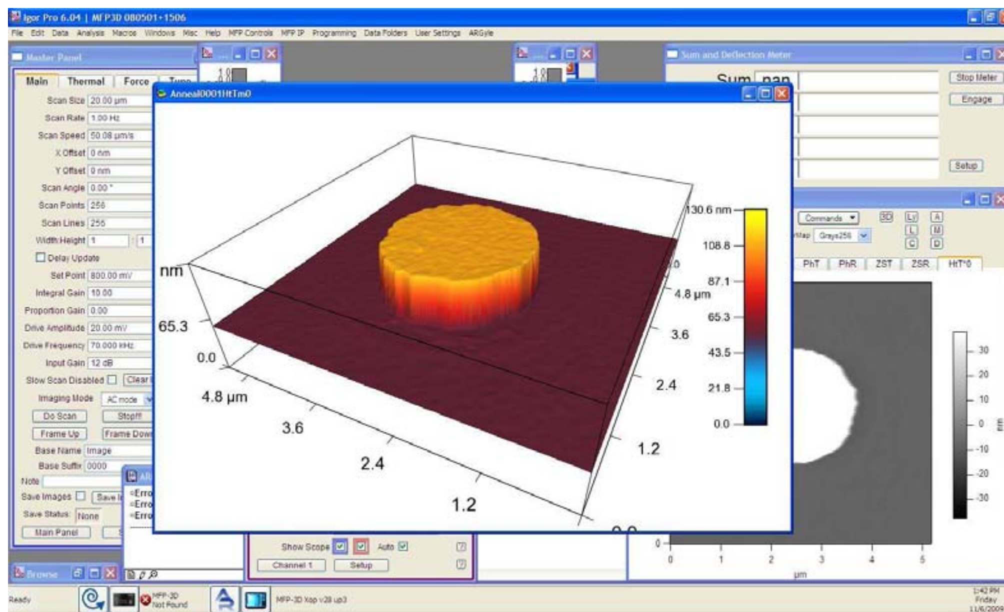
ASYLUM RESEARCH

Atomic Force Microscopes

SPM Applications Guide

USER GUIDE 3

Imaging and Spectroscopy Applications Guide



User Guide

including beta (complete, reviewed) chapters, including draft (nearly complete, not reviewed) chapters,

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Asylum Research

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Introduction

Volumes of the AR Imaging and Spectroscotpy 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 III *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 101010 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 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 Free at 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 *MFP-3D User Guide, Chapter: Installation*. Likewise, for the Cypher AFM a properly operating AFM system includes a PC with the AR SPM software installed.

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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

CHAPTER REV. 990, DATED 08/06/2012, 09:49. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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1.1. Preparation

1.1.1. Cantilever Selection

1.1.2. Do a Tutorial

Contact Mode imaging is sufficiently commonplace that a tutorial has been developed for it. If you own an MFP-3D AFM, please follow *MFP-3D User Guide, Chapter: Tutorial: Contact Mode Imaging in Air*. If you own a Cypher AFM, please follow *Cypher User Guide, Chapter: Tutorial: Contact Mode in Air*. This will familiarize you with the basics of loading 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. In case there are differences in how the software interacts with our different instruments, we'll cover that as necessary.

1.2. Set-Up and Initial 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 tip and 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 Micrsoscopy) discussed in detail in Section 1.3 on page 11. Please ignore these steps in case you are doing standard Contact Mode imaging.

1.

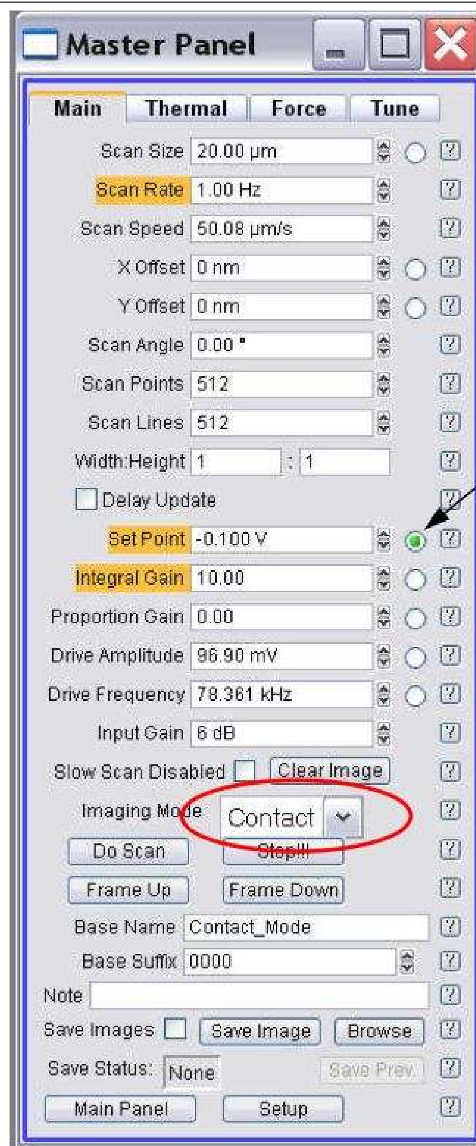
Set up Master Panel:

- In the Main tab of the Master Panel, select ’Contact’ from the Imaging Mode pull down menu.
- Set point: -0.1 V.
- Integral gain (I): 8 to 10.
- Scan rate: 1 Hz; for softer samples, choose a slower Scan Rate, perhaps 0.5 or 0.2Hz.
- Scan angle, resolution (scan points & lines) and image size is up to you. The figure to the right gives typical values.

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° for lateral force microscopy (LFM). This may be obvious, but some other commercial scopes scan perpendicular to the lever at 0°.



2.

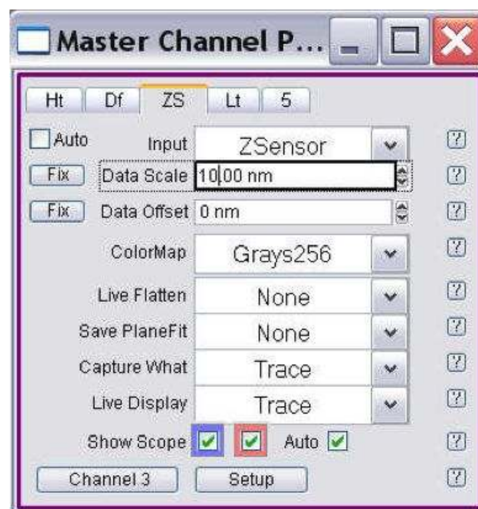
Image channel selection:

- Go to the Master Channel panel
- Select the leftmost tab and select 'Height' under the input pulldown menu.
- For the next two tabs do the same for 'Zsensor' and 'Deflection'.

Note If you do not change from the default AC mode image channels of amplitude and phase, these channels won't produce any meaningful data in Contact Mode. They must be changed prior to imaging in Contact Mode.

Lateral Force Microscopy Only:

- If doing Lateral Force Microscopy, choose Lateral in 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.

Deflection: the error signal of the feedback loop to maintain the user Set Point deflection (voltage), per X,Y scan point.

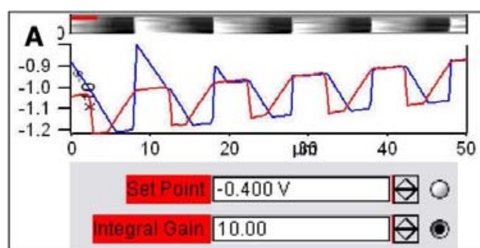
Z sensor: Closed loop sensor that monitors the movement of the optical lever detection assembly. Better for measuring accurate distances, typically >200nm.

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.

While not necessary, it's a good habit 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 it's a more precise Z measurement.

1.2.1. Fine Tuning Imaging Parameters

1. Click the 'Do Scan' button on the Main tab, and imaging will begin after a moment. The scan initiation first moves the tip to the starting point of the image, then lowers the tip onto the surface, and then begins an endless series of image scans.



2. **Determining Image Quality:**

- Start the learning process on a sample with a known topography, like an Asylum Research Calibration Grating (basically a matrix of square pits separated by raised alleys. Then you know immediately if the image looks sub-optimal.)
- Look at the 'Scope Trace' below the image. This graph represents the most recent line of the image. Blue indicates the tip moving left to right (a.k.a **trace**) and Red indicates tip returning from right to left (a.k.a **retrace**).

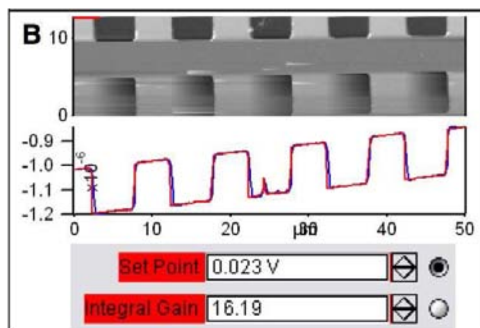
On most samples with relatively slowly changing features, trace and retrace should look the same. In other words, the landscape should look the same if you are flying the exact same route one way or the reverse. The image above shows the two quite different; an indication that imaging parameters need 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 (the left edge of each pit is quite sharp) 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 of 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**.

Hamster

The next steps will go into the details of strategies for tuning parameters in the main panel. Use the arrow clickers (to right of variable fields) to adjust parameters, rather than typing the values in. Alternatively, you can fine-tune the parameters using the 'Hamster' wheel on the front of the controller. Any parameter with a radio button next to it can be changed during a scan when it is activated (looks like black/ green dot in circle) with the 'Hamster'. The Hamster gives "digital control with analog feel". On the MFP-3D AFM controller the toggle switch to the left of the Hamster allows you to toggle between radio buttons in the panel. On the ARC2 SPM controller the outer hamster ring performs this function. This tactile experience lets you concentrate on the image while tuning parameters.



Adjusting Set-Point, i-gain, and scan speed:

3.

- The goal is to get trace and retrace to fall on top of each other (as shown to the right).
- Increase the **integral gain** or **i-gain** parameter (second highlighted item in Step 1 on page 7) and see what happens. The match between trace and retrace should improve. The feedback becomes more aggressive and the slope with which the tip “parachutes” down into the pits should increase.
- Keep increasing the integral gain until suddenly the trace and retrace start to oscillate wildly, a phenomenon called ringing. The feedback loop is now unstable and the i-gain needs to be lowered a bit.
- If increasing the i-gain does not seem to help, try to increase the set point voltage. This will cause the cantilever to press down harder on the surface when in contact, but will also improve its ability to track sudden steps on the sample.
- Finally decrease the scan speed.

Note Do not be alarmed if you have to crank up the Integral gain when using long floppy cantilevers- the gain is related to the optical lever sensitivity (more gain is required for less sensitive (longer) cantilevers).

4. The second parameters to adjust are the Scan Rate & Scan Angle. Having a vague idea of the orientation of the tip on the cantilever can make scanning at 90° more advantageous than 0° 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.

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

A The imaging parameters in the main tab of the main panel (See 1) will update as soon as you make a change. Note that some actions, such as changing variables points, lines, or rate will cause the tip to be lifted from the surface, mid scan.

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, without lifting the tip from the surface. 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.

1.3. 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 photodector which measures the cantilever deflection, except that different quadrants are subtracted to measure the lateral deflection or twist of the lever.

1.3.1. Zero-ing the Lateral Deflection Signal

To best measure the lateral deflection (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 Has no direct mechanical adjustment for later laser spot adjustment. The beam falls on the sensor as calibrated in the factory. Any offset away from center can be electronically adjusted. Please see for more information. This is the

least desirable way of zero-ing the signal and the noisiest. If LFM is your bread and butter, you will have one of the following pieces of equipment.

MFP-3D AFM with LFM Head Has special adjustments to mechanically center the laser spot on the detector. This is a superior to electronic zero-ing. Please see for more information.

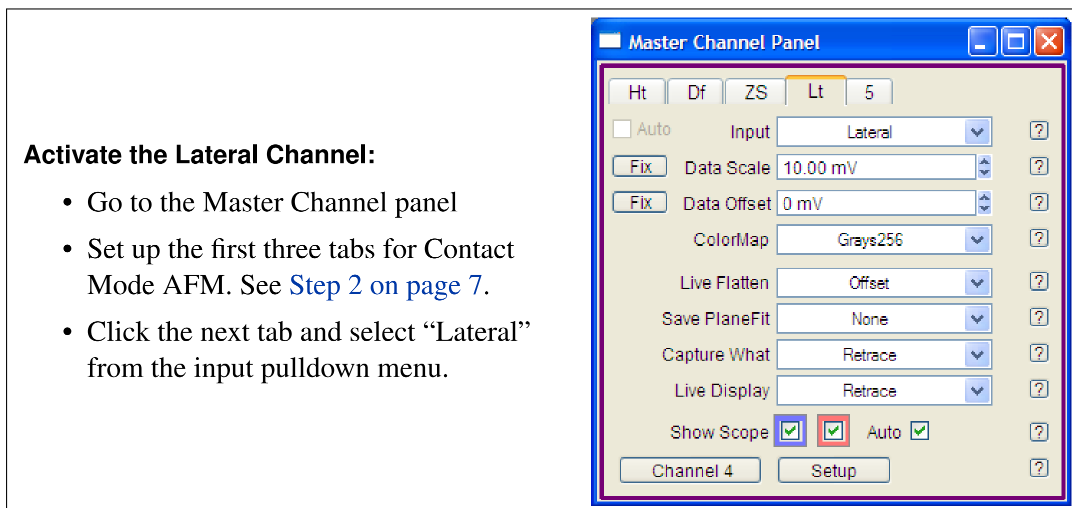
Cyper AFM Has built in motorized Zero-ing of the deflection and lateral signal. It all happens automatically. When the Cypher is prepared to do contact mode imaging, it is also ready to do LFM. Please see

1.3.2. Operating the Software in LFM mode

First we assume you have already have your system performing in Contact Mode, as described in the beginning of this section.

1. Stop the imaging process and retract the tip from the surface. Or simply engage the tip on the surface and retract it again so you know your AFM is ready to go. You'll have to consult the user guide specific to your AFM to do so.

2. **Activate the Lateral Channel:**
   - Go to the Master Channel panel
   - Set up the first three tabs for Contact Mode AFM. See Step 2 on page 7.
   - Click the next tab and select "Lateral" from the input pulldown menu.

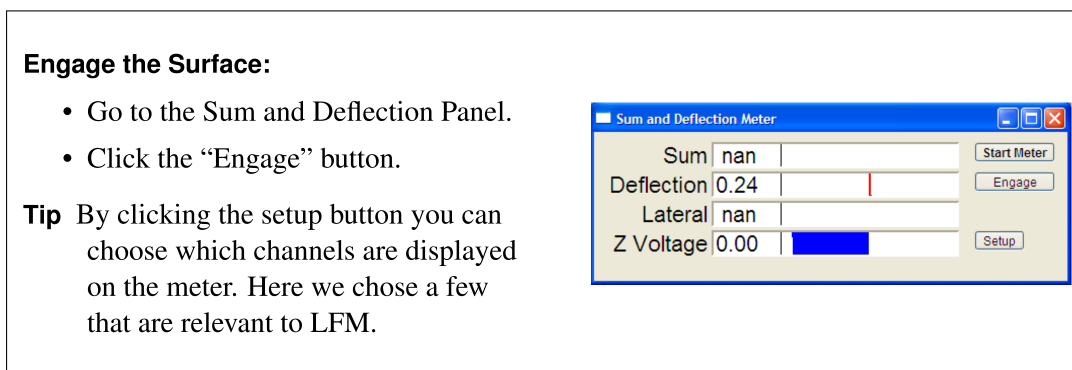


3. In the Main tab of the Master Panel (See Step 1 on page 7), confirm the Scan Angle variable is set to 90°.

4. In the same panel, confirm the imaging mode is set to Contact Mode.

5. **Engage the Surface:**
   - Go to the Sum and Deflection Panel.
   - Click the "Engage" button.

   Tip By clicking the setup button you can choose which channels are displayed on the meter. Here we chose a few that are relevant to LFM.

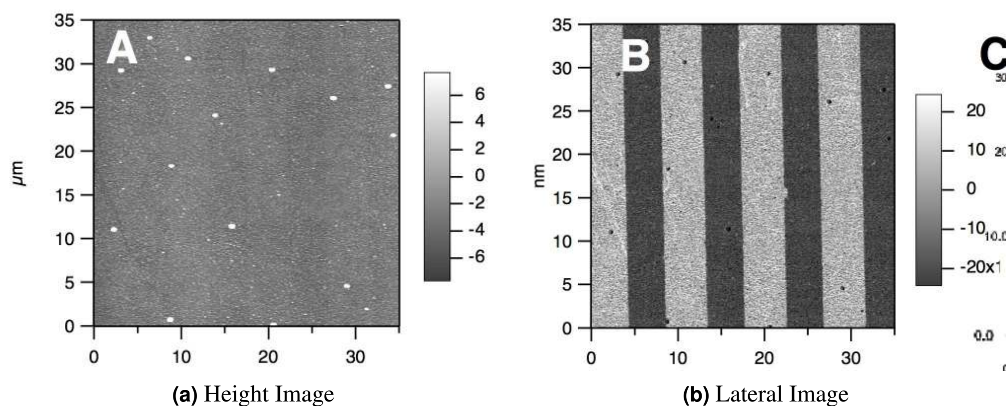


6. In the Main tab of the Master Panel (See Step 1 on page 7), hit the "Do Scan" button, as is customary for any type of AFM scan.

7. There should be four image windows on your screen. Amplitude, Deflection, and Zsensor should be familiar from Contact Mode Imaging. The fourth will be the lateral signal which will show contrast for areas of differing tip-sample frictional forces.

1.3.3. 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.



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

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.

1.3.4. ARUP (User Panel) LFM Friction Loop

You may have noticed in the AR user panel menu that there is something called LFM Friction Loop. In most cases, this is not useful to the average or even advanced MFP-3D user. To take advantage it requires an MFP-3D LFM head, which has special mechanical adjustments for laterally centering the laser spot on the photodetector and a special high bandwidth throughput for the lateral signal.

This user panel was designed by our programmers as an attempt to help a customer (who has the high bandwidth head with mechanical lateral zeroing) to figure out the lateral InvOLS from the turnaround area at the end of the scan line. Note that this approach is only valid for those rare occasions in which the lateral contact stiffness is significantly greater than the lateral (or torsional) stiffness of the cantilever. This situation might arise, for example, with colloidal probes under high applied load. If you have any intention of using this feature, please contact our support first. Please don't invest any time with it unless you understand what it can and cannot do.

2. Conductive AFM (ORCA)

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2.1. 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.

2.2. 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. Much stiffer, and your wear is to much.

- Metal coated Si probes. Pt, PtIr, Au, CoCr, Tungsten Carbide, and Ir coated probes from Asylum Research. These perform pretty close to each other. The Tungsten Carbide and Ir probes last a little longer. For Polymer samples or soft materials these are fine. radii are typically 50nm

- Conductive diamond coated probes. Use a diamond-like carbon film coated onto a Si probe. Available from Nanosensors. These last quite a lot longer than metal coated probes, but have a larger radius (~100nm). ADT makes a nano crystalline coating that wears at least as well as the Doped DLC, but typically has a smaller radius (<50nm).
- Solid W probes from Multiprobe. Very consistent. More expensive than most probes but last a long time. Forms an oxide over time that has to be worn away with a bit of imaging.
- Solid Pt probes from Rocky Mountain Nanotechnology. Best overall probes for CAFM. Small radius (5-20nm). Good wear characteristics. Even if the probe wears to larger sizes, it does not loose its ability to conduct.

2.3. Terminology

CAFM Conductive AFM. By my definition anything that looks at current passing through a sample and a tip in atomic force microscopy. I tend to include the following under this broader category.

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: Again, another form of CAFM, but here the user uses a tip with a tougher coating and drives up the 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.

Definition	What do the letters in ORCA stand for? Optimized Resistance Conductance Amplifier.

2.3.1. Spectroscopy vs. Imaging

Pretty obvious, but might as well be mentioned. In CAFM you can either collect a current map of a surface at a given bias, essentially giving you a single slice of the conductivity of your sample, or you can take current-voltage spectra at points either predefined by the user individually (i.e. ‘pick a point’), or on a grid of n×n points. Spectroscopy normally entails taking AC mode images of a surface, then changing over to contact mode and collecting the spectra where desired.

2.4. Repeatability in CAFM

- The most common is tip change. Metal coated probes are infamous for chipping, breaking, and otherwise acting in an obstinate manner. This normally shows up as a systematic asym-

metry 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.

- The easiest factor to diagnose is debris on the surface. 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. I make it a habit of taking an AC mode image of my surface whenever I am about to take a CAFM image.
- 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. By modification, I refer to some materials (CMO's come to mind) that can be modified by biases to be 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 CNT surfaces.
- Other problems that can lead to poor reproducibility involve the circuit, rather than the tip or sample. This is more in reference to sample to sample, but can also occasionally occur in multiple experiments on the same sample. Here, I refer to 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. These are often difficult to see because the user assumes that all is good because it has always been so. Always double check this even if you are sure the circuit is fine.
- This one really belongs in the previous item, but it is so common I am making it one on its own. On Si samples, you need to scratch away the SiO_2 from the surface, then as quickly as possible apply conductive paint to the surface (I recommend Ted pella leitsilber) to insure you don't have a metal-oxide-semiconductor (MOS) contact in your circuit.

2.5. The Hardware

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

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

These chapters will explain which cantilever holder to use, how to calibrate it, how to mount the sample, and how to connect the bias wire.

2.6. Current imaging

A schematic of CAFM is shown in 2.1.

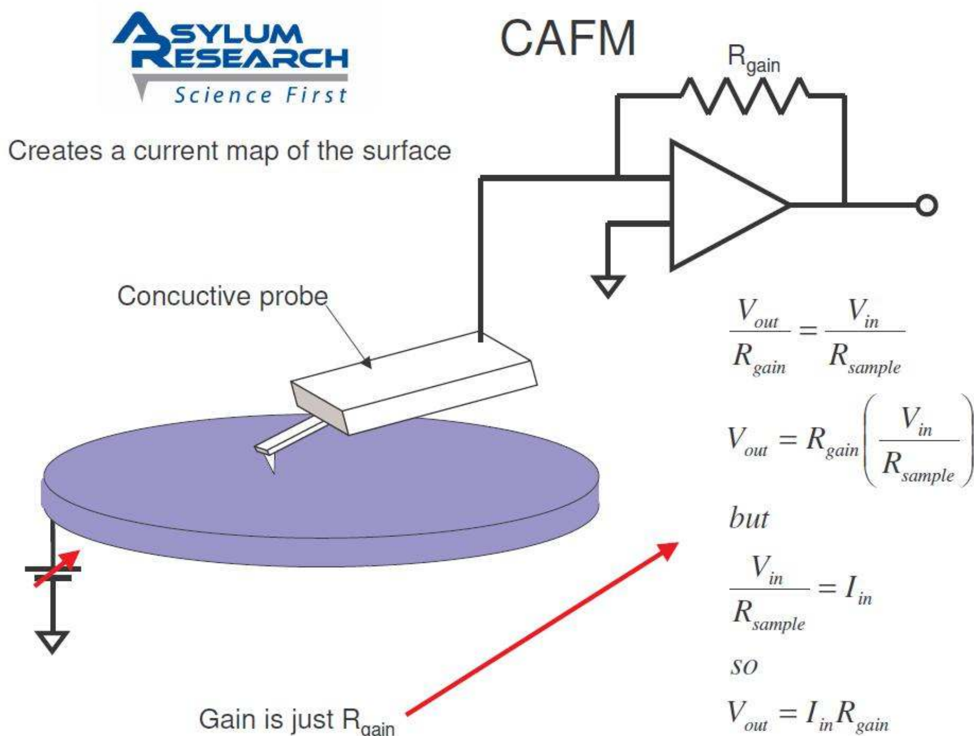


Figure 2.1.: Schematic of CAFM with relevant equations.

Current is passed through the tip and into a transimpedance amplifier, and this signal read on a user ADC. This completes the current loop.

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.

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.

Fig. 3.6 shows an example of a typical ORCA image.

Figure 2.2.: These controls, found on the DoIV panel, control the bias to the sample while the sample is imaging.

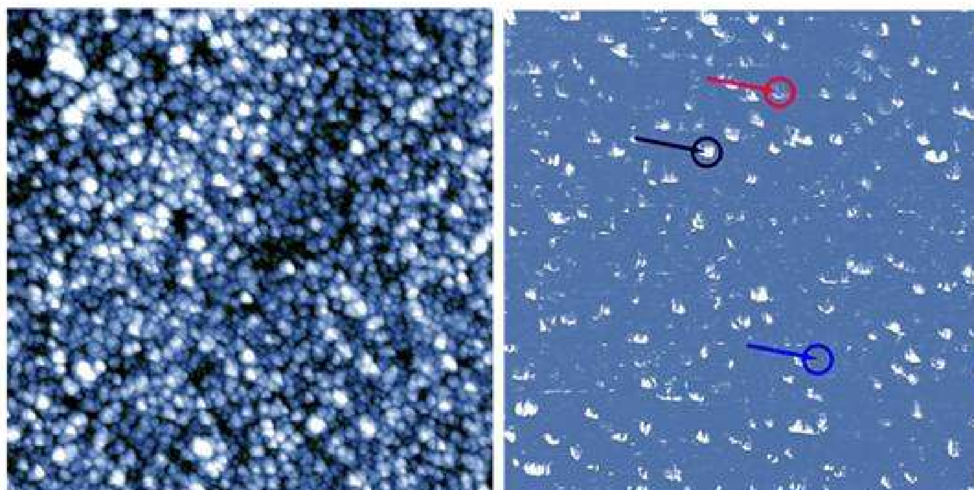


Figure 2.3.: This image is fairly typical of a thin film CAFM image. note that tip shape artifacts are visible on the current image, but not on the topgraphic image. This is fairly typical. As the metal coating on the probe tip wears, or the tip is contaminated, these artifacts will be more prominent. Rocky Mountain nanotech now makes a solid Pt probe that does not suffer from this problem.

2.7. Current-Voltage (I-V) Spectroscopy

IV curves on the MFP3D are done using the 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 in any of our existing functions, or any custom user functions that a user may wish to write. To access the Do IV panel, go to *MFP controls ▷ Do IV Panel*.

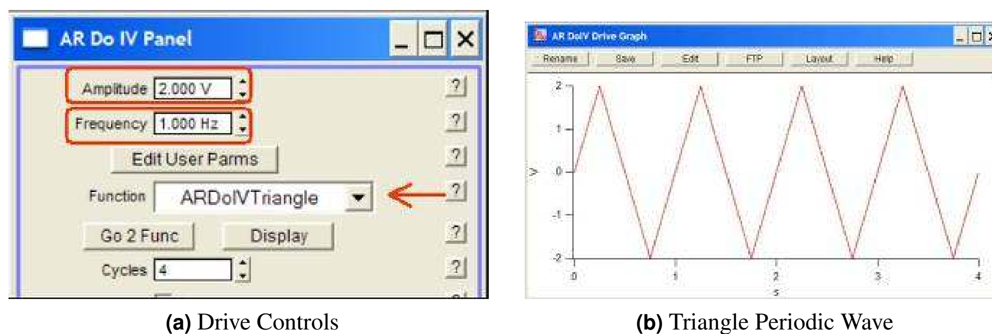
A description of all of the individual listings on the Do IV Panel can be found in the help file embedded in the software by clicking on any question mark to the right of the control in question.

Before attempting to do IV curves on a sample, it is recommended that you first run I-V curves on a test resistor. This procedure is described earlier in this document.

Running Current-Voltage curves on the MFP-3D generally involves first acquiring an image of your sample surface. For delicate samples that would be damaged or modified by contact mode, AC mode images are generally used.

Once you have an image on which to spot I-V curves, you are ready to set the parameters for your I-V curves. This is of course dependent on your experiment. To get reasonable start values you can consider the resistance of the sample with an Ohm meter, and figure that 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, Voltage = Current × Resistance. If you are not familiar with this law, you should probably not be doing CAFM.

I-V curves on the MFP-3D are treated like a periodic function. 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.



(a) Drive Controls

(b) Triangle Periodic Wave

Figure 2.4.: 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.

Checking the 'average' (see DoIV 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.

Checking the 'Apply during triggered Dwells' box will tie the IV curves to the force curve and force

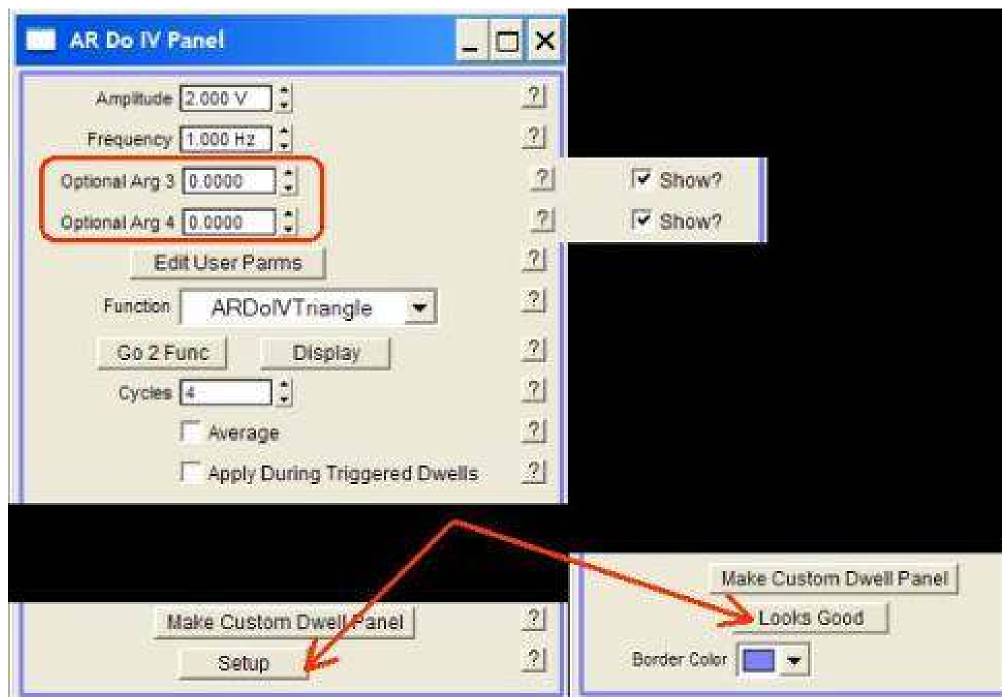


Figure 2.5.: [Setup on the DoIV panel] You can select the fields you would like to display using the setup button at the bottom of the DoIV panel.

Figure 2.6.: [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.

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 volume 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).

Next check the name of the curves and be sure you are happy with this. Changing the name on the curve will also clear any numbered markers that have been placed on the real time image. See below for more on this.

To adjust the filter on the analog to digital converter that will collect the output on the op-amp on the orca holder, simply change the field labeled 'low pass filter' on the AR Do IV panel. Values

Figure 2.7.: [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.

from 50 Hz to 1MHz 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, on the MFP3D AR Do IV Panel set Spot display to numbered markers. 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:

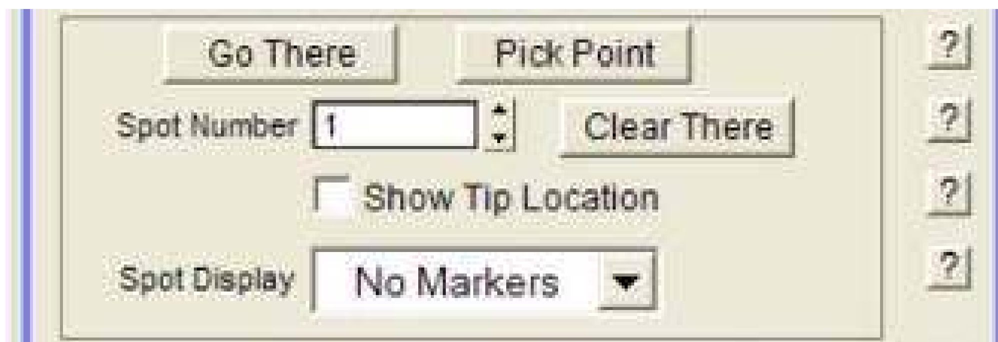


Figure 2.8.: [Go there] Use the pick a point, that's it, and go there buttons to move around the surface and collect IV curves. Use 'numbered markers' to graphically depict the selected locations on the AFM image.

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. 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'. Click this button. There should now appear a marker on the area where you would like your I-V curve. If you do not see one you may have gotten the order wrong, or some little bug may have flown into your software. Try it again. If it still does not work, try changing the name of the base name either in the main panel or the AR Do IV panel and repeat. Eventually you will get a marker where you want one. 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 Set Point , then check the box labeled 'Feedback on.' This will put the tip onto the surface and turn feedback on.

Caution	Do not use the simple engage button for IV curves. This button is for standard imaging, and uses an ADC for topographic feedback that is needed for current. Earlier versions of the software give strange IV curves. Later versions of the software handle it better, but there still may be bugs.

Now you are ready to get an IV curve. Simply click on the 'do it' button. The button label then changes to "stop". When it changes back to "do it" the curve is finished.

If you were using the 'Apply during triggered Dwells' box, instead of clicking 'do it', go ahead and do your force curves.

2.7.1. Dithering

It is also possible 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.

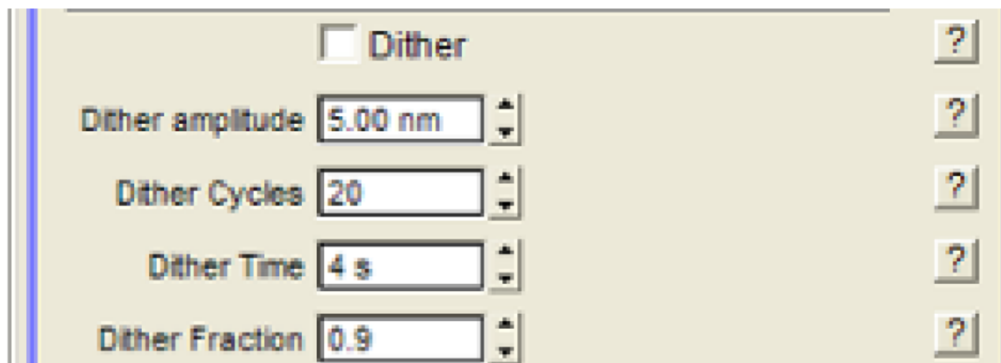


Figure 2.9.: [Dither] The dither function will create a spiral pattern the probe will trace out. This is intended to remove debris or to score the surface in order to get through oxides, thin films, or contamination on the sample surface.

The dither itself will be a spiral that starts at the point of the IV curve, then moves out by 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.

2.7.2. 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.

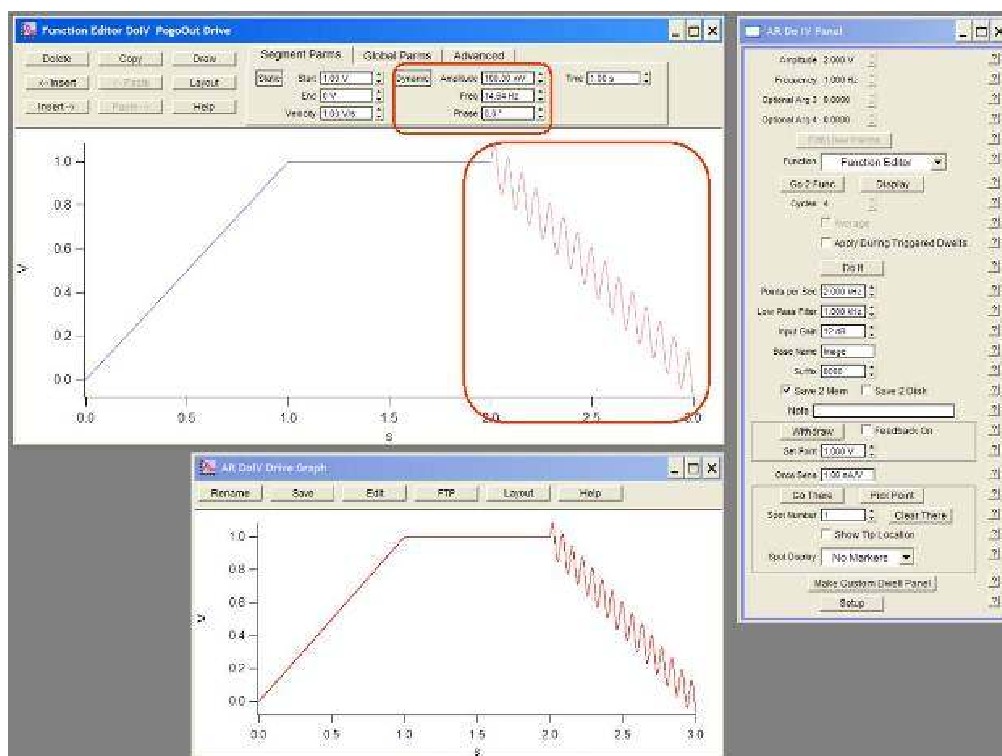


Figure 2.10.: [The Function Editor] The function editor can be used to make a variety of drive waves for current measurements. Insert new segments using the Insert button. Select different segments by clicking them. Each can be modified independently.

Under Global Parms, the entire drive wave can be modified with a multiplier, and offset, or a sine wave dither. By combining local dithering with global dithering, it is even possible to drive the wave with two sine waves.

The tools on the upper left can be used to insert segments Delete, copy, paste, etc., are all options. 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.

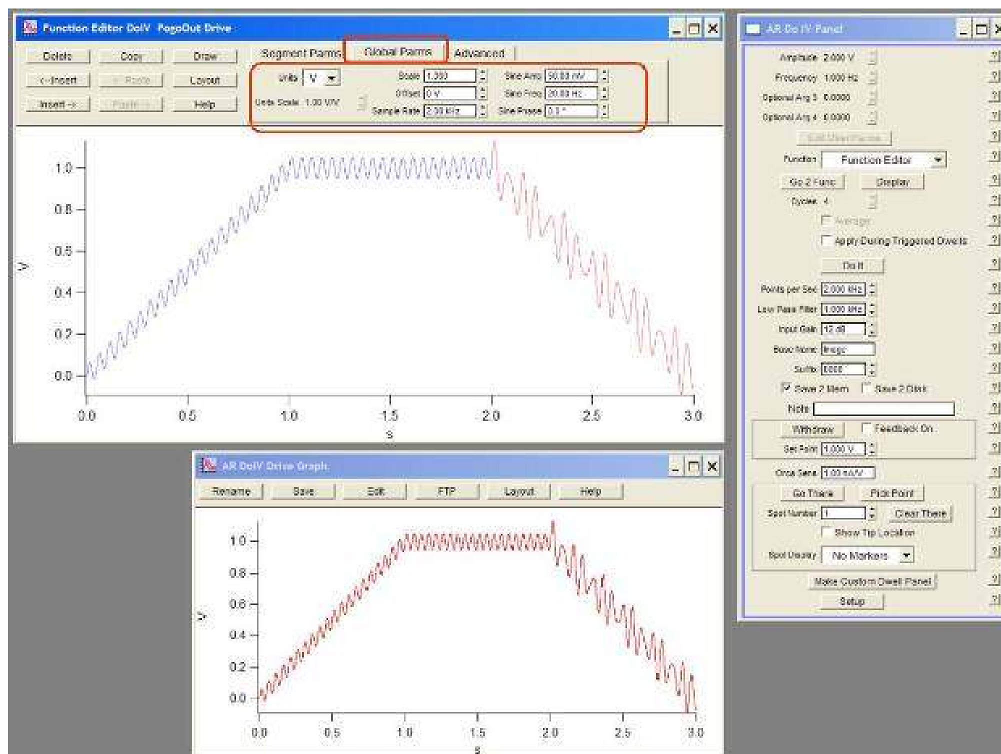


Figure 2.11.: [The Function Editor 2] Use the Global tab to modify all the individual segments at one time. Of particular use in this section is the Scale field. This allows you to scale up or down the entire wave function you have created. So if you had a custom wave from 0-1V with a dwel, scaling it by 5 would make it go from 0-5V.

