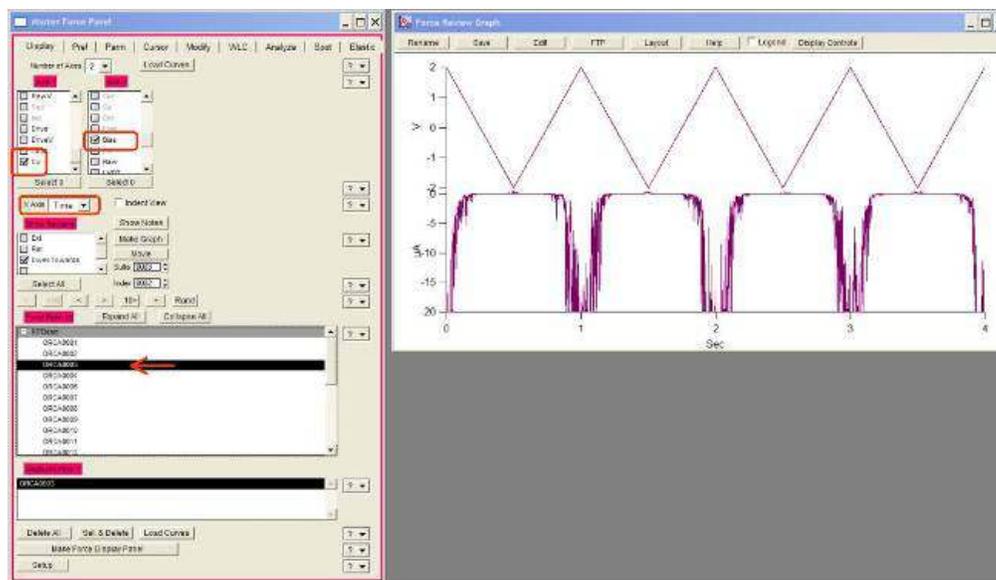


Figure 8.13.: [Current vs time] It is often useful to display the current vs time, particularly for samples that degrade or improve in conductivity with time. Si will oxidize as more current is passed through it if there is moist air near the surface.

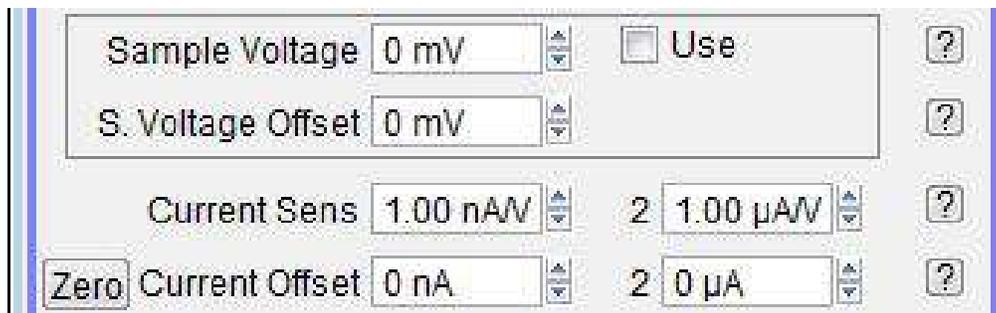
If you select the last IV curve in the list of loaded curves, then the force panel will automatically move to the next curve as it is taken so that you can view your I-V curves in real time. Simply select the last curve, then on the AR DoIV panel, click 'do it' and the force display panel will jump to the newest curve.

8.6.4. 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.



The screenshot shows a software interface for setting voltage and current offsets. It contains four rows of controls, each with a question mark icon to its right:

- Sample Voltage: 0 mV, with a checkbox labeled "Use".
- S. Voltage Offset: 0 mV.
- Current Sens: 1.00 nA/V, with a multiplier of 2 and a secondary value of 1.00 μ A/V.
- Zero Current Offset: 0 nA, with a multiplier of 2 and a secondary value of 0 μ A.

Figure 8.14.: [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.

9. Nap Mode

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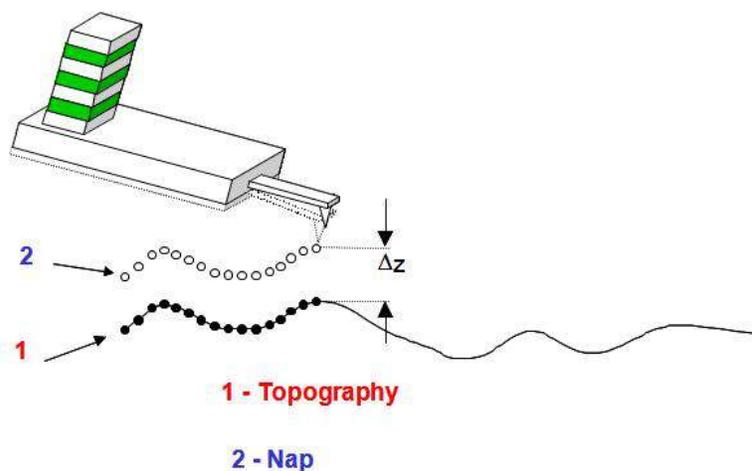


Figure 9.1.: During nap mode, the probe is first scanned across the sample surface, then raised to a delta height defined by the user. This allows for detection of long range forces between the tip and sample.

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 EFM, see [Chapter 11 on page 113](#). The nap panel can be accessed by selecting *AFM Controls* \triangleright *nap panel*(Ctrl + 9). The second is the nap channel panel, which allows the user to set display settings for the nap images. The Nap channel panel can be opened from the Nap Panel, or the menu *AFM Controls* \triangleright *nap Channel panel*.

Nap mode is a fairly simple technique. A 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. The second trace and retrace pass is the nap pass, and the method of this pass is set by the nap panel.

9.1. Nap Modes

The nap mode is set at the top of the nap panel, and can be set to:

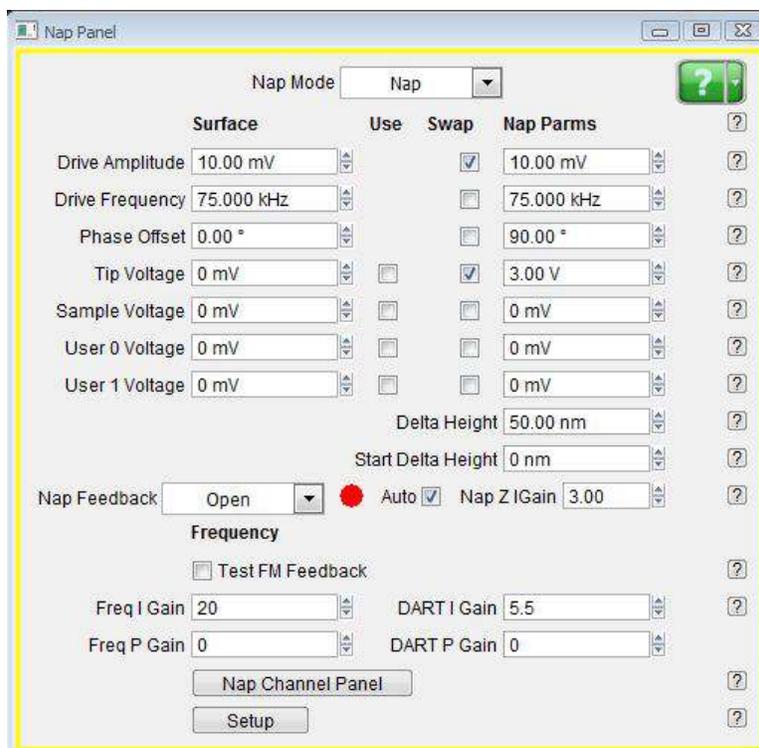


Figure 9.2.: Nap Panel, can be opened from the menu: *AFM Controls* ▷ *nap panel*(Ctrl + 9)

- 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 passes for every scan line as explained above see 9. 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 Nap techniques use this nap mode, see [Chapter 11 on page 113](#) and [Chapter 10 on page 106](#).

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.

9.2. Parameters

The following parameters can be switched, toggled between 2 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 4.3 on page 28](#)) 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 4.3.1 on page 31](#). This will require you to adjust the setpoints accordingly.

10. Scanning Kelvin Probe Microscopy (SKPM)

CHAPTER REV. 1578, DATED 08/29/2013, 22:20.

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10.1. Introduction

Scanning Kelvin probe microscopy (SKPM) is a technique that detects the potential difference between the probe tip and the sample. There are a few synonyms for this technique:

SKPM Scanning Kelvin probe microscopy, which will be used in this document, as it seems to be the most widely used descriptor for this technique.

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. The methodology is somewhat different, but the measured value is equivalent for both techniques. For clarity, this note will refer only to the microscopic technique SKPM.

10.2. Principles

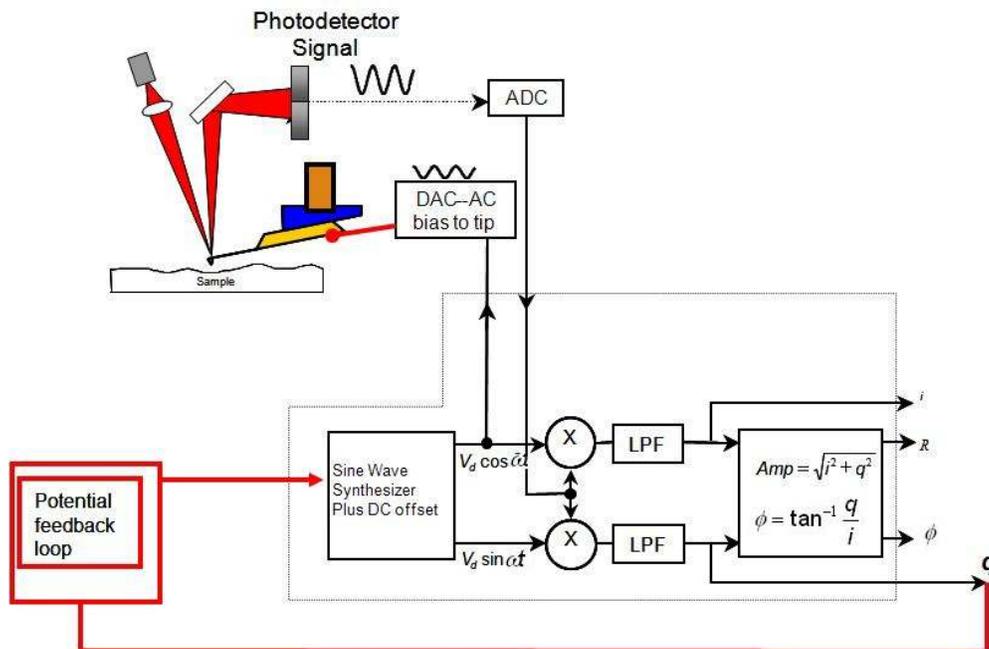


Figure 10.1.: Schematic showing how SKPM is done on Asylum AFMs. The probe is driven electrically with an AC bias. The potential difference between the tip and the sample causes the probe to oscillate. These oscillations are then canceled by a potential feedback loop. The voltage required to match the probe to the sample is captured as the surface potential channel in the 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 10.1 on page 107](#) 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:

$$F = \frac{1}{2} \frac{\partial C}{\partial z} V^2 \quad (10.1)$$

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}).

$$V = (V_{DC} - V_{sp}) + V_{ac} \sin(\omega t) \quad (10.2)$$

We need to square this to go into [Equation 10.1 on page 107](#).

$$V^2 = (V_{DC} - V_{sp})^2 + 2(V_{DC} - V_{sp})V_{ac} \sin(\omega t) + V_{ac}^2 \sin^2(\omega t) \quad (10.3)$$

and from

$$\sin^2 \theta = \frac{1}{2} (1 - \cos(2\theta)) \quad (10.4)$$

We have

$$V^2 = (V_{DC} - V_{sp})^2 + 2(V_{DC} - V_{sp})V_{ac} \sin(\omega t) + \frac{1}{2}V_{ac}^2 (1 - \cos(2\omega t)) \quad (10.5)$$

Which shows the important point, that there are DC, $1\omega t$ and $2\omega t$ components to the signal. Putting this into 10.1 and a little rearranging gives us:

$$F = \frac{1}{2} \frac{\partial C}{\partial z} \left(\left[(V_{DC} - V_{sp})^2 + \frac{1}{2}V_{ac}^2 \right] + 2[(V_{DC} - V_{sp})V_{ac} \sin(\omega t)] - \left[\frac{1}{2}V_{ac}^2 \cos(2\omega t) \right] \right) \quad (10.6)$$

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.