Warning	The drive wave is controlled by a 100 kHz digital to analog converter (DAC). This means that very fast changing waveforms can be created, but the output bandwidth is limited by nyquist theory to 50kHz. Take an FFT of your waveform if you need to know how high a bandwidth you would need to drive it.

2.7.3. Viewing I-V curves on the MFP3D

I-V curves on the MFP3D are treated like force curves. The actual data is treated as a dwell on a force curve. To display the curve, open the MFP3D Force Display Panel . If you have just taken some I-V curves, then the 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.

Next click on one of the curves. By default, the review panel selects deflection as the y axis and LVDT as the x axis. Since there is no deflection data for this, and since plotting current as a function of distance on a simple IV curve is not useful, you will change these values to 'current' for the y axis and 'bias' for the x axis.

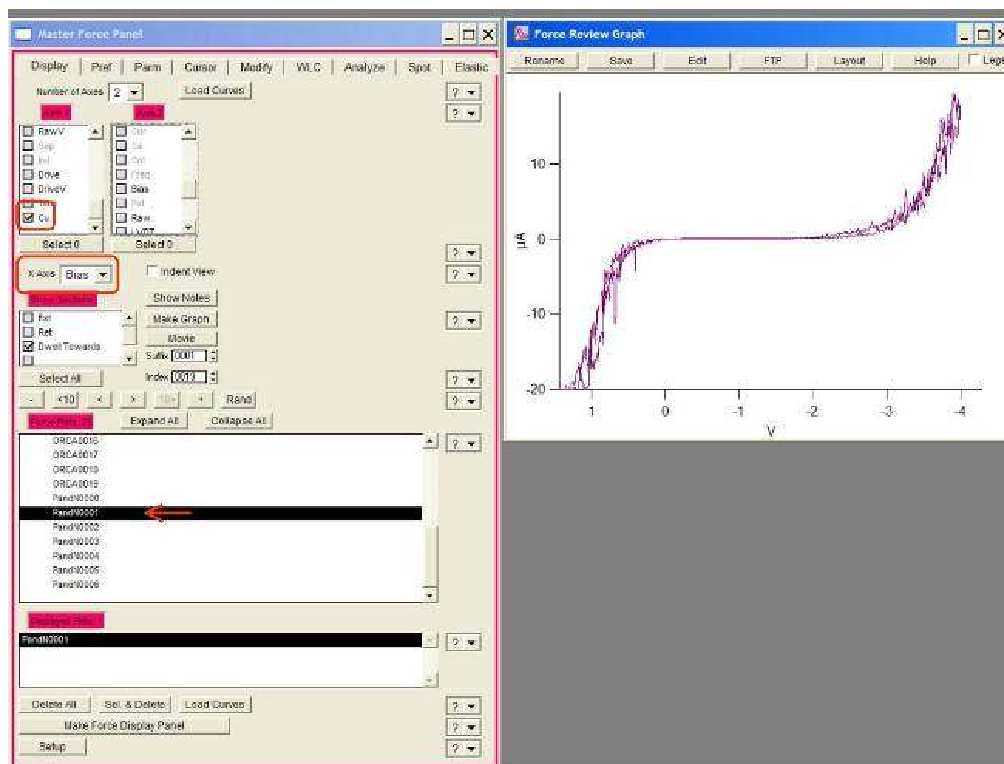


Figure 2.12.: [Current vs. Bias] To display IV curves, open the force review panel, view current (and current2 if you have a Dual Gain ORCA), Then set the x axis as bias, then select the dwell towards under the sections window. Click 'make graph' if the graph does not appear.

The different parts of force or I-V curves are listed under the window near the middle called 'show sections'. The section you want for I-V curves is 'dwell towards'. Uncheck 'Ext' and 'Ret' (these stand for extend and retract), and check 'Dwell Towards'.

You should now see your I-V curve as current vs voltage.

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).

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.

2.7.4. 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

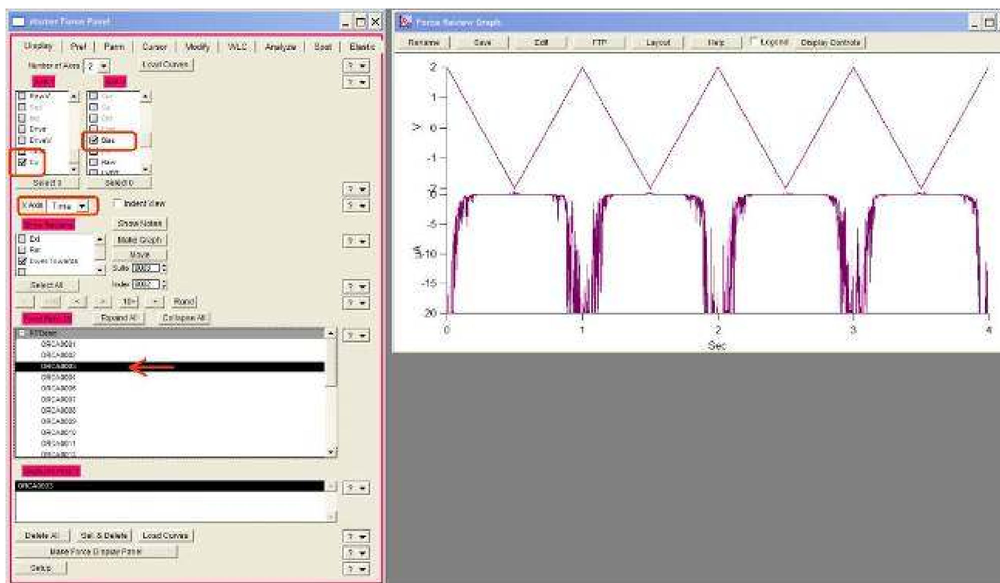


Figure 2.13.: [Current vs time] It is often useful to display the current vs time, particularly for samples that degrade or improve in conductivity with time. Si will oxidize as more current is passed through it if there is moist air near the surface.

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.

Next Set up the system in the test configuration listed section 1.3.1, and measure IV curves with the corrected voltage offsets. Adjust the current offsets on the DoIV panels until zero volts gives as close as possible to zero amps.

Figure 2.14.: [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.

3. Scanning Kelvin Probe Microscopy (SKPM)

CHAPTER REV. 977, DATED 07/23/2012, 12:19. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

by Keith Jones[1]

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3.1. Introduction

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

SKPM Scanning Kelvin probe microscopy

SSPM Scanning surface potential microscopy

SKFM Scanning Kelvin force microscopy

SPM Surface potential microscopy

SP-AFM Surface potential atomic force microscopy

SKPM will be used in this document, as it seems to be the most widely used descriptor for this technique.

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.

3.2. Principles

The principle aim of this technique is to determine the difference between the electrical potential of the probe and that of the sample. The data collected by this sample will represent a combination

[1] Asylum Research East Coast Office, Raleigh, NC, USA.

of three contributing factors: the work function difference, trapped charge, and any permanent or applied voltage between the tip and the sample. For this reason the technique is generally considered a pseudo-quantitative technique, in that it gives an accurate measurement of the potential difference, but that number likely consists of superposition of more than one physical quantity.

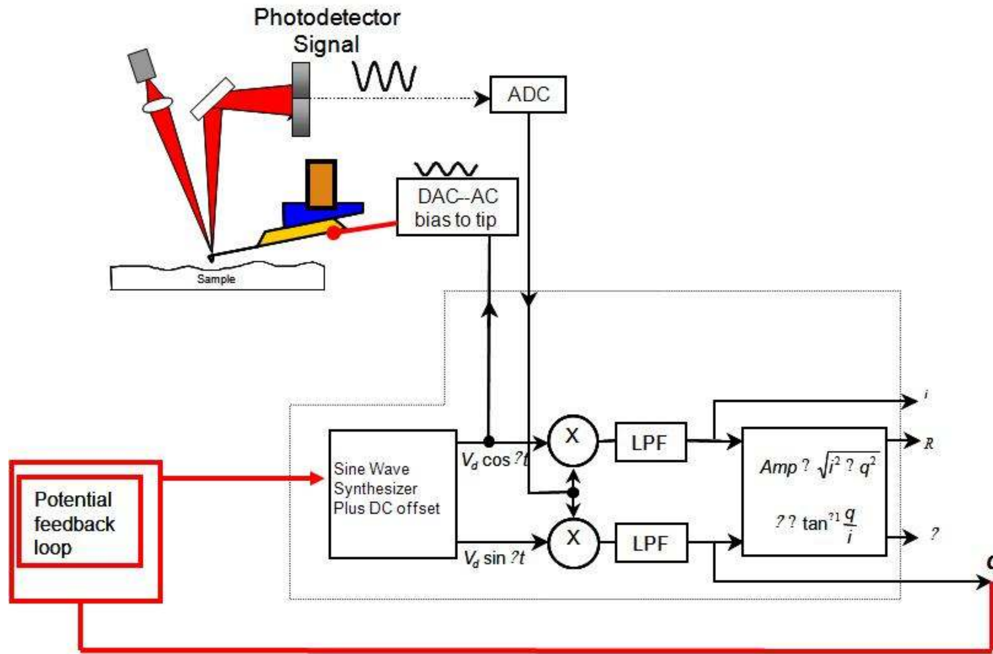


Figure 3.1.: Schematic showing how SKPM is done on the Asylum AFMs. The probe is driven electrically with an AC bias. Any potential difference between the tip and the sample causes the probe to oscillate. These oscillations are then cancelled by a potential feedback loop. The voltage required to match 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. 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 3.1 on page 28 shows a schematic diagram of the setup for the technique.

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

$$F = \frac{1}{2}\frac{\partial C}{\partial z}V^2 \tag{3.1}$$

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}).

$$V = V_{sp} + V_{DC} + V_{ac}\sin(\omega t) \tag{3.2}$$

If we substitute this into Equation 3.1 on page 28 and do some rearranging, we get:

$$F = \frac{1}{2}\frac{\partial C}{\partial z}\left(\left[(V_{DC}-V_{sp})^2+\frac{1}{2}V_{ac}^2\right]+2\left[(V_{DC}-V_{sp})V_{ac}\sin(\omega t)\right]-\left[\frac{1}{2}V_{ac}^2\cos(2\omega t)\right]\right) \tag{3.3}$$

Note that we have three force terms here summed together. The first force is static, meaning it has no frequency dependence. The second occurs at the AC voltage drive frequency. The third force oscillates at twice the drive frequency. The most important term here as far as surface potential is concerned is the second, since this depends not on the square of the voltage, but rather on the potential difference between the tip and the sample, multiplied by the magnitude of the applied AC voltage. This means that if there is a potential difference between the tip and the sample, then when an AC voltage is applied, there will be an oscillatory force at the frequency of the drive and proportional to the magnitude of the applied voltage and to the potential difference.

Further, if we make $V_{DC} = V_{sp}$, with a potential feedback loop between the tip and the sample, then the oscillations will be nulled. This is exactly how the technique is done.

3.3. Implementation of SKPM on the Asylum AFMs

The probes used will generally be conductive or heavily doped semiconductors. Insulating probes will not work on the MFP-3D, as the bias is applied to the tip. To implement this technique on the MFP-3D, we use a two pass technique. 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 is used to find the surface potential, and the tip is raised above the surface. To do this, a technique called nap mode is implemented. During a nap scan, the tip is raised over the surface on a point by point basis while the potential feedback loop is implemented. Figure 3.2 on page 30 shows a cartoon of nap mode.

The resulting data from this technique will have a data channel for height and another for surface potential. An example of such data taken on micro gels is shown in Figure 3.3 on page 31. The three discrete layers of surface potential are likely due to incomplete coverage of the three different micro gels. Note that in the section the actual potential differences are visible. In this case one is roughly at the same potential as the tip, one is about 10mV positive, and the other two levels are at -10mV and -30mV. It is important to be sure that data is collected with no processing in the save, since offsets in Surface potential data are relevant, unlike in some other techniques, where relative data is all that is needed.

3.4. Step by Step Instructions for SKPM

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

Note that this instruction set assumes the user is familiar with standard AC mode imaging, and that the user is opening a new MFP-3D template. This description matches software from Jan 2007. Earlier or later software may have differences.

Note that much of what is described here can be more automatically set up using the modemaster template for SKPM. To open this, navigate on the menu to 'user settings' –> Mode Master or [ctrl]+F2.

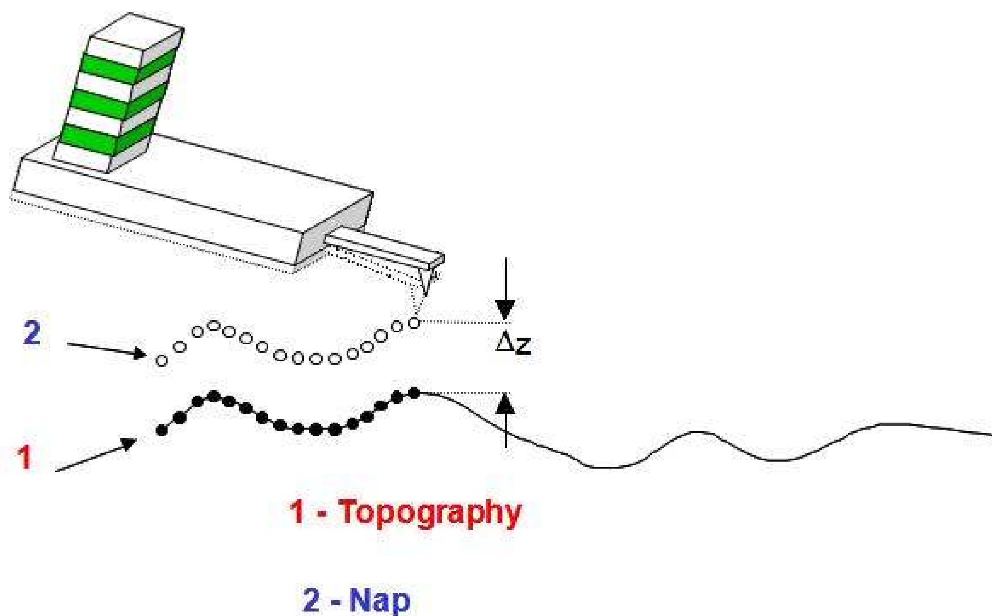


Figure 3.2.: Schematic of nap mode. For each scan line, the system captures the topography. The system then retraces that topography on the same line in order to keep a constant distance from the sample surface.

Note These instructions are still somewhat specific to the MFP-3D AFM. An update for more Cypher specific instructions will be added in the future.

1. 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.
2. Load a conductive AC mode probe into the AFM head, and set up the microscope for standard AC mode imaging.
3. Do a standard tune on the cantilever to find the resonance frequency. Set the target amplitude to 1.0 V and set the target percent to -5%.
4. Open the electrical tune panel by clicking *AFM Controls ▷ Other ▷ Electric Tune Panel menu*. This panel allows the user to find the resonance frequency of the probe when it is driven electrically, and more importantly, to set the phase properly for an electrically driven probe. This phase will be considerably different than that of the same probe driven mechanically with a piezo.
5. Engage on the surface in AC mode as if doing standard AC mode AFM.
6. 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 hovering just above the surface.
7. Click on the button labeled “–>” to copy the drive frequency over to the electric tune frequency.

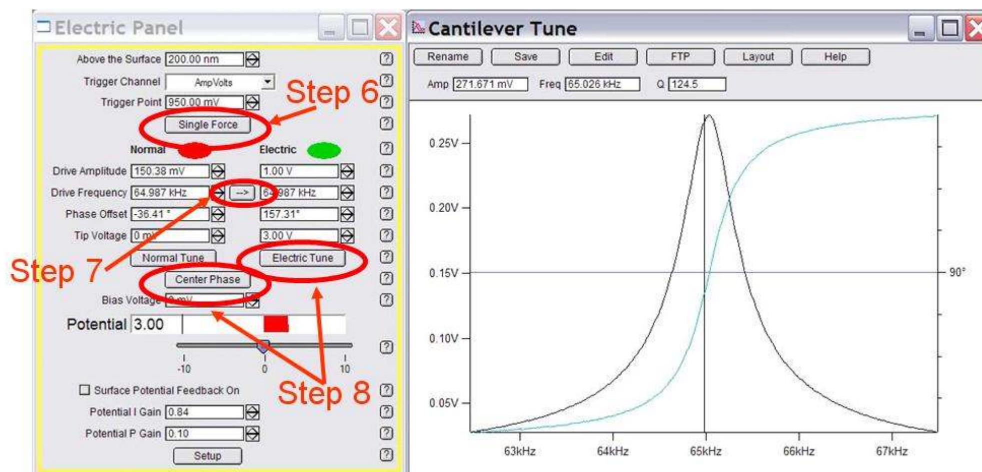


Figure 3.3.: Electric tune panel with the single force, electric tune and Center Phase buttons highlighted. The graph on the right is a properly electrically tuned cantilever.

8. 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 Figure 3.4 on page 32.

Note	The AFM software assumes that the tip is at a positive potential relative to the sample. 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.

9. From the main menu bar select *AFM Controls* ▷ *Nap Channel Panel* (Ctrl+8). The nap channel panel controls how the nap panels are displayed. Channels to be viewed must be selected in the main scan in the master channel panel.

10. Select Potential as a data channel in the master channel panel. Selecting this automatically turns the potential feedback loop whenever the scanner is imaging. The specifics of the nap channels should be selected in the nap channel panel. This includes setting live flatten to none, and deciding if both the trace and retrace should be selected for viewing or saving. Be sure that the nap potential channel has save planefit set to **none**.

11. From the main menu bar select *AFM Controls* ▷ *Nap Panel5* (Ctrl+9). The nap panel controls the real time operation of the system in nap mode. For nap mode, the system will make two passes back and forth (for a total of 4 individual scans across a single line) for every scan line. The first two passes gather topography, the next two, in the case of surface potential, raise the tip off of the surface on a point by point basis and collect the surface potential data. The

Nap Panel

Nap Mode: Nap

	Surface	Use	Swap	Nap Parms
Integral Gain	3.00			10.00
Proportional Gain	0.00			0.00
Set Point	800.00 mV			800.00 mV
Drive Amplitude	100.00 mV		✓	1.00 V
Drive Frequency	75.000 kHz		✓	75.000 kHz
Drive Amplitude 2	100.00 mV			100.00 mV
Drive Frequency 2	450.000 kHz			420.000 kHz
Phase Offset	0.00 °		✓	90.00 °
Tip Voltage	0 mV			3.00 V
Sample Voltage	0 mV			0 mV
User 0 Voltage	0 mV			0 mV
User 1 Voltage	0 mV			0 mV
Delta Height				50.00 nm
Start Delta Height				0 nm

Frequency

FM Feedback On

Freq I Gain	5	DART I Gain	5.5
Freq P Gain	0	DART P Gain	0

Nap Channel Panel

Setup

Figure 3.4.: Nap Panel configured for a surface potential scan. The numbers in the field may vary depending on your sample.

'surface' Column shows the settings for the main, or on the surface scan. The 'parm swap' Column shows the settings that will be used during the surface potential scan. Checking the box between the two columns (under the 'swap' column) that correspond to a row will cause the system to override the 'surface' settings with whatever is entered into the 'Parm Swap' column during the nap pass.

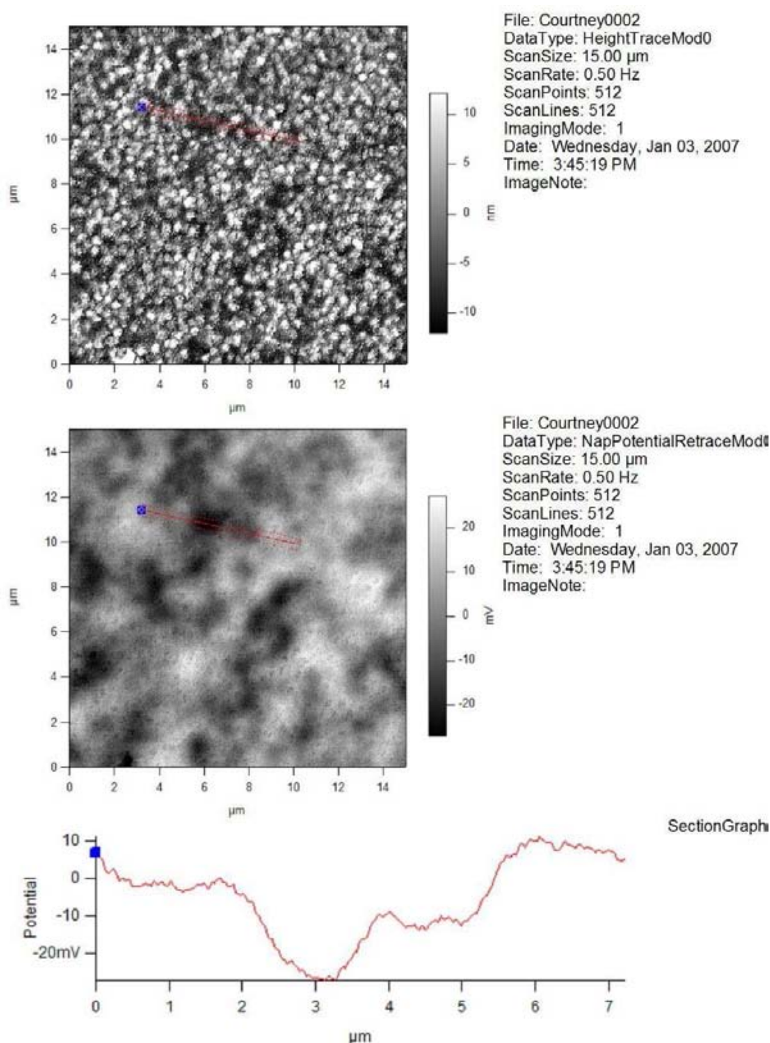
12. Select Drive amplitude, Drive Frequency, and Phase Offset under the swap column. See Figure 3.5 on page 33
13. Set the nap mode to 'nap'.
14. Set the delta height to 40nm. This will be lowered later if you want to try to get better lateral resolution. Because the reference height is based on the 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 significant, obvious scan line errors will occur.
15. Set the Start Delta height to 40nm. 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.
16. Set your scan speed and scan size to reasonable values for the sample you are imaging.

17. Start the scan.

18. 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.

19. 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.

20. Be sure to check the 'save images' so you save any good data you get.



Sample Courtesy Andrew Lyon, Ga Tech

Figure 3.5.: Height (top) and Surface Potential (bottom) image of a three-layer microgel sample. The variation in surface potential is likely attributable to incomplete coverage of the three different microgel layers. The section shows three discreet potential levels on the sample. Sample and images courtesy of C. Sorrell and A. Lyon, Georgia Institute of Technology.

4. Electrostatic Force Microscopy (EFM)

CHAPTER REV. 988, DATED 07/31/2012, 14:32. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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4.4 Step by Step Instructions for EFM . . . 39

4.1. Introduction

Electric Force Microscopy is relatively simple in concept and operation, but often requires some considerable thought in interpretation. The technique has the tip oscillating flexurally 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 areas of conductivity vary 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:

When a conductor is introduced into the electric field (see Figure 4.1 on page 35), 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. The phase lag of the response of the cantilever relative to the drive is sensitive to force gradient changes. Thus, conductors cause a phase shift that is detectable by the lock-in. This is particularly apparent when dealing with conductors in an insulating matrix. For insulators in a conducting matrix, the only contrast can be achieved when the conductors are on the surface, and that phase shift is minimal and often difficult to detect. If the insulating particles are below the surface, then they cannot be detected, as their presence is masked by the conducting surface (see Figure 4.2 on page 36).

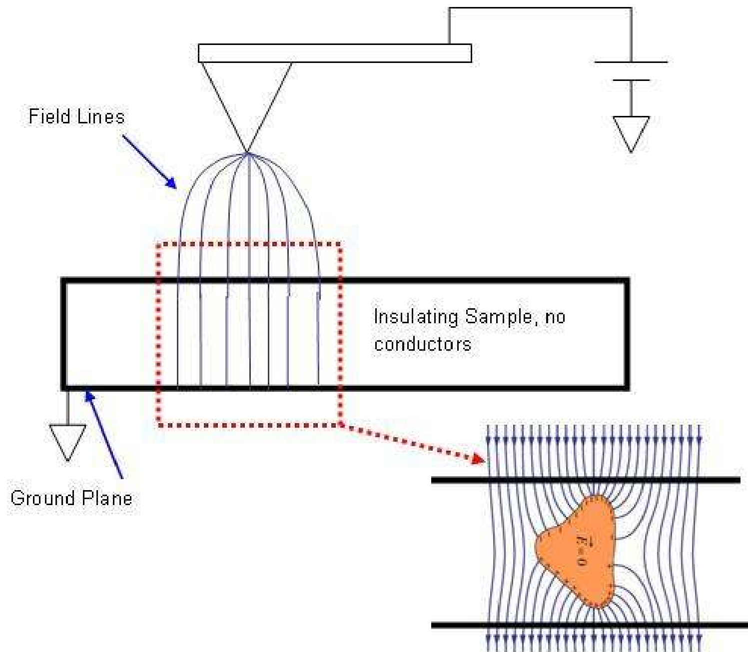

Figure 4.1.: This schematic shows how the introduction of a conductor into the electric field between the tip and sample can effect the field lines. The shift in the gradient due to the presense of the conductor causes a shift in the resonance frequency of the cantilever and therefore a shift in the phase and aplitude, which are then collected as the EFM data. The phase indicates whether the shift is attractive or repulsive. Attractive forces show a shift in the phase to higher than free air value. Repulsive forces shift the phase below the free air value.

4.2. 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 a capacitor. To bring the charge element dq to a positive electrode in a capacitor with potential V, you need energy:

$$dU = V dq \tag{4.1}$$

but for a parallel plate capacitor

$$V = \frac{q}{C} \tag{4.2}$$

where q is the charge and C is the capacitance. So

$$dU = \frac{q}{C} dq \tag{4.3}$$

Integrating gives the energy:

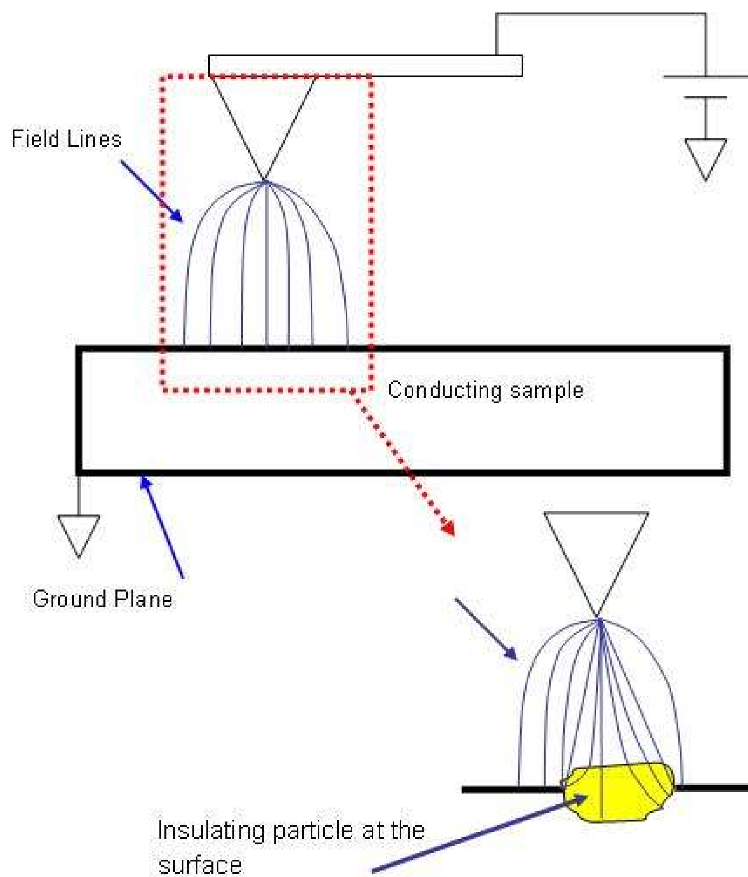

Figure 4.2.: Effects of introducing an insulator into a metal sample. EFM is generally not suitable for this sample configuration, as the field lines show little shift. Any shift would be difficult to discern from the changes in topography.

$$U = \int_0^Q \frac{q}{C} dq = \frac{1}{2}\frac{Q^2}{C} = \frac{1}{2}CV^2 \tag{4.4}$$

Differentiating with respect to distance gives the force on the cantilever due to the electrostatic field (simplifying to the field only in z, or normal to the sample surface):

$$F = \frac{dU}{dz} = \frac{1}{2}\frac{dC}{dZ}V^2 \tag{4.5}$$

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 4.1 on page 35, 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 3 on page 27.

EFM employs two techniques to deliver data. The first is phase detection. In phase detection, the cantilever is oscillated near resonance above the surface. Force gradients between the tip and the sample cause a shift in the phase between the drive and response of the cantilever. The principles of this interaction are derived from the simple harmonic oscillator (SHO) model, where the tip is a simple harmonic oscillator, and tip-sample force gradients act to damp the motion of the tip. The drive of the SHO system is the drive piezo that drives the cantilever substrate.

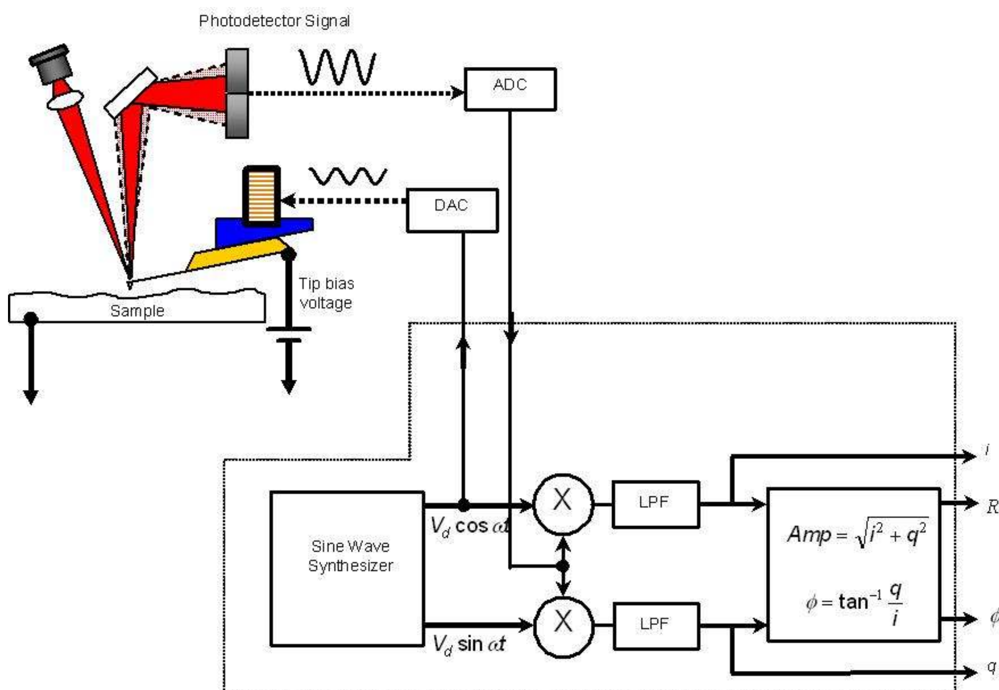

Figure 4.3.: Schematic of Electric Force Microscopy. The Probe is oscillated both during the main scan and during the nap scan. During the nap scan, the probe is lifted off of the surface, and a bias applied between the tip and sample to show the location of areas that are conductive or strongly charged.

The electric field is decoupled from topography by performing two scans over each scan line. In the first, the normal topography is taken. On the second, on a point by point basis, the tip is raised above the surface and a phase measurement taken to see how the electric field lines vary as a function of lateral x-y position. This is done while a bias is applied between the tip and the sample. An illustration of this is shown in Figure 4.4 on page 38.

4.3. Nap Mode

Nap mode on the MFP-3D 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. The second

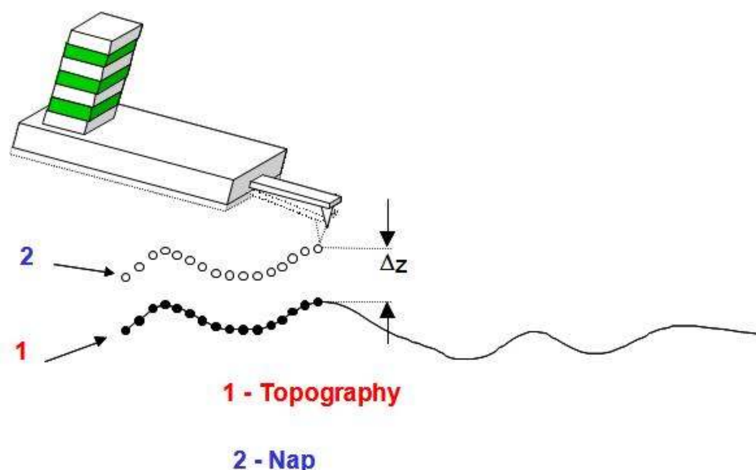

Figure 4.4.: During nap mode, the probe is first scanned across the sample surface, then raised to a delta height defined by the user. This allows for detection of long range forces between the tip and sample.

is the nap channel panel, which allows the user to choose channels that will be displayed while running EFM. The nap panel can be accessed by selecting *MFP Controls ▷ nap panel.*

Nap mode on the AFM is fairly simple in principle. Two passes each of a trace and retrace are made on the sample surface. The fist is a normal topographic scan, and in general can be in either contact or AC mode (for EFM AC mode is used). The second trace and retrace set are definable by the user as one of four options in the nap panel. Nap mode is selectable on the nap panel under the drop down selector menu listed as 'nap mode'.

The first is normal, and simply does an ordinary scan. This setting is the equivalent of setting nap mode to disabled.

The second is nap, and causes the system to scan above the surface on a point by point basis. The resulting motion of the probe will be to trace out the topography a fixed distance from the surface on a point by point basis. Tip-sample force feedback is off during this type of nap scan. This technique is generally used for EFM, and normally does the best job of reducing topographic features affecting the EFM scans. This technique is shown in Figure 4.4 on page 38.

The third setting is called 'parm swap', and will leave the tip and sample in feedback on whatever channel is being used as the primary scan. It will, however, use a different, user definable set of parameters. The parameters that may be changed for the main and nap scan are listed in the upper right corner of the nap panel and are:

Integral Gain, Proportional Gain, Set Point, Drive Amplitude, Drive Frequency, Tip 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 a user wants to have two scans taken over the same area of the sample with interleaved scan lines. One scan is to be in repulsive mode, and the other is to be in attractive mode. The user may select a set point and drive amplitude as the two parm swap parameters. For the first scan the user may chose a frequency that is to the low side of the resonance peak, and an amplitude setpoint that is 50% lower than the free air amplitude. This will tend to drive the cantilever into repulsive mode. For the parm swap scan the user may choose a drive frequency that

is slightly higher than the resonance peak, and an amplitude setpoint that is 85 or 90% of the free air amplitude. The user will then select the channels in the main scan that are to be displayed. The user will then open the nap channel panel, and select the channels that are to be displayed for the nap mode trace and retrace. Note that in order to bring up a channel in nap mode it must be selected in the main scan.

The fourth setting in nap mode is 'snap'. This refers to linear, or 'stupid' nap mode. In this case, the tip is raised above the surface, and rastored back and forth during the nap trace and retrace, ignoring topography, except to compensate for the slope of the sample. Generally, either 'nap' or 'snap' mode will be used for EFM and surface potential, or scanning Kelvin probe (SKPM) imaging, and the more common technique is that using 'nap' mode.

The delta height parameter is used for snap and nap mode. During the nap and snap scans, the tip will be raised above the surface for each point by this amount.

The 'FM feedback On' box allows the user to have a phase lock loop enabled that will monitor the phase, and adjust the frequency so that the phase stays constant. This technique is used both in MFM and EFM. This box should not be checked for normal EFM operation.

4.4. Step by Step Instructions for EFM

1. 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 from ted pella. 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.
2. Load a probe that is conductive, and align the superluminescent diode (laser) as for normal AC mode. The standard cantilever used for EFM is typically an Olympus Electrilever; 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.
3. Open the nap panel, then open the nap channel panel.
4. Tune the cantilever as normal for an AC mode image. Once this is done, it is wise 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.
5. On the main and nap channel panels, choose height, amplitude, phase, Inputi and Inputq. More will be discussed later regarding Inputi and Inputq.
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

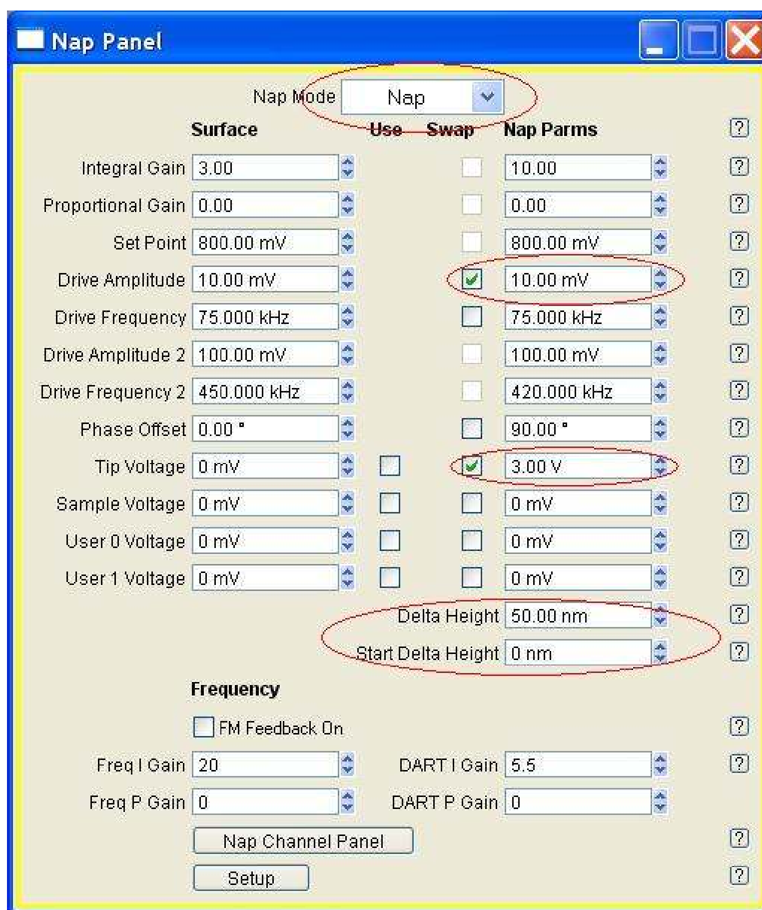

Figure 4.5.: Here the Nap panel is set for EFM operation. The relevant fields have been circled. The Use column in the middle left has check boxes that, when checked, cause that parameter to be used during the main scan. The swap column is has check boxes that, when checked, allow those fields to be used during the parm swap/nap scan.

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 1V. This value should be linearly higher if the free amplitude is higher. Figure 4.5 on page 40 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. As the tip is scanned over the sample for the first frame, these values can be gradually raised until the desired EFM image is obtained. Once the values are set, begin a scan.

9. Once the images are situated so that all of the desired images can be seen, adjustments can be made to the EFM parameters. Note that initially it is helpful to concentrate on the trace and retrace scopes rather than on the images, as it is typically easier to modify the parameters while monitoring real time changes there than in the image.

10. The first priority is to be sure that the topographic scan is good, as the nap scans will rely on this to find each of the nap points. These adjustments are not different from normal imaging, so refer to the standard AC mode imaging chapter for details on this.

11. Once the topography looks good, the drive amplitude and tip voltage can be adjusted under the parm swap parameter column on the nap panel. Slowly raise these two numbers until data begins to appear in the nap phase, nap i and nap q channels.

12. The amplitude during the main and nap scans can be viewed in real time on the sum and deflection meter. By watching the amplitude in real time, the user can ascertain fairly quickly if the tip is striking the surface during the nap scan. To do this, watch the amplitude during the nap scan. The drive amplitude during the nap scan should generally be lower than the drive amplitude during the main, or surface scan. The real time amplitude display for the nap and main channels can be distinguished by noting that the amplitude for the real time should match the setpoint under the surface column on the nap panel, and the setpoint parameter on the main scan panel.

13. At this point, the operation section of performing EFM is finished. From here, the user must adjust parameters to improve contrast to a point where the images are usable. To be sure that images are related to the electrical properties of the sample and not the topography, the tip bias can be slowly changed during scanning. If the signal is related to electrical properties, then the phase contrast between the different features should increase with increasing voltages, rather than a general offset of the phase for the entire surface.

Note	Note also that the EFM contrast is not just a result of the interaction between the tip and the sample. The cantilever, tip cone, and tip apex all contribute to the total contrast in EFM. If the tip apex is the dominant contributor, the highest resolution will result. It has been shown that this is best achieved if the tip is very close to the surface, so as minor adjustments are made, the tip should always be kept as close to the surface as possible. Further, a larger tip radius tends to increase the distance at which the major contributing feature is the tip apex, so it is recommended that the tip be no smaller than the smallest features on the surface that will be imaged.

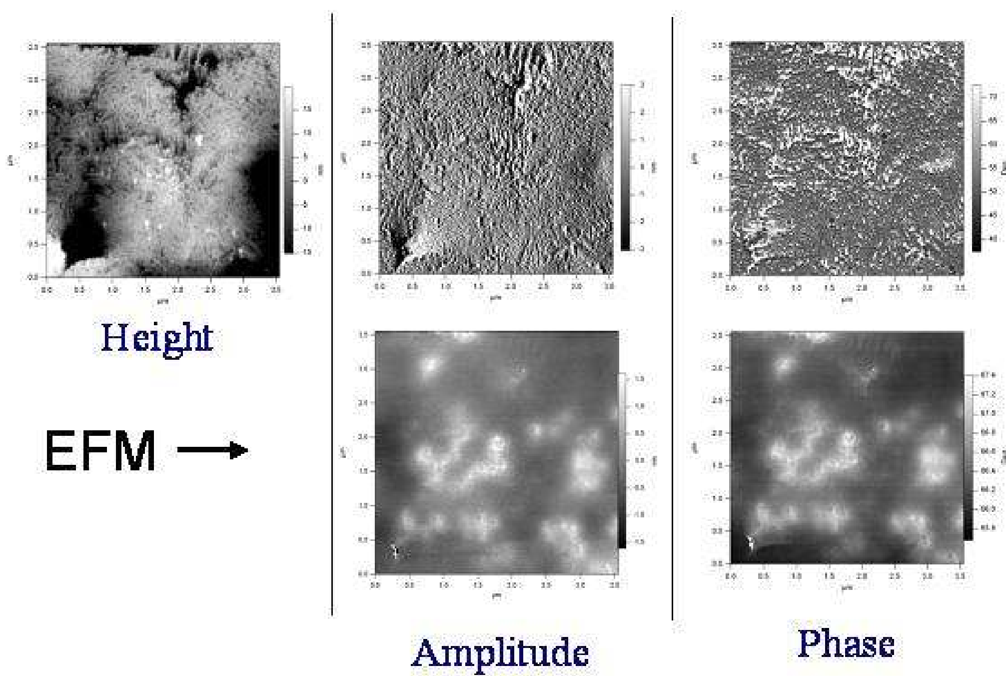

Figure 4.6.: EFM image of polypropylene with carbon black particles (CB) imbedded into it. The areas of contrast in amplitude and phase are due to the presence of CB particles either at or below the surface of the polypropylene.

5. AM-FM Viscoelastic Mapping

CHAPTER REV. 1003, DATED 08/28/2012, 12:08. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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5.1. Overview

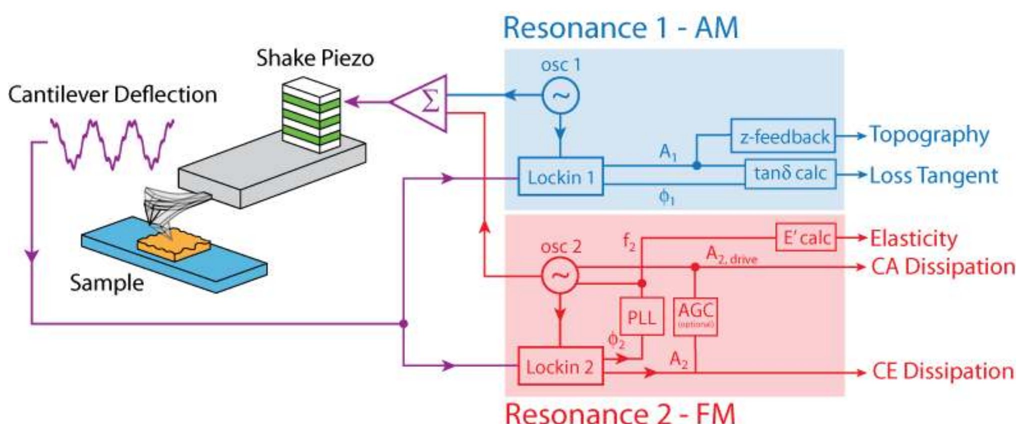


Figure 5.1.: AM-FM Mode. The first mode amplitude is controlled to create a topographic image of the sample (blue). The second mode is used to calculate elasticity and tip-sample stiffness (red).

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 degrees, 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 maintains a constant second mode oscillation amplitude which is used to calculate the tip-sample stiffness. This parameter is called Drive Set Point in the software.

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:

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

5.2. How-To Guide

1. For the most recent features use the latest 111111 MFP3D software version. As of July 16, 2012, this would be 111111+0805. If you are a registered user on the Asylum Research Forum, you can download the software at https://support.asylumresearch.com/forum/content.php?14-Beta-Releases.

2.

Select AM-FM Mode:

- The ModeMaster Panel appears when you first open up the MFP3D software. Select *AR Modes* ▷ *NanoMechPro* ▷ *AMFM*

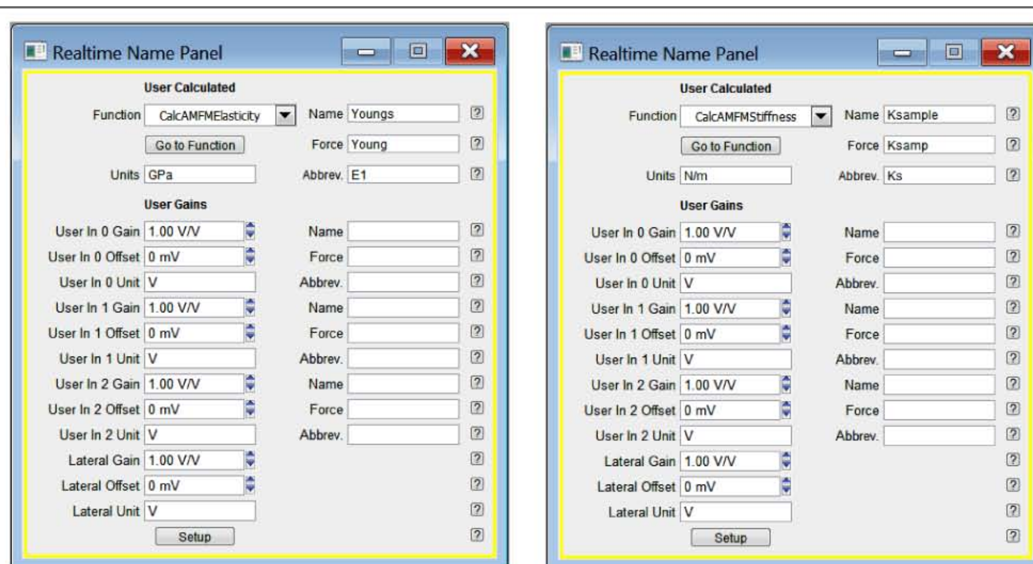
Note that the asterisk in the last button indicates that this mode has been edited and saved by the user. If your AMFM button has this, you can revert back to the factory settings by shift-clicking on the button.

Note: Earlier 111111 software versions can be used; however, the AMFM Modemaster button is located under "Contact Mechanics" button.

Note: Some of the first AMFM Viscoelastic Mapping cantilever holders were shipped with incorrect ID resistors, meaning that the software does not recognize them. In this case, an error message may pop up when clicking on the final AMFM ModeMaster button. To get around this, open up the Sample and Cantilever Holder panel and explicitly choose the "High Frequency Cantilever Holder". After choosing this, clicking on the AMFM ModeMaster button should work properly.

3. The Usual Master Panel and Master Channel Panel will appear.

4.



Note the appearance of the Realtime Name Panel:

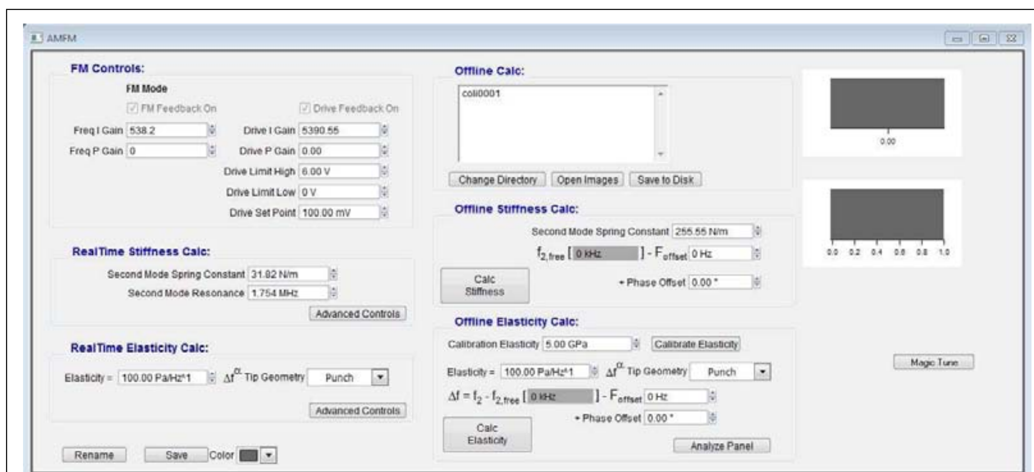
- This panel selects User Calculated Functions (i.e. Stiffness/Elasticity). This panel is used to choose the function that calculates either stiffness or elasticity. This panel should remain open but can be minimized to reduce desktop clutter.
- If you need to re-open it, go to *AFM Controls ▷ Other ▷ Realtime Name Panel.*

5.

Uses of the Realtime Name Panel

- This panel is used to choose the function that calculates either stiffness or elasticity. If you change the User Calculated Function, make sure you have the appropriate channel selected. For example for Stiffness, the 5th channel should be Ksample and for Elasticity it should be Youngs.
- Verify that the units are correct. They should be GPa and N/m for CalcAMFMElasticity and CalcAMFMStiffness, respectively.
- The tip-sample stiffness is calculated and imaged by selecting *Function ▷ CalcAMFMStiffness* (left).
- The Elasticity is calculated by selecting *Function ▷ CalcAMFMElasticity* (right). The rest of the parameters should appear as shown in Step 4 on page 44.

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, a second feedback loop will be enabled that keeps Amplitude2 at a setpoint. In the Step 6 on page 45, it is set to 100mV. The best results are from keeping this value as small as possible, while still maintaining good signal to noise. Typical values are 20-50mV. More than that will result in large higher mode amplitudes which may perturb the cantilever dynamics.

Note: In the earlier versions of this panel, there is a units bug for the Drive Set Point that converts mV to volts the first time a new value is entered. Make sure that the value is set in mV before imaging. If you end up running the higher mode at a volt or more, you may end up damaging the tip and then will need to replace the cantilever. This bug has been fixed in 111111+0805.

- Choosing between Stiffness and Elasticity is accomplished through the real time name panel as shown in Step 4 on page 44.

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 AC240TS cantilevers appear to work the best so start with them and switch to AC160TS if you need a stiffer cantilever.

9. If you plan on measuring the tip-sample stiffness (Ks), 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”. To calculate the second mode spring constant, we are using the equation .

After tuning the 1st and 2nd resonances the software automatically calculates the Second Mode Spring Constant. However, there is currently a bug where the calculate value is only the frequency ratio squared. For a more accurate value multiply this number by the first mode spring constant, and manually enter it into the software.

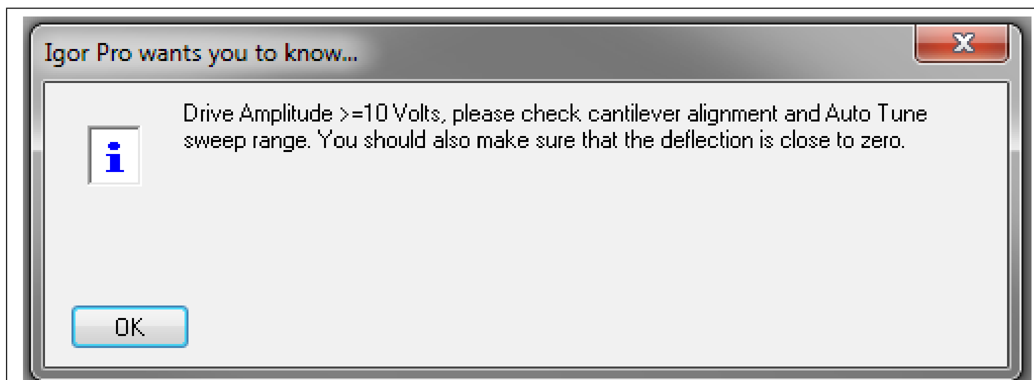
Note on Stiffness (subject to change): We are actively working on improving the stiffness calibration for the higher modes. For now, please assume that the higher mode stiffness can be described by , where is the higher mode stiffness, is the first mode stiffness calibrated in the normal manner, is the higher mode resonance frequency and is the first resonance mode frequency.

10. Auto tune the cantilever on the first resonance.

- Check Auto Tune Low and High for both first and second resonances – adjust accordingly. Note: The Target Amplitude is 2.00V and Amplitude Setpoint is 800mV. This will ensure that you will be imaging in Repulsive Mode, i.e. phase < 90 degrees, ideally < 60 degrees. Since you are selecting a frequency on the peak, do not use a Target Percent offset.
- Make sure that your second mode free amplitude is significantly lower than your first mode free amplitude (i.e.< 500mV); otherwise, the software will not be able to find the first resonance and adjust the free amplitude to 2V.
- fIf you get an error seen in Figure 6, check the switch on the back of the holder. Move it to the other end and retry the tune.

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.

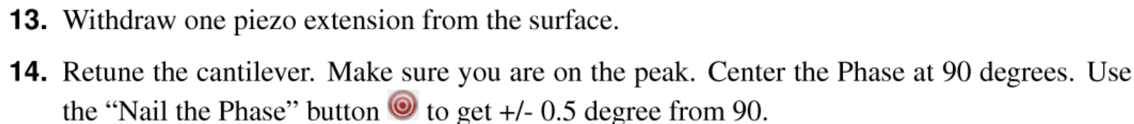


Autotune Error:

- Auto Tune error when the switch on the back of the holder is on the wrong side. In this case your cantilever holder has a switch on it. Please change the position of the switch and tune again.

12. Engage on the surface.

13. Withdraw one piezo extension from the surface.

14. Retune the cantilever. Make sure you are on the peak. Center the Phase at 90 degrees. Use the “Nail the Phase” button  to get +/- 0.5 degree from 90.

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

15. Re-engage on the surface and begin scanning the surface. It is also recommended that you scan at 90 degrees 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 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 that 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, so don’t be too worried about going above the instability point.

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

> “I’ve found it’s often easier to kill the amplitude 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 in the AMFM Panel.14. To display accurate values for the elasticity channel, adjust the “Elasticity” factor, located under “RealTtime Elasticity Calc” in the AMFM Panel.

- First, select a reference sample with known modulus.
- Change the elasticity factor until the value in the Youngs image reflects the known modulus.
- Place your “unknown” sample on the AFM and scan it without changing the elasticity factor. Assuming that the tip shape hasn’t 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.

6. iDrive Imaging

CHAPTER REV. 1013, DATED 09/21/2012, 15:36. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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6.1. Theory

6.1.1. 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 on the following page. 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 $\vec{B}$:

$$\vec{F} = \vec{i} \times \vec{B} l \tag{6.1}$$

$$F = ilB \sin\theta \tag{6.2}$$

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

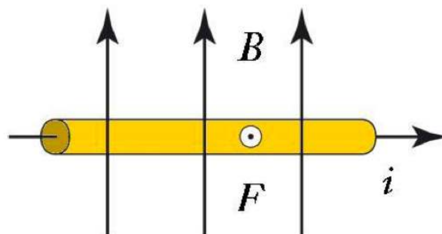


Figure 6.1.: The Lorentz Force F is orthogonal to both current i and the magnetic field B and is pointing out of the page.

Since the force is proportional to the current, if the current is oscillated, the force acting on the wire also oscillates. In 2001, Silberzan and colleagues realized this would make an ideal cantilever actuation mechanism that directly drives the cantilever[1]. Their patented invention avoids the well-known complications of driving the cantilever acoustically and allows for magnetic actuation without the need for coating the cantilever with a ferromagnetic film. Ferromagnetic films can be problematic for fluid work since they are subject to oxidation (rust!) and may contain material that is toxic for various biological samples. These and other iDrive advantages are listed below:

- Avoids magnetic materials in the solution. Magnetic materials typically include rare earth ions that easily oxidize and perhaps worse, can be toxic. The iDrive cantilevers are coated with inert gold, avoiding this complication.
- Avoids the use of an oscillating magnetic field. Experimental studies have shown that an oscillating magnetic field can inadvertently lead to acoustic driving of the cantilever[2]. This unwanted acoustic energy often manifests itself as bumps and dips in the cantilever tune[3]. Because the current loop is made by the cantilever itself, the drive is completely localized. This means that the tunes are smooth and that other parts of the microscope mechanics are not being excited.
- Because the drive is well defined and is a smooth function of frequency, 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 6.2 on page 52, Insulin Fibrils) and even allows advanced techniques such as Q-control.

Despite the relative simplicity of this technique, there were some engineering challenges that needed to be surmounted. These included:

- Improved response with the addition of a small permanent magnet. Referring to Equation 6.1 on page 50, the drive current through the cantilever can be smaller if the static magnetic field is larger. Although the Earth's ambient field is sufficient for some cantilevers, we were able to reduce the required current to just a few mA by including a permanent magnet in the design of the cantilever holder.
- A cantilever with an integrated circuit loop and terminals that direct the current flow through the legs.

[1] Buguin, A./Roure, O. Du/Silberzan, P., Active atomic force microscopy cantilevers for imaging in liquids. Applied Physics Letters, 78 2001, Nr. 19 ⟨URL: http://link.aip.org/link/?APL/78/2982/1⟩.

[2] Revenko, I./Proksch, R., Magnetic and acoustic tapping mode microscopy of liquid phase phospholipid bilayers and DNA molecules. Journal of Applied Physics, 87 2000, Nr. 1.

[3] Han, Wenhai/Lindsay, S. M./Jing, Tianwei, A magnetically driven oscillating probe microscope for operation in liquids. Applied Physics Letters, 69 1996, Nr. 26 ⟨URL: http://link.aip.org/link/?APL/69/4111/1⟩.

Figure 6.2.: Insulin Fibrils imaged in water. Phase signal overlaid onto topography. The "white" regions in the image are from the phase channel and correspond to higher energy dissipation. 1μm scan.

- A cantilever holder that allows quick, easy and reliable connection of the electrical contacts when the cantilever is swapped out.

Using a patent-pending technique, we are able to mass-produce cantilevers with predefined conductive paths and contacts that only require a thin and biologically safe gold coating. Other metals such as Platinum are available upon request.

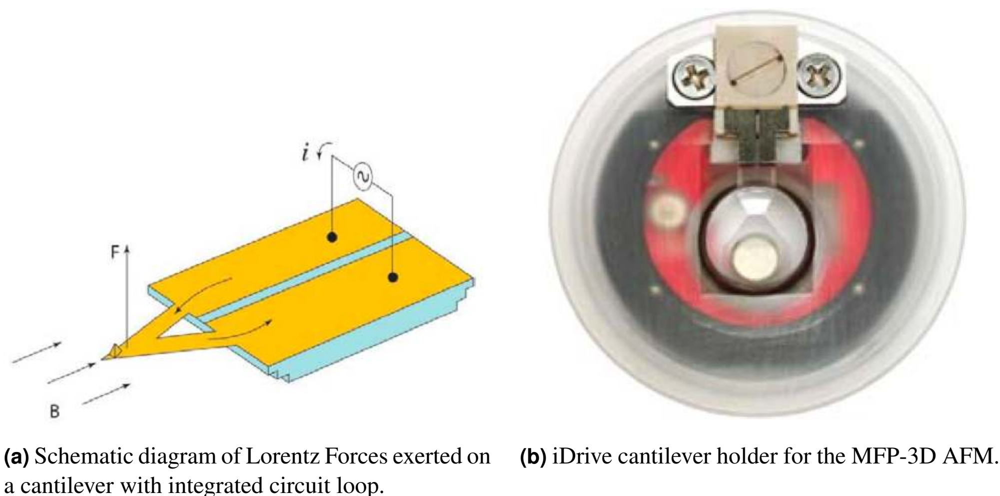
A schematic diagram of the cantilever chip and a picture of the iDrive cantilever holder can be seen in Figure 6.3 on page 53.

6.2. Cantilever Holder Design

For iDrive, a standard cantilever holder with piezo actuation is modified to include a NbFeB magnet in the glass insert, to provide that magnetic field (Figure 6.3b on page 53). 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 clips are connected to the SPM controller and clamp down on the cantilever terminals on the back of the chip. This arrangement allows the controller to supply necessary current to actuate the cantilever. The iDrive cantilever holder still retains its piezo actuation capability so users can switch between piezo-driven (acoustic) AC mode and iDrive AC mode via the software.

6.3. Compatible Cantilevers

iDrive also requires the use of special probes. During the fabrication process, a circuit that includes the legs of the cantilever and terminals are formed. A close-up view of the probe clearly shows each terminal, along the length of the cantilever chip, connected to one leg of each of the two short 100μm long cantilevers (Figure 6.4 on page 54). Only these two 100μm long cantilevers, not the 200μm long cantilever, can be used for iDrive AC mode. This selection is consistent with fluid bio imaging using standard piezo-driven AC mode, where one does not typically use the 200μm long lever. The iDrive cantilever still retains its full functionality as a standard non-iDrive cantilever and can be operated in both contact mode (air and fluid) and piezo- driven AC mode in fluid.



(a) Schematic diagram of Lorentz Forces exerted on a cantilever with integrated circuit loop.

(b) iDrive cantilever holder for the MFP-3D AFM.

Figure 6.3.

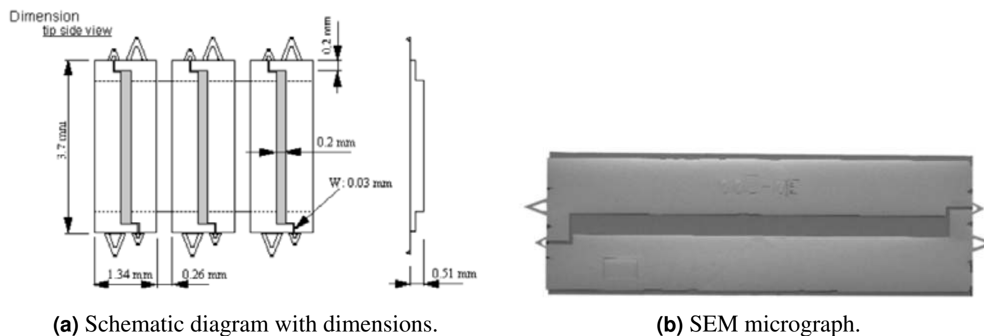
We currently have two iDrive compatible probes, Model Numbers AR-iDrive-N01 and AR-iDrive-N02. The specifications for both cantilevers can be found at the Asylum Research Probestore (http://www.AsylumResearch.com/ProbeStore). Model Number AR-iDrive-N01 is the standard cantilevers included with the iDrive accessory. The resonant frequency of these cantilevers in fluid is approximately 6-10 kHz in fluid. These cantilevers have a spring constant of approximately 0.09N/m and are ideal for imaging soft samples. A stiffer cantilever is also available (Model Number AR-iDrive-N02) if a higher spring constant of 0.61N/m is desired.

The resistance across the cantilever chip should be between 8-20 ohms, depending on whether the two 100μm long cantilevers are intact or not. The value will be higher if there is only one cantilever attached to the chip. Although it is not necessary to check the resistance every time a new cantilever has been placed in the holder, you may want to make this measurement to troubleshoot the iDrive holder (i.e. no peak appears in the cantilever tune).

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

1. Select “resistance” on your multimeter and, if necessary, the low resistance range.
2. Place the probes on each side of the cantilever chip (i.e. one to the left and the other to the right of the terminals).
3. 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.

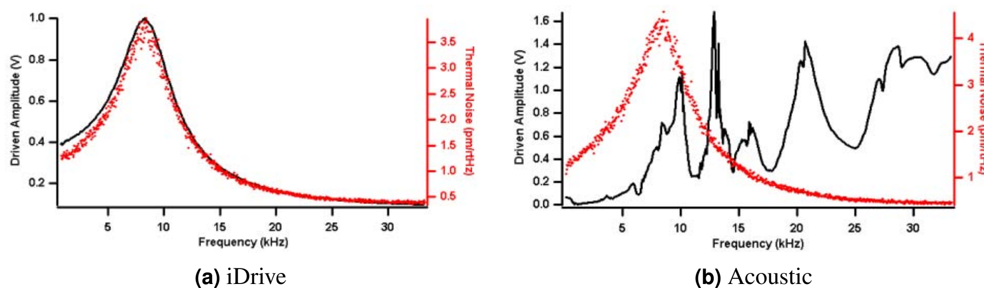


(a) Schematic diagram with dimensions. **(b)** SEM micrograph.

Figure 6.4.: iDrive cantilevers (Model AR-iDrive-N01)

6.4. Applications

Direct actuation of the cantilever has many benefits including simplified fluid imaging, improved interpretation of the phase images and implementation of Q-control. Fluid Imaging iDrive was specifically designed for imaging soft biological and polymer samples in fluid. iDrive simplifies fluid imaging by eliminating the multitude of resonance peaks due to the mechanical coupling of the shake-piezo to both the cantilever and fluid; and therefore, allows the user to auto tune the cantilever in fluid. A comparison between cantilever tunes acquired with iDrive AC mode and piezo-driven (acoustic) AC mode can be seen in Figure 6.5 on page 54, where both methods are compared to the Brownian "thermal" tune.

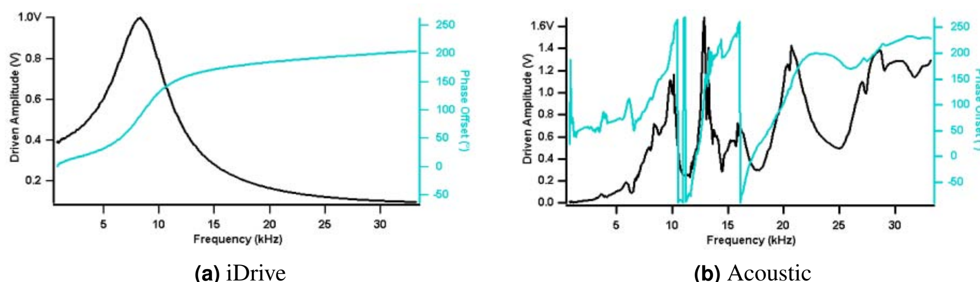


(a) iDrive **(b)** Acoustic

Figure 6.5.: 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).

6.4.1. Phase Imaging

When using iDrive AC Mode, the phase response in fluid is very similar to that seen in air. This is quite different compared to piezo-driven acoustic AC mode. This allows researchers to obtain quantitative phase data. A comparison of the phase response can be seen in Figure 6.6 on page 55.

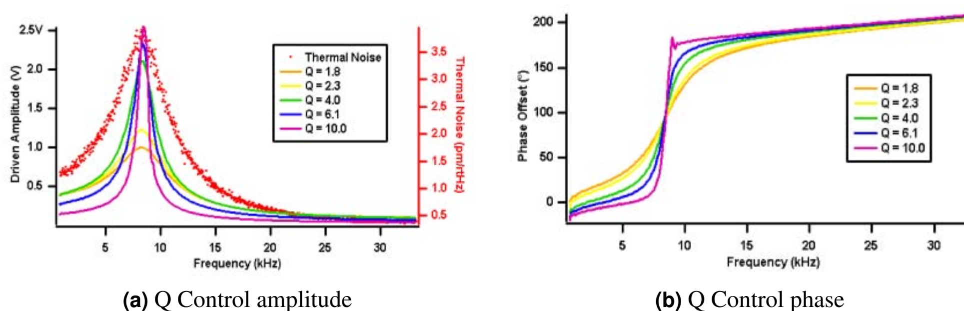


(a) iDrive **(b)** Acoustic

Figure 6.6.: 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).

6.4.2. Q-Control

With a "clean" iDrive cantilever tune, it is feasible to use Q-control (See **??** on page **??**) in fluid 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 didn't always increase. Similar to air, the phase contrast with iDrive in fluid increases with greater Q (Figure 6.7 on page 55).



(a) Q Control amplitude **(b)** Q Control phase

Figure 6.7.: Cantilever tunes (left) and phase offset (right) acquired using iDrive AC mode with increasing Q.

6.4.3. 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 6.8 on page 56, where the cantilever tune is measured at fixed distances above the sample surface. This decrease in the cantilever's oscillation amplitude can be explained by the Simple Harmonic Oscillator Model. The equation is as follows:

$$\omega_{\max} = \omega_0 (1 - {}^1/_{4Q^2}) \tag{6.3}$$

where $\omega_{\max}$ is the maximum resonance, ω_0 is the free cantilever resonance and Q is the quality factor.

These results are consistent with theoretical and experimental data that explains the observed decrease in cantilever resonance and shift to a lower value[4]. The hydrodynamic effect on the cantilever is not seen with piezo-driven acoustic mode since the multitude of peaks that are observed in the cantilever tune correspond to non-cantilever resonances, typically due to the resonances of various hardware components.

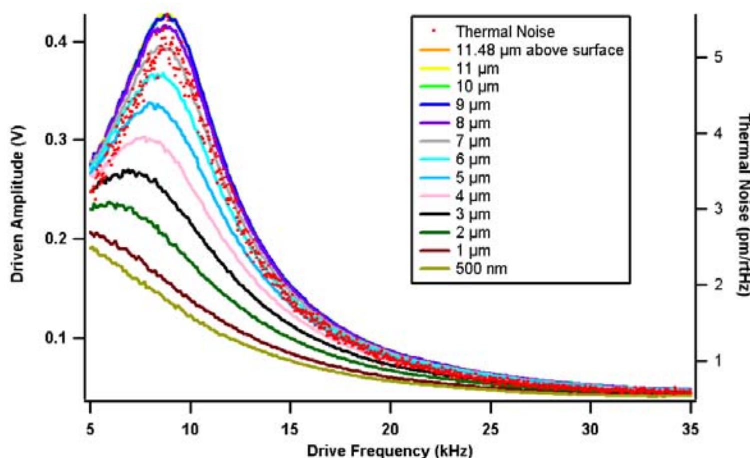


Figure 6.8.: Cantilever amplitudes at different Z positions using iDrive AC mode

6.5. Installation and Operation

6.5.1. Bill of Materials

There are two Asylum Research Model Numbers for the iDrive Accessory:

1. iDrive (new systems)

2. iDrive-R (retrofits)

1. Model Number iDrive (new systems) includes the following items:

iDriveassemblykit 900.236 o Cantilever holder assembly 900.235 o Miscellaneous extras see shipping packing list o iDrive compatible probes AR-iDrive-N01 o Latest version of MFP-3D software 070926 or later

2. Model Number iDrive-R (retrofits) includes the following items:

iDriveassemblykit 900.236 o Cantilever holder assembly 900.235

o Miscellaneous extras o iDrive compatible probes o Pull Down Resistor Board and Install Instructions 458.164 o Latest version of MFP-3D software 070926 or later

The iDrive and standard cantilever holders are easily discernable with red and green circuit boards respectively. The iDrive holder is also serialized for easy reference.

[4] Rankl, Christian et al., Hydrodynamic damping of a magnetically oscillated cantilever close to a surface. Ultramicroscopy, 100 Aug 2004, Nr. 3-4 ⟨URL: http://dx.doi.org/10.1016/j.ultramic.2003.12.014⟩.

6.5.2. Mandatory Controller Upgrade

This mandatory controller upgrade only applies to MFP3D systems that include older controllers. As of April 2008, all new systems are shipped with our latest controller, Model Number ARC2. The ARC2 design already has electronics to protect the cantilever from shorting.

For older (black) controllers, the Pull Down Resistor (PDR) circuit board was designed to ground six output circuits in order to prevent applying too much voltage to the cantilever holder and damaging the cantilevers while in iDrive AC mode. Located in the Crosspoint Panel in the MFP-3D software, these six circuits are Out0, Out1, Out2, PogoOut, Chip and Shake. This grounding also ensures that any re-boot of the controller will not damage the cantilevers. This includes turning the controller OFF then ON as well as rescanning the BUS (by clicking the icon).

If iDrive has been purchased as an upgrade, then the PDR circuit board will need to be added to the controller by the customer.

Note	Follow the step-by-step instructions included with this document called PDR Board Install Note before operating in iDrive AC mode.

6.6. Software Configuration

The iDrive cantilever holder has an ID resistor. This identification allows the software to reconfigure certain parameters for full iDrive functionality.

Note	Igor 6.02 (or later) and a current version of MFP-3D software (070926 or later) are necessary to recognize the ID resistor and provide additional parameters to run iDrive.

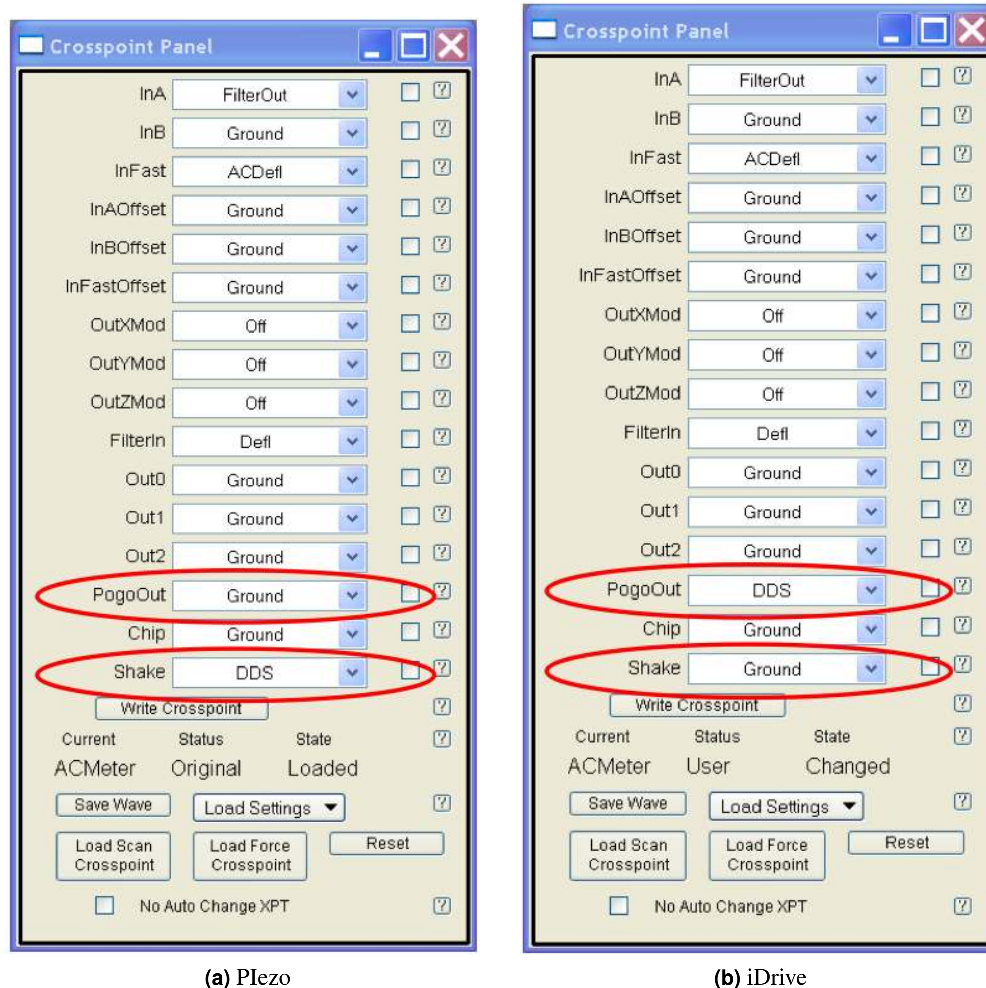
To properly drive the cantilevers in iDrive AC Mode, changes to the PogoOut and Shake parameters in the Crosspoint Panel are required. Although this can be manually done in the Crosspoint Panel (Programming-Crosspoint Panel), the latest version of software simplifies iDrive operation by automatically changing pertinent Crosspoint and Tuning parameters when iDrive is selected (Figure 6.9 on page 58).

6.7. 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. A. Tunethecantilever(TunePanel)
2. B. Select the appropriate amplitude setpoint value and approach towards the surface (Main Panel)



(a) PIezo **(b)** iDrive

Figure 6.9.: Crosspoint Panel configured for piezo-driven AC mode (left) and iDrive AC mode (right).