10.3. Methodology

The probes need to be conductive, insulating probes will not work, as the bias needs to be applied to the tip. A good first lever to try would be the [electri lever](http://www.asylumresearch.com/ProbeStore/APPLICATION?Entry=JustLooking#EFM,%20KPFM), see also <http://www.asylumresearch.com/ProbeStore/APPLICATION?Entry=JustLooking#EFM,%20KPFM>. This technique uses a two pass method called nap mode. The first pass is used to determine the topography of the surface, and is done exactly like a standard AC mode scan line. The second pass the tip is raised above the surface and is used to find the surface potential, see Chapter 9 on page 103 for more details.

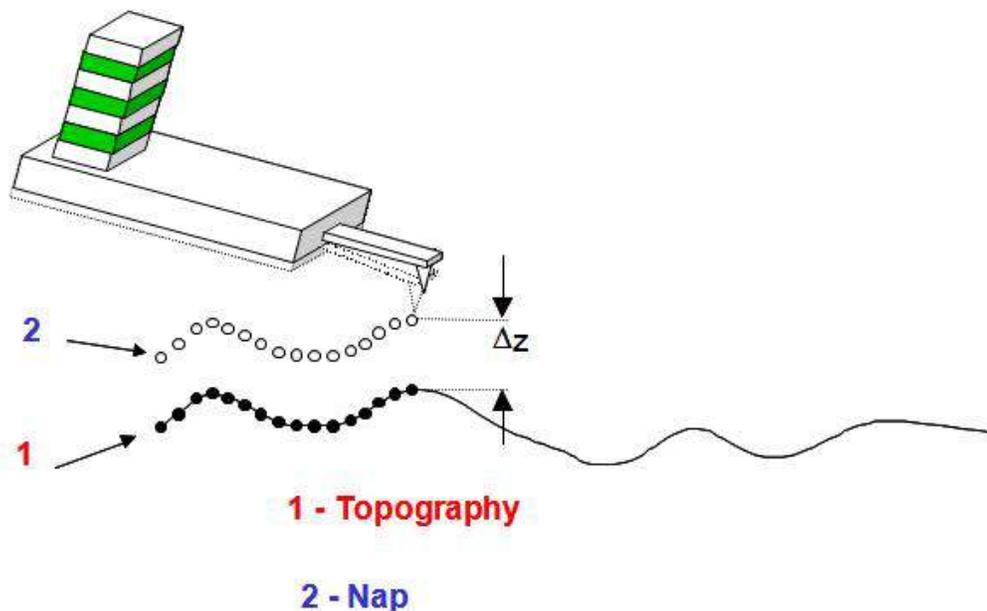
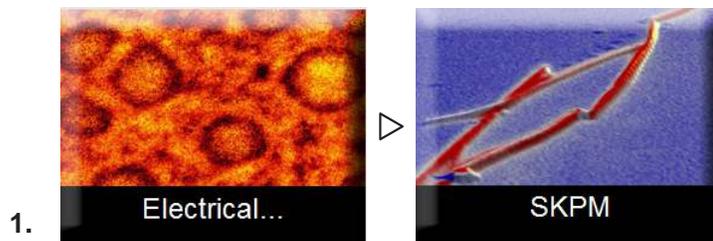


Figure 10.2.: Schematic of nap mode. For each scan line, the system captures the topography. The system then retraces that topography on the same line in order to keep a constant distance from the sample surface.

10.4. How to Guide

Step by step implementation of SKPM imaging on the MFP-3D

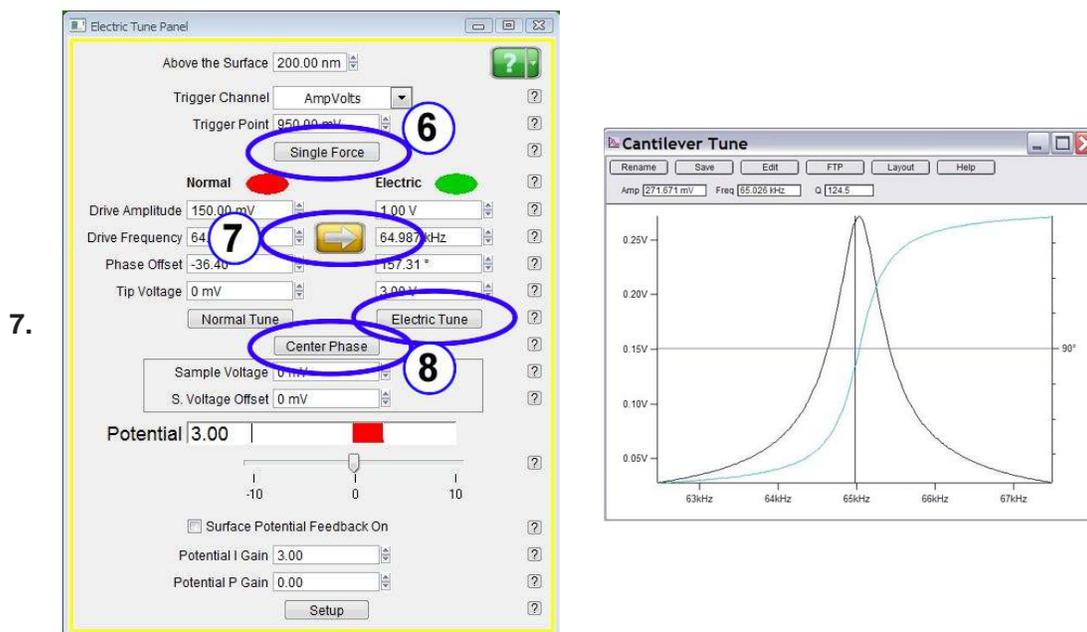
Note that this instruction set assumes the user is familiar with standard AC mode imaging, see Chapter 3 on page 18.



Select SPKM Mode:

- The ModeMaster Panel appears when you first open up the AFM software. Select *Electrical* \triangleright *SKPM*
2. Load the sample. Insulating and semiconducting samples need not be grounded, but metal samples should be grounded to the scanner plate to avoid arbitrary offsets in the potential that might mask the work function between the tip and the sample.
 3. Load a conductive AC mode probe into the AFM head.
 4. 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%.

- Open the electrical tune panel by clicking *AFM Controls* > *Other* > *Electric Tune Panel* menu (this menu is only enabled when in AC mode). This panel allows the user to find the resonance frequency and phase of the probe when it is driven electrically. This phase will be considerably different than that of the same probe driven mechanically with a piezo, and it very important for the correct determination of potential.
- Engage on the surface in AC mode as if doing standard AC mode AFM.



- Electric tune panel with the single force, electric tune and Center Phase buttons highlighted. The graph on the right is a properly electrically tuned cantilever. At the top of the Electric Tune panel set the height above the surface to 300nm. Set the trigger to AmpVolts, and the trigger point to 800mV. Then click the button marked *single force*. When the force curve is done the tip will be positioned just above the surface.
- Click on the arrow button to copy the drive frequency over to the electric tune frequency.
 - Now click the 'electrical tune' button. This will sweep an AC bias to the probe and show the response in the tune graph. The resonance frequency will be very close to that of the electric tune. Be sure that the tip voltage field under the Electric column reads 3.0V 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 7 on page 110.

Note

The AFM software assumes that the tip is at a positive potential relative to the sample. If this assumption is wrong, 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 3V. The easiest way to see if the sample has too high a voltage is to tune everything as described here, then collect an image. Normally, if the tip is at too high a potential, the Nap surface potential channel will be railed either at positive or negative 8-10 V (depending on the drive voltage). If this happens set the tip bias to a higher voltage and try again. Repeat if necessary.

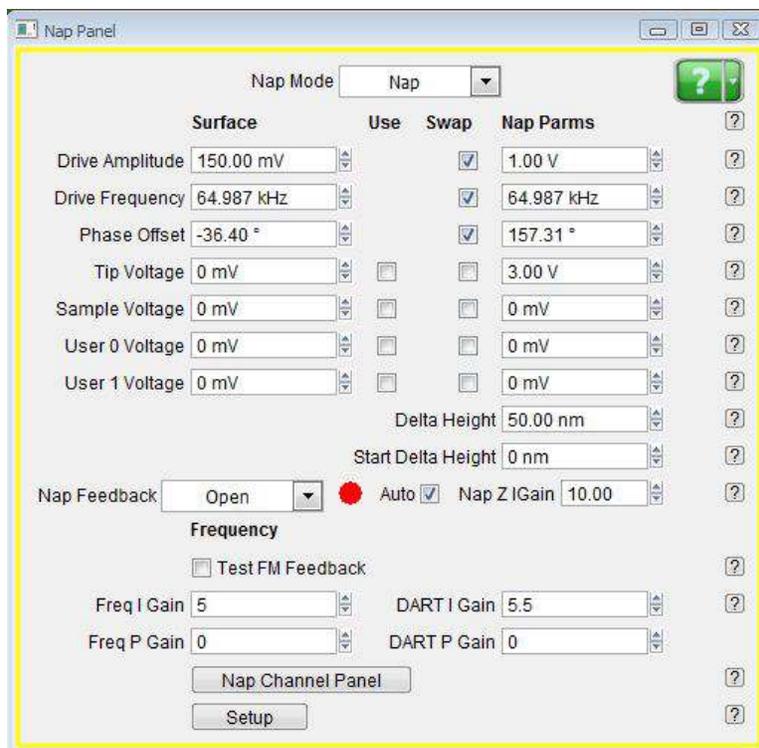
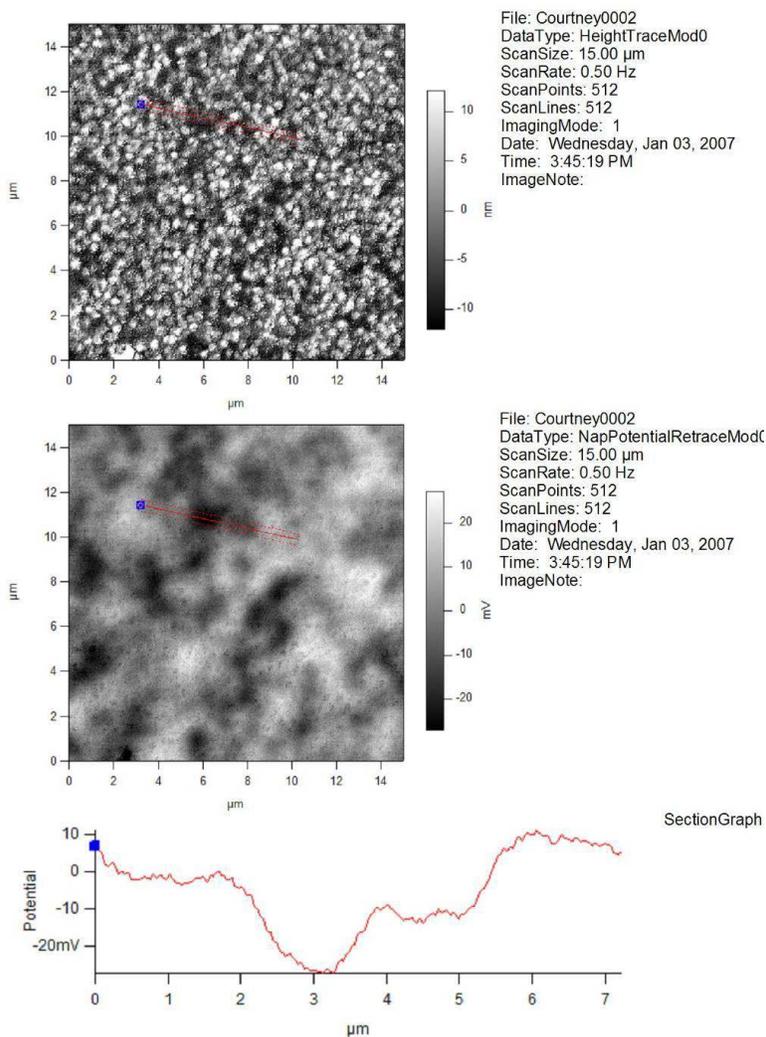


Figure 10.3.: Nap Panel configured for a surface potential scan. The numbers in the field may vary depending on your sample.