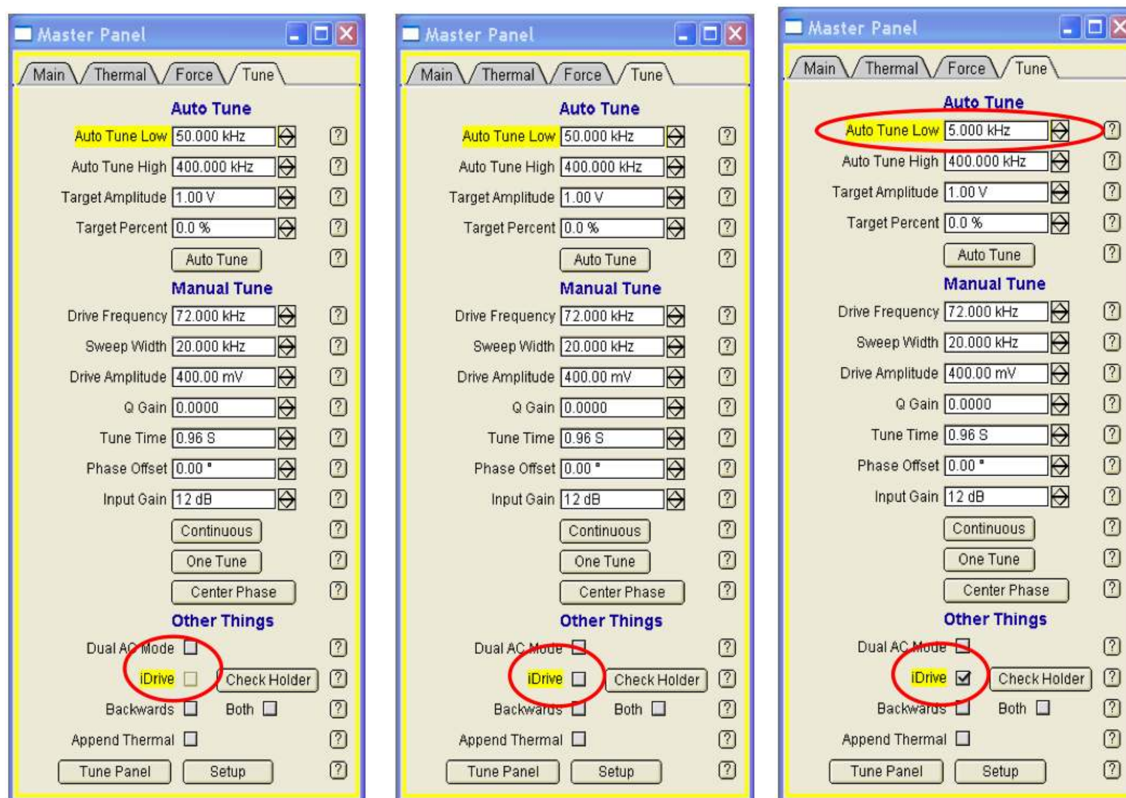
6.7.1. Tuning the cantilever (Master Panel - Tune Tab)

Existing users who have Igor 5 or 6 and older versions of the MFP-3D software will notice some general changes to the Tune Panel in the latest, iDrive compatible MFP-3D software. The parameters have been reorganized into three distinct groups: 1) Auto Tune, 2) Manual Tune and 3) Other Things. Included under "Other Things" are the iDrive box and the 'Check Holder' button (Figure Figure 6.10 on page 59).

If the MFP-3D 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 6.10a on page 59). To un-shade the iDrive box, attach the iDrive cantilever holder to the MFP-3D head and click on the "Check Holder" button (Figure 6.10b on page 59). To select iDrive, click on the iDrive box (Figure 6.10c on page 59). By clicking on (checked) or off (unchecked) the iDrive box, you can toggle between iDrive and piezo-driven AC modes.

When iDrive is selected, two changes take place:

- 1. The PogoOut and Shake parameters are reconfigured.



(a) standard cantilever holders

(b) iDrive cantilever holders but iDrive not selected

(c) iDrive cantilever holders with iDrive selected

Figure 6.10.: Tune Panel configurations.

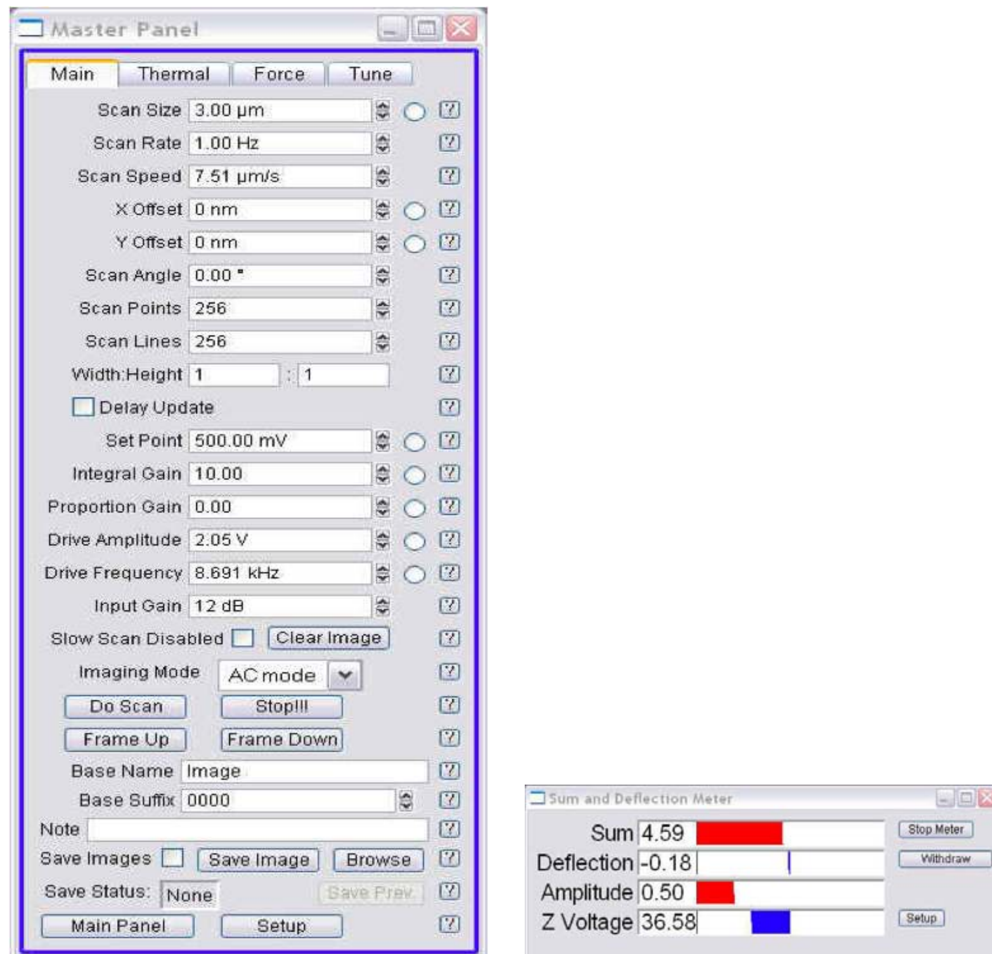
- 2. The Auto Tune Low parameter will decrease to 5 kHz so that the resonance of the AR-iDrive-N01 cantilevers, which is approximately 6-10 kHz, will be within the range for the Auto Tune function. Once the iDrive box has been selected (Figure 6.10c on page 59), verify that the Target Amplitude is set to 1V then click on the "Auto Tune" button. Confirm that the software-selected Drive Frequency is between 6-10 kHz and that the Free Amplitude in the Sum and Deflection Meter Window is 1V. Return to the Main Panel.

Selecting the amplitude setpoint and approaching towards the surface (Main Panel)

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 (Figure 6.11a on page 60). Engage onto the surface as usual. When the free amplitude in the Sum and Deflection Meter window equals the amplitude setpoint and the audible beep is heard, continue to lower the head until the Z Voltage position is approximately 35V (i.e. slightly in the BLUE, Figure 6.11b on page 60). 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, it is recommended that you re-tune the cantilever once the tip is on the surface. To do this, click on the 'Stop' button, go to the Tune Panel and click on the 'Auto Tune' button. Alternatively, the use can choose to enter a 'Target Percent'

of negative (-) 5-10% which will automatically select a resonant frequency that is 5-10% off and to the left of the resonant peak. This offset will compensate for the shift in resonant frequency. This step is recommended since the jump into contact is quicker due to the attractive forces; and therefore, less force is exerted onto the sample.



(a) Master Panel with recommended Amplitude Setpoint Parameter.

(b) Sum and Deflection Meter Window with the Z Voltage position at 36.58V (right).

Figure 6.11.: Double Side by Side Figure General Caption

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. The oscillation amplitude of the cantilever is a parameter to adjust to optimize resolution and may be different depending upon the sample.

6.8. 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.

7. Piezo Force Microscopy: Scientific Background and Theory

CHAPTER REV. 960, DATED 07/12/2012, 14:07. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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

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

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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™ AFM and Cypher™ AFM from Asylum Research.

Figure 7.1.: PFM amplitude channel overlaid on AFM height (top) and phase image overlaid on height (bottom) of lead zirconium titanate (PZT), 20µm 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. 10µm 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 phenomena[3]. The applications of electromechanically active materials include sonar, ultrasonic and medical imaging, sensors, actuators, and energy harvesting technologies. In the realm of electronic devices, piezoelectrics are used as components

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

of RF filters and surface-acoustic wave (SAW) devices[4]. The ability of ferroelectric materials to switch polarization orientation – and maintain polarization state in a zero electric field – has lead to emergence of concepts of non-volatile ferroelectric memories and data storage devices[5]. Electromechanical coupling is the basis of many biological systems, from hearing to cardiac activity. The future will undoubtedly see the emergence, first in research labs and later in industrial settings, of the broad arrays of piezoelectric, biological and molecular-based electromechanical systems. Progress along this path requires the ability to image and quantify electromechanical functionalities on the nanometer and molecular scale (Figure 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 materials[6].

7.3. Principles of PFM

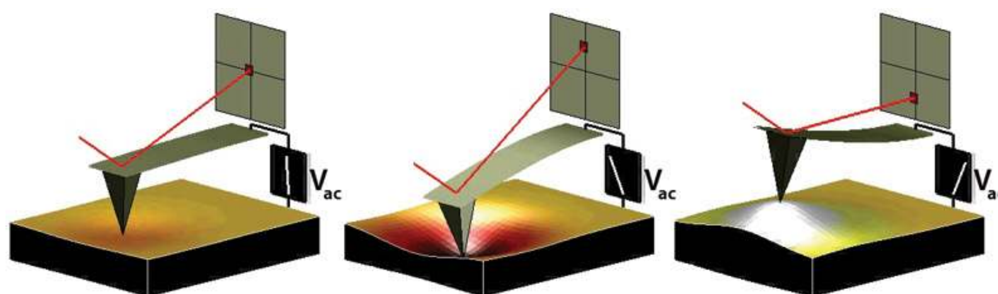

Figure 7.3.: Depiction of PFM operation. The sample deforms in response to the applied voltage. This, in turn, causes the cantilever to deflect, which can then be measured and interpreted in terms of the piezoelectric properties of the sample. Image courtesy S. Jesse, ORNL.

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

When the tip is in contact with the surface and the local piezoelectric response is detected as the first harmonic component of the tip deflection, the phase φ, of the electromechanical response of the surface yields information on the polarization direction below the tip. For c- domains (polarization vector oriented normal to the surface and pointing downward), the application of a positive tip bias results in the expansion of the sample, and surface oscillations are in phase with the tip voltage, $\varphi = 0$. For c+ domains, the response is opposite and $\varphi = 180°$. More details are given in Section 2 (below).

[4] Uchino, K., *Ferroelectric Devices*. Marcel Dekker, 2005.

[5] Scott, J., *Ferroelectric Memories*. Berlin: Springer Verlag, 2006.

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

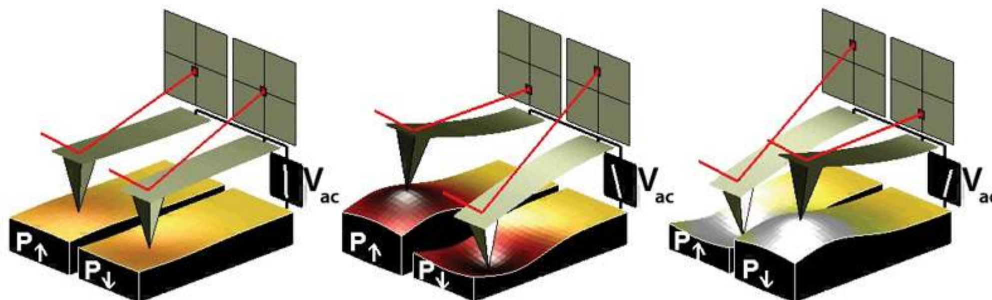

Figure 7.4.: Sign dependence of the sample strain. When the domains have a vertical polarization that is pointed downwards and a positive voltage is applied to the tip, the sample will locally expand. If the polarization is pointed up, the sample will locally contract. The phase of the measured response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

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

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

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} component[11], since it couples directly into the vertical motion of the cantilever. The voltage applied to the tip is

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

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

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

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

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

Table 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
$L_iN_bO_3$ single crystals	Electro-optical devices	10-20	10V-1kV	N/A
Quartz	Balances, frequency standards	3	N/A	N/A
Polar Semiconductors	RF devices, switches	0.1-0.2	N/A	N/A
Calcified Tissues		0.5-3	N/A	N/A
Collagen		0.5-3	N/A	N/A
Thin Films and Capacitor Structures				
1-5 micron PZT	Capacitors	10-30	1-100	100
100-300nm PZT	FeRAM elements	3-10	1-10	10-20
30-100 nm $B_iF_eO_3$	FeRAM	3-10	1-10	10-20
Ultrathin Films				
1-5nm $B_iF_eO_3$	Tunneling Barriers	1-10	1-5	10 (can be below switching voltage in air)
10nm PVDF	Actuators	20	2-5	10

† The PFM signal is given by Equation 6, A=d_{33}VacQ where d_{33} is material property, Vac is driving voltage, and Q is the quality factor. Q=1 for low frequency PFM, and Q = 20-100 if resonance enhancement (DART or BE) method is used. Vac is limited by material stability and polarization switching. The microscope photodetector sensitivity, thermal noise and shot noise impose the limit A > 30pm. The ultimate limit is A = thermal noise.
††Quantitative spectroscopic measurements require probing bias to be one to two orders of magnitude smaller than coercive bias, limiting the voltage amplitude.

††† Measurements are not possible above this limit due to sample and tip degradation.

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

resulting in piezoelectric strain in the material that causes cantilever displacement

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

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

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

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

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 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.

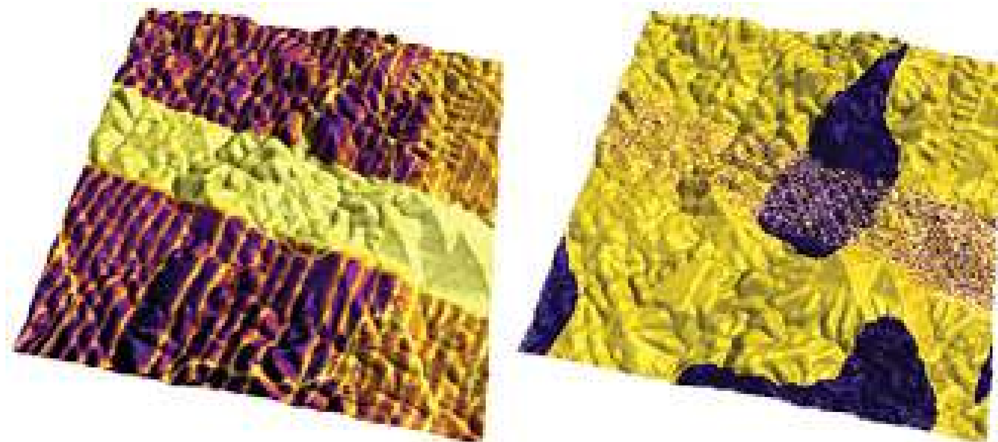

Figure 7.5.: Vertical PFM amplitude overlaid on AFM topography (left) and PFM phase overlaid on AFM topography (right) images of lead titanate film, 5µm scan. Images courtesy of A. Gruverman and D. Wu, UNL. Sample courtesy H. Funakubo.

As mentioned above, the direction of sample polarization determines the sign of the response. Figure 7.4 on page 65 demonstrates this idea. If the polarization is parallel and aligned with the applied electric field, the piezo effect will be positive, and the sample will locally expand. If the local sample polarization is anti-parallel with the applied electric field, the sample will locally shrink. This sign-dependent behavior means that the phase of the cantilever provides an indication of the polarization orientation of the sample when an oscillating voltage is applied to the sample.

The relationship in Equation 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 <100kHz. This and the lack of sophisticated control options precluded the use of resonance enhancement (see sections below on DART and BE) in PFM since typical contact resonance frequencies are >300kHz.

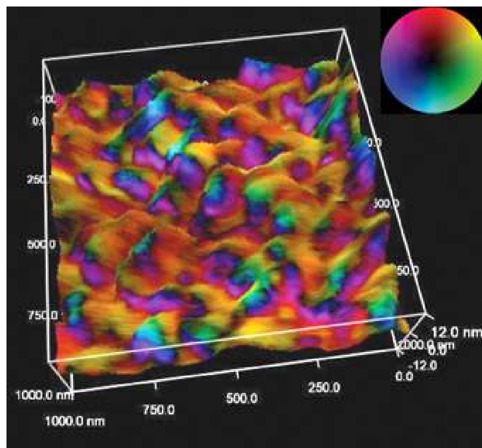

Figure 7.6.: BST film with vector PFM overlaid on AFM topography, 1µm scan. Image courtesy of C. Weiss and P. Alpay, Univ. of Conn., and O. Leaffer, J. Spanier, and S. Nonnenmann, Drexel University. Color wheel indicates PFM vector orientation.

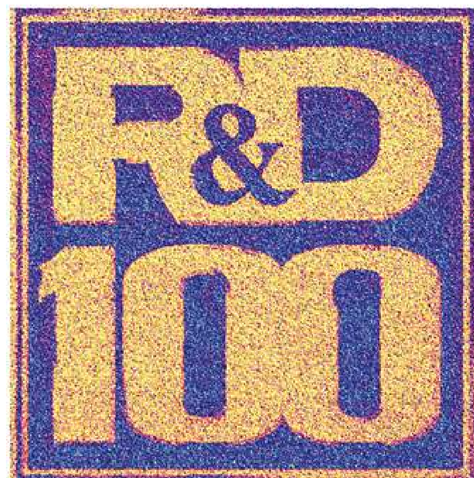

Figure 7.7.: R&D 100 logo written on a sol-gel PZT thin film by PFM lithography. PFM phase is overlaid on top of the rendered topography, 25µm scan. Oak Ridge and Asylum Research were awarded an R&D100 award for Band Excitation in 2008.

7.3.3. 3. PFM Imaging Modes

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

7.3.3.1. A. 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

7.3.3.2. B. 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

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

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

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

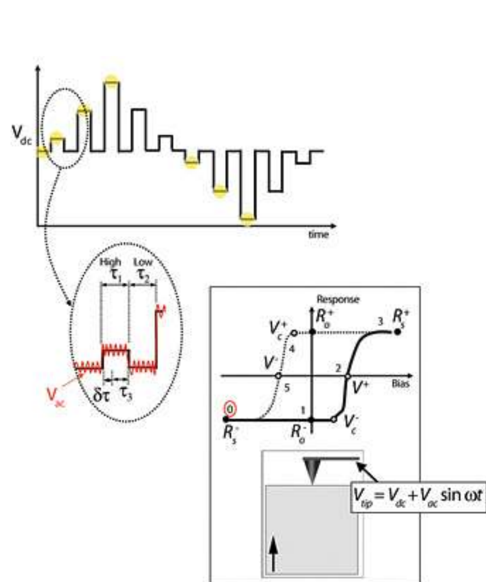

Figure 7.8.: Switching spectroscopy PFM diagram (see text for discussion). Reused with permission from Jesse, Baddorf, and Kalinin, Applied Physics Letters, 88, 062908 (2006). Copyright 2006, American Institute of Physics.

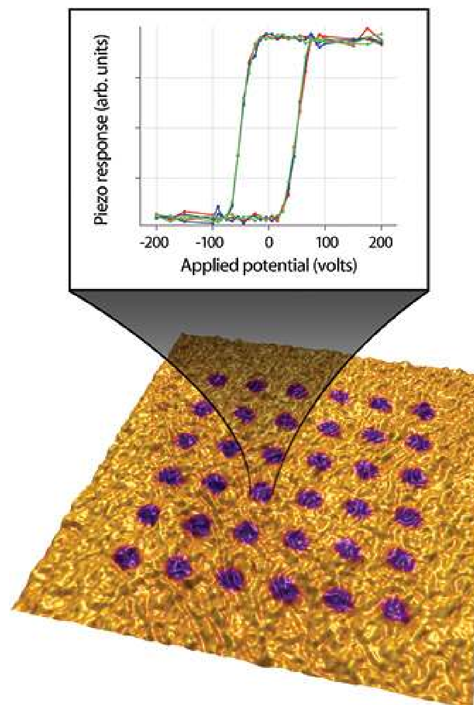

Figure 7.9.: Rendered topography of a LiNbO3 sample with the PFM signal overlaid on top, 4µm scan.

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 films[16].

7.3.3.3. C. 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.6 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 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.

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

D. 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.

4. Spectroscopy Modes

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

Understanding the switching behavior in ferroelectrics on the nanometer scale is directly relevant to the development and optimization of applications such as ferro-electric non-volatile random access memory (FRAM), and high-density data storage. Multiple studies have addressed the role of defects and grain boundaries on domain nucleation and growth, domain wall pinning, illumination effects on the built-in potential, and domain behavior during fatigue.15 The origins of the field date back to the seminal work by Landauer, who demonstrated that the experimentally observed switching fields correspond to impossibly large (~103 - 105kT) values for the nucleation activation energy in polarization switching. Resolving this 'Landauer paradox' requires the presence of discrete switching centers that initiate low-field nucleation and control macroscopic polarization switching[17]. However, difficulties related to positioning of the tip at a specific location on the surface (due in part to microscope drift), as well as time constraints related to hysteresis loop acquisition, limit these studies to only a few points on the sample surface, thus precluding correlation between the material's microstructure and local switching characteristics.

A. Switching Spectroscopy Mapping

A new spectroscopy technique, Switching Spectroscopy PFM (SS-PFM), has demonstrated real-space imaging of the energy distribution of nucleation centres in ferroelectrics, thus resolving the structural origins of the Landauer paradox[18]. These maps can be readily correlated with surface topography or other microscopic techniques to provide relationships between micro- and nanostructures and local switching behavior of ferroelectric materials and nanostructures. Figure 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

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

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

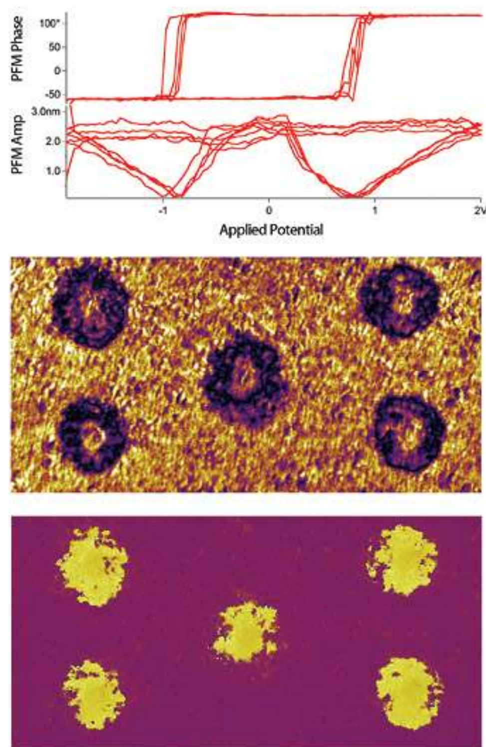

Figure 7.10.: Sol gel PZT sample where local hysteresis loops were measured and displayed (representative phase and amplitude loops shown at top). After the switching spectroscopy measurements, the area was imaged, the DART amplitude (middle) and phase (bottom) are shown, 3.5µm scan.

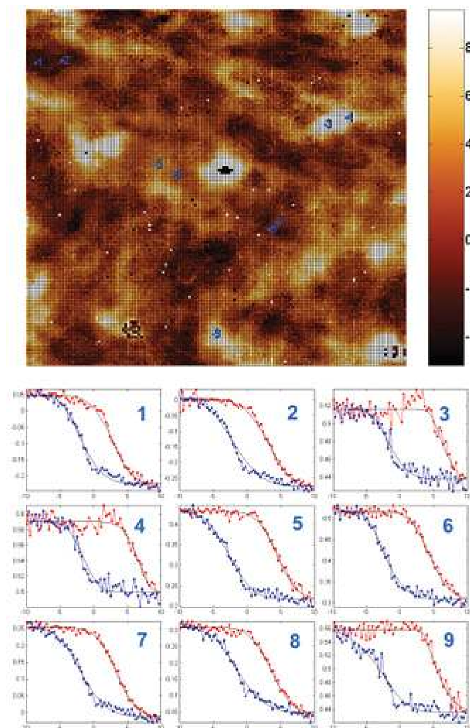

Figure 7.11.: SS-PFM and hysteresis loops of capacitor structures. Data courtesy K. Seal and S.V. Kalinin, ORNL. Sample courtesy P. Bintacchit and S. Trolier-McKinstry, Penn State Univ.

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 71 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 71 shows SSM-PFM of capacitor structures and Figure 7.12 on page 72 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.

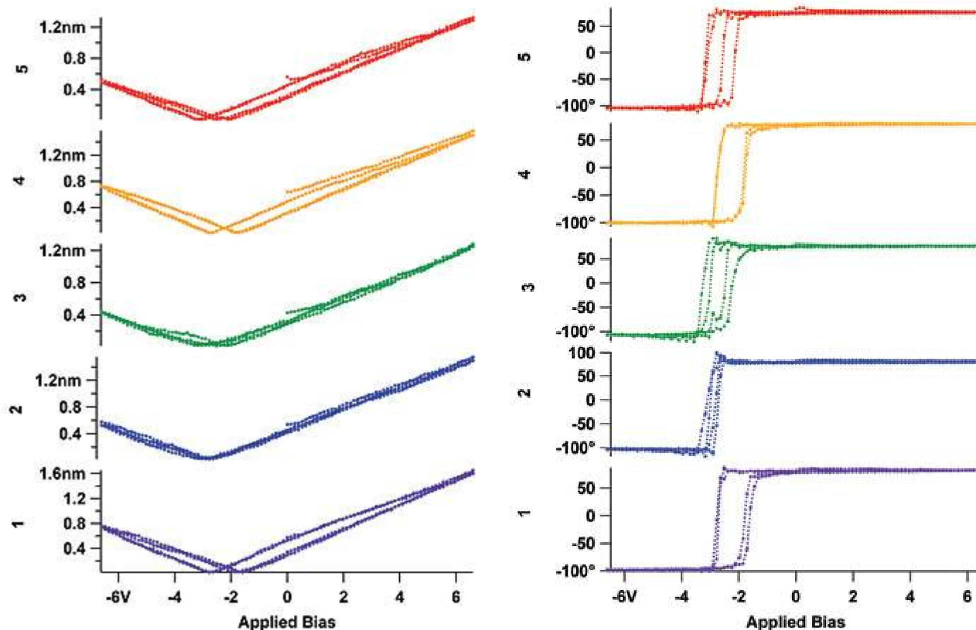

Figure 7.12.: Amplitude (left) and phase (right) hysteresis loops measured at five different locations on a PZN-PTi thin film.

7.4. Limitations of Conventional PFM Methodologies

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$100pm/V for PZT, 10pm/V for LiNbO3) allowed direct imaging and spectroscopy of ferroelectric materials sufficient for applications corresponding to a detection limit of 50pm at ~100kHz. 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 1-2x below the magnitude of standard, low-frequency PFM capabilities. Finally, the use of PFM as an electrophysiological tool necessitates operation in the mV regime, as required to prevent damage to biological systems, as well as stray electrochemical reactions[19].

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

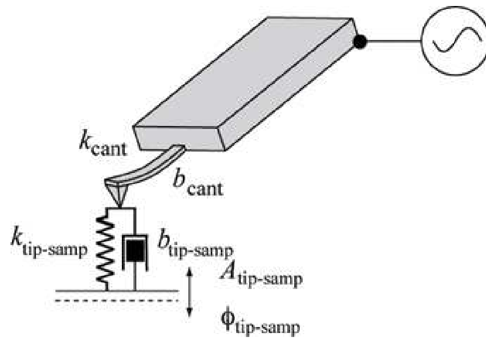

Figure 7.13.: In PFM, the cantilever voltage is modulated, usually at some fixed frequency. This causes the sample to distort at some amplitude and phase. Mediated by the contact mechanics, this drives the tip which, in turn, is monitored by the AFM sensor.

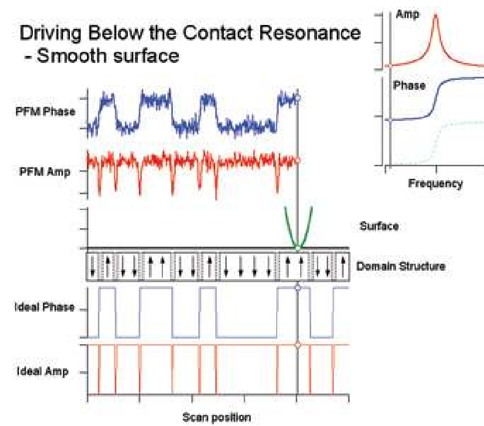

Figure 7.14.: This figure shows the ideal and measured PFM response of an idealized tip (green) scanning over a smooth surface (black line below the "tip"). The domain structure of the ferroelectric sample is shown below the surface where the arrows correspond to the sample polarization direction. The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

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

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

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

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

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

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

$$A = d_{33}V_{ac}Q \tag{7.6}$$

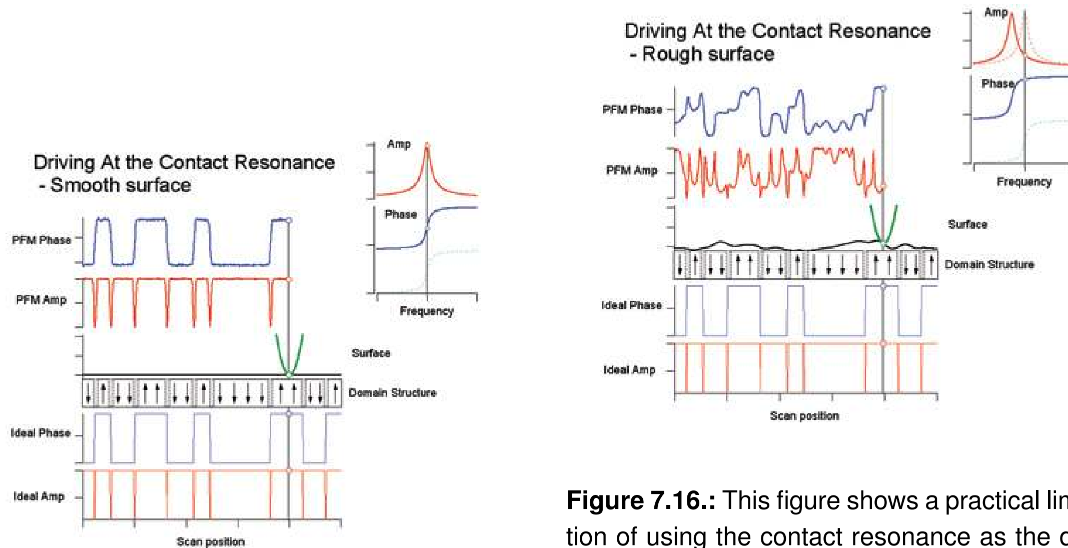

Figure 7.15.: This figure shows the same situation as described in Figure 7.14 on page 73, except that here we are using resonance enhancement to boost the small PFM signal. The inset frequency tune in the upper right corner shows the drive frequency. In this case, since the Q-value of the resonance 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 10-30kHz over the course of imaging a rough sample. Typical cantilevers have a full-width half max of 4-10kHz meaning the phase shift due to the changing contact resonances will easily be near 180° over the scan. The PFM phase shift will be added to the phase of the cantilever contact resonance, yielding a convolution that makes practical interpretation of domain structures very difficult. This is clear in comparing the PFM phase signal to the sample domain structure. In contrast to the off-resonance smooth sample, it is quite difficult to correlate the domain structure with the PFM phase.

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

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

($A_{\text{tip-samp}}, \varphi_{\text{tip-samp}}$). The cantilever in contact with the surface has a resonance defined by the mechanical properties of the cantilever and the stiffness of the tip-sample contact. This resonance can have a high quality factor (Q) for typical PFM samples that effectively amplifies the piezo signal by a factor of $\sim Q$ near the resonance. For samples with small piezo coefficients, this is potentially a very important effect and could mean the difference between only noise or a measurable signal. Unfortunately, because the cantilever resonance frequency depends on the tip-sample contact stiffness, the resonance frequency is very unstable. As the tip scans over the sample topography, the stiffness of the mechanical contact (($k_{\text{tip-samp}}$).) will typically change significantly. This, 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}≈100pm/V). The noise visible in the PFM amplitude and phase curves were calculated to be the ideal thermal (Brownian motion) noise of a cantilever at typical room temperature (300K). Here, the domain structure is shown in the middle of the image with purely vertical polarization vectors. The sample is treated as perfectly smooth, meaning that the contact stiffness remains constant as a function of position. The simulation reproduces many of the features present in a real scan where the measured phase reproduces a map of the domain structure, and the amplitude goes to zero at the domain boundaries. This occurs as the tip is being driven by two oppositely oriented domains, each canceling the other since they are 180° out of phase. As discussed below, real-world samples have behaviors that make extracting unambiguous domain maps much more complicated.

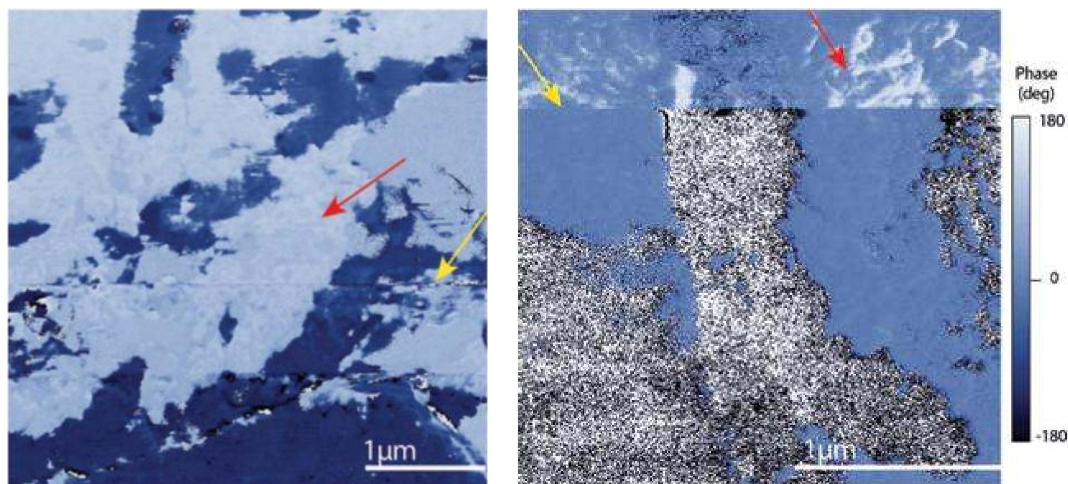

Figure 7.17.: PFM phase channel on a polished PZT sample. The cantilever was driven near the contact resonance to enhance the SNR. There is significant crosstalk between the sample topography and the PFM signal. Red arrows indicate "roughness" where the contact stiffness modulates the phase. In addition to the surface roughness changing the contact resonance and therefore the measured phase, changes in the tip can also cause large phase shifts. The yellow arrows indicate a sudden tip change caused a change in the contact resonance. 4µm scan (top), 2µm scan (bottom).

The gain in the signal from the Q-factor when operating near resonance improves the SNR for the PFM amplitude and the phase. This is illustrated in Figure 7.15 on page 74 which shows

the same sample as in Figure 7.14 on page 73 but now imaged with the cantilever voltage being modulated at the cantilever resonance. This should not come as a surprise; as with many other types of dynamic SPM, oscillating at the cantilever resonance greatly benefits the SNR. However, the experimental conditions shown in Figure 7.14 on page 73 are very rare. Usually, the sample will have some roughness. This roughness 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.

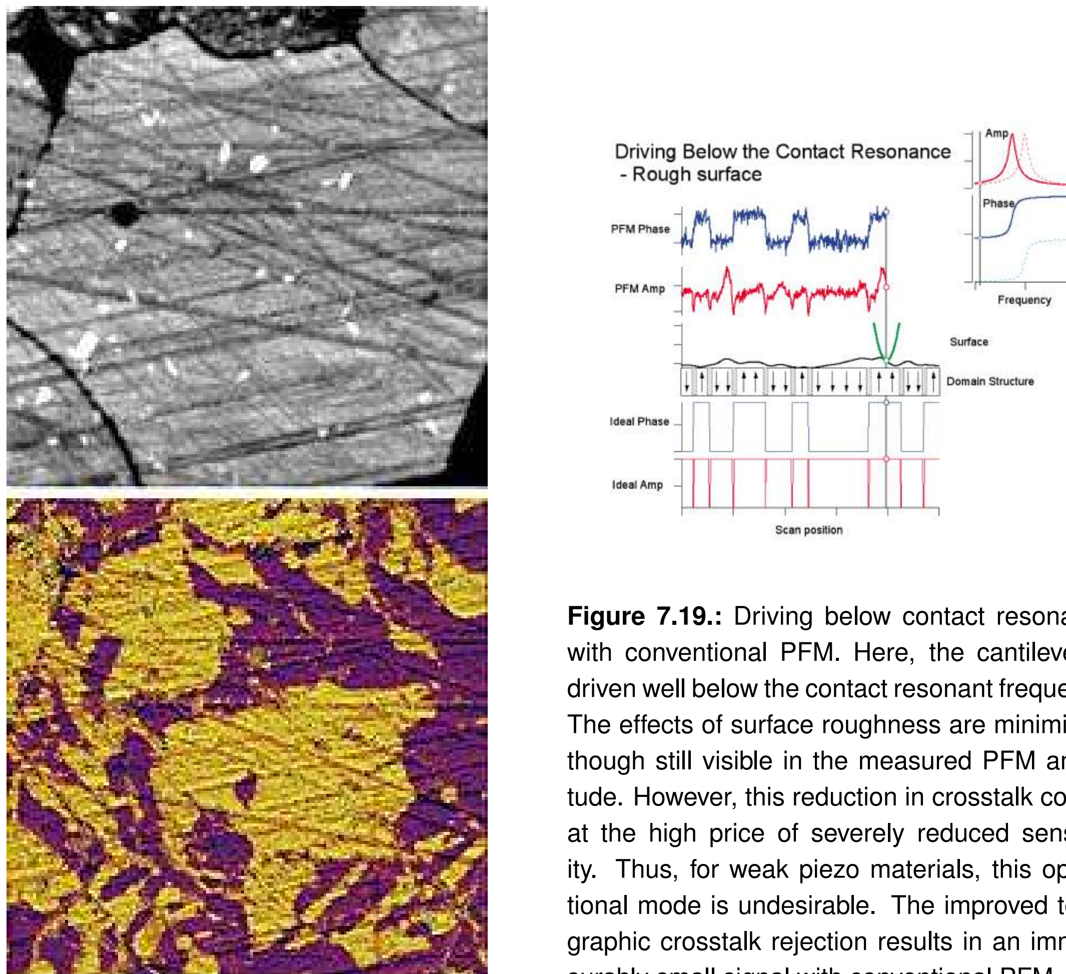

Figure 7.19.: Driving below contact resonance with conventional PFM. Here, the cantilever is driven well below the contact resonant frequency. The effects of surface roughness are minimized, though still visible in the measured PFM amplitude. However, this reduction in crosstalk comes at the high price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

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

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.

Figure 7.20.: MFP-3D Piezo Force Module software menu allows easy point and click navigation.

Another source of phase shifts can come from irreversible changes to the cantilever itself. PFM is a contact mode technique and therefore can exert large forces on the tip. If the tip fractures or picks up a contaminant, the contact resonance can experience a sudden jump, usually positive, since tip wear tends to blunt the tip. The resonance jumps are typically of the order of a few kHz. This causes large, discontinuous changes in the measured phase. Figure 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.

To summarize the discussion in this section, with 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.

In the following sections we discuss new solutions for improving our PFM options with Asylum’s PFM and SPM capabilities.

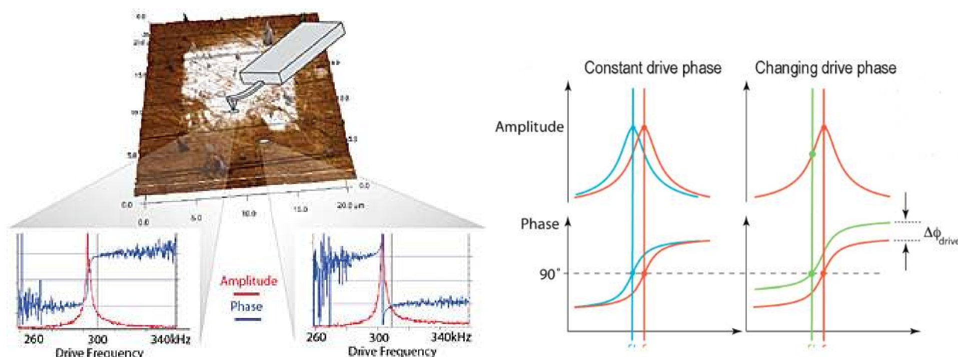

Figure 7.21.: For domains with an antiparallel (180°) orientation, conventional PLLs drive the PFM frequency away from resonance. (Top) Amplitude, red, and phase, blue, cantilever response over antiparallel domains. In the measurement, phase is offset by 180° over anti-parallel domains (see curves on the right). (Bottom) PFoptioanl titleIM 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. Printed with permission[22].

7.5. Solutions to Limits of Conventional PFM

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 below under Emerging Applications for PFM).

Asylum's Piezoresponse Force Module is currently the only commercially-available AFM that enables high voltage PFM measurements. A programmable bias of up to +220V for the MFP-3D and up to +150V for the Cypher AFM is applied to the AFM tip using a proprietary high voltage amplifier, cantilever and sample holder. The amplitude of the response measures the local electromechanical activity of the surface while the phase yields information on the polarization direction. High probing voltages can characterize even the weakest piezoelectric sample and insure that you have the ability to switch the polarization of high-coercivity materials. The fully integrated system allows both PFM imaging modes and spectroscopy modes. All PFM imaging and spectroscopy modes are fully integrated with the AFM system software and Piezoresponse Force Module hardware. An easy-to-use PFM menu panel (7.20) provides users with point-and-click navigation to the operation they wish to perform. For advanced users, custom panels can by created within the flexible IGOR Pro environment.

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

The most common kind of resonance-tracking feedback loop is called a phase-locked loop (PLL). It utilizes the phase sensitive signal of a lock-in amplifier to maintain the system at a specific phase value, typically 90°. The PLL is generally limited to techniques where the phase and amplitude of the driving force is constant (e.g. the mechanical excitation of a cantilever resonance using an external actuator). This is manifestly not the case in PFM, where the relationship between the phase of the excitation force and driving voltage strongly depends on material properties.[23,24] The amplitude and phase of the local response are a convolution of material response to the external field and cantilever response to the material-dependent local force, which cannot be separated unambiguously. Figure 7.21 on page 78 is an example where, for antiparallel domains, a conventional PLL will actually drive a PFM away from resonance.

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 7.22 on page 80 shows how DART works. The potential of the conductive cantilever is the sum of two oscillating voltages with frequencies at or near the same resonance. The resulting cantilever deflection is digitized and then sent to two separate lock-in amplifiers, each referenced to one of the drive signals. By measuring the amplitudes at these two frequencies, it is possible to measure changes in the resonance behavior and furthermore, to track the resonant frequency. Specifically, by driving at one frequency below resonance (A1), and another above (A2), A2-A1 gives an error signal that the ARC2™ controller uses to track the resonance frequency changes.21

DART-PFM studies of polarization switching are illustrated in Figure 7.23 on page 80, where the resonant frequency (A), amplitude (B) and phase (C) images of a lithium niobate surface are shown Figure 7.23A. 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 7.23 (G-I) are DART-PFM images of domains switched by the application of three 176V magnitude pulses for ~10 seconds in three adjacent locations. Note the significant change of resonant frequency and the strong amplitude depression in the newly fabricated domain.21

Additional DART images of ferroelectric materials are shown in Figure 7.24 on page 81 and Figure 7.25 on page 81. Figure 7.24 on page 81 shows a series of images of PFM on multiferroic $B_iF_eO_3$ nanofibers. Figure 7.25 on page 81 shows a short relaxation study on a sol-gel sample.

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

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

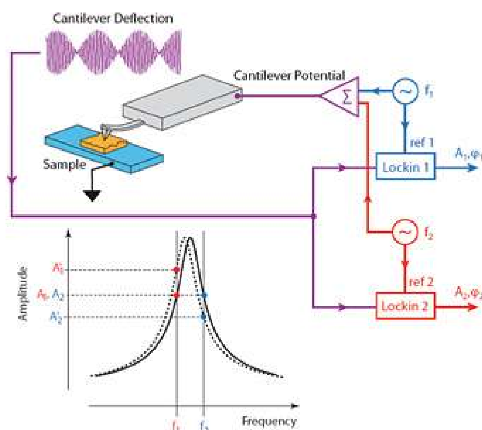

Figure 7.22.: Schematic diagram of Asylum Research's new DART showing a drive phase independent feedback signal. Printed with permission (see reference 21).

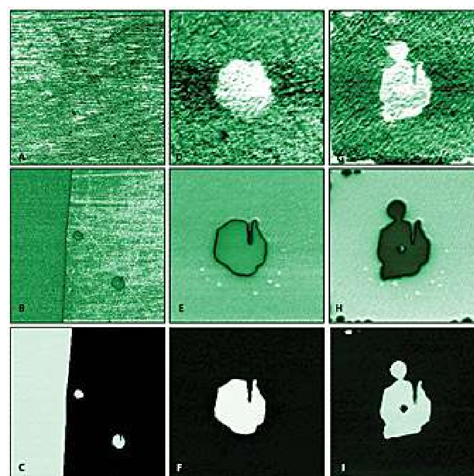

Figure 7.23.: (A), (D), (G) Resonance frequency, (B), (E), (H) piezoresponse amplitude and (C), (F), (I) piezoresponse phase images of antiparallel domains in lithium niobate. Shown are images of the (A)–(C) native domain structure, (D)–(F) an intrinsic domain and (G)–(I) domains switched by ±176V (locations marked in (E)). The images are obtained at wf = 4kHz and Vac = 66V. The frequency images have been flattened to account for minute changes of contact radius from line to line. Reprinted with permission (see reference 21).

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 a new option that can be utilized with PFM. The technology is exclusively available with Asylum Research SPMs under license from Oak Ridge National Laboratory[25] and has received the R&D 100 award for 2008. The Band Excitation controller and software extend the capabilities of Asylum's MFP-3D and Cypher 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 un-

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

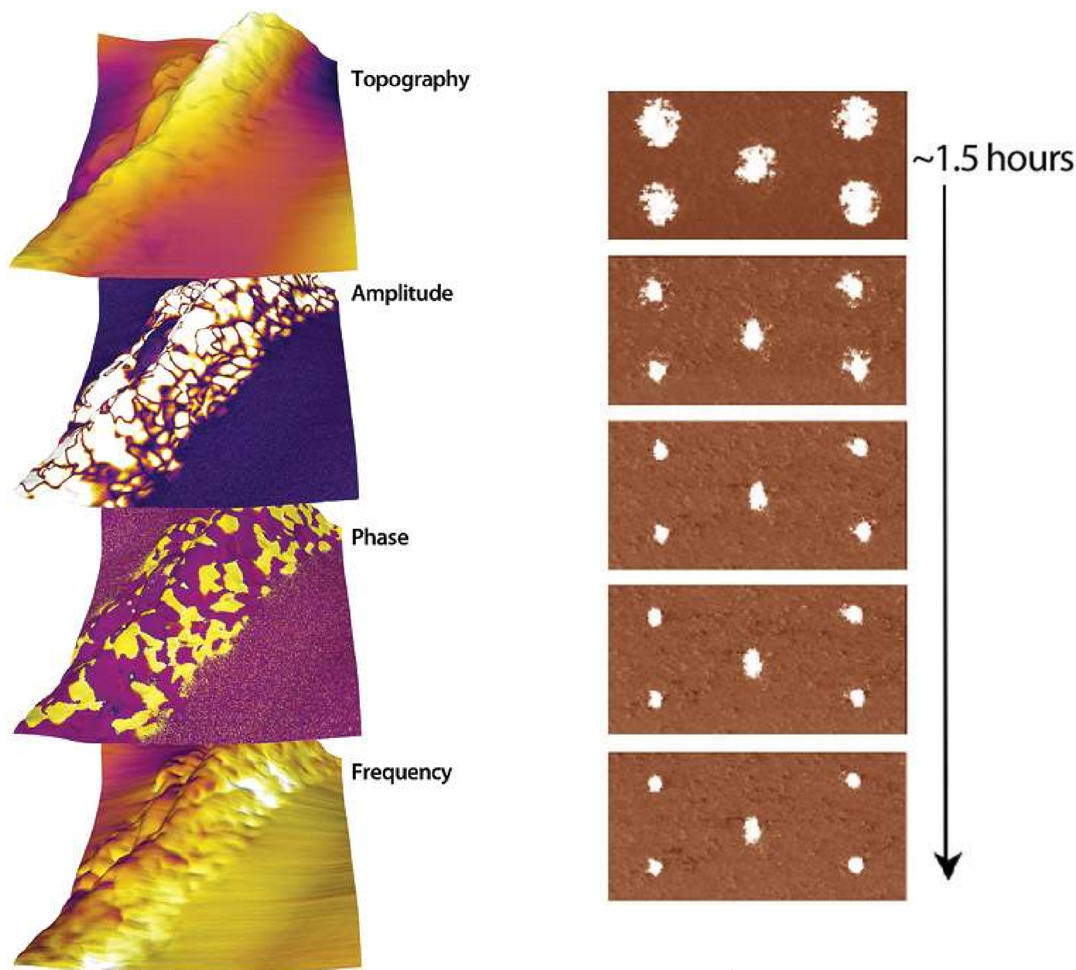

Figure 7.24.: PFM of multiferroic BiFeO3 nanofibers, 1µm scan. Collaboration with Shuhong Xie, Xiangtan University, China and JiangYu Li, University of Washington.

Figure 7.25.: 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. 3.5µm scan.

certainties than there were measured quantities (see Equations (4) and (5)).23 BE surmounts this difficulty by detecting responses at all frequencies simultaneously. BE introduces a synthesized digital signal that spans a continuous band of frequencies, and monitors the response within the same frequency band. This allows ~100x improvement in data acquisition speed compared to other commercially-available technologies.

The immediate benefit of this approach is that a full response spectrum can be collected (with insignificant [30-50%] decrease in signal to noise ratio) in the amount of time required for obtaining a single pixel in conventional single-frequency SPM. BE allows quantitative mapping of local energy dissipation in materials on the nanoscale.23 Figure 7.27 on page 83 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:

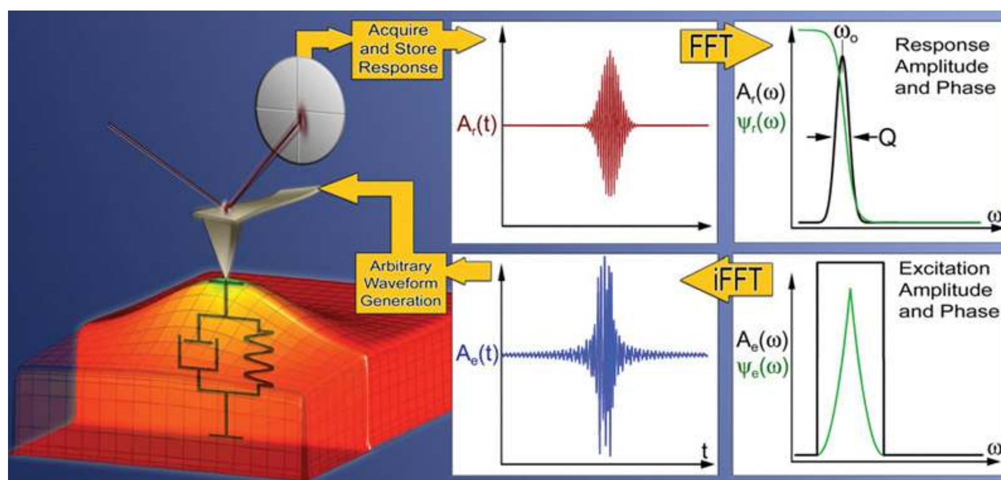

Figure 7.26.: Operational principle of the BE method in SPM. The excitation signal is digitally synthesized to have a predefined amplitude and phase in the given frequency window. The cantilever response is detected and Fourier transformed at each pixel in an image. The ratio of the fast Fourier transform (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.23 Reprinted with permission (see reference 23).

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

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.

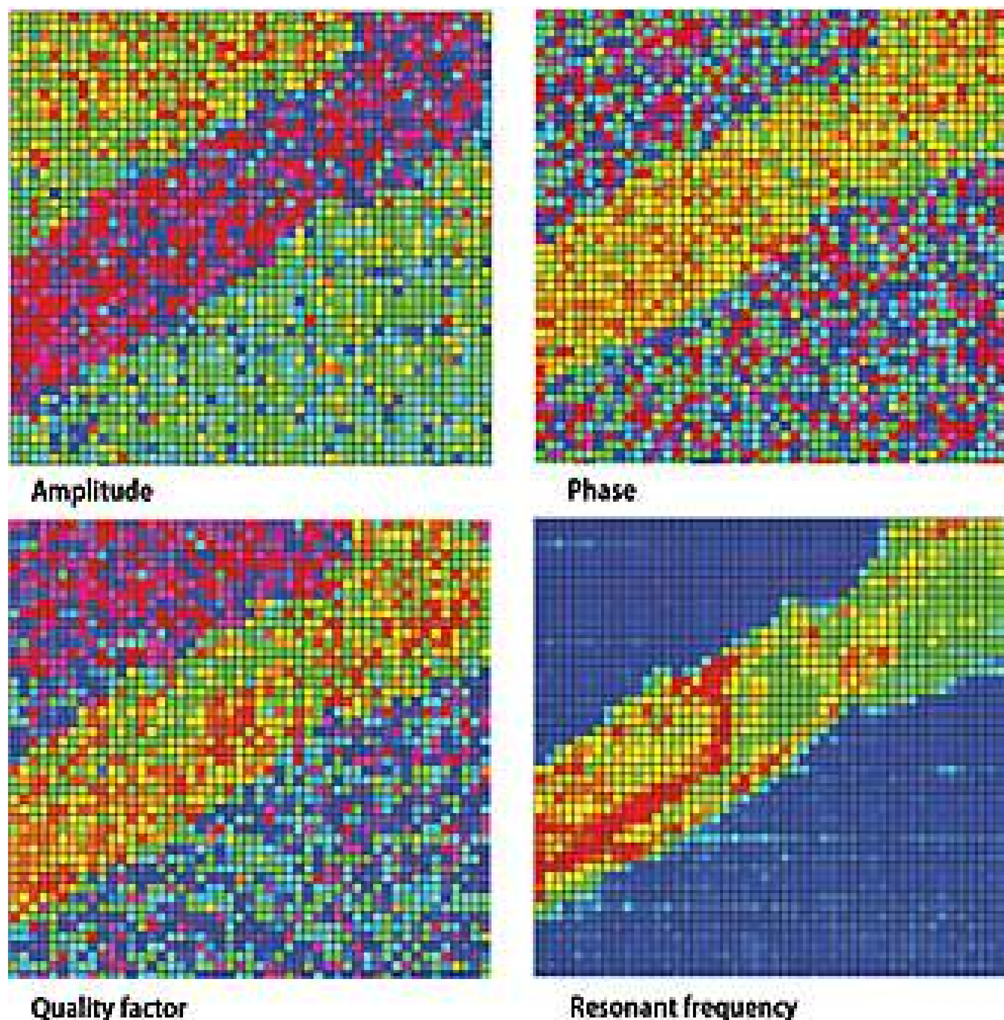

Figure 7.27.: Amyloid fibril (bovine insulin) on mica imaged in water using BE-PFM technique, 250nm x 250nm. Image courtesy of G. L. Thompson, V. V. Reukov, A. A. Vertegel, M. P. Nikiforov, Clemson University, Dept. Bioengineering, and S. Jesse, S. V. Kalinin, Oak Ridge National Lab.

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 10-100MHz range. Asylum's microscopes allow cut-off at ~2-8MHz and potentially higher, opening a pathway for high frequency studies of polarization dynamics. Figure 7.28 on page 84 illustrates the different information that is revealed by imaging a ceramic PZT material at various frequencies.

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 100Hz. The strong amplitude and

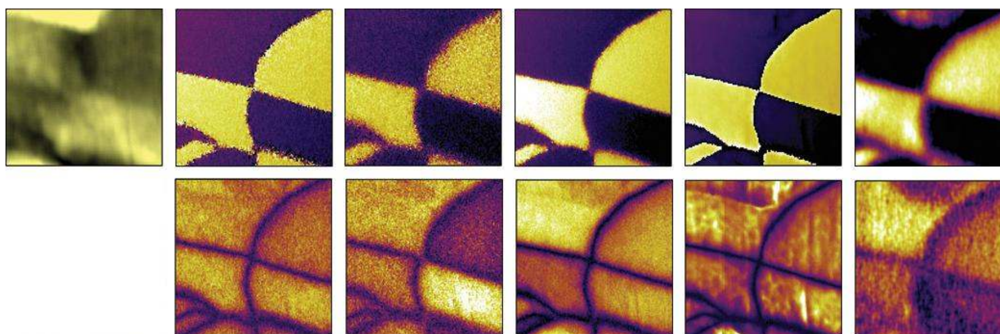

Figure 7.28.: High Frequency PFM using Asylum's fast photodiode on a ceramic PZT sample at different frequencies (phase left, amplitude right) – below first resonance (top row) and at cantilever resonances (all others) using a MikroMasch NSC 35B cantilever. 1µm scans. Image courtesy of K. Seal, S. Kalinin, S. Jesse, and B. Rodriguez, Center for Nanophase Materials Science, ORNL.

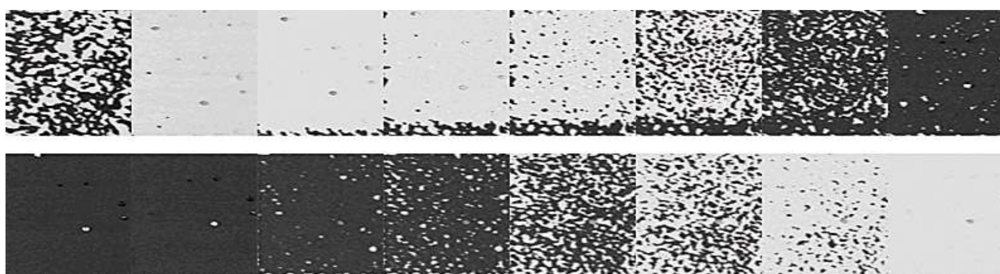

Figure 7.29.: This image sequence (left to right, top to bottom) is excerpted from a movie of 244 consecutive High Speed PFM images (4µm scans) depicting in situ ferroelectric memory switching. For the first half of the movie, the tip is biased with a positive DC offset throughout the measurements. By monitoring the phase of the piezoresponse, this allows direct nanoscale observation of ferroelectric poling, in this case from white to black contrast (a 180 degree 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.

phase contrast achievable in PFM, as well as the resolution enhancement provided by this contact-mode based method, have allowed 10nm spatial resolution even at image rates of up to 10 frames per second[26].

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 7.29 on page 84 and Figure 7.30 on page 85.

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 surfaces[27].

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

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

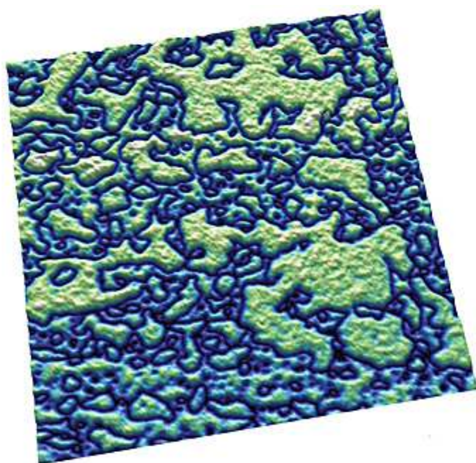

Figure 7.30.: (001) domains in a PZT thin film, 3.8µm scan. Image courtesy N. Polomoff and B. D. Huey, University of Connecticut Institute of Materials Science. Sample courtesy R. Ramesh, UC Berkeley.

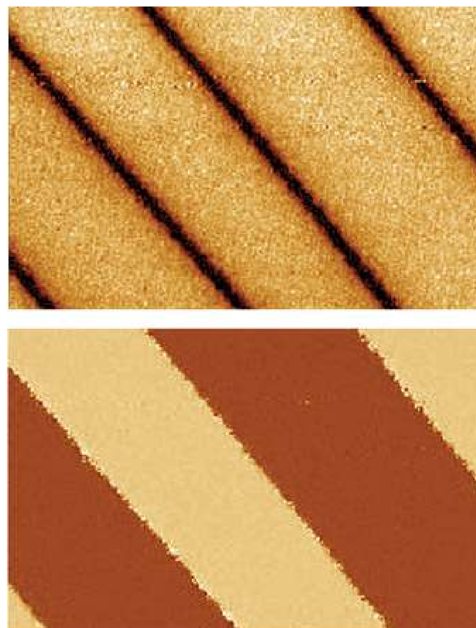

Figure 7.31.: PPLN amplitude (top) and phase image (bottom) acquired with the MFP NanoIndenter, 50µm scan.

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 are at best problematic. Another issue is that the cantilever geometry means that the motion of the cantilever tip is not well defined. Specifically, when the cantilever deflects, there is motion along the vertical axis (z-axis) that is well defined, but there is also motion parallel to the sample surface. This motion is not well characterized and in most cases is not even measured.

The ability to probe forces and directly image the piezo response of a sample with the Asylum Research MFP NanoIndenter is an emerging application area[28]. 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. Displacement of the indenting flexure is performed with a piezo actuator (head) and measured with a patented nanopositioning sensor (NPS™). The force is computed as the product of the spring constant and the measured indenter flexure displacement. This measurement is done by converting the vertical flexure displacement into an optical signal measured at 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 7.31 on page 85 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

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

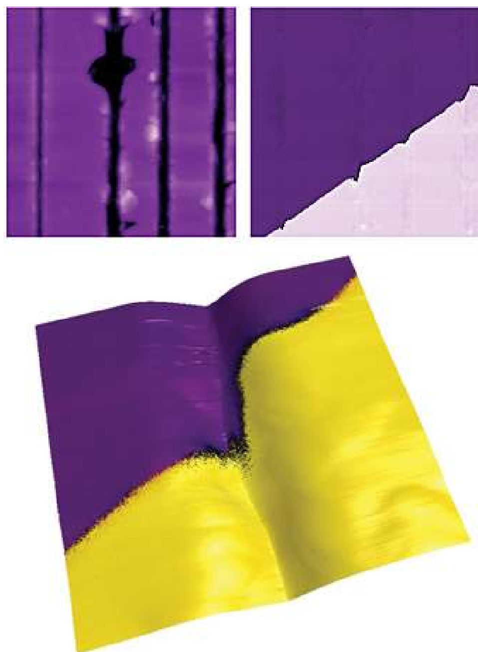

Figure 7.32.: Surface topography of PPLN after it has been purposefully scratched with different loading forces using the NanoIndenter, 10µm scan (top images). 1µm scan (bottom).

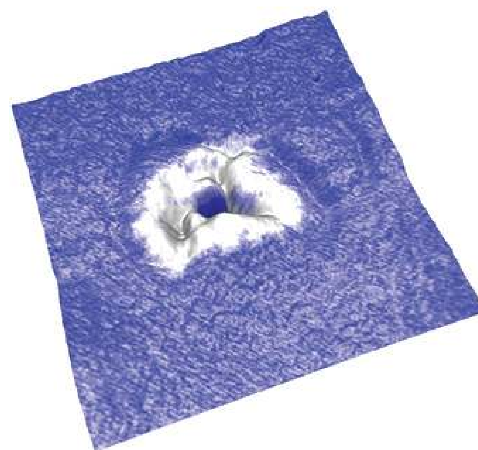

Figure 7.33.: Zoom of the top surface of a red blood cell. The surface shape was rendered to show the topography while the phase channel is overlaid on top to show piezo response. A small sub-micron region on top (white) of the cell exhibited a much different piezo response than the surronding cell surface. 2µm scan. Image courtesy of B. Rodriguez and S. Kalinin, ORNL.

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 7.32 on page 86. The top image shows the surface topography of PPLN after it has been purposefully scratched with different loading forces using the NanoIndenter tip.

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

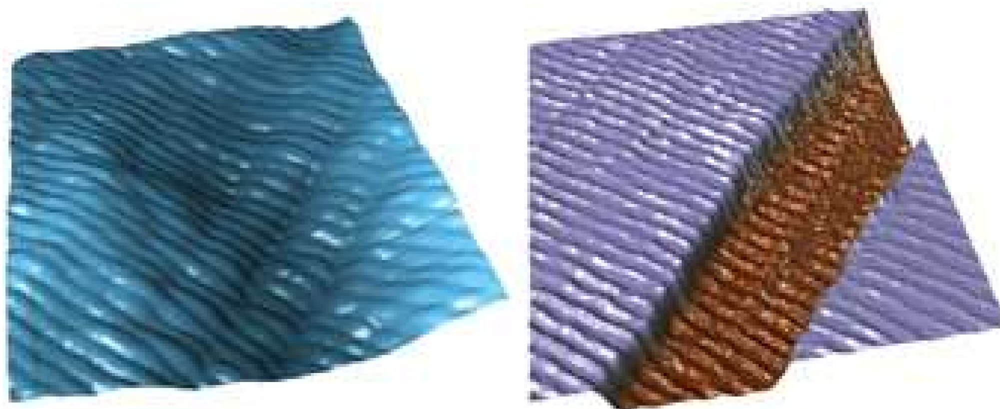

Figure 7.34.: Topographic (top) and PFM phase (bottom) images of collagen fibers, 1.4μm scan. Image courtesy D. Wu and A. Gruverman, UNL. Sample courtesy G. Fantner.

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 polymers, bacteriorhodopsin, and connective tissue[29,30]. Figure 7.33 on page 86 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 7.33 on page 86[31]. This image shows a zoom of a red blood cell with the PFM phase channel painted on top to show piezo response.

7.7. Applications of Piezoresponse Force Microscopy

7.7.1. Fundamental Materials Science

- Domains

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

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

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

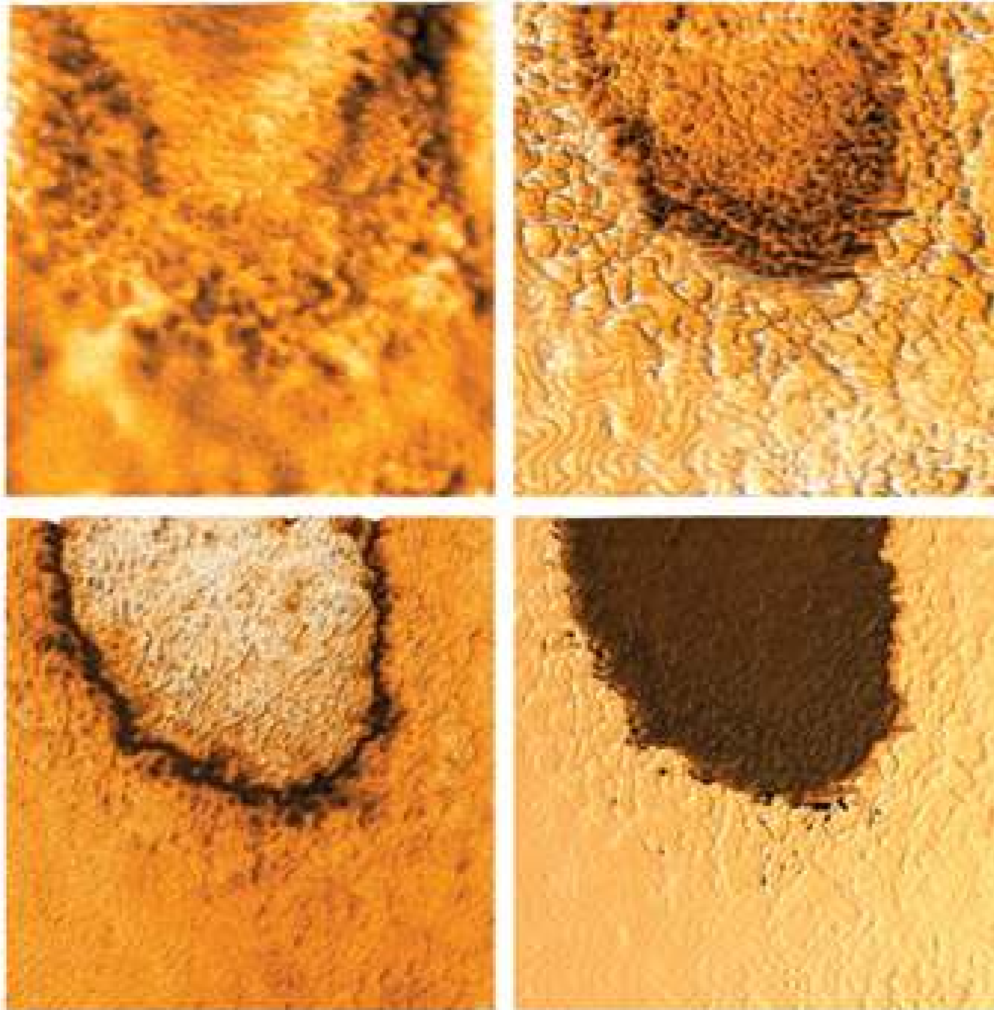

Figure 7.35.: DART image of C-domains in lead titanate thin film, 5µm scan. Image courtesy D. Wu and A. Gruverman, UNL.

- Phase Transitions and Critical Phenomena
- Size Effects
- Nucleation Dynamics
- Multiferroics
- Ferroelectric Polymers
- Liquid Crystals
- Composites
- Relaxor Ferroelectrics

7.7.2. Piezoelectric Materials

- Micro ElectroMechanical Systems (MEMS)

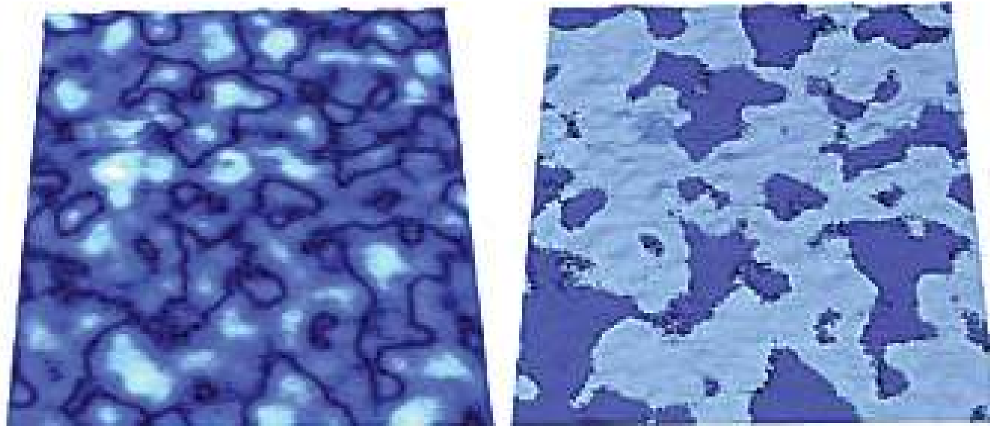

Figure 7.36.: PFM amplitude overlaid on AFM topography (left), and phase overlaid on topography (right) of 1µm thick PZT film with 50nm Pt capacitor electrode. A bias was applied between the bottom and top electrodes and the tip was electrically isolated. Taken at a frequency of ~1MHz, 5µm scan. Image courtesy of K. Seal, S. Kalinin, S. Jesse, ORNL, and P. Bintachitt, S. Trolier-McKinstry, Pennsylvania State University.

- Sensors and Actuators
- Energy Storage and Harvesting
- RF Filters and Switches
- Sonar
- Balance and Frequency Standards
- Giant k Dielectrics
- Capacitors

7.7.3. Ferroelectric Materials

- Domain Engineering
- Non-volatile Memory
- Data Storage Devices
- Domain Energetics and Dynamics

7.7.4. Bio-electromechanics

- Cardiac
- Auditory
- Cell Signaling
- Structural Electromechanics
- Biosensors

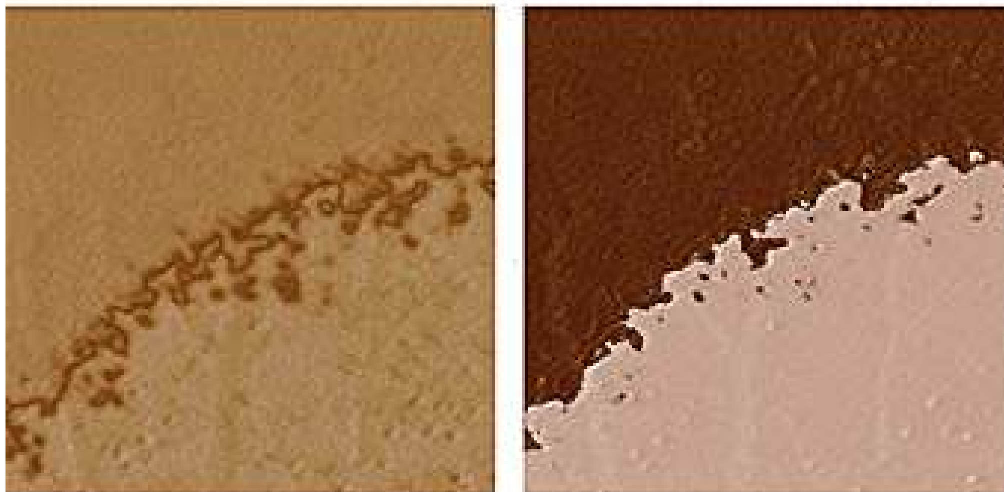

Figure 7.37.: DART image of lead titanate showing domains, amplitude (left) and phase (right), 4µm scan.

7.8. Conclusion

Characterizing electromechanical responses in a variety of materials will be crucial for understanding and improving technologies ranging from bioscience to energy production. Scanning probe microscopy has emerged as a universal tool for probing such structures and functionality at the nanometer scale. Asylum's Piezoresponse Force Microscopy capabilities now allow characterization of an endless variety of materials and devices that previously could not be measured using conventional piezoresponse force microscopy. Research with this new tool will enable new advancements in many disciplines from biology to semiconductors, while yielding improvements for ongoing work in diverse areas from data storage devices and molecular machines to improved materials for renewable energy.

7.9. Additional Reading

7.9.1. Scientific Articles of Interest

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

- *Theory of indentation of piezoelectric materials*[32]
- *Indentation of a transversely isotropic piezoelectric half-space by a rigid sphere*[33]

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

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

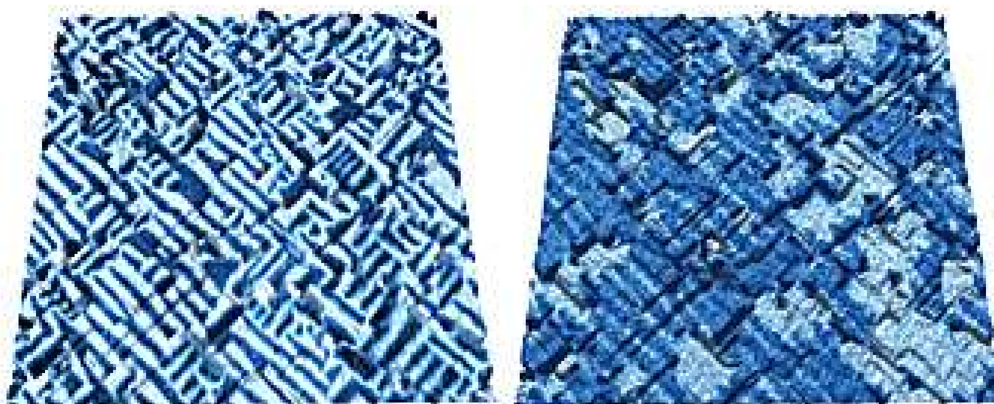

Figure 7.38.: PFM amplitude overlaid on topography (left) and PFM phase overlaid on topography (right) of in-plane images of 50nm BFO/LMSO/STO(001), Uac = 2V, f = 25kHz. The in-plane images show stripe-like domains, 5µm scan. Image courtesy of N. Balke, Department of Materials Science and Engineering, University of California, Berkeley.

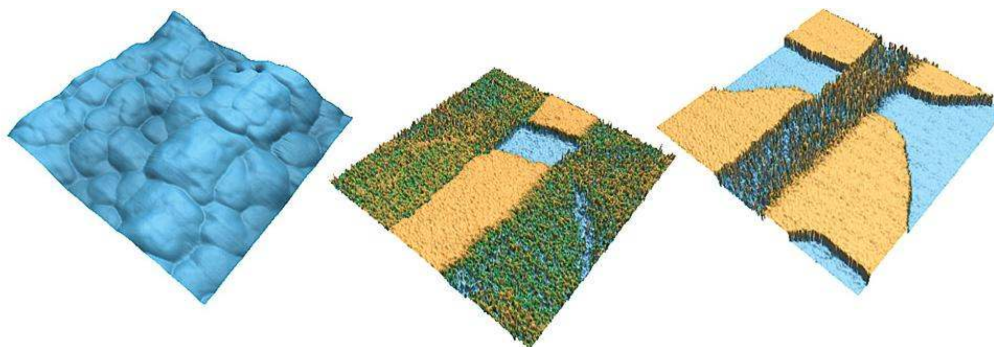

Figure 7.39.: Topography (top), lateral PFM phase (center), and vertical PFM phase (bottom) images of lead titanate film, 3µm scan. Images courtesy A. Gruverman and D. Wu, UNL. Sample courtesy H. Funakubo.