10. Open the nap panel from the main menu bar by selecting *AFM Controls* ▸ *Nap Panel* (Ctrl+9), see Chapter 9 on page 103.
11. Select Drive amplitude, Drive Frequency, and Phase Offset under the swap column. See Figure 10.3 on page 111
12. Set the nap mode to *nap*.
13. Set the delta height to 40nm. This will be lowered later if you want to try to get better lateral resolution. Because the reference height is based on the zero point of the cantilever oscillations, and because the potential feedback loop will keep the amplitude at zero during the potential scan, it is actually possible to enter a value lower than 0 nm as a delta height. If too low a value is entered, the tip will strike the surface, and you will have a poor image.
14. Set the Start Delta height to 40 nm. If you are imaging a sticky sample, this might need to be raised to get the tip off of the surface for the nap scan.
15. Click on Nap Channel Panel button at the bottom of the nap panel.
16. Make sure Potential as a data channel in the master channel panel (Ctrl + 7). Selecting this automatically turns on the potential feedback loop when scanning. Be sure that the *nap* potential channel (set on Nap Channel Panel) has Real time and save plane fit set to **none**.
17. On the main tab of the master panel (Ctrl + 5), set your scan speed and scan size to reasonable values for the sample you are imaging.
18. Start the scan.

19. Look at the surface potential 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 is unstable or very chaotic, try lowering the gains quite a bit. A good starting value is around 1 for integral and 0.5 for proportional.
20. To improve lateral resolution slightly, try lowering the delta height. It might even be possible for this number to be negative, since the zero point is based on AC mode imaging, where the cantilever is oscillating.



Sample Courtesy Andrew Lyon, Ga Tech

Figure 10.4.: Height (top) and Surface Potential (middle) image of a three-layer microgel sample. The section (Bottom) shows three discrete potential levels on the sample, which is mostly likely attributable to incomplete coverage of the three different microgel layers. Sample and images courtesy of C. Sorrell and A. Lyon, Georgia Institute of Technology.

11. Electrostatic Force Microscopy (EFM)

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11.1. 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:

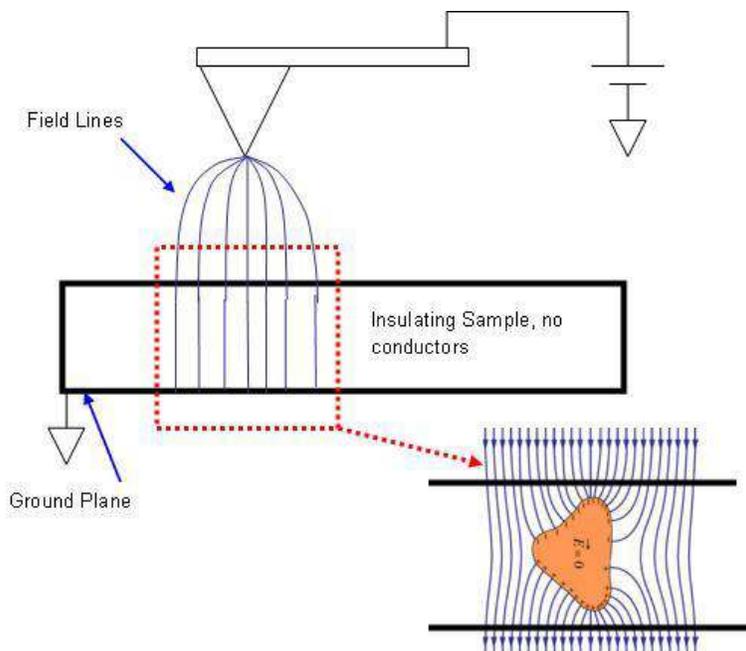


Figure 11.1.: This schematic shows how the introduction of a conductor into the electric field between the tip and sample can effect the field lines. The shift in the gradient due to the presence of the conductor causes a shift in the resonance frequency of the cantilever and therefore a shift in the phase and amplitude, which are then collected as the EFM data. The phase indicates whether the shift is attractive or repulsive. Attractive forces show a shift in the phase to higher than free air value. Repulsive forces shift the phase below the free air value.

When a conductor is introduced into the electric field (see [Figure 11.1 on page 114](#)), 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 11.2 on page 115](#)).

11.2. Theory

Theory

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

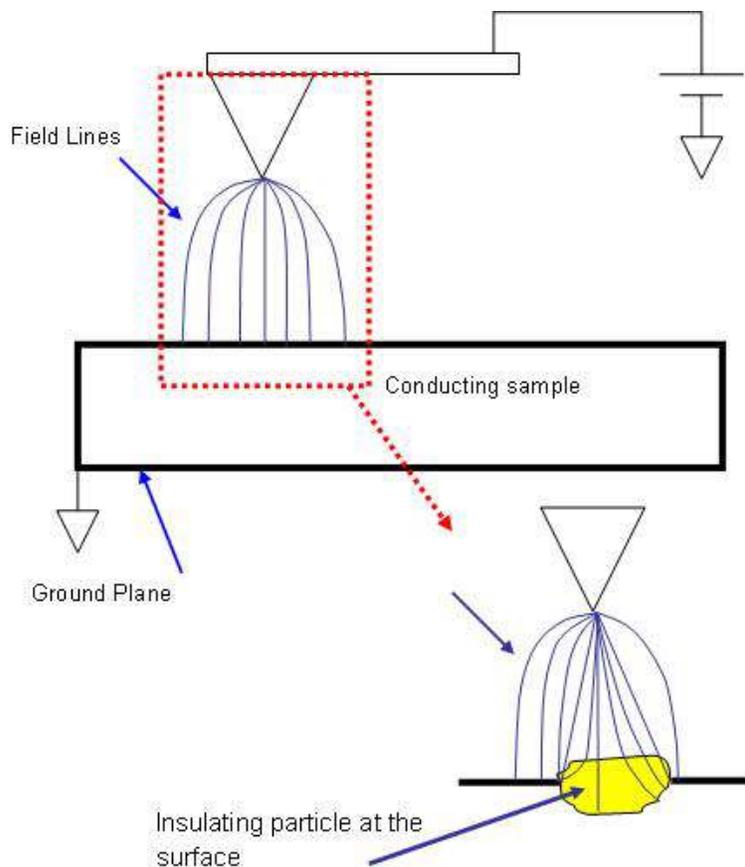


Figure 11.2.: Effects of introducing an insulator into a metal sample. EFM is generally not suitable for this sample configuration, as the field lines show little shift. Any shift would be difficult to discern from the changes in topography.

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

$$F = \frac{1}{2} \frac{dC}{dZ} V^2 \quad (11.1)$$

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 11.1 on page 114](#), before the conductor is introduced into the insulating sample, the capacitor can be considered to be the tip to the ground plane, with two dielectrics between. One is the sample, and the other is the air between the tip and the sample (of course this is much more complicated if you consider the contamination layer). When the conductor is introduced, you now have essentially two capacitors: the tip to the conductor and the conductor to the ground plane. Areas where there is no conductor will have a different dC/dV than areas where conductors exist.

It is also possible to get contrast due to the presence of charge, but this is normally very faint compared to the contrast due to the capacitor geometry under the tip. For samples that have varying charge, a much better technique to use is scanning Kelvin probe microscopy (SKPM), described in detail in [Chapter 10 on page 106](#).

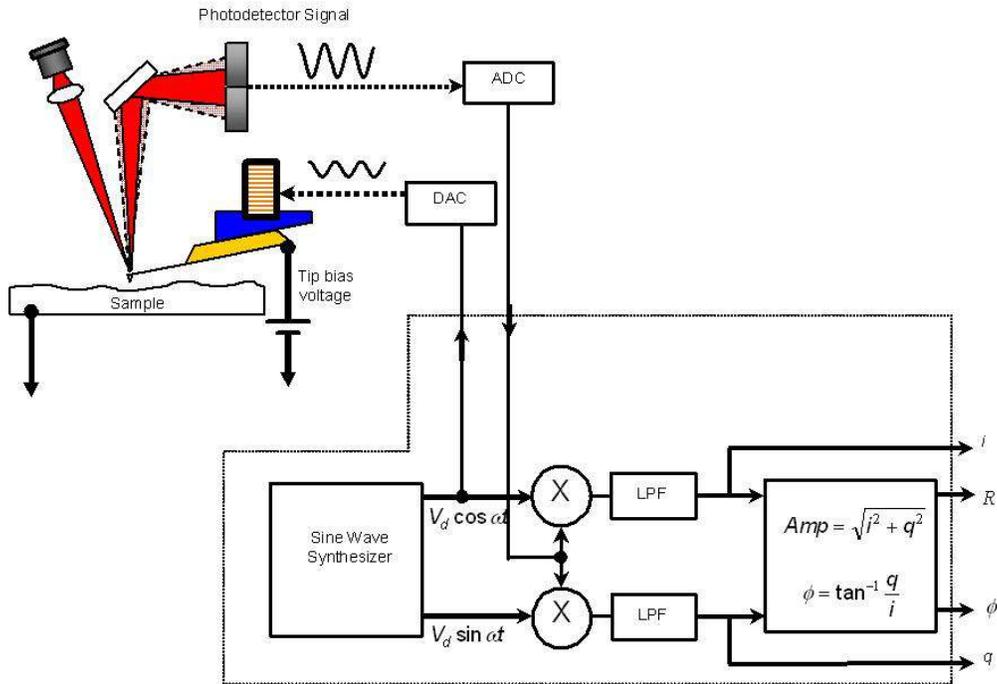


Figure 11.3.: Schematic of Electric Force Microscopy. The Probe is oscillated both during the main scan and during the nap scan. During the nap scan, the probe is lifted off of the surface, and a bias applied between the tip and sample to show the location of areas that are conductive or strongly charged.

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 9.1 on page 103. This is done to maintain the separation between the tip and the sample constant while collecting the EFM data. You note from equation 11.1 that the force is dependent on changes in Z .

11.3. 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 ??.

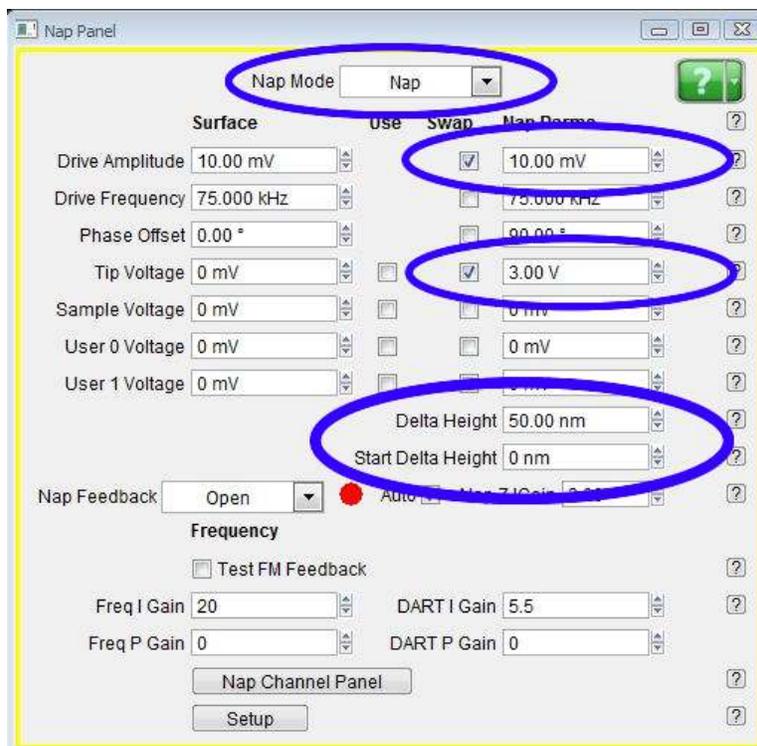
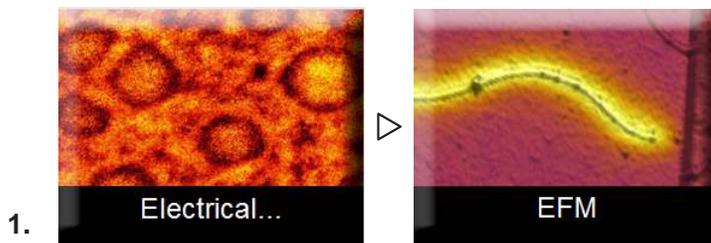


Figure 11.4.: Here the Nap panel is set for EFM operation. The relevant fields have been circled. The Use column in the middle left has check boxes that, when checked, cause that parameter to be used during the main scan. The swap column is has check boxes that, when checked, allow those fields to be used during the parm swap/nap scan.

11.4. How to Guide



Select SPKM Mode:

- The ModeMaster Panel appears when you first open up the AFM software. Select *Electrical* ▷ *EFM*
2. 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.

3. 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.
4. Open the nap panel (Ctrl + 9), then open the nap channel panel.
5. 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.
6. 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.
7. 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 AmpInvol). This value should be linearly higher if the free amplitude is higher. Figure 11.4 on page 117 shows the nap panel with typical settings for EFM.
8. 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.
9. 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 3.
10. 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.
11. 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.
12. 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.

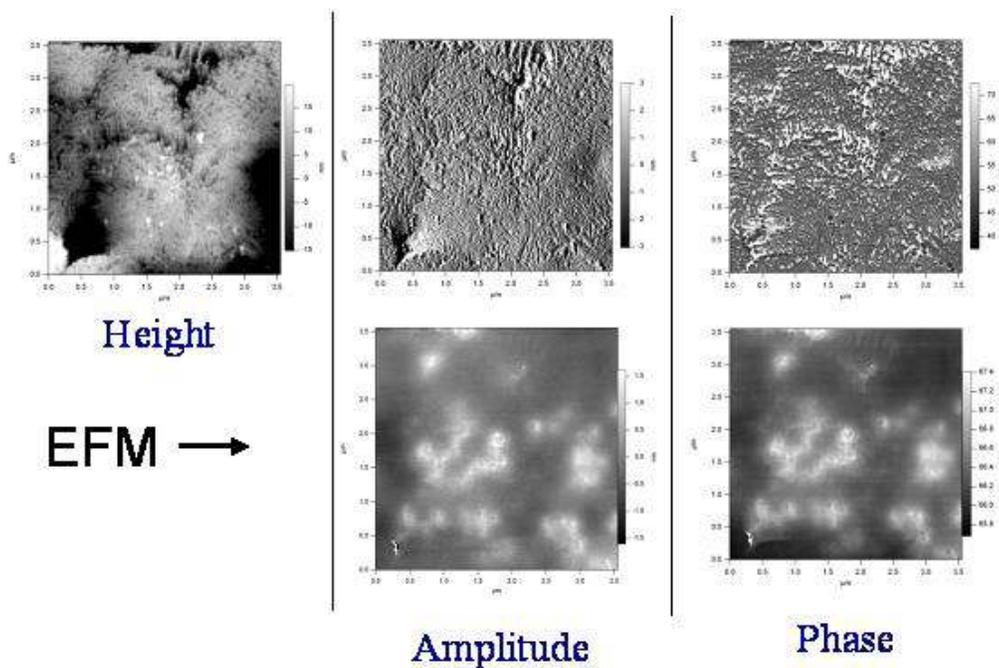


Figure 11.5.: EFM image of polypropylene with carbon black particles (CB) imbedded into it. The areas of contrast in amplitude and phase are due to the presence of CB particles either at or below the surface of the polypropylene.

12. AM-FM Viscoelastic Mapping

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12.1. Overview

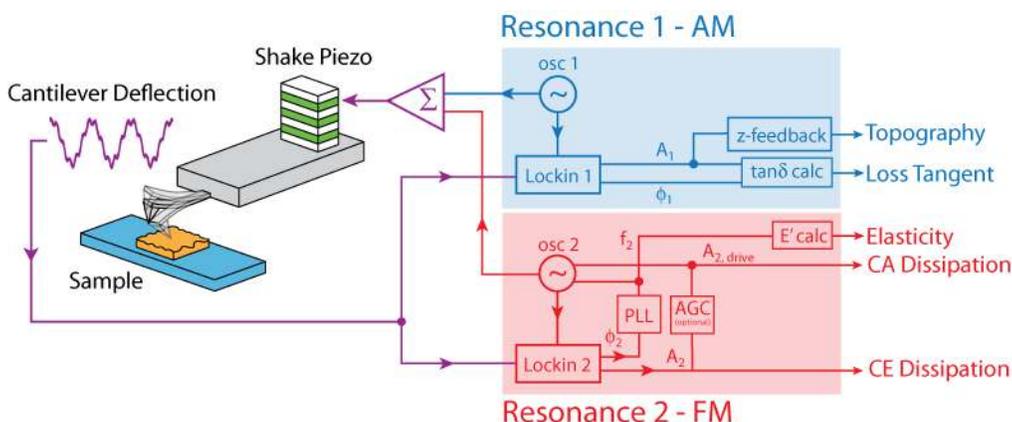


Figure 12.1.: AM-FM Mode. The first mode amplitude is controlled to create a topographic image of the sample (blue). The second mode is used to calculate elasticity and tip-sample stiffness (red).

AM-FM imaging combines the features and benefits of normal tapping mode (also called AM) with fast scanning and quantitative, high sensitivity Frequency Modulation (FM) mode. When operating in standard AM-FM mode, there are three feedback loops. The topographic feedback on the cantilever oscillation amplitude operates in normal tapping mode, providing non-invasive, high quality imaging. The *second mode* drive frequency is adjusted to keep the phase at 90° , on resonance. This resonant frequency is a sensitive measure of the tip-sample interaction. 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 final feedback is optional, but when enabled, maintains a constant second mode oscillation amplitude which is used to calculate the tip-sample stiffness. This parameter is called Drive setpoint in the software.

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

$$K_2 = \left(\frac{\omega_2}{\omega_1} \right)^2 K_1 \quad (12.1)$$

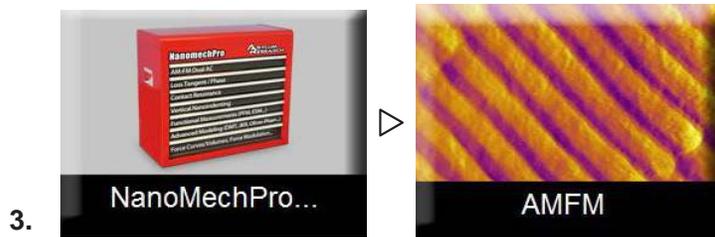
As with conventional FM mode, AM-FM is a quantitative technique in which the conservative and dissipative tip-sample interactions can be separated. However unlike FM, the feedback loop in AM-FM is completely decoupled from the FM loop, both greatly simplifying and stabilizing operation.

AM-FM imaging is possible on the MFP-3D and Cypher AFMs. In both cases a specialized AM-FM Viscoelastic Mapping Cantilever Holder is required. Depending on your model of AFM, please refer to:

- ?, .
- ?, .

12.2. How to Guide

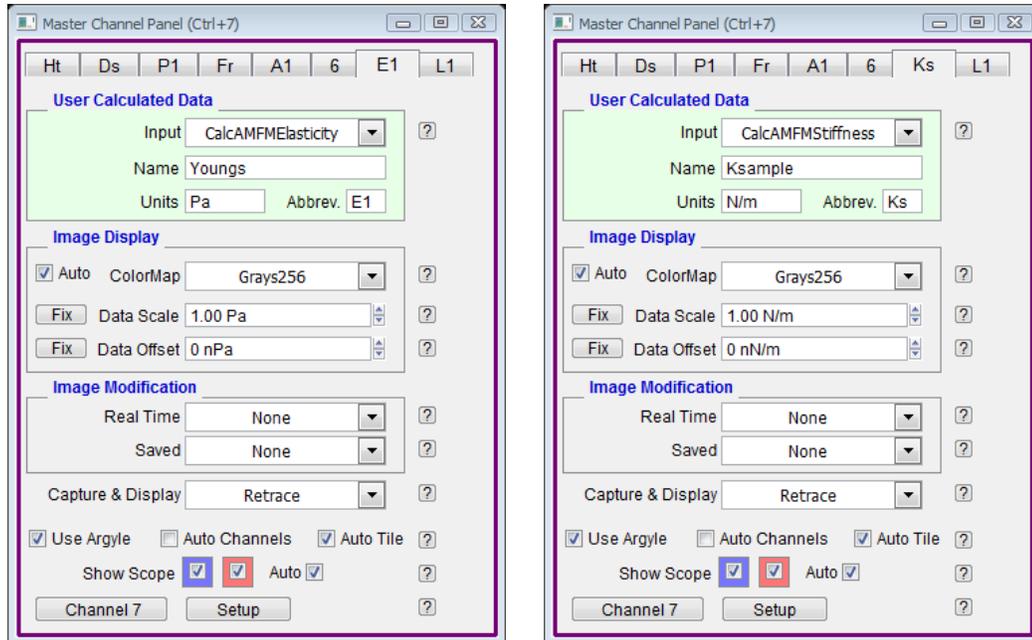
1. For the most recent features use the latest software version. If you are a registered user on the Asylum Research Forum, you can download the software at <https://support.asylumresearch.com/forum/content.php?4-Software>.
2. You can also view the AMFM video on our forum: <https://support.asylumresearch.com/forum/content.php?488-AM-FM>



Select AM-FM Mode:

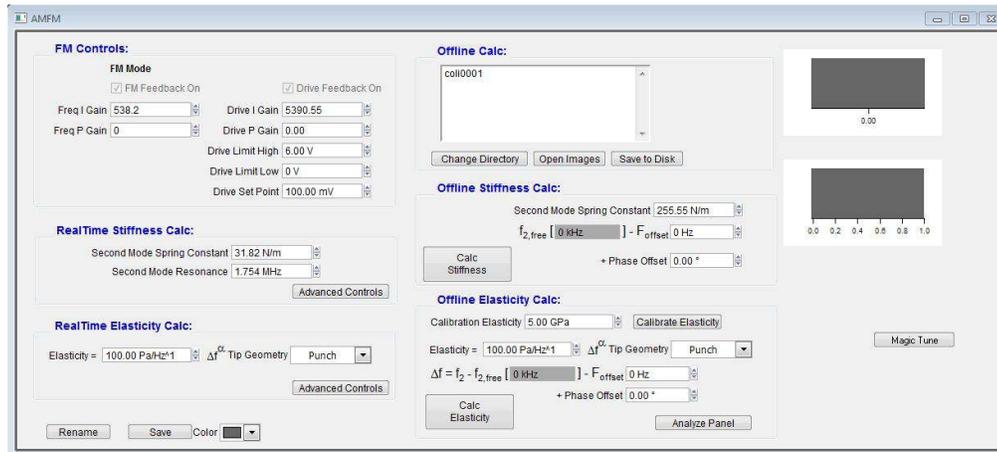
- The ModeMaster Panel appears when you first open up the AFM software. Select *NanoMechPro* ▷ *AMFM*
4. The usual master panel and master channel panel will appear.