- *Point force and point electric charge in infinite and semi-infinite transversely isotropic piezoelectric solids*[34]
- *Nanoelectromechanics of piezoresponse force microscopy*[35]
- *Nanoelectromechanics of piezoelectric indentation and applications to scanning probe microscopies of ferroelectric materials*[36]

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- *Modeling and measurement of surface displacements in BaTiO3 bulk material in piezoresponse force microscopy*[37]
- *Nanoscale piezoelectric response across a single antiparallel ferroelectric domain wall*[38]
- *Materials contrast in piezoresponse force microscopy*[39]
- *Electromechanical detection in scanning probe microscopy: Tip models and materials contrast*[40]
- *Local probing of ionic diffusion by electrochemical strain microscopy: Spatial resolution and signal formation mechanisms*[41]
- *Spatial resolution, information limit, and contrast transfer in piezoresponse force microscopy*[42]
- *Nanoscale phenomena in ferroelectric thin films*[43]
- *Encyclopedia of Nanoscience and Nanotechnology*[44]
- *Nanocrystalline multiferroic BiFeO3 ultrafine fibers by sol-gel based electrospinning*[45]
- *Local bias-induced phase transitions*[46]

7.9.2. Comprehensive Material

These references provide key papers and comprehensive reviews on PFM:

- *Scanning probe microscopy : electrical and electromechanical phenomena at the nanoscale*[47]
- *Nanoscale characterisation of ferroelectric materials : scanning probe microscopy approach*[48]
- *Imaging and control of domain structures in ferroelectric thin films via scanning force microscopy*[49]

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7.10. Glossary

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

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

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

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

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

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

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

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

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

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

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

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

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. Single Frequency Piezo Force Microscopy (PFM)

CHAPTER REV. 960, DATED 07/12/2012, 14:07. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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8.3 PFM Lithography with ModeMaster . . . 99
8.4 Vector PFM . . . 101

Single Frequency Piezo Force Microscopy Author's Full Name, Asylum Research 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 in the surface which in turn causes a periodic deflection of the cantilever.

This document 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 8.1 on page 95 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 blush in that the contact resonant frequency depends strongly on details of the contact mechanics – the elastic modulus, tip shape and sample topography can conspire 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 on. For advanced users we suggest using Dual AC Resonance Tracking (DART) as discussed in tech note 13.

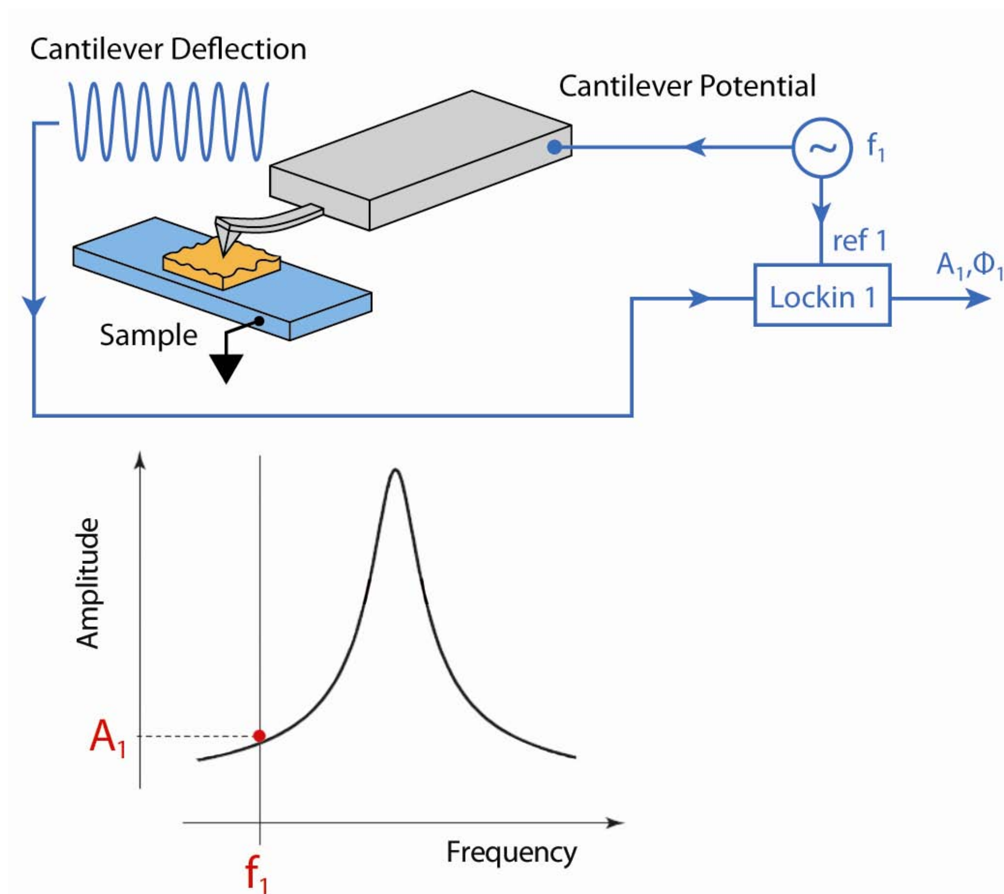


Figure 8.1.: A diagram of Single Frequency PFM

8.1. 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 3-5x 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. See Table 8.1 on page 95.

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

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

8.2. 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 the HV cantilever holder if you have the optional HVA220 unit. Other cantilever holders may not be compatible without cross-point adjustments.

8.2.1. 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 , 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.

8.2.2. Software Instructions

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

1. To run Single Frequency PFM, open a new MFP template by starting the software from scratch, or by clicking *File* ▷ *New MFP3D Template*. The Mode Master window should pop up. If it does not, click [ctrl+F2], or navigate to *User Settings* ▷ *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 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.
3. Maximize the sum, and then put deflection at ~ -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.
   - 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.

6.

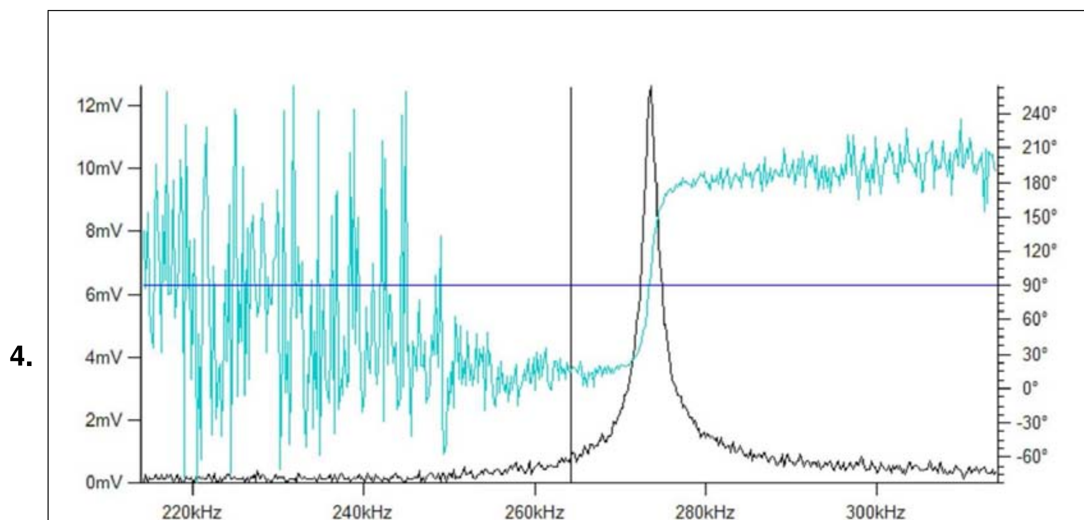
Engage the Surface

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

8.2.3. Tuning

1. Once you are engaged, it is time to tune. Table 8.1 on page 95 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 at 200kHz. Frequency width should be set to 5kHz (most of the time). You should see a peak. If not, try widening the sweep range and gently increasing the drive voltage. For an AC240, once the drive goes above ~250mV if you have the optional high voltage amplifier or ~5 volts if you have a standard holder, you may start to degrade the tip quality. For PPLN and PZT samples, 10mV (into the HV amp) or 200mV (with the standard holder) 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 (50mV to 140mV with high voltage amplifier) will give a decent peak.
3. Right click close to the peak and click on *Set Drive Frequency*. Your goal is to select a frequency close to but not on the peak to allow enough amplification but to avoid effects by changes in contact resonance.

4.



Center Phase

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

8.2.4. Imaging

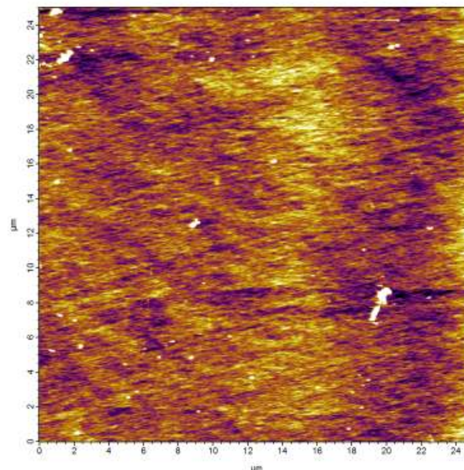
At this point, we're ready to image. Since the domains are typically many microns across, it can be advantageous to scan a larger range. Also note – you will have more reproducible results if you scan at 90°.

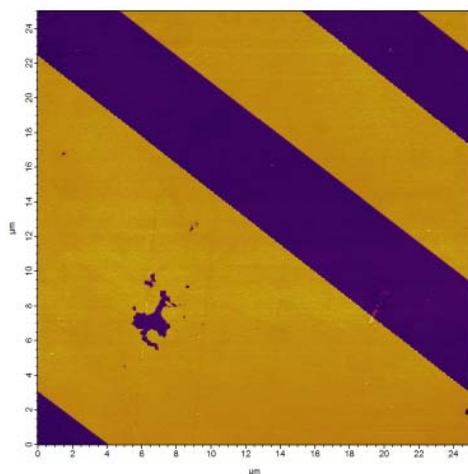
1. Hit *Do Scan* to start imaging.

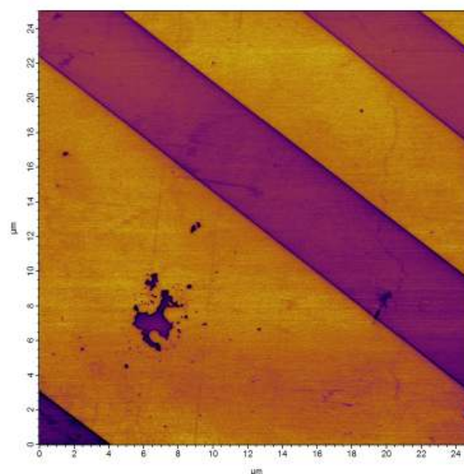
2. **Inpsecting data**

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

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

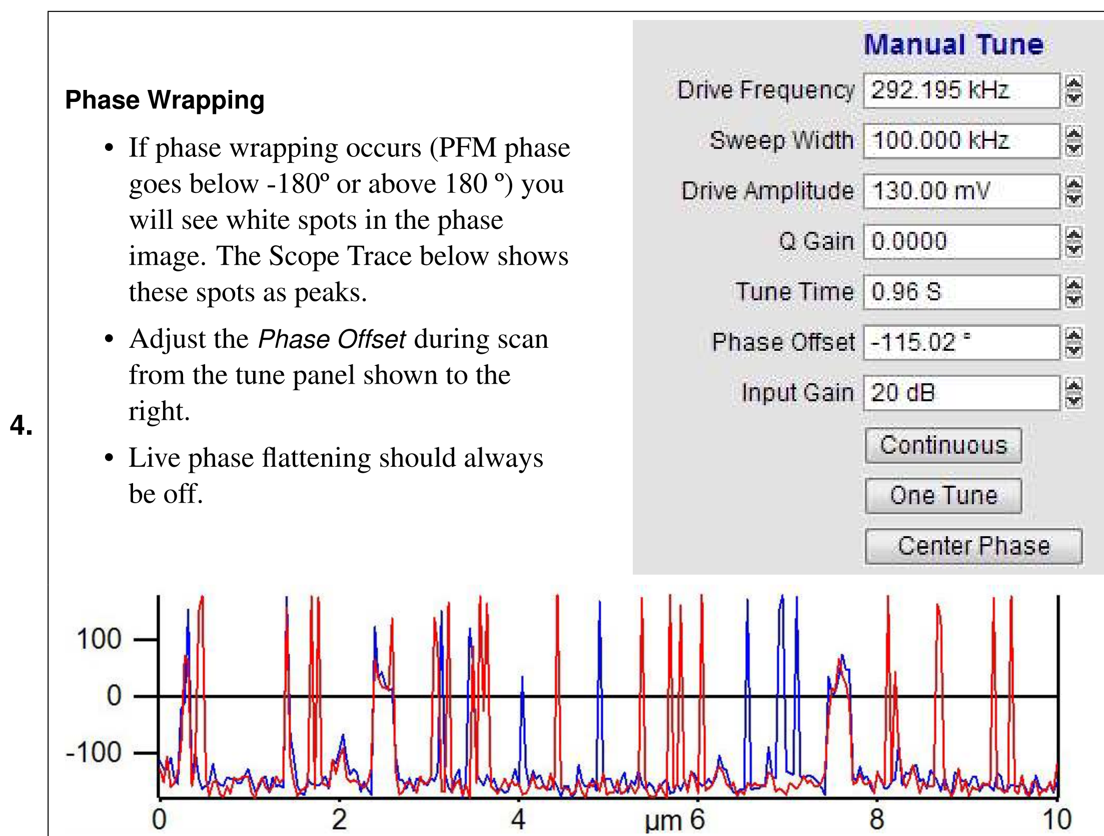
   

3. A common problem is the loading force being too small. If you have tried adjusting the drive voltage and still do not see good domain contrast, try increasing the loading force. Click *Stop* and increase the setpoint, then retune on the surface. The contact resonance changes depending on the setpoint.

4. **Phase Wrapping**

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



8.3. 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, Piezo Force Microscopy using Dual AC Resonance-Tracking.

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.

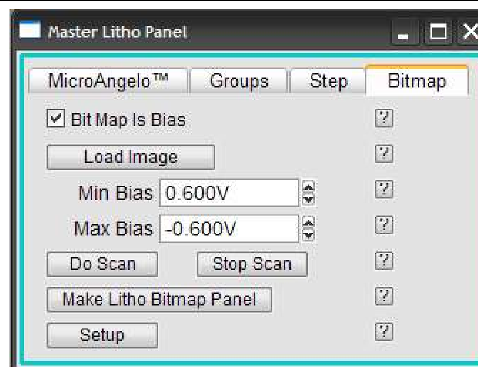
Software Instructions

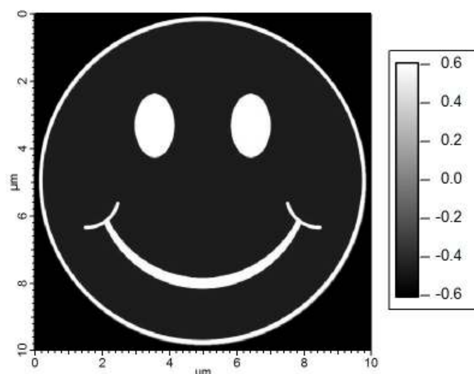
1. To run LithoPFM, click [ctrl+F2], or navigate to *User Settings* ▷ *Mode Master* Click *PFM*, then click *LithoPFM*.

2\.

Loading Bitmap Image

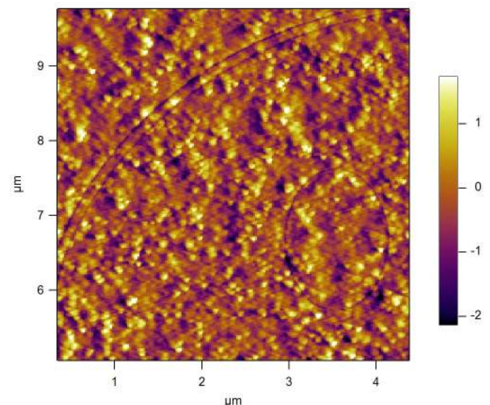
- Click *Load Image* and select a file. The bit-mapping lithography assumes a 256 x 256 or 512 x 512 image. A file named *Smile.jpg* is used for this example.





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

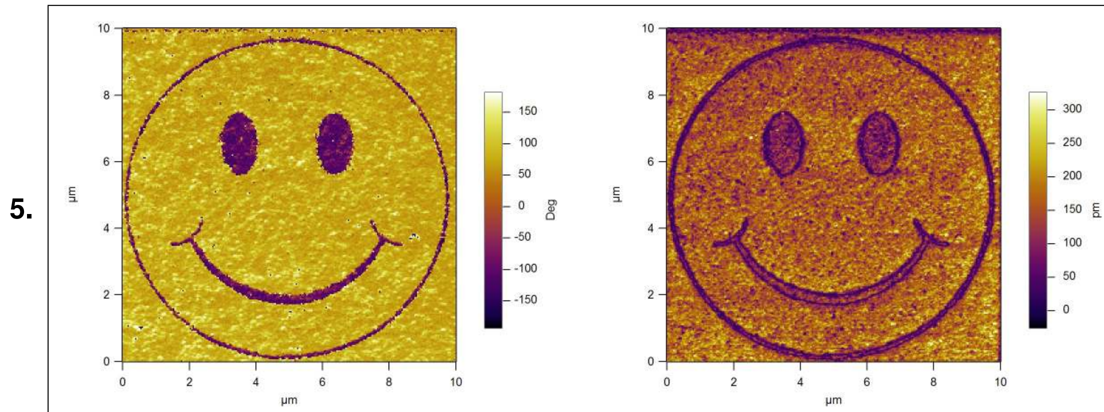
4\.



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

5.



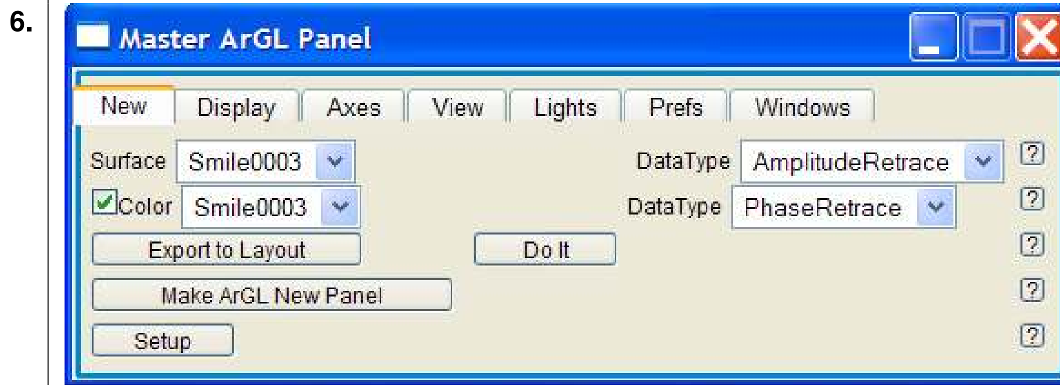
- Switch to the tune panel and tune for single frequency PFM and image as normal. Left image is Amplitude, right Phase.

6.

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



8.4. Vector PFM

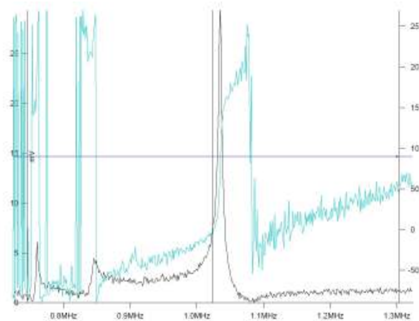
Lateral PFM (Vector PFM) is a two pass technique, first measuring the vertical PFM signal in one pass and then the lateral PFM signal in the second pass. The enhanced LFM head option is required to mechanically center the lateral signal to take advantage of higher gains. The sample used in this example 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

- 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.



Crosspoint Panel

InA	FilterOut		☐
InB	Ground		☐
InFast	Lateral	Lateral	☑
InAOffset	Ground		☐
InBOffset	Ground		☐
InFastOffset	Ground		☐
OutXMod	Off		☐
OutYMod	Off		☐
OutZMod	Off		☐
FilterIn	Defl		☐
BNCOut0	DDS		☐
BNCOut1	Ground		☐
BNCOut2	Ground		☐
PogoOut	Ground		☐
Chip	DDS		☐
Shake	Ground		☐

Write Crosspoint

Current	Status	State
PFMMeter	User	Loaded

Save Wave | Load Settings
Load Scan Crosspoint | Load Force Crosspoint | Reset
No Auto Change Crosspoint

Nap Panel

	Surface	Use	Use	Parm Swap
Integral Gain	10.00		☐	10.00
Proportional Gain	0.00		☐	0.00
Set Point	0.000 V		☐	0.500 V
Drive Amplitude	3.00 V		☑	3.00 V
Drive Frequency	1.024 MHz		☑	1.024 MHz
Drive Amplitude 2	100.00 mV			100.00 mV
Drive Frequency 2	0 Hz			420.000 kHz
Phase Offset	-32.93 °		☑	-32.93 °
Tip Voltage	0 mV	☐	☐	0 mV
Sample Voltage	0 mV	☐	☐	0 mV
User 0 Voltage	0 mV	☐	☐	0 mV
User 1 Voltage	0 mV	☐	☐	0 mV

Nap Mode: Parm Swap — Delta Height: 50.00 nm
☐ Toggle Laser — Start Delta Height: 0 nm

Frequency

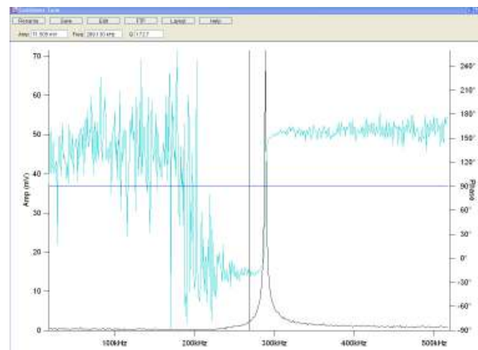
☐ FM Feedback On
Freq I Gain: 5 — DART I Gain: 5.5
Freq P Gain: 0 — DART P Gain: 0

Setup

3.

Vertical Tuning

- To tune the vertical deflection signal, change *Infast* to *ACDefl*, click *Write Crosspoint* and uncheck *No Auto Change Crosspoint*.
- 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.

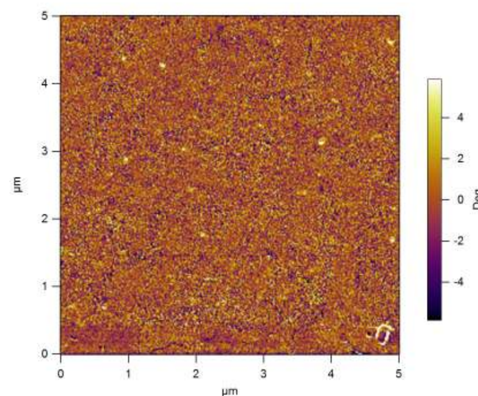


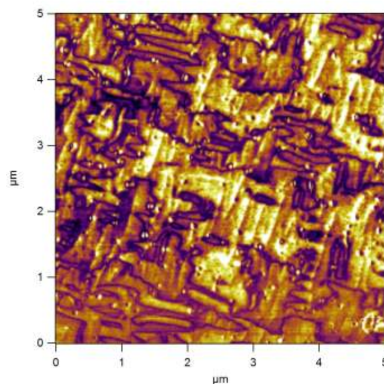
4.

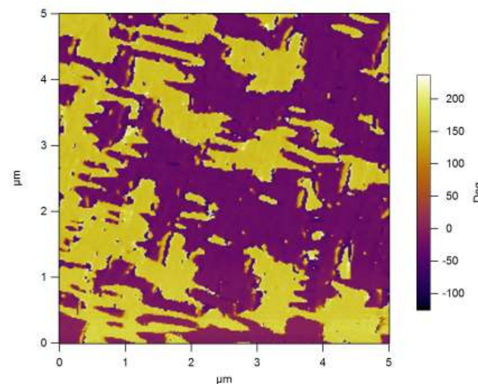
Scan

- Hit *Do Scan* to start imaging.

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







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

CHAPTER REV. 960, DATED 07/12/2012, 14:07. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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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 Laboratory[2]. This technique is sensitive to ion transport into and out of the lattice in energy storage (battery) materials such as $LiCoO_2$[3,4,5]. For more information on this powerful new technique, please refer to

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

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

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

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

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

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

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

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

In the following pages we will go through:

1. A basic introduction to the Dual AC Resonance Tracking (DART) concept . . . . . Page (105)
2. Choosing cantilevers and imaging with DART software . . . . Page (105)
3. DART Hysteresis loop measurement (aka Switching Spectroscopy PFM) . . . . . . Page (113)

9.1. DART concepts

DART is a recently developed 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 9.1 on page 106 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.

9.2. 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 cantilever holder if you have the optional HVA220 unit. Other cantilever holders may not be compatible without cross-point adjustments.

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

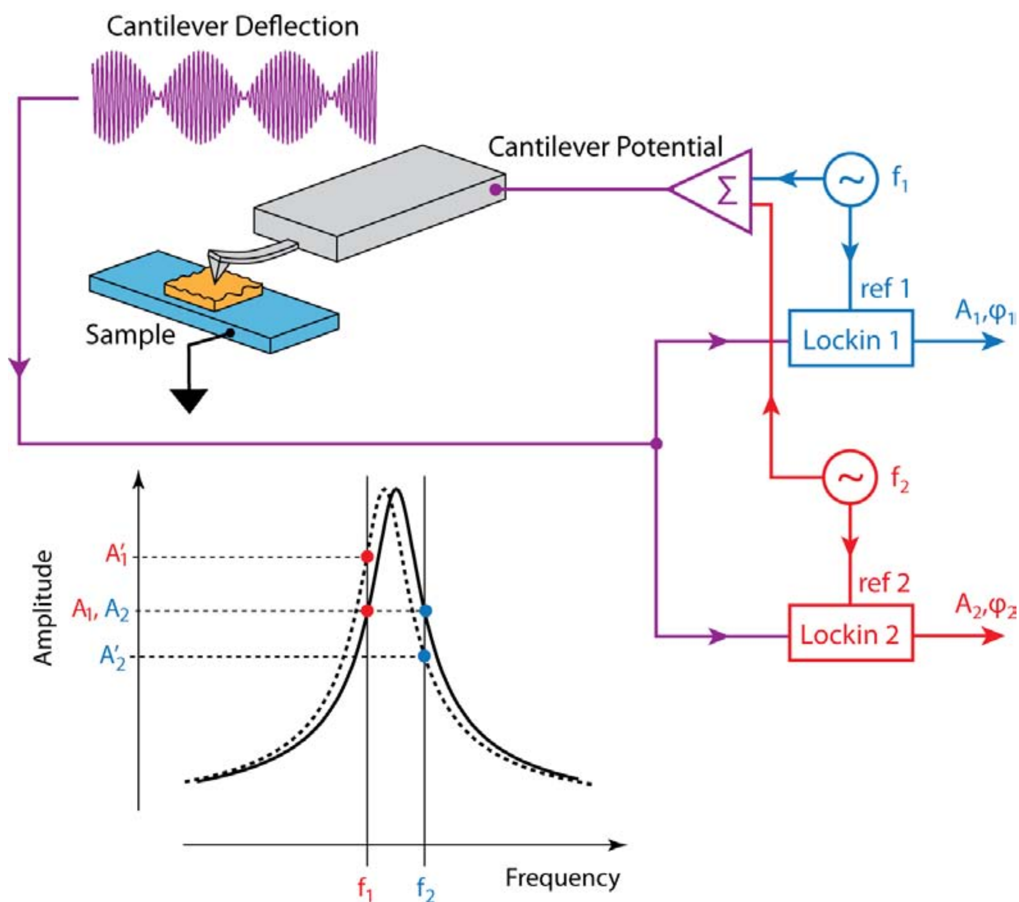


Figure 9.1.: Dart Operation

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, 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 3-5x 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.

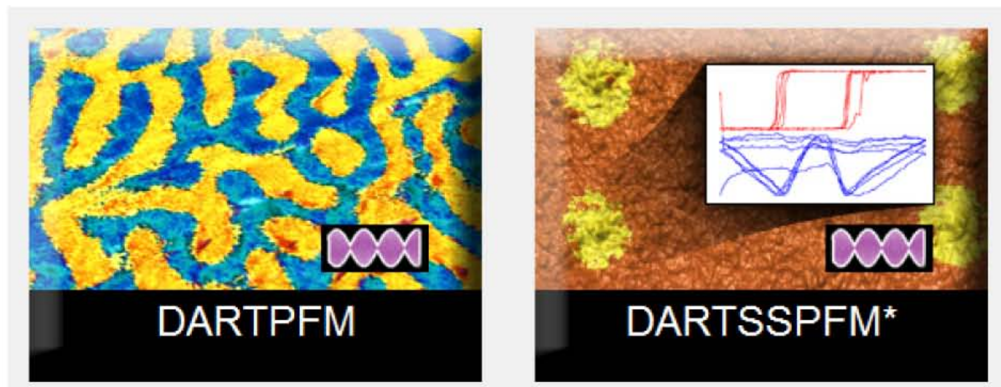


Figure 9.2.: OperationModeMaster DART buttons. Selecting the left option will bring up a template for DART imaging and selecting the right will bring up a template for both imaging and point-spectroscopic (SS-PFM) measurements.

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

9.2.1. Software Instructions

Note These instructions are for software version 101010+0106 or later. Please update your software accordingly. You can find the latest software at www.support.asylumresearch.com.

1.

DART PFM: Getting Started

- Open a new MFP template by starting the AR SPM software from scratch, or by selecting *File ▷ new MFP3D template* from main menu bar.
- From theMode Master window click on *PFM.*
- From the next window click on *DART-SSPF-PFM* (See Figure 9.2 on page 107).

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

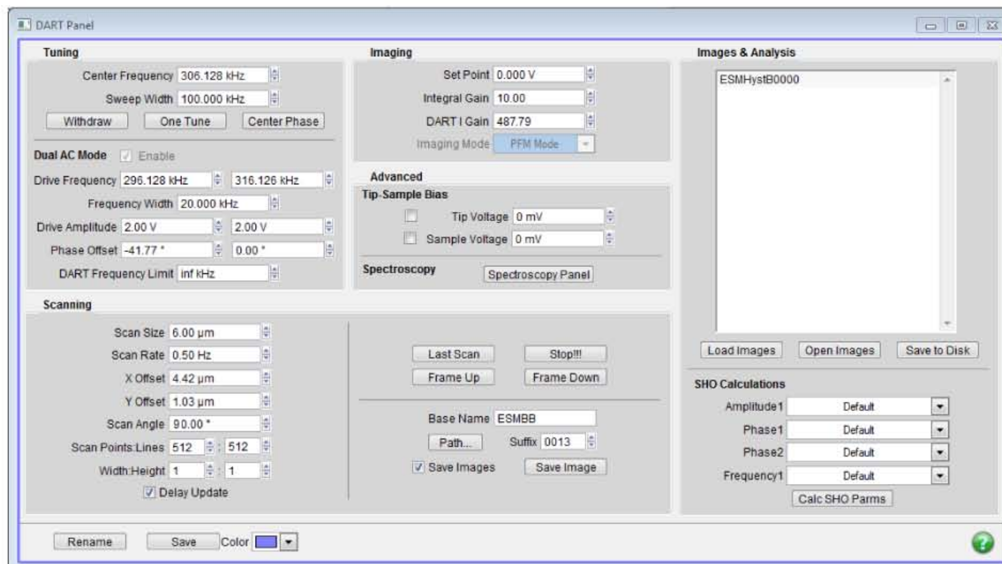


Figure 9.3.: The DART panel.

2.

The Dart Panel:

- The panel in Figure 9.3 on page 108 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. If it does not, or if it was not listed in the User Panels, contact Asylum Research support for help.
- Close the ModeMaster window to make more room on your desktop. You can always get it back by pressing Ctrl-F2.

3. **PFM Initialization**

   - In the *Tuning Area* of the DART panel:
     - Set both drive amplituded to 10mV. If you start with larger values, you may end up unnecessarily degrading your tip.
   - In the channel panel:
     - Typically, you will want to record frequency (important to have this on channel 1), *Height*, *Phase1*, *Phase2* and *Amplitude1*. You can use the difference between the phase channels to evaluate the dissipation (Q) of the tip-sample interactions.
   - In the crosspoint panel (Menu->Programming->Crosspoint Panel):
     - Make sure that the DDS is routed correctly:
       * for the HVA220, it should go to *BNCOut0*
       * for a conventional cantilever holder, it should go to *Chip*
       * for other DART techniques, it may be routed to other actuators such as the contact resonance holder
     - *InA* should be connected to *Filter Out*.
     - *InFast* should be connected to *AC Deflection*.

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 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. Maximize the sum, and then put deflection at ~ -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).

6. 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.

7. Position the head above the sample.

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

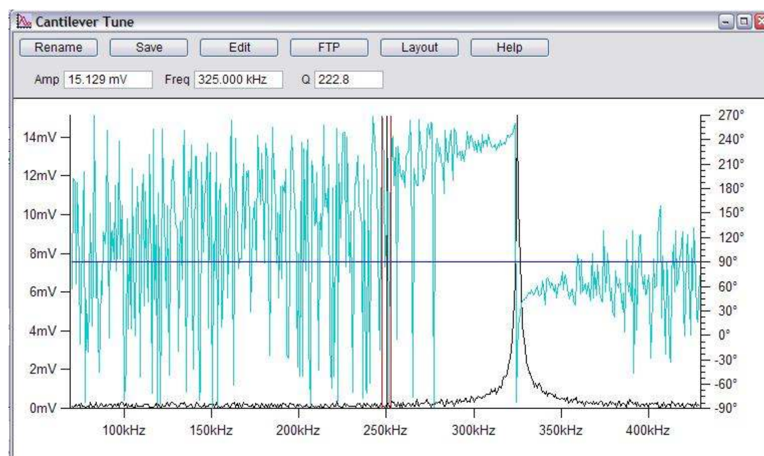
   - Check that the red wire is seated into the HV connector slot glowing red on the sample holder. The magnet should suck the end of this wire into the HV connection slot when the head is positioned above the sample.
   - Take care to insure that the cantilever holder and the sample clip do not interfere 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.
- 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 9.1 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 umfamiliar cantilever, do at thermal tune off the sample surface (Master Panel->thermal tune tab->Start thermal). Look for the first and second resonance–they should appear after 10-100 averages of the thermal tunes. The contact resonance will be between these two values.

- In the DART panel in Figure 9.3 on page 108 try a center frequency of 320 kHz and sweep width of 200kHz.
- The frequency width refers to the width of the separation between f2 and f1around 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 5kHz.
- 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 range 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

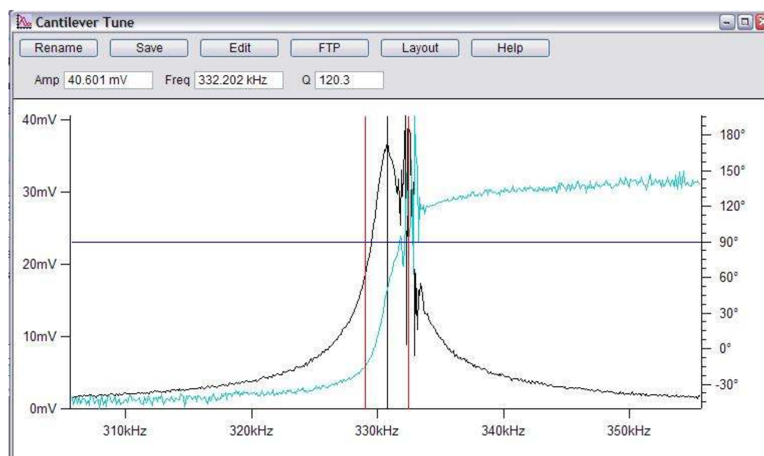
sufficient to see the peak but you may need more. Your goal is to see a contact resonance peak of ~10-50mV. Going higher than that will typically cause problems with feedback stability and will lead to rapid tip degradation.

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. Zoom to 50kHz and do another tune. The "noise" centered on the high frequency red bar is normal interference between the two lockins and means things are functioning normally.

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

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

13.



Successful tuning result

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

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

14.

Set Scanning Parameters

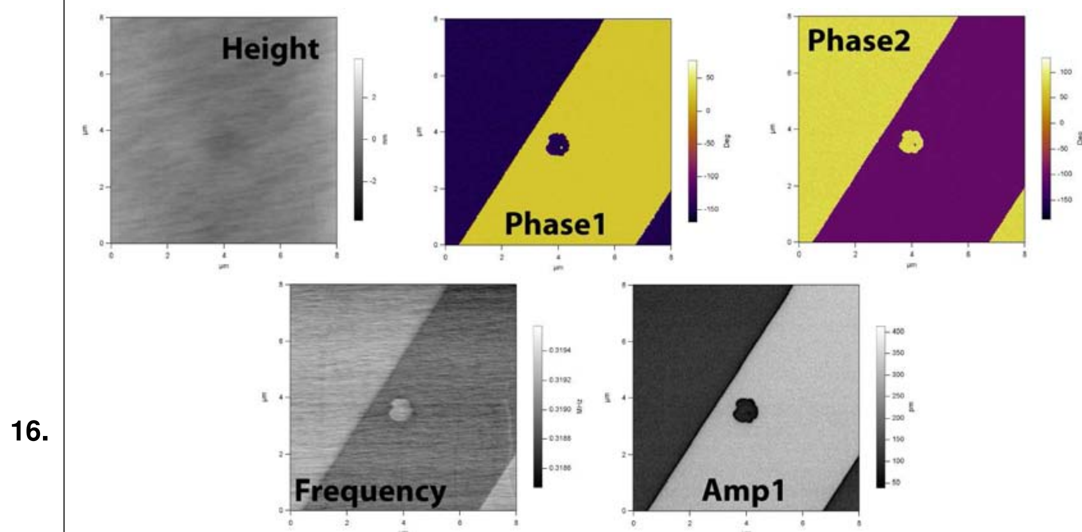
- In the Scanning section of the DART panel 9.3 we find it useful to start with a resonably large scan range (perhaps 20 by 20 microns).
- Set the scan angle of 90° (the latter generates better reproducable results).

15.

Set Imaging Parameters and Start Imaging

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

16.



Typical Images, PPLN sample

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

17.

Optimizing Images

- Right-Click on one of the images and choose *Fix All Scales*.
- Depending on the balance of electrostatic and piezo forces, you may see more or 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 this not by changing 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.

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

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. To do this, you will interact with the DART Spectroscopy Panel, shown in Figure 9.4 on page 114. We will go through the process of measuring SS-PFM hysteresis loops as described by Jesse et al. First we'll review some of the terminology related to the triangle step functions which will excite the tip voltage.

There are four parameters for the Triangle step function. The effects of varying them are illustrated in Figure 9.5 on page 115 below.