5.



In version 120804, the last 2 tabs of the master channel panel are for user calculated channels (which are used by AM-FM). You can set the calculation to be either stiffness or Young's modulus, as shown above.

6.



Note the appearance of the AMFM Panel:

- This panel includes realtime gains parameters, realtime Stiffness/Elasticity factors, offline Stiffness/Elasticity calculations.

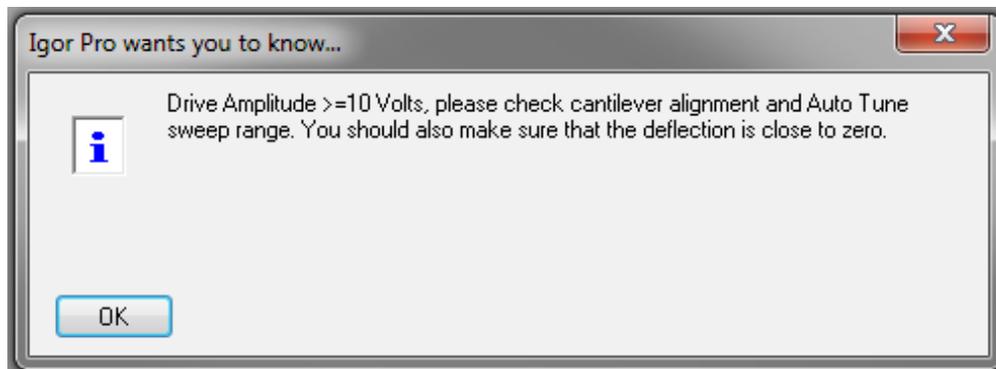
7. In the master channel panel, the default channels are Height (Ht), Dissipation (Ds) or Amplitude2 (A2), Phase1 (P1), Frequency (Fr) and either Stiffness (Ks) or Elasticity (E1).

- If you choose Dissipation instead of Amplitude2, the third feedback loop will be enabled that keeps Amplitude2 at a setpoint. In the Step 6 on page 122, it is initially set at 25 mV. The best results are from keeping this value as small as possible, while still maintaining good signal to noise. Typical values are 20-50 mV. More than that will result in large higher mode amplitudes which may perturb the cantilever dynamics.

8. Recommended cantilevers for air: AC240TS, AC160TS. Select an appropriate cantilever depending on the sample (i.e. estimated sample stiffness). Use: AC240TS (use 3rd mode for second resonance, typically ~1.2MHz) or AC160TS (use 2nd mode for second resonance, typically ~1.8MHz). Right now the AC160TS cantilevers appear to work the best so start with them and switch to AC240TS if you need a softer cantilever.
9. If you plan on measuring the tip-sample stiffness (K_s), you should first calibrate your cantilever using the standard protocol, i.e. use an infinitely hard substrate (glass, mica, silicon) and the built in thermal calibration method. Then switch to your sample and tune the cantilever.
 - In the AMFM Panel, the *second mode spring constant* is listed under *realtime stiffness calc.* After tuning the 1st and 2nd resonances the software automatically calculates the second mode spring constant using equation 12.1.
10. Auto tune the cantilever on the first and second resonances.
 - Check Auto Tune Low and High for both first and second resonances – adjust accordingly. Note: The *target amplitude* is 2 V and *amplitude setpoint* is 800 mV. This will ensure that you will be imaging in repulsive mode, i.e. phase $< 90^\circ$, ideally $< 60^\circ$. Since you are selecting a frequency on the peak, set the *target percent offset* to 0%.
 - Make sure that your second mode free amplitude is significantly lower than your first mode free amplitude (i.e. < 25 mV).
 - If you get an message seen below, check the switch on the back of the holder. Move it to the other side and retry the tune.
 - It has been found that more reproducible results are obtained when you do your tunes the same height above the surface. We recommend you do triggered force plots with a consistent force distance (500 nm seems to work well) before each retune.

Note on Spot Position You want the sensitivity of the optics to be as high as possible for the second mode. To do this, after you have tuned, you should move the spot as far out onto the end of the lever as possible. Observe the higher mode amplitude while you do this and pick a location where you've maximized A2. You may pay a small price in first mode sensitivity, that is ok.

11.

**Autotune message:**

- If your MFP3D cantilever holder has a switch on it, this message may mean that switch is in the wrong position.

12. Engage on the surface.

13. Do a triggered force plot with a force distance of 500 nm to set the distance above the surface.

14. Retune the both modes of the cantilever. Make sure you are on the peaks. Center the phase at 90°. Use the *Nail the Phase* button  to get $\pm 0.5^\circ$.

Note: For accurate measurements, it is very important to be on resonance and the phase as close to 90°.

15. Re-engage on the surface and begin scanning the surface. It is also recommended that you scan at 90° due to the dimensions of the AC240 tip.

16. It is important to image in **repulsive mode**. If the phase is mode hopping between attractive and repulsive, it isn't going to work well. Initially the *amplitude setpoint* is 40% of the free amplitude but you may need to change this to keep the AFM in repulsive mode. Start small and get larger i.e. gradually increase the *drive amplitude* to maintain a completely repulsive regime.

17. Adjust the parameters to optimize topography, frequency and, if collected, dissipation. For best results adjust these channels in the following order:

Topography (Ht): Most likely the tip is in repulsive mode so you don't need to adjust the setpoint. But to improve tracking you may need to adjust the integral gain – typically, increase the gain until you see ringing then decrease it just until the noise disappears.

Frequency (Fr): Adjust the Freq I Gain in the AMFM Panel until the Trace and Retrace lines are overlapping in the scope trace and there is contrast in the Fr image.

Dissipation (Ds): Adjust the Drive I Gain, also located in the AMFM Panel, to improve tracking and contrast.

Note: The Frequency and Drive gains can have enormous dynamic range, from ~10 to 100,000. Don't be afraid to turn these up. If you start to see ringing in either the amplitude or frequency channels, just back down the gains a little. This is only causing instabilities in the small amplitude higher mode drive frequency and should have essentially no effect on the first mode and should not damage the tip.

Note: See comments above for setting gain values. Also, Mick suggests the following approach:

“I’ve found it’s often easier to kill the drive loop (by setting the gains to zero), tune the frequency loop until it’s just short of ringing, then increase the drive loop gains steadily (reducing the frequency loop gains a little, as necessary).”

18. To display accurate values for the elasticity channel, adjust the *Elasticity factor*, located under *RealTime Elasticity Calc* in the AMFM Panel.

- First, select a reference sample with known modulus.
- Scan that sample, and load the image into the AR software.
- Enter your known Young’s modulus into the *Calibration Elasticity* under *Offline Elasticity Calc*.
- Select the geometry of the tip.
- Click on *Calibrate Elasticity*.
- Place your “unknown” sample on the AFM and scan it without changing the elasticity factor. Assuming that the tip shape has not changed the calculated modulus should be a fairly accurate estimation of your sample modulus.

Note: Ideally, select a reference sample whose modulus is close to your experimental sample.

13. iDrive Imaging

CHAPTER REV. 1573, DATED 08/27/2013, 16:28.

USER GUIDE REV. 1578, DATED 08/29/2013, 22:20.

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13.1. Theory

13.1.1. 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 13.1 on page 127. 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} :

$$\vec{F} = \vec{i} \times \vec{B}l \tag{13.1}$$

$$F = ilB \sin \theta \tag{13.2}$$

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

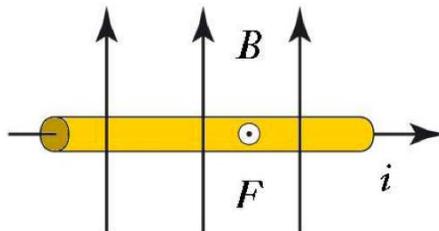


Figure 13.1.: The Lorentz Force F is orthogonal to both current i and the magnetic field B and is pointing out of the page.

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 cantilever¹. 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 cantilever². This unwanted acoustic energy often manifests itself as bumps and dips in the cantilever tune³. Because the current loop is made by the cantilever itself, the drive is completely localized. This means that the tunes are smooth and that other parts of the microscope mechanics are not being excited.
- 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 13.2 on page 128, Insulin Fibrils) and even allows advanced techniques such as Q-control.

¹?, .
²?, .
³?, .

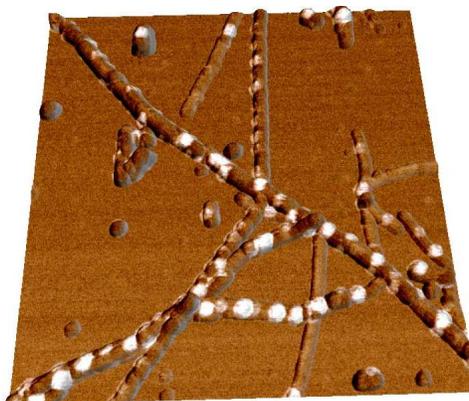


Figure 13.2.: Insulin Fibrils imaged in water. Phase signal overlaid onto topography. The “white” regions in the image are from the phase channel and correspond to higher energy dissipation. 1 μ m scan.

13.2. iDrive Requirements

iDrive requires a specific cantilever holder, and specific cantilevers.

13.2.1. Cantilevers

A generalized schematic of a iDrive cantilever is shown in 13.3a. This shows 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 13.4 on page 129](#)) clearly shows each electrical contact along the length of the cantilever chip, connected to one leg of each of the two short 100 μ m long cantilevers. Only these two 100 μ m long cantilevers, not the 200 μ m 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 200 μ m 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](#).

13.2.2. 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 13.3b on page 129](#)). 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.

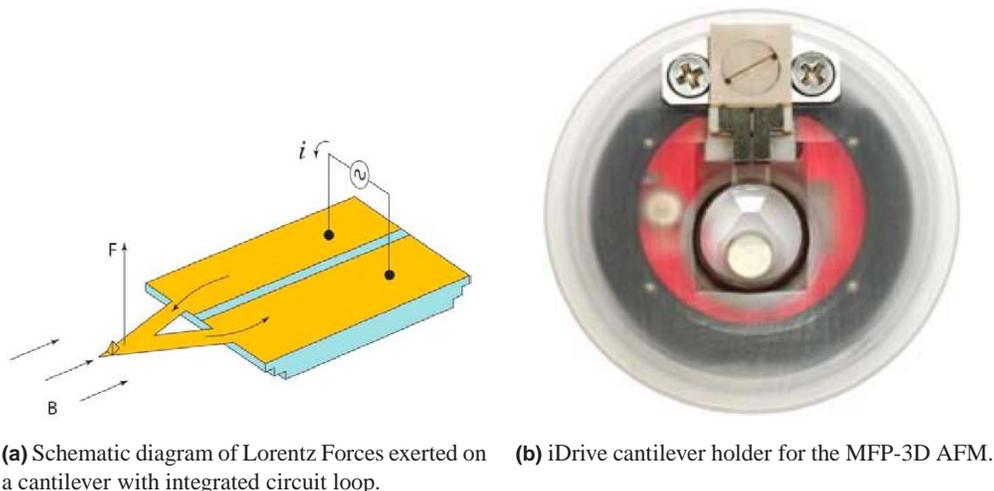


Figure 13.3.