Amplitude The zero to peak value of the drive wave. Care should be taken that the sum of this amplitude plus the drive (oscillating) amplitudes never exceeds 10V for the standard holder or 220V for the high voltage option (HVA220) amplifier. Also, remember that since DART applies two sinusoidal drives, you must use 2xOscillator drive amplitude when calculating this. It is generally better to start small and increase the number gradually, say ~1V.

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

Optional Argument 3 This controls the phase of the overall hysteresis measurement. A value of 0.5 means the voltage starts at 0 and ramps in the positive direction first. For some examples see Figure 9.6 on page 115.

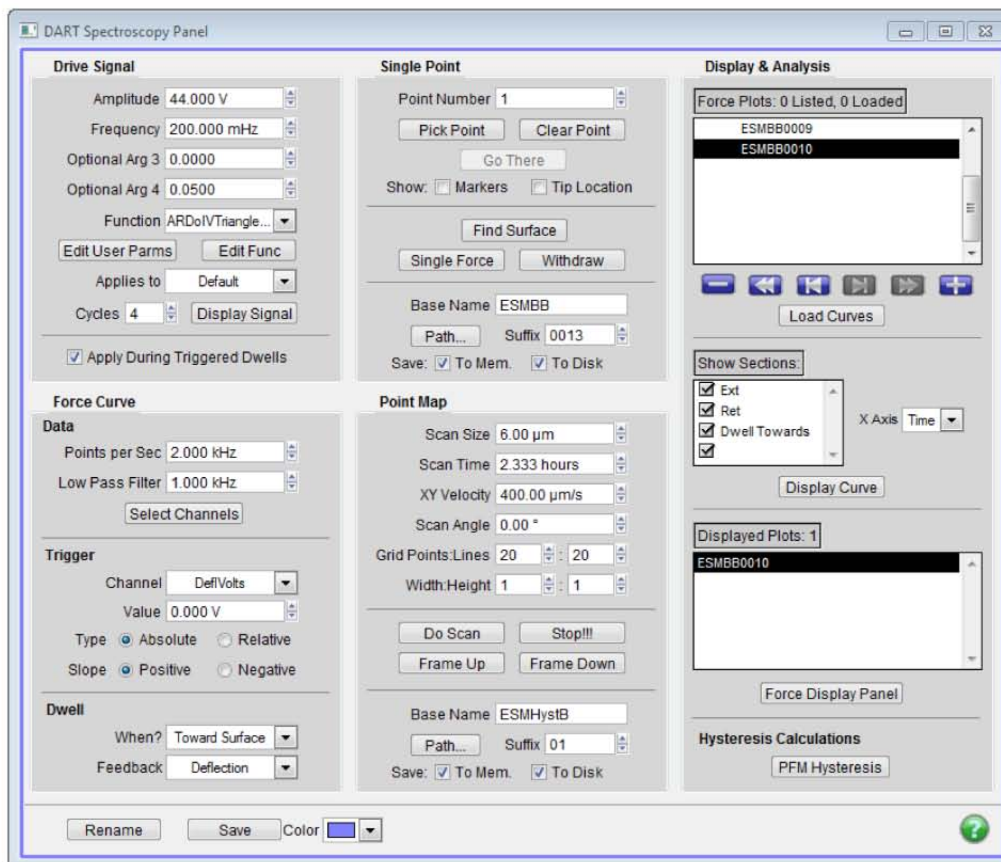


Figure 9.4.: The DART Spectroscopy Panel

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 250m seconds total. Of that, 125m seconds will be at the applied voltage and 125m seconds will be at 0 volts.

Note	The switching voltage for PPLN is >10V, you will need the HVA220 module to see hysteresis loops. With the standard configuration you will not see the loops.

9.3.1. Step by Step Software Instrucitons

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 9.4 on page 114), go to the *Drive Signal* group of controls.
3. Click on the *Display Signal*. This brings up a voltage versus time display for the loop measurements. This is useful for insuring you are applying the proper waveform.

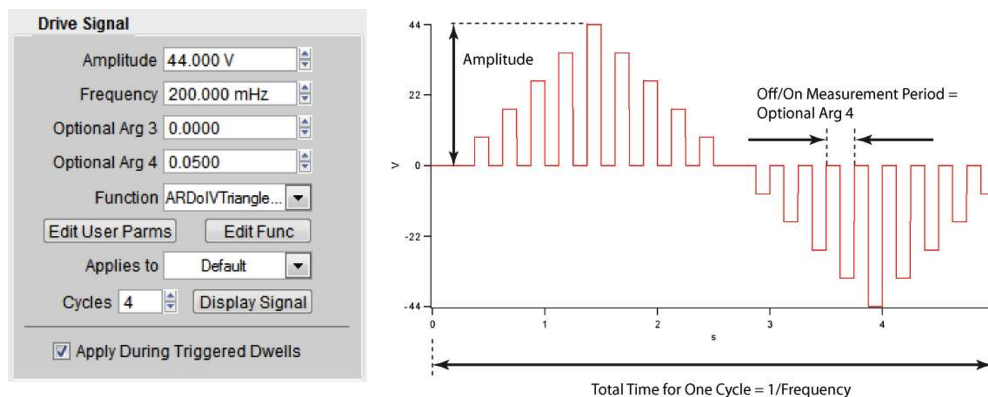


Figure 9.5.: PFM drive waveform parameters

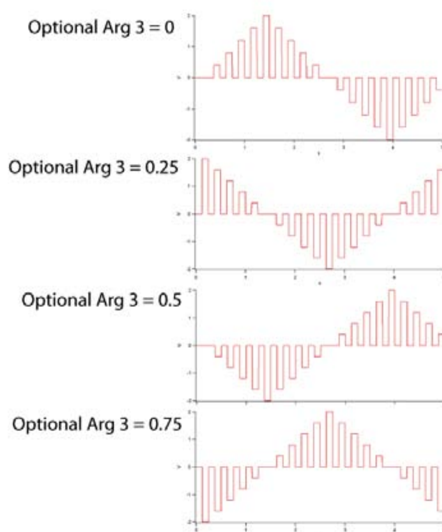


Figure 9.6.: Some examples of the effect of Optional Argument 3

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, *Amplitude 1*, *Phase 1* 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.
   c) *Dwell* 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.

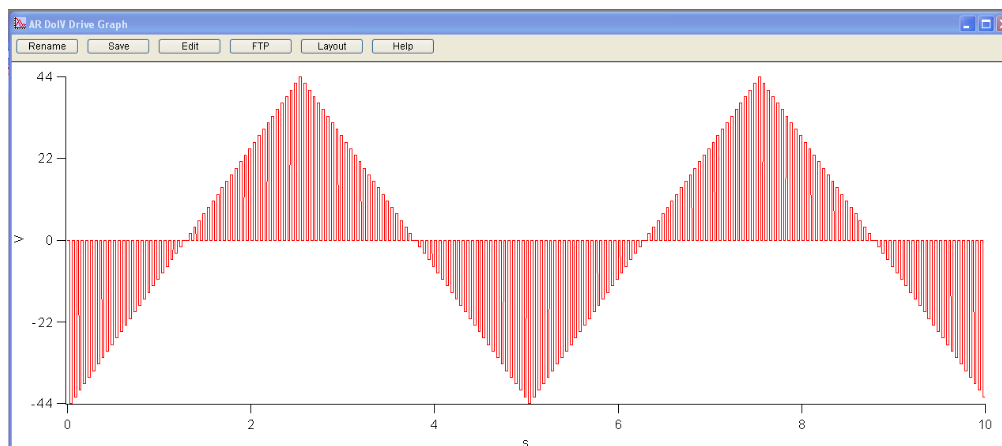


Figure 9.7.: A typical scan waveform for measuring in-field and remnant hysteresis loops for SS-PFM.

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. Select the popup *Show Markers* to bring up the locations and indices of the various locations you've selected.

e) Check the box *Tip Location*. This should place a red ball on the current tip position. Using the *Go There* button and the position index, you can put the tip on any of the locations you have selected.

f) Go to one location and do a surface tune. As before, center the peak between the red bars.

g) Click on *Find Surface*, this will engage the tip and find where the surface. This only needs to be done once, by default the tip will pull away 1μm from the surface after a force curve. If the surface is rougher than 1μm then *Find Surface* should be clicked occasionaly such that the tip does not crash into the surface if the surface is above the start point of the force curve.

h) Click on *Single Force*, the data displayed in the Force Graph 9.8 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 9.10 below for a nice example of data extracted from the time waves shown in Figures 9.8 and 9.9.

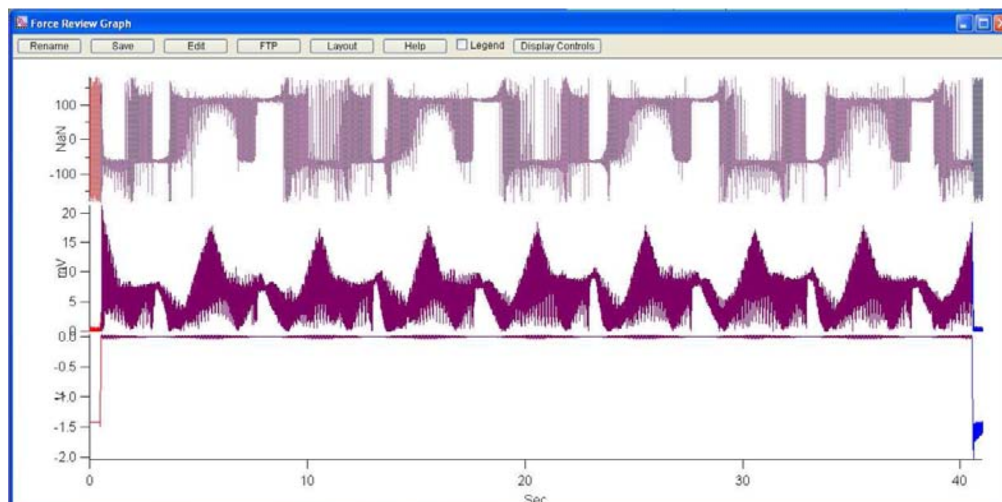


Figure 9.8.: The SS-PFM raw data set. The phase1, amp1 and deflection traces for an 8-cycle, 40 second hysteresis loop. The details of the switching are difficult to discern from the time data and need to be processed to produce hysteresis loops.

9.3.2. 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) Hit *Do Scan*. The red ball should start moving on the image and you should see data start to appear in the Force Graph window. Force volumes 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. At the time of this writing, the estimated time grossly underestimates the time required for an array of hysteresis loops to complete.

e) If there's a problem, hit the *Stop!!* button, make adjustments to the parameters and start again.

9.4. DART Guidelines and Troubleshooting

9.4.1. Amplitudes of Drive and Response

Start out with a drive voltage of 250mV. If you need to drive the tip at >5.5 volts for the standard cantilever holder, it is likely the tip will experience some degradation. These drive amplitudes should result in response peak amplitudes between 5-50mV. Generally, amplitudes less than that

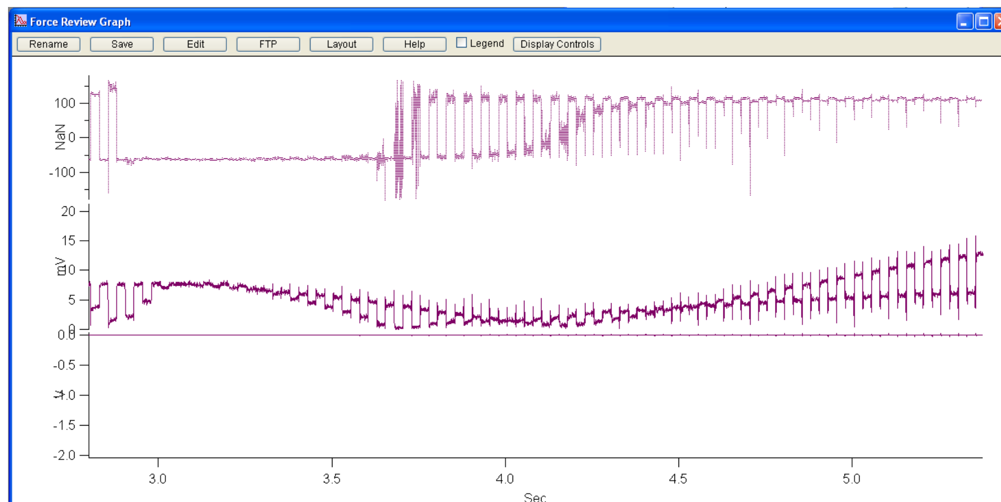


Figure 9.9.: Zoomed in region from Figure RawData. Here, the individual steps from the stepped SS-PFM method of Jesse et al. are evident. h.Repeat the above steps as many times as you would like, adjusting the drive parameters as you wish. i.If you notice noise during the higher voltage portions of the curves, you may have your 'Freq Igain' set too large. Reduce it until the effect disappears.

may not result in stable feedback and amplitudes larger than ~50mV lead to instabilities in the frequency feedback loop.

9.4.2. Image Size

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

9.4.3. 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°.

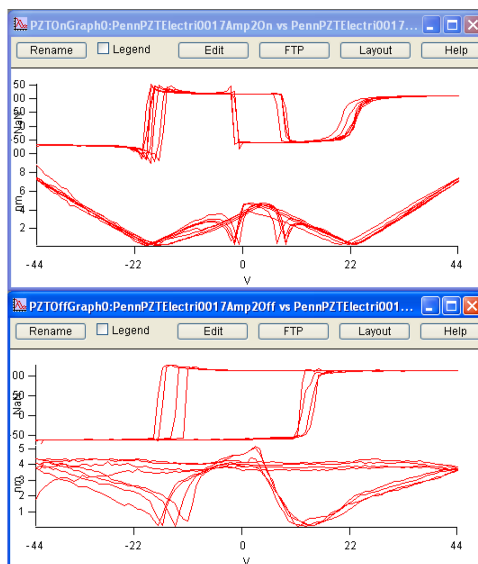


Figure 9.10.: The applied ('On', top graph) and remnant ('Off', bottom graph) hysteresis loops obtained from the processed time waves of Figures 7 and 8. The phase loops are on the top of each window and the amplitude "butterfly" loops are on the bottom.

9.4.4. "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.

9.4.5. Loss of Frequency Lock

If the sample is weak 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.

9.4.6. 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.

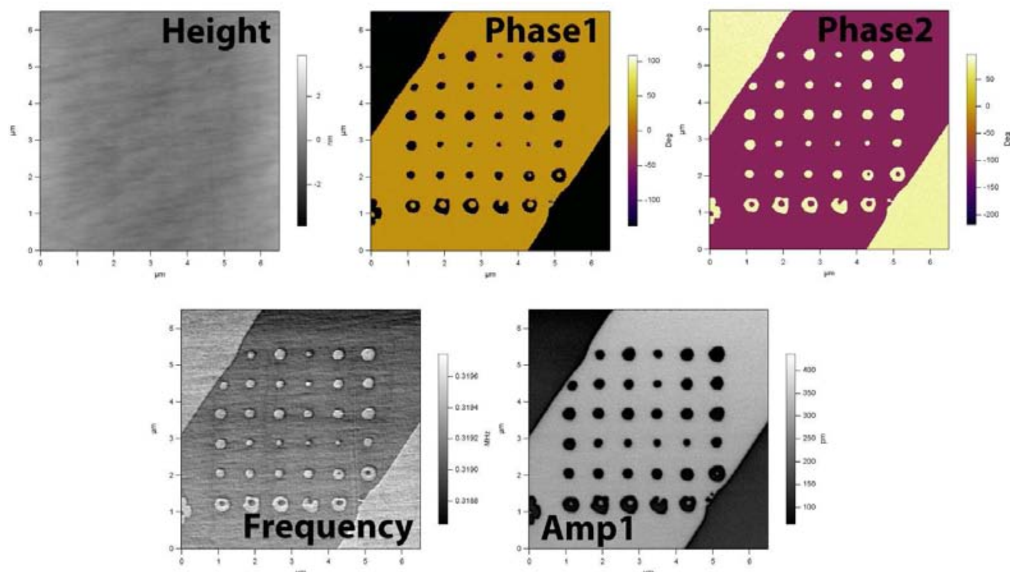


Figure 9.11.: A DART PFM image of PPLN after a 6x6 array of hysteresis loops have been measured.

9.5. 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 highly safe and secure manner. The tip itself is not energized until the head is securely in place over the sample, through the use of a simple magnetic interlock. Once the head is in place, the high voltage at the sample still needs to be enabled through a SmartStart™ command in the Asylum software.

To access the HVA220 enable command, first make sure that the SmartStart system has detected the high voltage module by clicking on the controller icon at the bottom of the screen. A list of detected devices should appear, including the HVA220, as shown in Figure 9.12 on page 121.

If the HVA icon does not appear, rescan the SmartStart bus by clicking on the 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 in the on position. If you still have problems, contact Asylum Research support for help.

Once you have located the HVA220 icon in the SmartStart list, you need to tell it to enable the high voltage relays supplying the signal to the sample. Go to *Default -> Mode* and select *On*. Click on the *Write* button. You may hear a small click in the HVA220 as the safety relay closes. You should also see the green HV LED on the HVA220 front panel turn on and the red warning lights on the HV sample holder come on. You are now ready for HV PFM imaging and spectroscopy.

If the lights do not come on, power the unit down, check the connections and repeat the above instructions. If you still have issues, please contact Asylum Research support for further help.

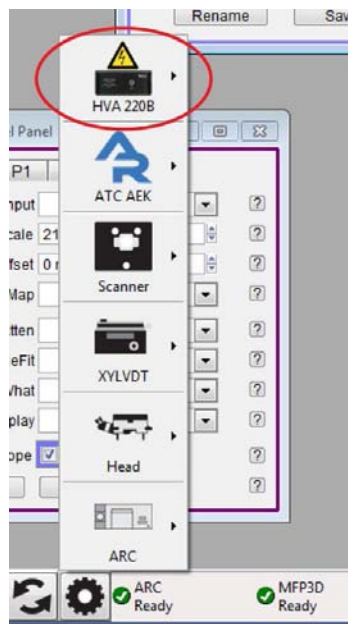


Figure 9.12.: Testing communication with the HVA using the SmartStart Device interface. The HVA icon indicates that the HVA is present and active.

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

Part II

Force Curves and Maps, Spectroscopy, and Lithography

Part II: Who is it for? Succinct step by step instrucitons for force curves, force volume mapping, lithography, and other Spectroscopies. Quick instructions with a minimum of theory.

Part Contents

10 Spring Constant Determination **125**
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10. Spring Constant Determination

CHAPTER REV. 777, DATED 08/27/2011, 00:27. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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10.1. 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 10.1.1 on page 126.
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 10.1.2 on page 130.
3. Withdraw tip & perform a thermal tune to determine the cantilever's resonant frequency. An algorithm computes the spring constant using the equi-partition theorem. See Section 10.1.3 on page 134.

This protocol can also be found in the Spring Constant Tutor: to view it in the AR SPM software, select from the main menu bar: *Programming ▷ Start User Panel ▷ Spring Constant Tutor.* It has an all in one panel set up that contains everything you need to do to complete the k determination, see Figure 9.9

A simplified technical note on spring constant calibration via the thermal method entitled Spring Constant Calibration by Roger Proksch, PhD. can be found at the end of this chapter.

NOTE: If at all possible, it is best to do the spring constant calibration in air due to the higher cantilever Q (relative to the generally low cantilever Q in fluid).

10.1.1. Determine Virtual Deflection in Optical Path

The virtual deflection is a mechanical coupling of the deflection signal with the Z movement, 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 in return aids force curve analysis because the free air baseline will have a constant deflection value until it makes contact or some other force is imparted upon the tip. In many dissociation based force measurements, analysis generally averages some section of the free air to find the zero contact point of the retract portion of the curve; having a baseline with a constant deflection value makes this more precise.

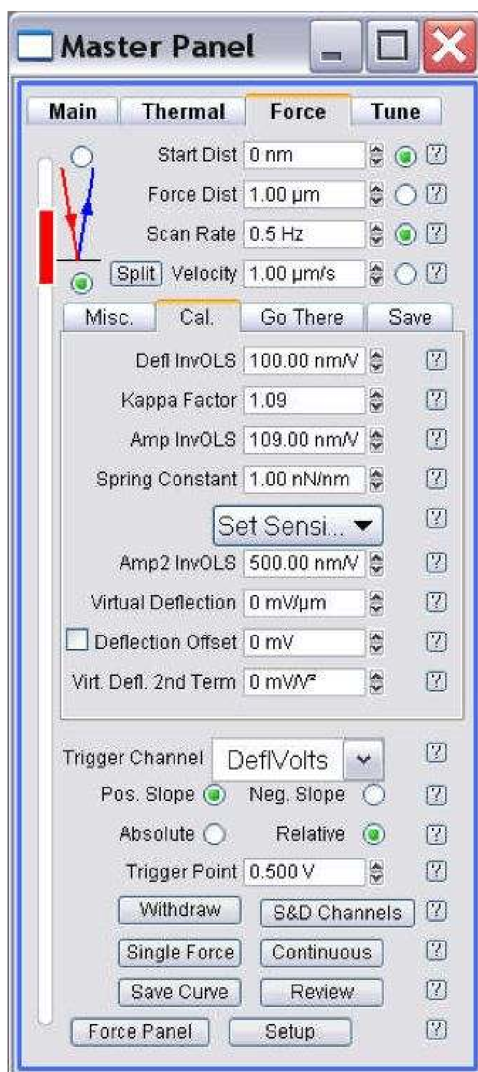


Figure 10.1.: *Master Panel ▷ Force Tab ▷ Calibration Tab.*

1. Put the tip on the surface by engaging in Contact Mode on a clean, (infinitely) hard surface[1] (i.e., freshly cleaved mica, clean glass slide, cleaved graphite).

   Note Avoid bumpy surfaces such as an evaporated metal film like Au on glass, or a dirty sample, because the tip may experience some slipping on the surface if on an asperity.

2. Select 'Contact' from the Imaging Mode pull-down menu on the *Master Panel ▷ Main Tab*
3. Select a Set Point voltage (force). A low Set Point voltage is slightly more positive (in volts) than the value of the 'Free Air' Deflection (in Volts) in the S&D meter; a higher Set Point voltage (force) is much more positive than the free air deflection voltage value. It's best to use a low set point forces to avoid truncating the tip's apex.
4. Integral gain = 7 to 10 is usually sufficient.
5. Click the 'Engage' button on the *Sum and Defletion Panel* meter.
6. Engage tip by slowly turning the thumbwheel (counterclockwise) until the Deflection voltage matches the Set Point voltage, a bell sound will chime if speaker volume available. Continually approaching with the thumbwheel, the Z-voltage value goes from railed (150 V, red), to some other lower voltage value. Adjust this down to around 70 V (no color in Z- voltage meter).

   NOTE: If you need to engage as gently as possible, it is best to do a false engagement in AC Mode first. Increase the Set Point force until the tip hard engages at the minimum possible force, then switch over to Contact Mode once engaged.

7. Select *Master Panel ▷ Force Tab*: This panel is set up so the user can define the specific distance the piezo travels during the force-distance cycle (called 'Force Distance'). SeeFigure 10.1 on page 126.
8. The white bar on the left side of the window represents the entire Z range of the Z-piezo in the AFM head. The red bar represents the 'Force distance'- a user defined approach/ retract cycle distance that the Z-piezo moves during the force-distance curve acquisition. As you change the value of the force distance, notice that the size of the red bar's length changes. This can also be activated with the Hamster wheel, or by clicking on the red bar and dragging the mouse.
9. Initial Parameter adjustment- set parameters similar to those in Figure 10.1 on page 126.
10. Activate the radio button for 'red' mode (force distance bar is red)- this means the tip starts the approach/ retract cycle off the surface .
11. Slide the red force distance bar all the way to the top of the white vertical Z range bar.
12. Choose a 'Force' distance (0.5 to 1 µm).
13. Velocity of 1 µm/s. Activate the 'Trigger' from the Trigger channel pulldown menu. Choose the 'DeflVolts' channel. The trigger point value is the amount of positive deflection (or force, or Z distance) the tip experiences in contact with the surface before the piezo switches direction for the retraction part of the curve.
14. Activate the 'relative' trigger radio button.
15. Enter a 'trigger' point value of 0.4 to 0.6 V.

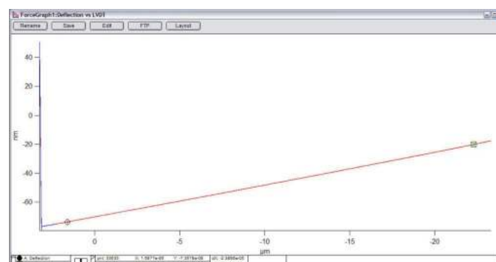
[1] An infinitely hard surface will have a slope of unity in a distance vs deflection plot

16. Click 'Single Force' button. Notice the Z-piezo voltage extends the tip to approach the surface, reverse direction when it finds the surface, then retracts and eventually stops at a voltage value that correlates with the user defined Force Distance.

17.

Inspect the Resulting Force Curve

- The resulting curve should have a long sloping section on the right, preferably spanning nearly the full Z range of the AFM.
- Since this long approach curve is needed to perform the virtual deflection correction, please adjust your force curve parameters until you achieve such a result.



18. With the force plot window active, hit Ctrl + I which brings up a panel on the bottom of the plot that has square and circular cross hair cursors.

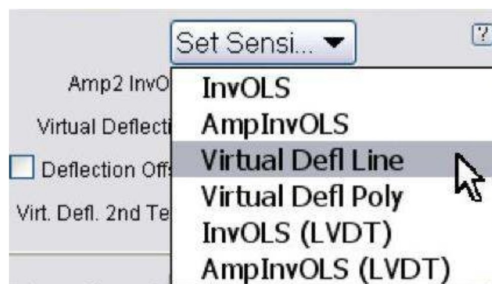
19. Drag and drop the both cursors onto the linear part of the (red) approach curvePlace the Igor cursors on linear part of free air approach (red) curve as shown in Step 17 on page 128.

20. Make sure both cursors are on the same (approach/ retract) trace by using the keyboard arrow keys to assure they both move in the same direction. If they move away from (or towards each) other, they are not on the same trace.

21.

Perform Line Fit

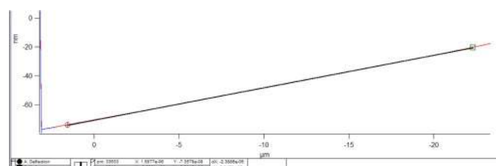
- Double check that the 'Cal' tab of the *Master Panel* ▷ *Foce Tab* is active, as seen in on page 126.
- Select 'Virtual Defl Line' from the 'Set Sensitivity' pull-down menu.
- When you "release" the mouse button, a fit will be performed.



22.

Inspect Fit Result

- The portion of the curve between the Igor cursors gets fit, represented with a black line.
- Also notice the virtual deflection value has updated in the calibration sub tab.



23. **(Optional) Remove Fit**

- To remove this line fit, place the mouse cursor on the black fit line, right click; choose 'Remove fit_DeflVotls' .

Note Removal of this curve allows you to perform another force curve for the next step. If you don't remove the fit, then the updated virtual deflection parameter will have the next curve off scale and you won't see the new curve in the plot window.

Note Alternatively, just close the Force graph window- a new one will open the next curve acquistion and will be properly rescaled.

24. 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..

10.1.1.1. Tips and Tricks

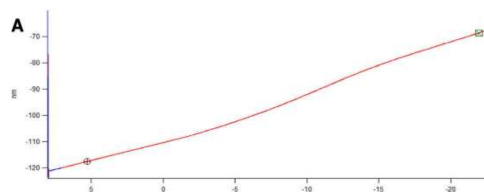
Tip Keep your distance!

Alternatively, you can calibrate the virtual deflection with the tip so far off the surface that the tip never touches down. This ensures that the entire curve qualifies as the "approach".

- In case you were already engaged on the surface, first hit the withdraw button.
- On the MFP-3D head, turn the front leg wheel by at least one full Z range.
- On the Cypher AFM, use the ???? panel to motor up 10 microns.

Tip Extended Range MFP-3D Head

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

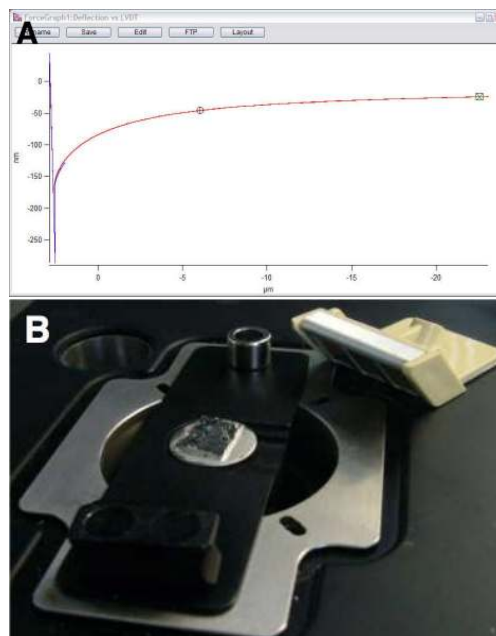


- In this case you should select 'Virtual Defl Poly' in Step 21 on page 128 to account for the curvature of the approach curve.

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.
The graph shows the effect of long range electrostatic forces wich can vary strongly over time. Hence even a polynomial fit is not a good solution.
Solutions are shown to the right:

- Choose a conductive flast surface such as freshly cleaved graphite (HOPG) or silicon on a conducting substrate.
- Place a static master ionizer near your sample.



10.1.2. 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.

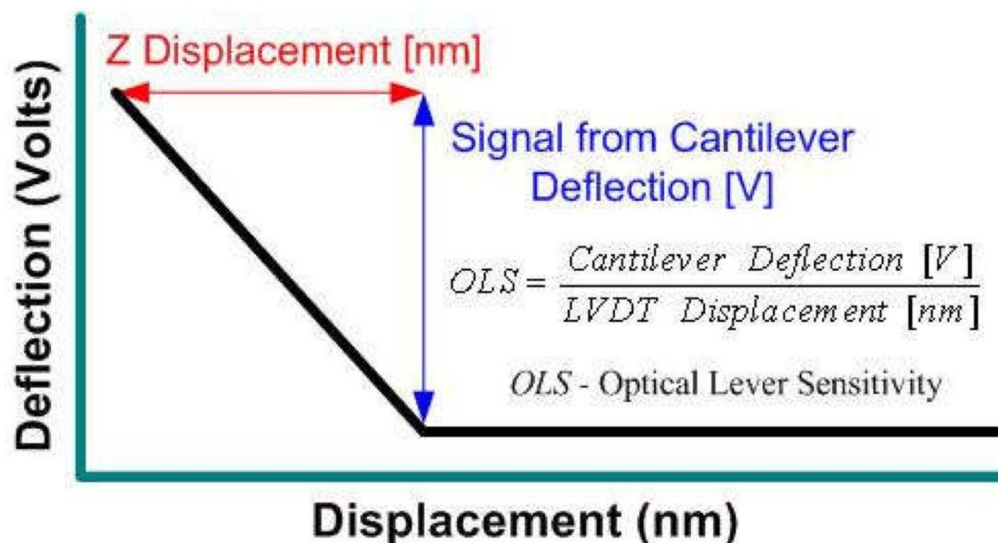


Figure 10.2.: Inverse Optical Lever Sensitivity derived from a force curve on a hard surface.

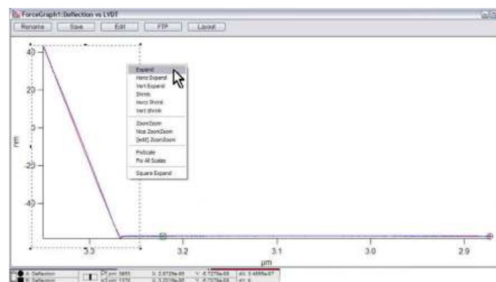
Figure 10.2 on page 130 gives a sense of what the optical lever sensitivity is, given in units of V/nm, and the software inverts it for the algorithm.

1.

Perform Force Curve

- Click the ’Single Force’ button again. This time the force distance will be equal in both approach and retract cycle because the instrument now knows where the surface is.
- Optionally click and drag a box around the sloped region of the curve and right click to expand on it for better a better view.

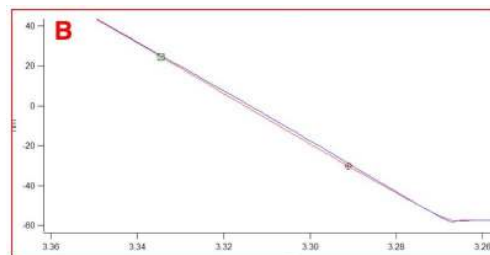
Note the non-contact region of the curve should be horizontal if the virtual deflection correction is working properly.



2.

Place the Crosshair Cursors

- As in Step 18 on page 128 and the following two steps, place the crosshair cursors on the straight part of the line.
- Perform the check with the arrow keys to see both cursors are on the same line segment.



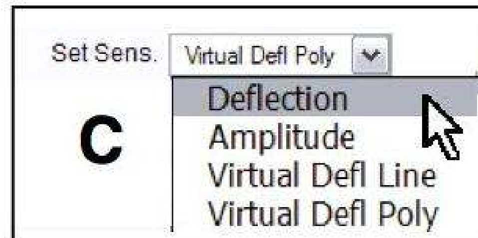
3.

(Optional) Fine Tune Cursor Positions

- Deactivate one of the cursors on the curve by going into the lower margin of that Igor window and clicking on the open circle/square.
- When it’s deactivated, it will turn black allowing you to finely position the other cursors on the curve. When done, reactivate and check that both cursors are on same trace.

4. **Perform Line Fit**

- Double check that the *Cal tab* of the *Master Panel* ▷ *Force Tab* is selected.
- Select 'Defl InvOLS' from the 'Set Sensitivity' pull-down menu.
- When you "release" the mouse button, a fit will be performed and the InvOLS value will update.



5. **Disengage the Tip**

- Disengage the tip by clicking the 'Stop!!!' button on the bottom of the *Master Panel* ▷ *Main Tab*,
  -OR-
- by clicking the 'Stop' button to the right of the deflection meter on the Sum and Deflection Meter window
  -OR-
- by clicking the 'Withdraw' button in the *Master Panel* ▷ *Force Tab*.

All do the same thing: the S&D meter should show a piezo voltage of 0.0 V in its 'withdrawn' position.

10.1.2.1. Averaging InvOLS

For more precise measurements of the InvOLS (because they can be slightly different from curve to curve), statistical representation can easily be performed using AR SPM software.

1. At the bottom of the *Master Panel* ▷ *Force Tab* (See Figure 10.1 on page 126), click the 'Set Up' button.

2. **Add the 'Limit Cont' Control**

   - Check the 'Limit Cont. to:' Show? checkbox.

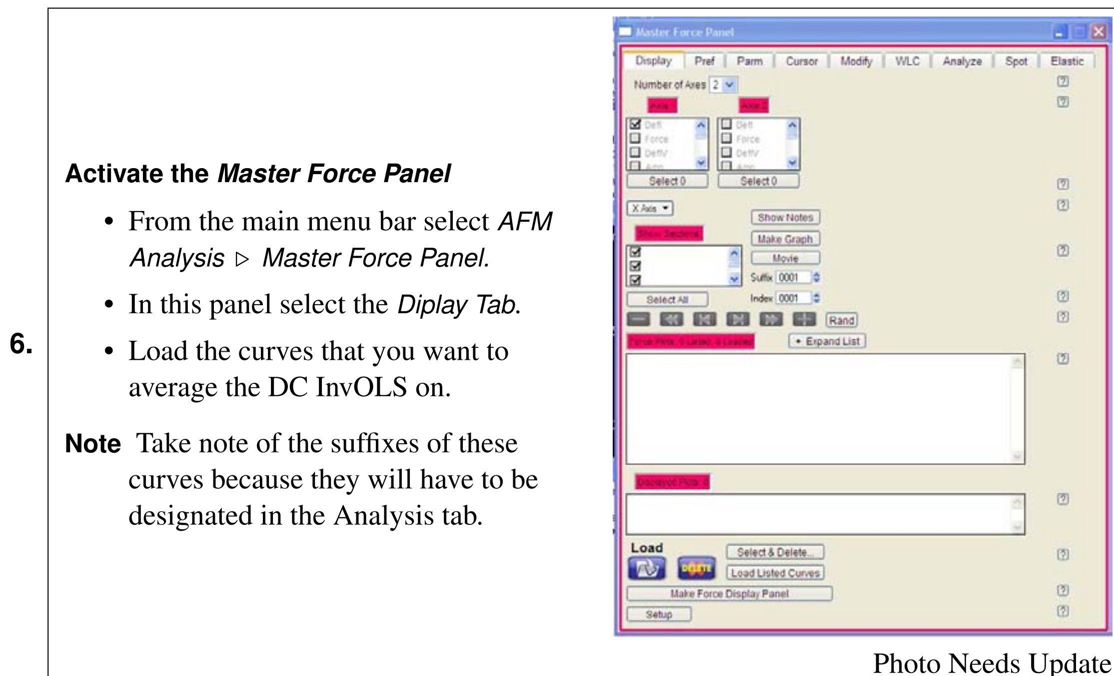
   Note This will allow a user defined number of force curves to be sequentially acquired at the same XY location, without clicking the 'Single Force' button repeatedly.

3. Click the 'Looks Good' button to return to the *Master Panel* ▷ *Force Tab*, but now with the additional control item.

4. Enter a value for how many force curves to be acquired in one spot. Lets try 100 curves.

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

6.



Activate the *Master Force Panel*

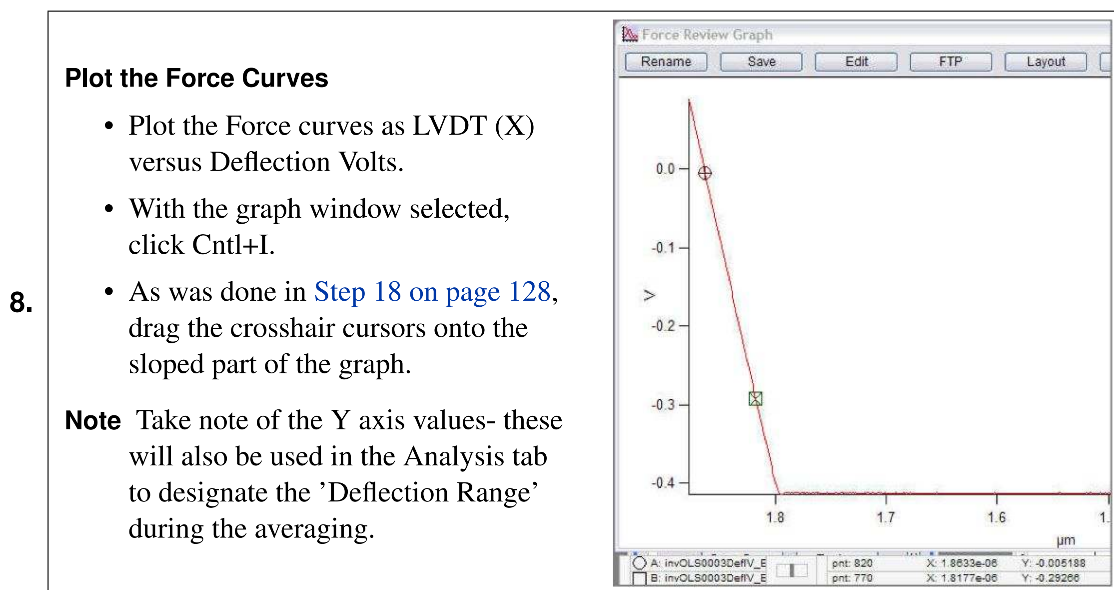
- From the main menu bar select *AFM Analysis ▷ Master Force Panel.*
- In this panel select the *Diplay Tab*.
- Load the curves that you want to average the DC InvOLS on.