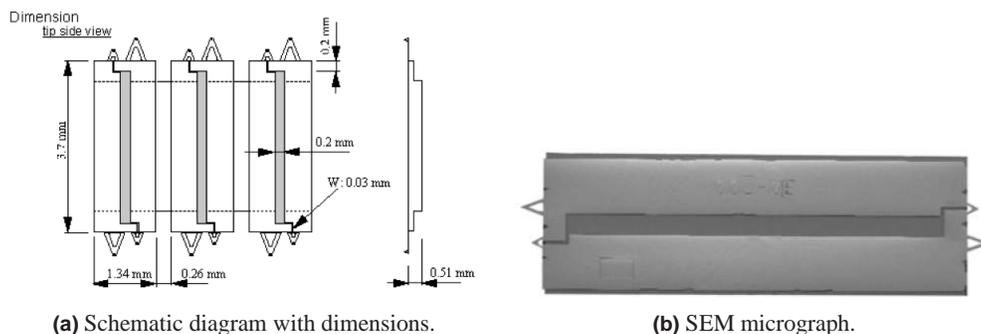


Figure 13.4.: iDrive cantilevers (Model AR-iDrive-N01)

13.3. 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 100 μ m 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.

13.4. iDrive works in water

iDrive was specifically designed for imaging soft biological and polymer samples in liquid. 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 13.5 on page 130, where both methods are compared to the Brownian “thermal” tune.

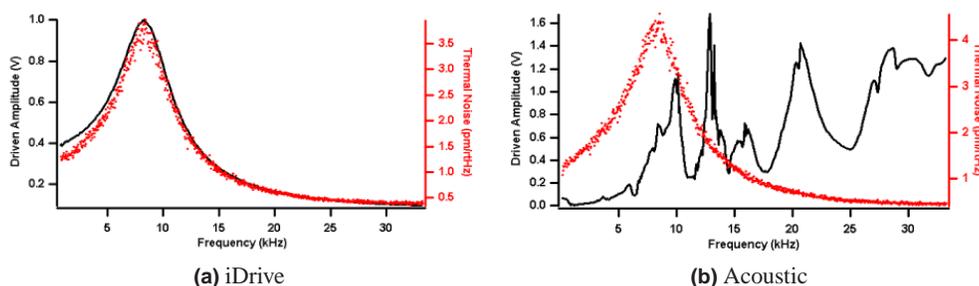


Figure 13.5.: Cantilever tunes acquired with iDrive AC mode (left) and piezo-driven (acoustic) AC mode (right) showing both the mechanical response (black curves) and the thermal noise power spectrum (red curves).

13.4.1. 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 13.6 on page 131.

13.4.2. Q-Control

With a “clean” iDrive cantilever tune, it is feasible to use Q-control (See Section 3.5.2.3 on page 22) 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 13.7 on page 131).

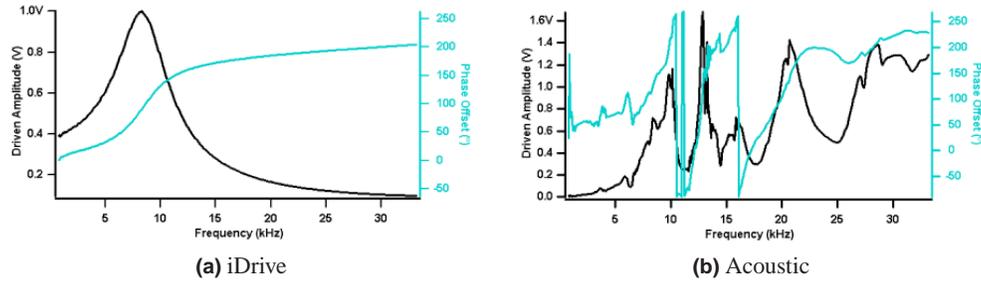


Figure 13.6.: Cantilever tunes acquired with iDrive AC mode (left) and piezo-driven AC mode (right) showing both the mechanical response (black curves) and the phase response (teal curves).

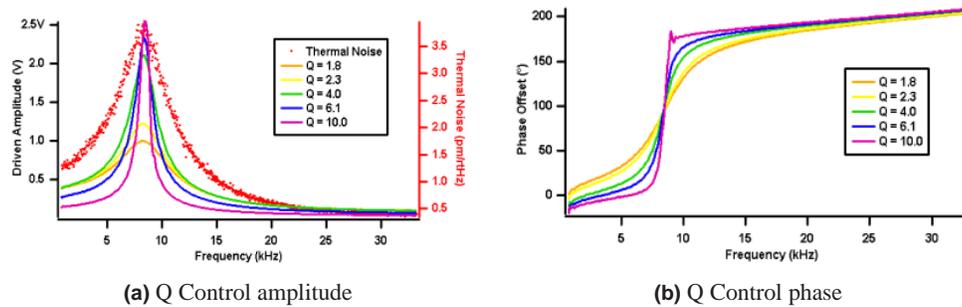


Figure 13.7.: Cantilever tunes (left) and phase offset (right) acquired using iDrive AC mode with increasing Q .

13.4.3. 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 13.8 on page 132, 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:

$$\omega_{\max} = \omega_0(1 - 1/4Q^2) \quad (13.3)$$

where ω_{\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 value⁴. 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.

⁴?, .

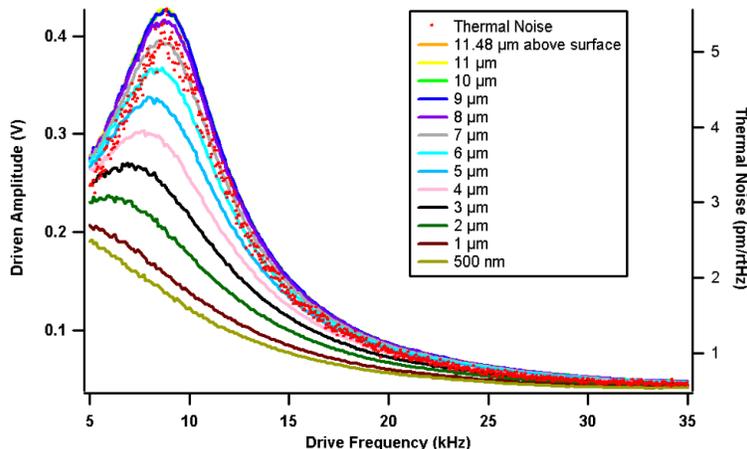


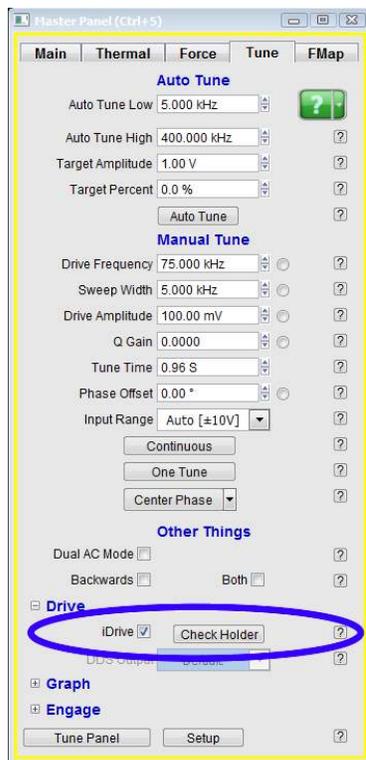
Figure 13.8.: Cantilever amplitudes at different Z positions using iDrive AC mode

13.5. iDrive Operation

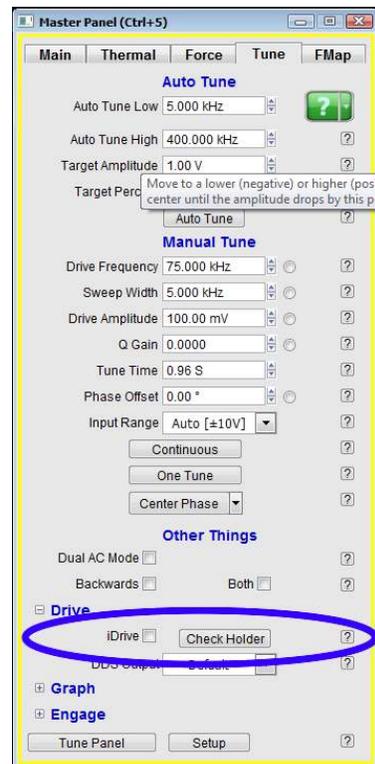
There is no special sample preparation required for iDrive. Standard glass slides with mica, gold or any other substrate can be used. First, install an iDrive compatible cantilever (Model AR-iDrive-N01) into the cantilever holder pocket, as usual, and tighten the PEEK screw until the cantilever chip 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 [Chapter 3 on page 18](#). The exception is that you need to make sure that the iDrive checkbox is checked as shown below [13.9a](#).
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 13.9.: Tune Panel configurations.

13.5.1. 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 13.9a on page 133). To enable the iDrive box, attach the iDrive cantilever holder to the MFP-3D head and click on the *Check Holder* button (Figure 13.9b on page 133).

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.

13.5.2. 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 13.4.3 on page 131](#), 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 user 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.

13.6. 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.

Part II

Spring constant calibration and Thermals

Part II: Who is it for? Succinct step by step instructions for calibrating spring constants

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14. Spring Constant Determination

CHAPTER REV. 1571, DATED 08/27/2013, 11:36.

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14.1. 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 14.1.1](#) on page 138.
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 14.1.2](#) on page 141.
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 15](#) on page 148.

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 14.2](#) on page 146.

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.

14.1.1. Virtual Deflection Correction

The virtual deflection is a mechanical coupling of the deflection signal with the Z movement seen on MFP3Ds. It is a result of the mechanical path not being quite perfect, resulting in a slight slope

in the force curve. Although this may only be a few nm's over several microns, if not corrected, it can skew the accuracy in the measurement and 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 lever as it travels through the 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.

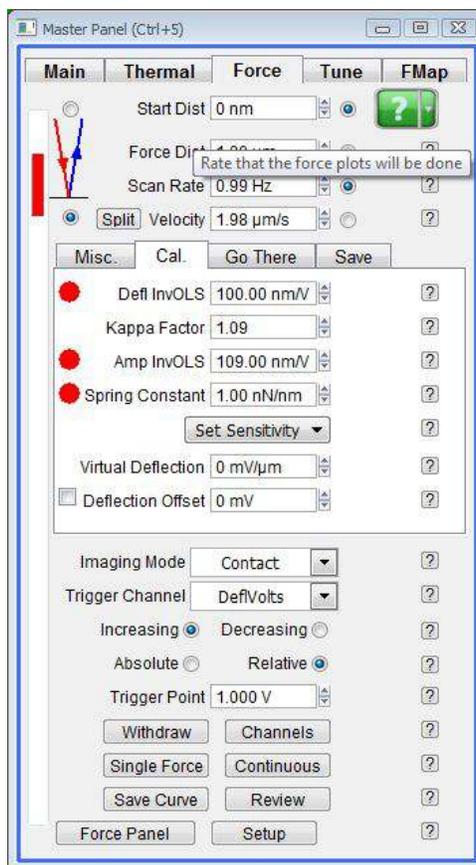
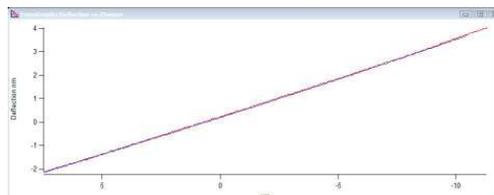
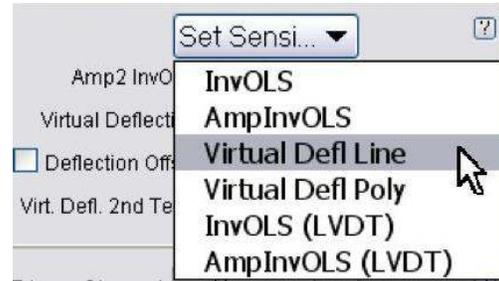


Figure 14.1.: Master Panel > Force Tab > Calibration Tab.

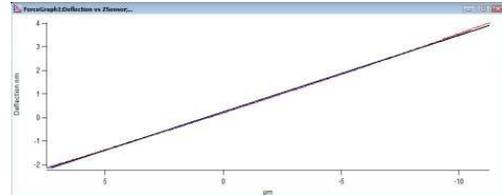
1. You want the tip far from the surface, i.e. the tip will not contact the surface even when the Z voltage is at 150 volts. This virtual deflection is a good thing to calibrate before engaging.
2. Set the trigger channel to none
3. Set the start dist to -inf, or drag the red bar to the top
4. Set the force dist to inf, or expand the red bar all the way to the bottom
5. **Do a force plot**



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

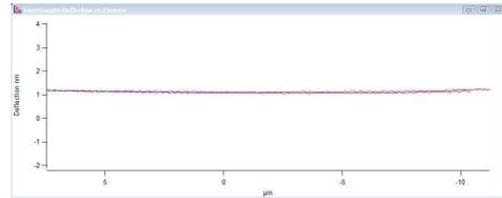


7. **Check the fit**



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 MFP3D head, you may need to do a second order poly 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.



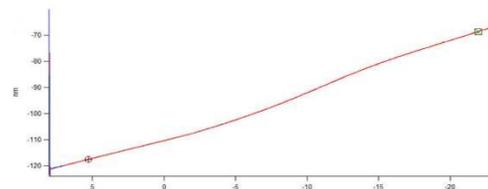
14.1.1.1. Tips and Tricks

Tip Extended Range MFP-3D Head

If you are using an MPF-3D extended Z range head (~28 μm Z range for early models and ~40 μm for current MFP-3D), the long approach of a force curve may not appear linear at all.

- In this case you should select 'Virtual Defl Poly' in Step 6 on page 140 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 inaccurate.

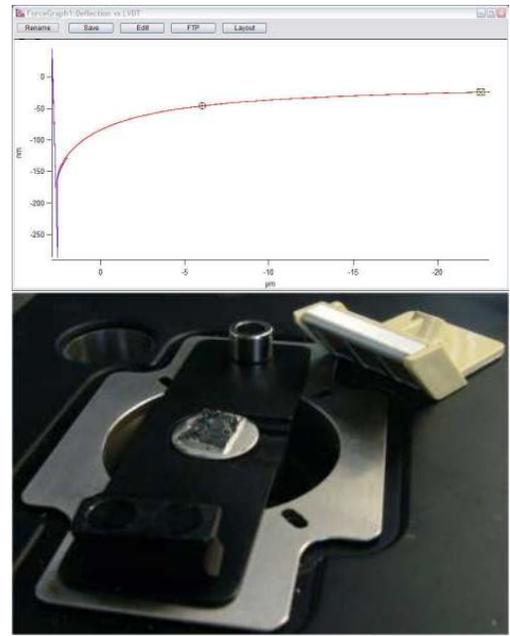


Tip Eliminating Surface Charge Effects.

Certain flat substrates (glass, mica) have inherently charged surfaces. Combined with a soft cantilever (0.03 N/m) this can cause some odd non-linear deflections in the force curve approach.

Possible solutions:

- Choose a conductive surface such as freshly cleaved graphite (HOPG) or silicon on a conducting substrate.
- Place a static master ionizer near your sample, see Section 3.5.3.1 on page 24.

**14.1.2. InvOLS**

The objective of the second step is to measure the slope of the contact region (called the inverse optical lever sensitivity or InvOLS), a parameter necessary for the spring constant algorithm. In the previous section we eliminated possible instrument errors of this measurement which might otherwise contribute to inaccuracies of the spring constant measurement.

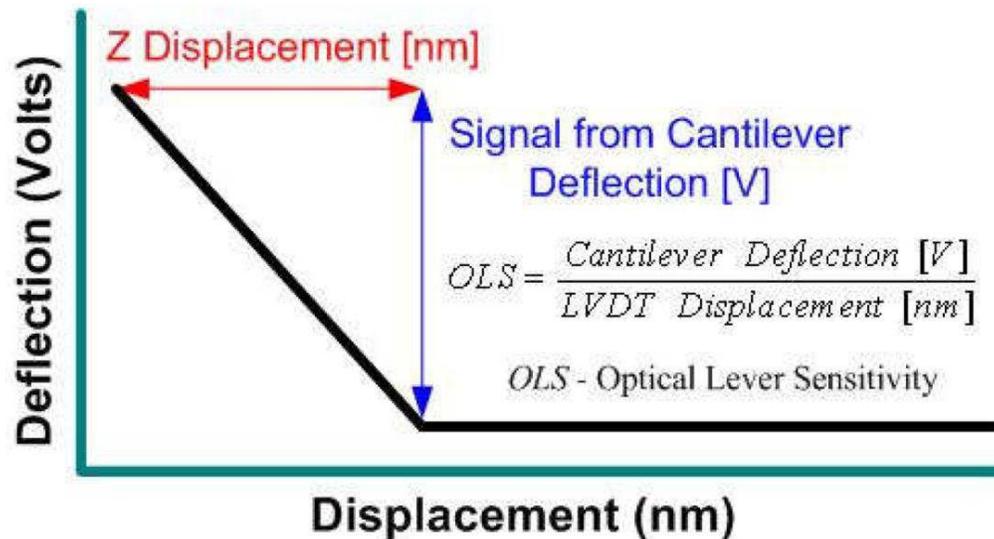
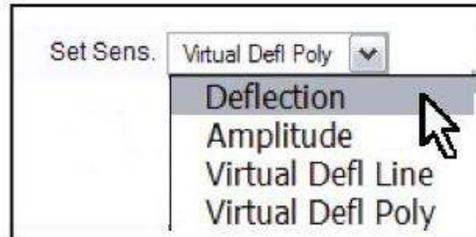


Figure 14.2.: Inverse Optical Lever Sensitivity measured from a force curve on a hard surface.

Figure 14.2 on page 141 gives a sense of what the optical lever sensitivity is, given in units of V/nm, and the software inverts it for the algorithm.

Perform Line Fit

4.
 - 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.

**14.1.3. Averaging InvOLS**

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 14.1 on page 139), click the 'Set Up' button.

[Optional] Add the 'Limit Cont' Control

2.
 - 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.

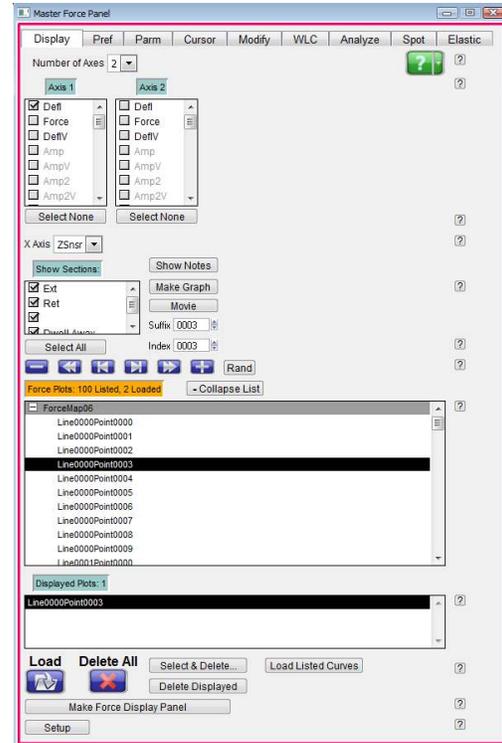


Note This will allow a set number of force curves to be sequentially acquired at the same XY location, without clicking the 'Single Force' button repeatedly.

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

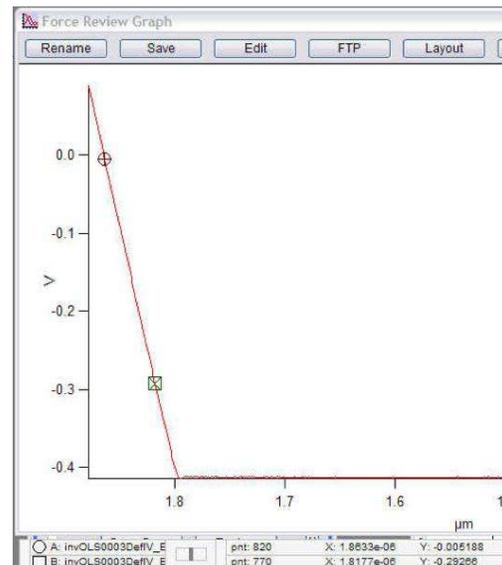
4. **Activate** the *Master Force Panel*
- From the main menu bar select *AFM Analysis* ▸ *Master Force Panel*.
 - In this panel select the *Display Tab*.
 - Load the curves that you want to average the DC InvOLS on, if you saved the curves to memory, they will already be loaded.

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



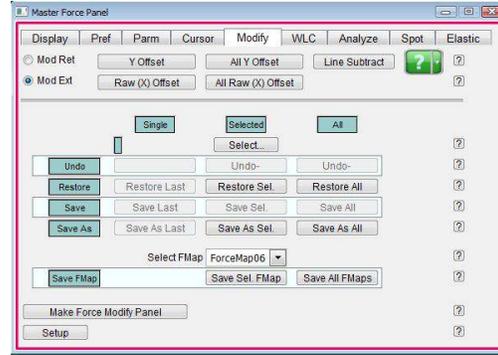
5. **Plot** the Force Curves
- Plot the Force curves as Deflection volts versus ZSnsr.
 - With the graph window selected, click **Ctrl + i** to show the info area on the bottom of the graph.
 - Drag the cursors from the info area onto the sloped part of the graph.

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.



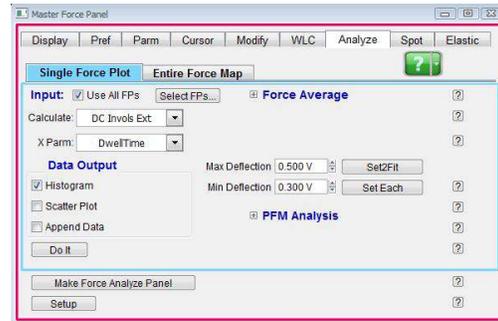
Offset the Data

6.
 - 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 Involts analysis looks at the same voltage range, so deflection drift needs to be corrected.



Prepare for Analysis

7.
 - Go to Analyze tab of the force review.
 - Under the 'Calculate' pull-down, select 'DC InvOLS'.

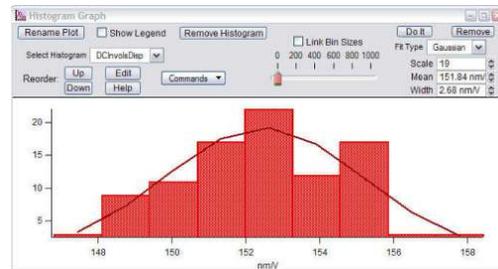


8. [Optional] Click on the Select FPs... button to select which force plots you want to analyze.
9. Under the 'Max Deflection' control, enter the value of the higher cursor.
10. Under the 'Min Deflection' control, enter the value of the lower cursor.

Create the Histogram

11.
 - 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.



14.1.4. Do a thermal

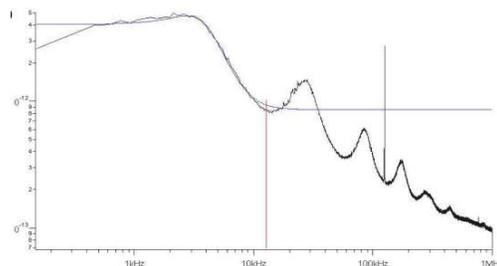
Once you have your invols calibrated, you can collect the thermal data, which is needed to fit for the spring constant. Thermals are outlined in the next section 15. Advanced note, you don't need to collect the thermal last. You can collect the thermal first, then when the Involts value is updated, the last collected thermal data is rescaled. You can then fit the rescaled thermal, and obtain the correct spring constant.

14.1.5. Tips and Tricks

Tip Thermal fitting in liquids.

Sometimes when using a 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.



14.2. Spring Constant Tutor

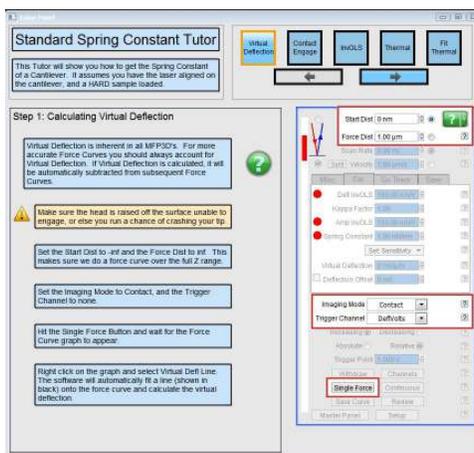


Figure 14.3.: Spring Constant Tutor

The Spring Constant Tutor allows all three steps of the spring constant (k) determination steps Section 14.1 on page 138 to be performed in a single software panel.

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 determination.

14.3. Get Real™

GetReal will calibrate your Invols and spring constant without touching the surface.

On the thermal panel, click on GetReal, select the lever you have loaded on the lever panel. This loads constants that describe that specific lever geometry. Then click GetReal again and the software will 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 15.3 on page 151 for more details.

14.4. Sader method

The software also has the option of using the Sader method to determine the spring constant. This technique uses the dimensions of the cantilever (in meters), the Q 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, the following references may be helpful:

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.

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.

15. Thermals

CHAPTER REV. 1569, DATED 08/27/2013, 09:15.

USER GUIDE REV. 1578, DATED 08/29/2013, 22:20.

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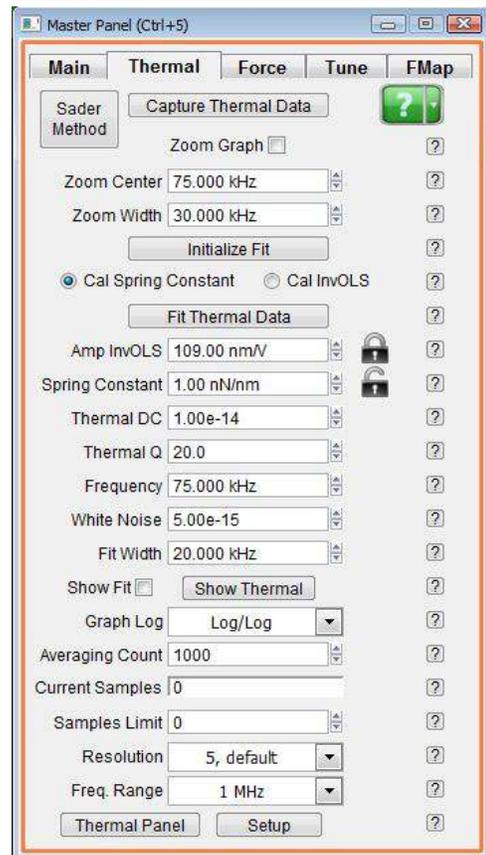
Capturing a thermal spectrum consists of pulling back data at a high rate and doing a FFT to determine the spectral components. The MFP3D can run thermals up to 2.5 MHz on MFP3D and 32 MHz on Cypher.

Thermal Tunes are mostly used for a few specific tasks:

- Determining the spring constant OR Involts.
- 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).

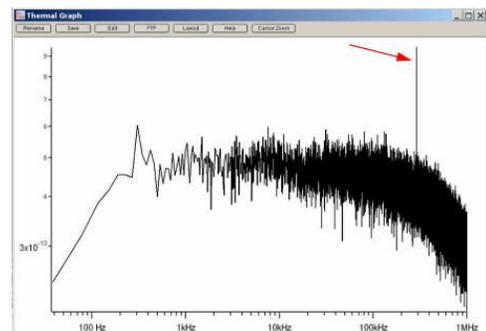
15.1. Capturing the Thermal

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).

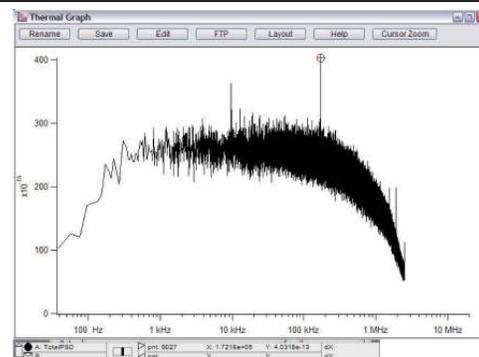
Resolution of the acquisition can be changed in the resolution pull down menu of the Thermal tab. Larger numbers go faster (less resolution); while smaller values acquire slower, but give less noise. The way the data is plotted (i.e., linear vs. logarithmic) can also be changed in the Graph Log pull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



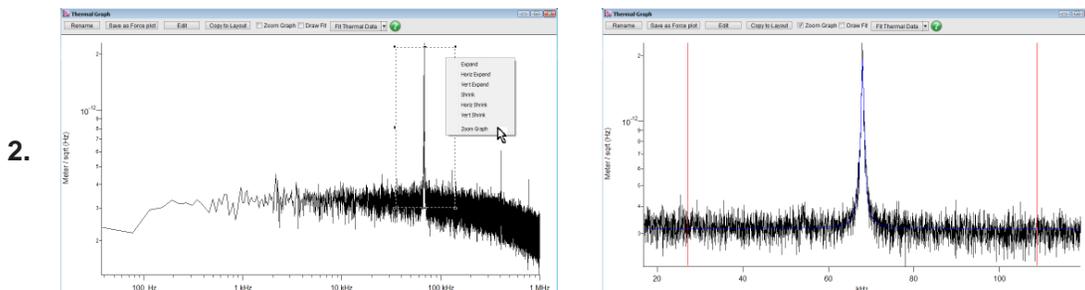
Save Thermal tune plots by

- 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 $m/\sqrt{\text{Hz}}$ 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.

15.2. Thermal fitting for spring constant

1. You need to have calibrated AmpInvs before doing this stage, see Section 14.1.2 on page 141 for more details.



- 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 AmpInvs is locked on the thermal panel see 1. This means when you do a fit, you are holding AmpInvs constant, and fitting for the spring constant of the lever.

15.3. Thermal fitting for Invsols

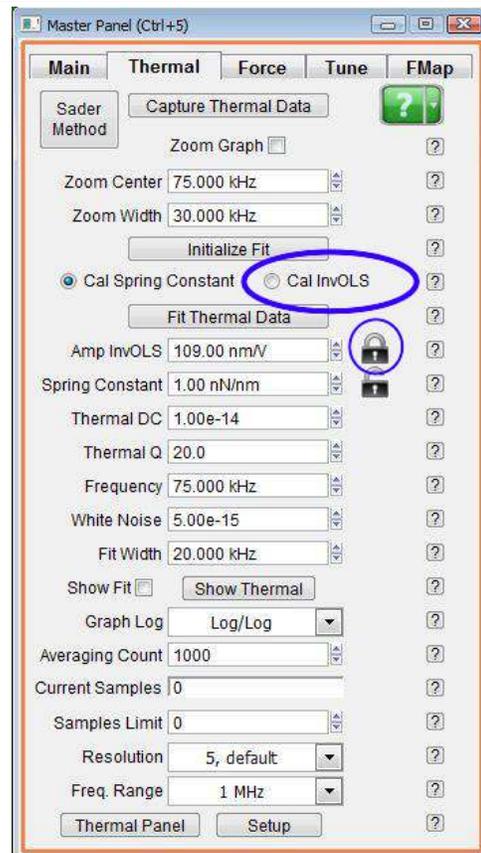
This is mostly useful for recalibrating Invsols after changing the sample or adding liquid. The basic idea is that you have calibrated the spring constant in air, on some clean hard surface. The spring constant should not change. Invsols will depend on numerous factors, temperature, spot position, liquid, etc, so if you know the spring constant, you can collect thermal data, and find what Invsols value would give you the same spring constant.

General outline:

1. Calibrate Invsols and or Amp Invsols 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.

15.4. Thermal Tuning a Cantilever

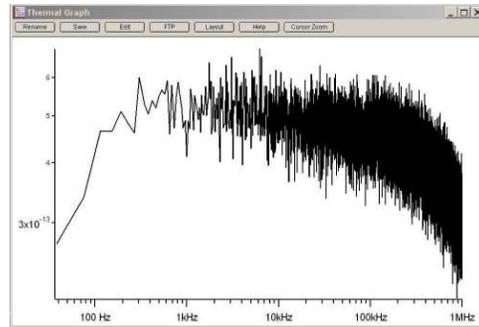
- Thermals work very well for determining the resonant frequencies (and ultimately drive frequencies) of cantilevers in fluid.
 - After you have fit the thermal peak, you can right click on it and select Move Freq, Phase and amp to tune. This will set the drive amplitude, drive frequency and Phase offset 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.

15.5. Thermals for tip checks

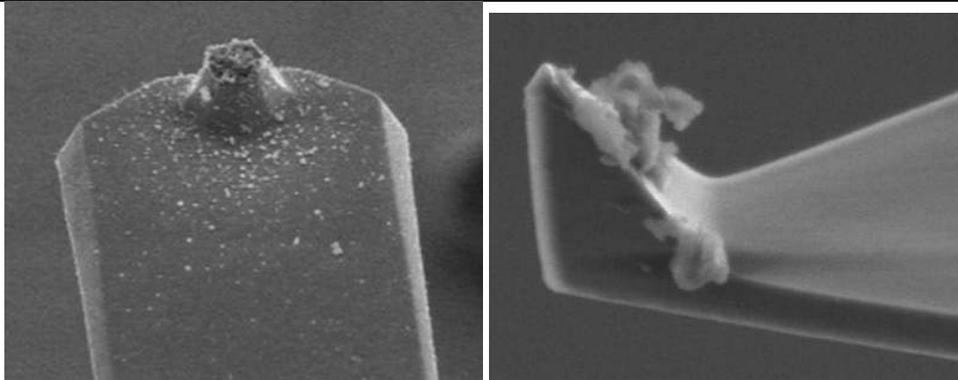
The thermal is also a very convenient way to check whether the light source spot is actually on the cantilever. When aligning spot via IR card, sometimes a nice sum is given when the spot is on the probe substrate, thermals are a good way to double check that the spot is on the lever.

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_0), or if there is an increase in mass on the cantilever/ tip from adhered debris (lowering f_0).