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

Photo Needs Update

7. Confirm that only the ‘Ext’ checkbox is activated, indicating that you only want to look at the extension part of the force curve.

8.



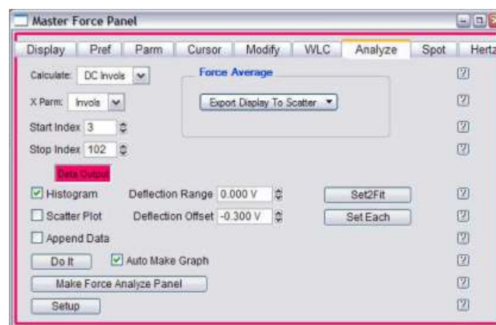
Plot the Force Curves

- Plot the Force curves as LVDT (X) versus Deflection Volts.
- With the graph window selected, click Cntl+I.
- As was done in Step 18 on page 128, drag the crosshair cursors 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.

9. **Prepare for Analysis**

- Go to *Master Force Panel* ▷ *Analyze Tab*.
- Under the 'Calculate' pull-down, select 'DC InvOLS'.
- Under the 'X Parm' pull-down, select 'InvOLS' (InvOLS is what you want the histogram to have on its X axis).



10. Enter the appropriate suffix range in the 'Start' and 'Stop' Indices.

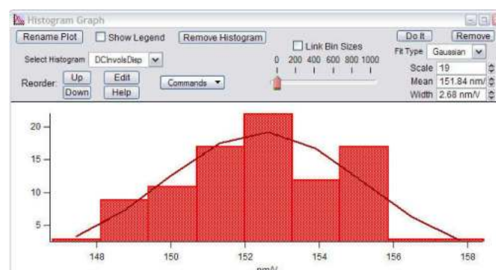
11. Under the 'Deflection Range' conrol, enter the value of the crosshair cursor closer to the trigger point.

12. Under the 'Deflection Offest' conrol, enter the value of the other crosshair cursor.

13. **Create the Histogram**

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

Note The Mean and Width of the histogram fit is displayed, and that the Bin size can be changed using the slider bar.



10.1.3. Perform a Thermal Tune

1. Select the *Master Panel* ▷ *Thermal Tab*.

2. (Optional) To be very precise, retract the cantilever a hundred or more microns from the surface. On the MFP-3D use the large wheel on the head. On Cypher use the ???.

3. **Zero the Deflection**

- Adjust the deflection so it reads ~0V deflection in the Sum and Deflextion meter.

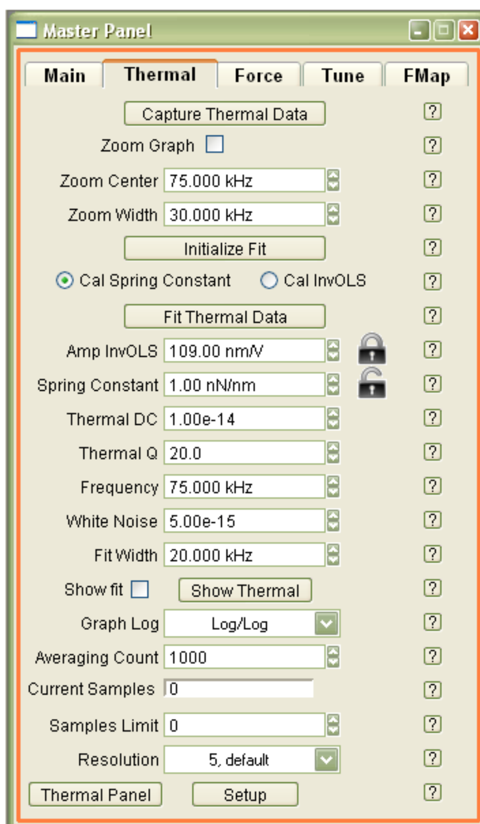


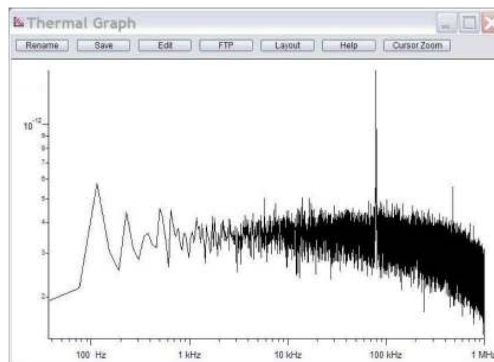
Figure 10.3.: *Master Panel* ▷ *Thermal Tab.*

4.

Capture Thermal Data

- Click the 'Capture Thermal Data' button at top of the *Master Panel* ▷ *Thermal Tab.*

This measures the deflection signal as a function of time and presents the Fourier transform of these data in the form of a power spectrum. The spectrum peaks at the first and higher order resonant frequencies of the cantilever. Over time, the measured spectra are averaged and you will notice the curve become less noisy over time.



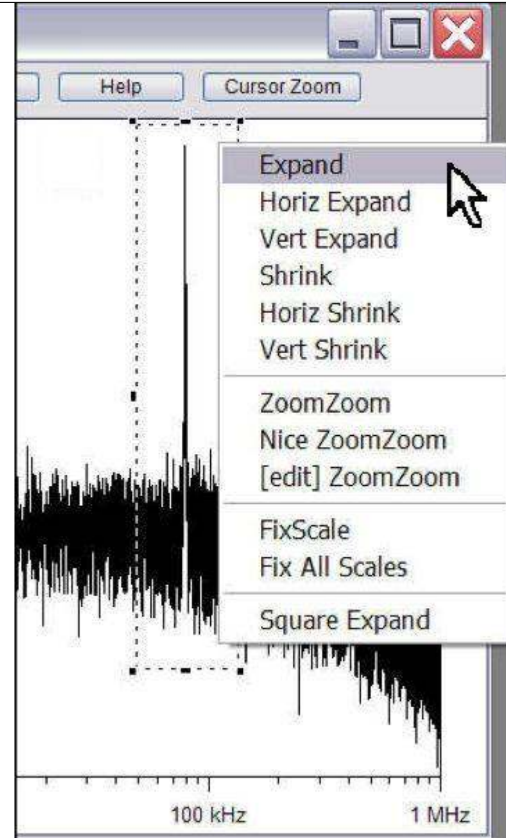
Note How fast the Thermal tune proceeds is dependent on the resolution value: 3 is very good, but slow; 7 is efficient but produces a noisier plot.

5.

Stop Thermal Capture

- Click the 'Stop Thermal Capture' button at top of the *Master Panel* ▷ *Thermal Tab* to cease sampling when plot looks like it has definition enough to fit a curve to it.
- Click and drag a box around the peak in the data and right clik and select 'Expand'.

Note To get the full graph back, type Ctrl+A.



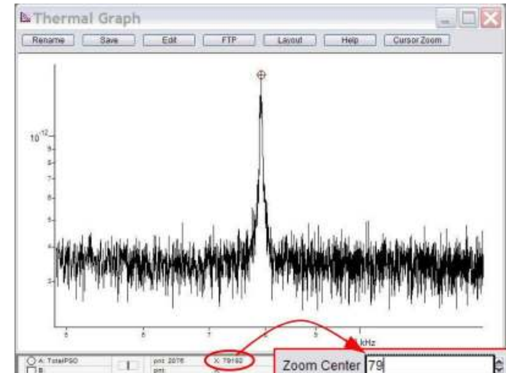
6. Hit Ctrl + I to get the crossahair cursors below the grapha and drag and drop one on the maximum of the first resonant peak.

7.

Zoom Center

- Type the value from the cursor (X value, which is in Hz) into 'Zoom Center' parameter (in kHz) on the *Master Panel* ▷ *Thermal Tab*.

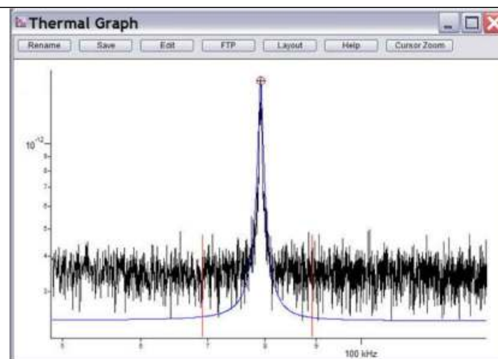
Note: If working in fluid, resonant peaks are much broader , and their resonant frequencies drop to about 1/2 of the "air" values.



8.

Initialize Fit

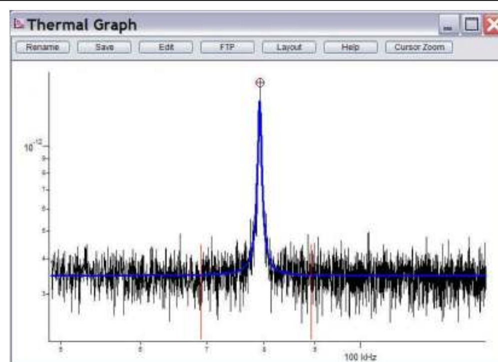
- In the *Master Panel* ▷ *Thermal Tab*, check the 'Show Fit ?' checkbox.
- Click the 'Fit Guess' button (this brings up a blue Gaussian shaped curve very near to the thermal resonant peak).



9.

Initialize Fit

- In the *Master Panel* ▷ *Thermal Tab* click the 'Fit Thermal Data' button.
- Observe a blue curve fit to this resonant peak .
- Note the updated spring constant and resonant frequency values in the *Master Panel* ▷ *Thermal Tab*.



At this point the software has determined the cantilever's spring constant by the equi-partition method. This is very useful when performing force distance curves, or needing to know how much force the Set Point is being applied by the tip. This value will be store in any saved data parameter file.

10.1.4. Tips and Tricks

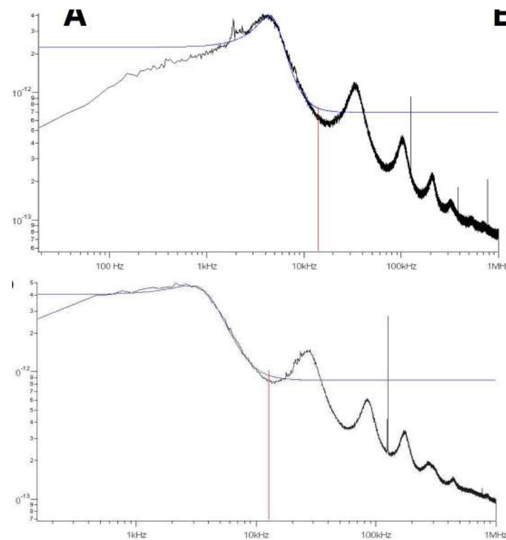
Tip Thermal fitting in fluids.

Sometimes when using a floppy cantilever in fluid, it can be difficult to determine the first resonance peak position. Two options for succes are:

- Take the frequency spectrum before engaging/ taking force curve; perform a try fit once you have the InvOLS value (there will be some greater error in k determination with this approach).

  -OR-

- Disengage the tip and manually retract of the tip a turn or two of the thumbwheel (20 µm to 40 µm) before taking the frequency spectrum. This seems to work much better, with a low number of iterations.



10.2. Sader method

The AR SPM 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.

10.3. Spring Constant Tutor

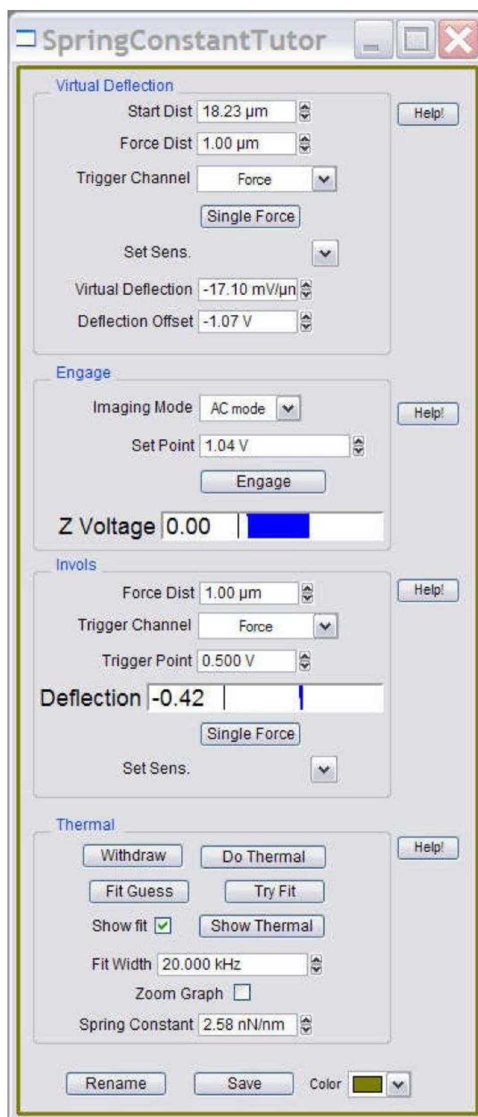


Figure 10.4.: Spring Constant Tutor

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

1. Go to Programming ▷ Start User Panel ▷ Spring Constant Tutor. This panel is set up where the progression of determining the spring constant goes from top to bottom.
2. With Spring Constant Tutor virtual deflection is intended to be determined when the tip doesn't engage the surface- the entire range of the piezo is fit.
3. Engage the tip; notice the active Z-piezo voltage meter.
4. 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.

5. Thermal tune: first withdraw the tip, then click 'Do Thermal'; bring up Igor cursors to fit the fundamental resonant peak to complete the spring constant determination.

11. MicroAngelo^{TM} Lithography and Manipulation software

CHAPTER REV. 951, DATED 07/10/2012, 17:51. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

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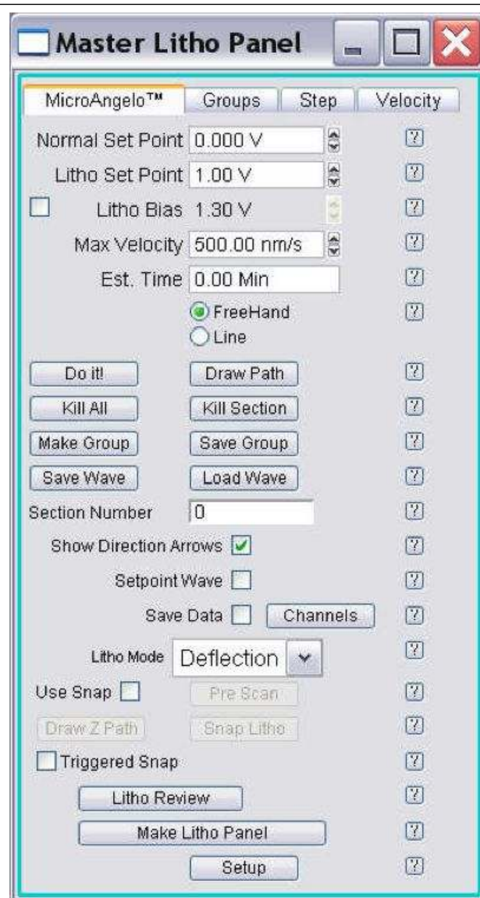
The MFP-3D closed loop system makes it ideal for accurate and complex scanning probe based lithography, and nanometer to micron scale tip manipulations. Typically, an area of the substrate is first imaged non-destructively (in AC mode or gentle contact) to determine what the 'real estate' looks like; then mouse strokes representing the tip's path(s) can be added to the image using the MicroAngelo software. Images can be imported and scaled appropriately, set point voltage (or tip bias) ranges defined for the execution, and the scripting performed.

These instructions assume that the MFP-3D is set up with desired tip and substrate, and is engaged. TEST

11.1. Introduction to Software Panel

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



The MicroAngelo tab allows the user to draw lines on the image representing tip paths, determine the set point during the manipulation and the set point between paths (kind of like a retraction set point); make groups of paths; define constant velocity during the path; determine mode (contact or AC); apply constant tip bias (if performing an oxidative or reductive manipulation; other features of this window will be described later in this procedure text.

The Groups tab allows the user to import an image, rescale a saved path (or group of paths) that you have previously drawn, or select between saved groups in memory. There are slider bars that allow X& Y rescaling preserving the aspect ratio; offsetting the group in X and/or Y; and group rotation.

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

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

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

2. Image the substrate to determine an area suitable for lithography or the manipulation. Depending on your substrate material or application, it may also be a good idea to have the

spring constant determined prior to the manipulation, so the user is aware of the amount of force applied at a given Set Point voltage.

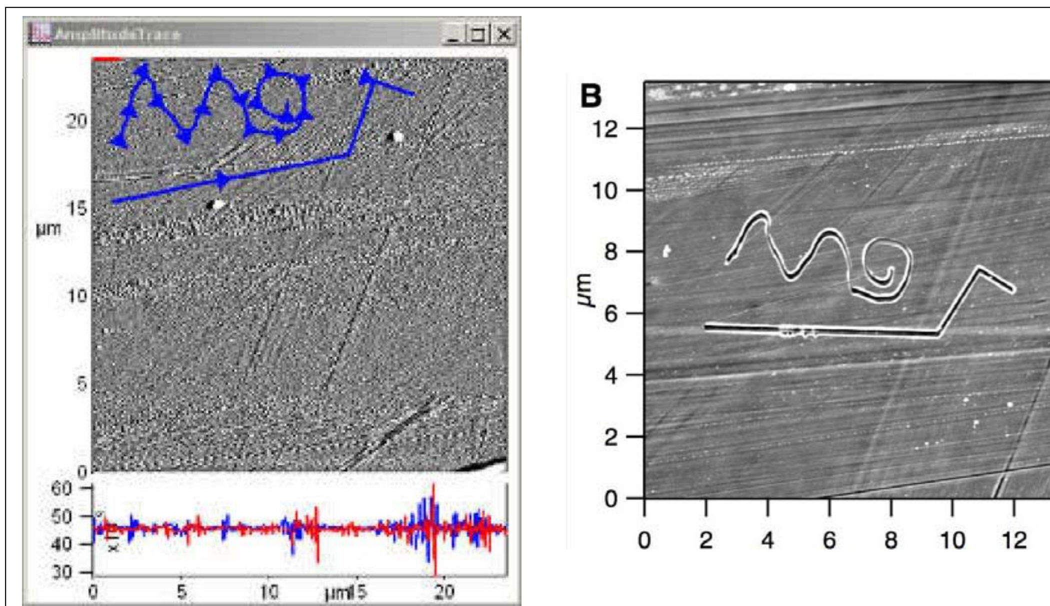
11.2. Drawing lines

11.2.1. Basic Operation

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

1. In the MicroAngelo tab, activate the FreeHand or Line radio button depending on what kind of line you want to draw.
2. Click on the 'Draw Path' button. Notice the button now reads 'Stop Draw', the free hand and line selections become faint because they can not be switched between each other, and a cross hair cursor will be present when the mouse is on one of the image channel windows.
3. Draw a path on the image window with mouse/cursor. If drawing more than one path (with the line tool or free hand tool), left click mouse button at each segment; double click mouse button when finished drawing line segment.
4. Paths can be grouped together: If you want to save the pattern for future use, Click 'Make Group', then 'Save Group', name it in the dialogue that shows up. Notice this name will now appear in the list in the Groups tab.
5. If you want to switch between the FreeHand & Line tools, click 'Stop Draw', switch to the other; then click 'Draw Path' again. Notice the drawn line turns from red to blue once clicking 'Stop Draw'.
6. For scratching based lithography or manipulations, switch the imaging mode to Contact in that Main tab imaging mode pull down menu of the Master panel, or in the 'Litho Mode' pull down in the Litho Panel.
7. Select a 'Normal Set Point' that will not damage the surface while the tip is moving between paths. In contact mode, a deflection value that is the same as the free air deflection (or more negative than that value) won't damage the surface between litho paths.
8. Determine/ select a 'Litho Set Point' value that will apply enough force to perform the desired lithography result (Scratch, oxidative/reductive, diffusion based direct write, etc.), or have enough force to perform the manipulation (without riding over what is intended to be pushed).
9. Click 'Do It!' to execute the litho event. Notice this button now reads 'Stop Litho'.
10. The tip will be withdrawn when completed with the lithography/ manipulation execution.
11. The 'Show Directions Arrow' checkbox will show the direction the path will traverse, and will become apparent on the image. To adjust the number of arrows on the path pattern, double click on the path and a dialogue comes up allowing you to reduce or increase the number of arrows, similar to how plots are customized in Igor.

12.



The figures above show a simple set of lines drawn with MicroAngelo: one was by free hand, one by line. The two paths were grouped by clicking the 'Make Group', then 'Save Group' buttons- a dialogue came up asking what the group is to be named. The Free Air deflection (from S&D meter) was -0.57V, so the 'Normal Set Point' was defined as -0.5V, while choosing the Litho Set Point as 0.5 V. The velocity was 600 nm/s.

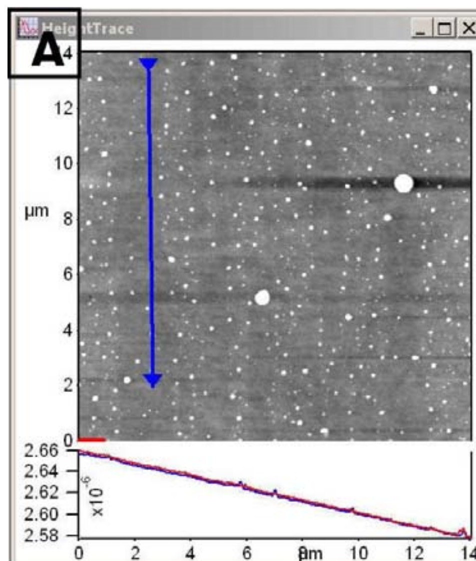
- Similarly, this same approach can be applied for sample manipulation via the tip.
- If you rescale the image/ scan area to view results, you will probably have to kill the section by clicking 'Kill All' button.

11.2.2. Using the Setpoint Wave Checkbox

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

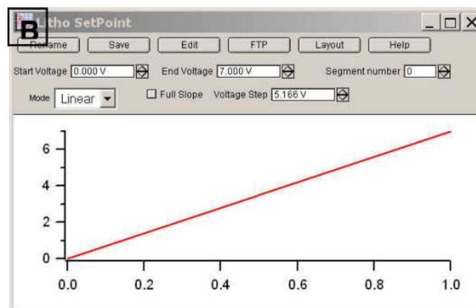
1. When Setpoint Wave is checked, a new dialogue is presented that allows Set Point voltage ranges to be applied along an individual line/path, in either a linear or staircase waveform. Note that is not able to distinguish between objects in a group.

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



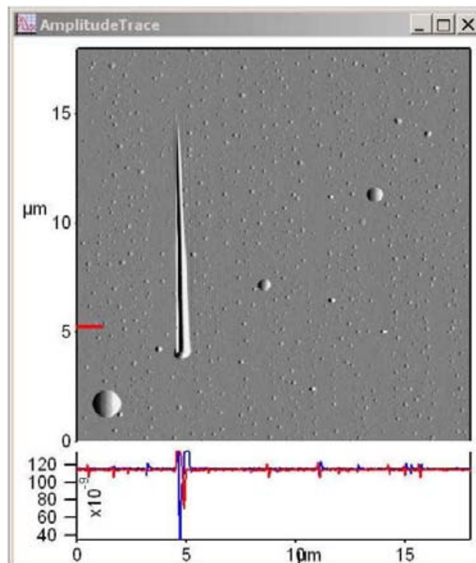
3. Check the 'Set Point Wave' checkbox , which brings up the Litho SetPoint window. The control parameters at the top of this window allow the user to define a start and ending Set Point voltage, and whether that ramp is stepped linearly, or as a step function.

- In this example, the Set Point range was from 0 V to 7 V with a linear ramp wave. The 'Voltage Step' setvar value is insignificant when using linear waveform ramps- it only applies when using step wave forms.



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

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

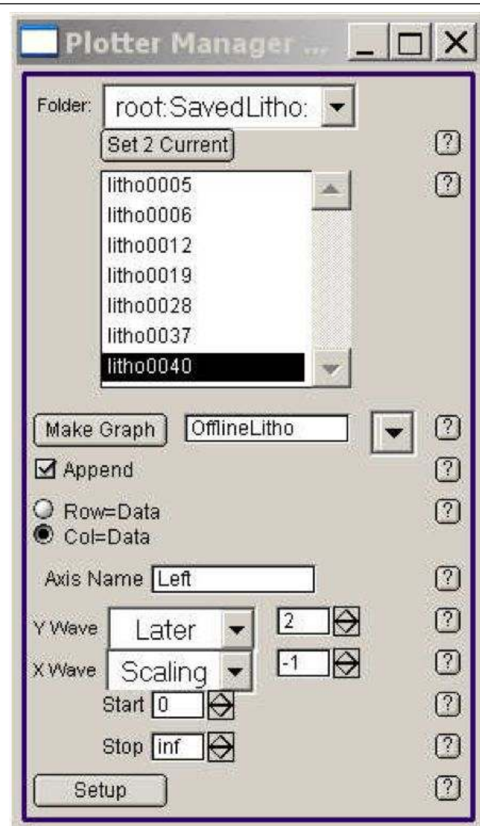


11.2.3. Save Data checkbox

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

1.

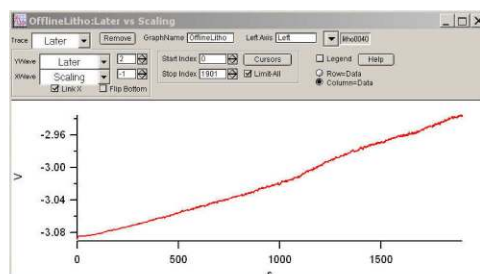
- To view the deflection, lateral and time signals during the manipulation event, click the 'Save Data' checkbox BEFORE the event (before clicking the 'Do It!' Button).
- After the event, click the 'Litho Review' button, which will bring up a panel similar to the one seen in the example. This panel shows a list of the litho events that were performed in Igor's memory.
- Choose the event you want to see the saved data for during the event.
- Choose X & Y wave desired from the respective pull down menus.



2.

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

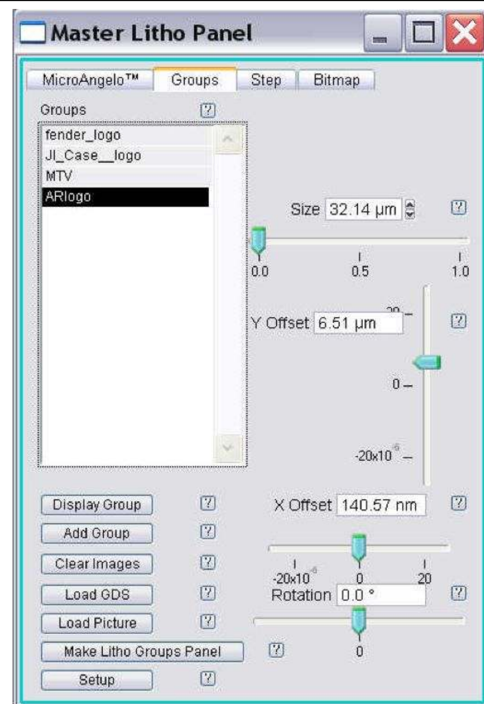
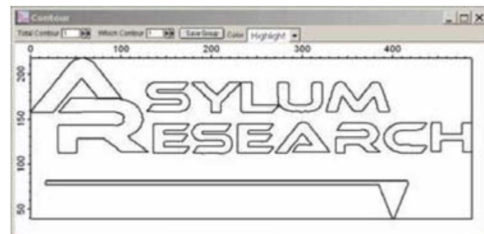
Note that the lateral signal during the manipulation is limited by the bandwidth of the ADC used for the lateral signal (~100 kHz). The bandwidth on the deflection signal uses the Fast ADC at 2.5 MHz.



11.3. Importing Images as Patterns

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

imported.

1.	• Open the Groups tab. • Click on the 'Load Image' button; a browse dialogue comes up letting you select the stored file.	
2.	• The image loads and Igor immediately makes a 'Contour' of the image. This figure shows the results of loading the AR logo • The contour image defines the edges of the imported image as the path the tip will traverse during the manipulation.	

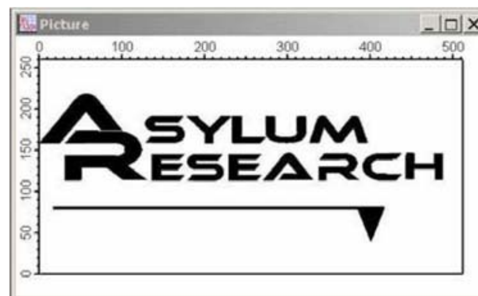
3.

- Notice at the top of the Contour window, there are some parameters you can adjust. These parameters are discussed further towards the end of this section. For now, this simple image will be contour value '1'. Contour effects are more pronounced in imported images that are grayscale or have color. It's best to try it on your own.

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

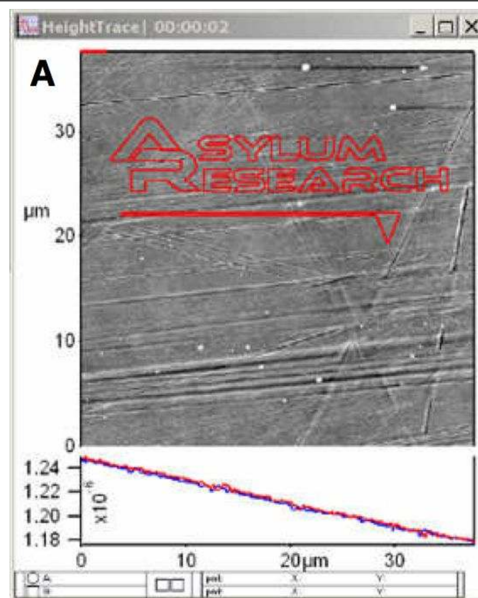
'Which contour' chooses which contours will be included in the group ultimately used as the path.

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

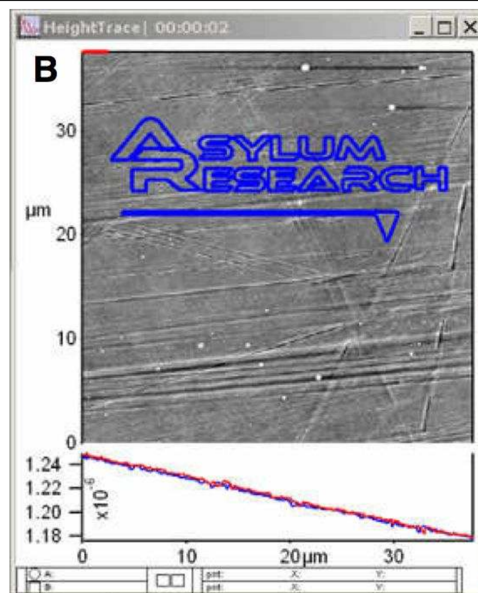


4.

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



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



6. Go back to the MicroAngelo^{tm} tab.

7. Define the 'Normal' & 'Litho Setpoint' voltages and tip velocity.
   - Choose a 'Normal Set Point' voltage value that doesn't damage the surface between paths.
   - Choose a 'Litho set point' value that will induce enough force to scratch the surface.
   - Choose a tip velocity that will be at a rate sufficient enough to do what is intended.

8. If the manipulation requires an oxidative or reductive potential click the 'Litho Bias' check-box and define an appropriate bias to the tip.

9. Make sure 'Contact' is selected from the pull-down menu (if that is the desired mode). Incidentally, if you change the imaging mode in the Main tab of the Master panel it will be updated here as well.

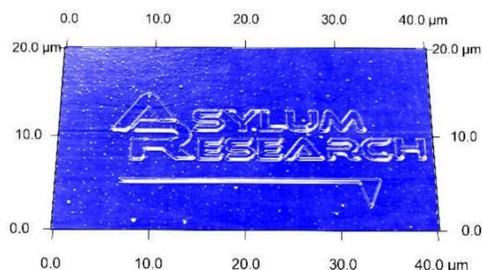
10. The 'Show Directions Arrow' checkbox will show the direction the path will traverse, and will become apparent on the image. To adjust the number of arrows on the path pattern, double click on the path and a dialogue comes up allowing you to reduce or increase the number of arrows, similar to how plots are customized in Igor.

11. When all parameters are defined, click the 'Do It!' Button;

12. During the manipulation, A red dot that represents the location of the tip, based on the values from the X,Y LVDTs is shown on the screen. You'll also notice the 'Draw Path' button now shows what section of the group path is traversing. If you click this button during the litho procedure, it won't do anything.

13.

- At the end of the patterning, the tip will be withdrawn.
- In the pattern example shown in step 5 above, an AC 160 Si cantilever was used to scratch a polycarbonate surface. The resulting scratch can be seen in this figure.



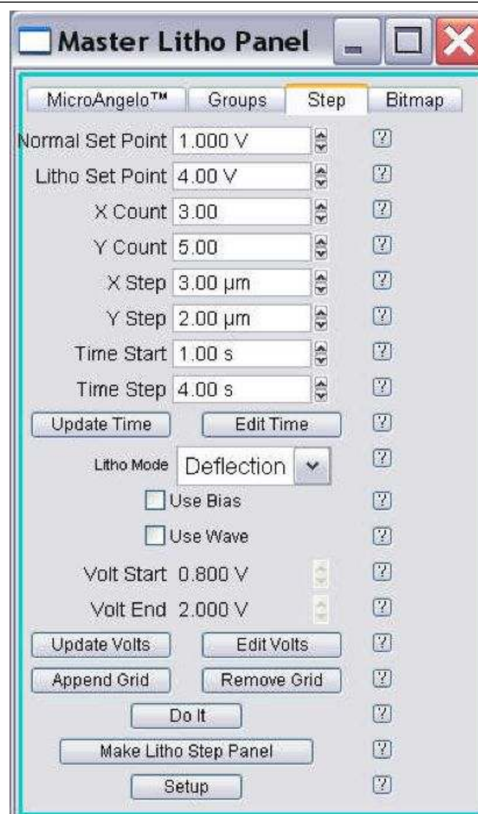
11.4. Creating Array Patterns: The Step Tab

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

11.4.1. Creating simple arrays

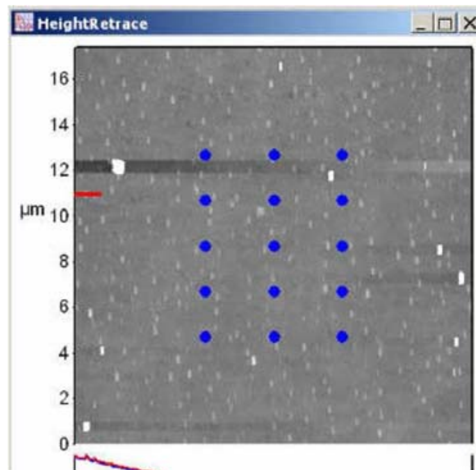
1.

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



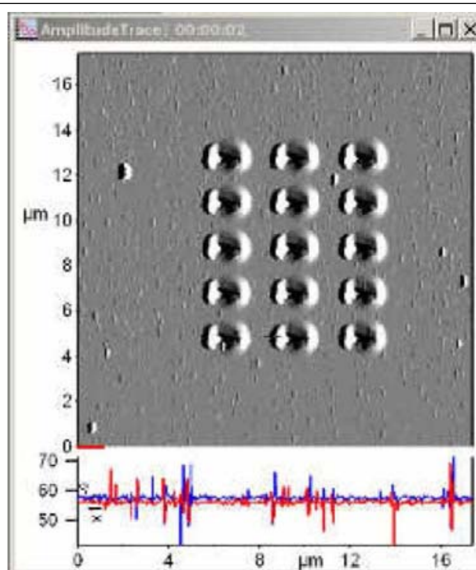
2.

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



3.

- The array will be centered in image window; the XY offsets in the group tab doesn't move the array to a user defined area in the image (like it does when using a pattern).
- In the example shown a 3 x 5 array was created using the parameter values shown in Step 1. Large forces were applied using a rather dull tip, resulting in the poor quality image below.



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

- To systematically vary the dwell time at each subsequent point in the array-

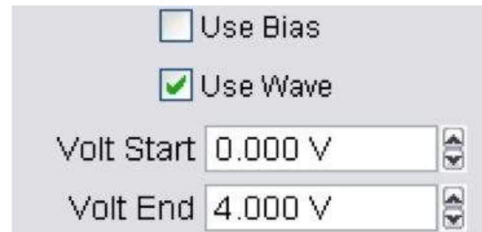
  1. Define a 'Start Time' value which represents the amount of time the tip dwells at point 1.
  2. Define a 'Time Step' value: this is the amount of additional time spent at each subsequent point. For example, if you want the tip to spend 3s longer at each subsequent point, enter 3s in 'Time Step'; (Start Time being 1s). Point 1 (lower left of the array) would spend 1s; point two would spend 4s; point three, 7s, etc. Make sure to click the 'Update Time' button to ensure this change takes effect.
  3. The individual time values at each point can be viewed in spreadsheet form by clicking the Edit Time button.

- To independently vary the dwell time at each subsequent point in the array-

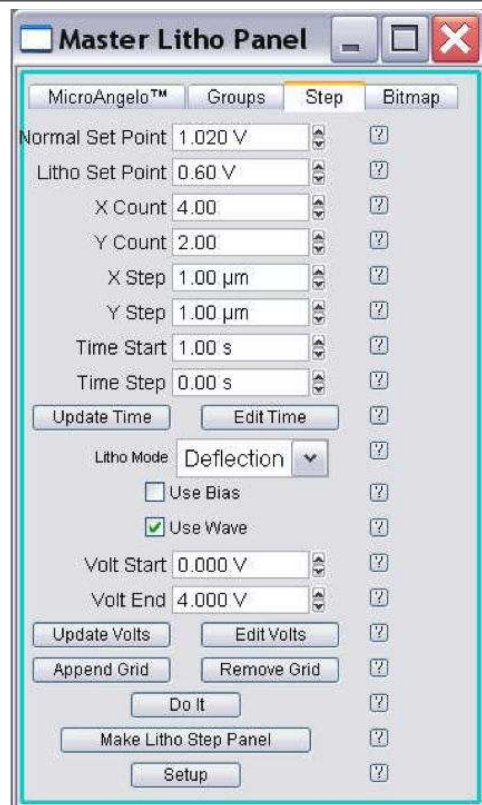
1. When a more custom variation is required, click the 'Edit Time' button. This will bring up a spreadsheet that allows the user to manually change the time at each respective point.

Varying Tip Set Point Voltage at each Array Point The amount of applied force the tip imparts to the surface the surface at each array point can be independently or systematically varied using the Steps tab. This can be helpful when doing a series of indents at different applied forces in an array.

1. Check the checkbox above the 'Volt Start' setvar input (this is actually called 'Use Voltage Wave). This will disregard the 'Litho Setpoint' input value in the MicroAngelo tab.

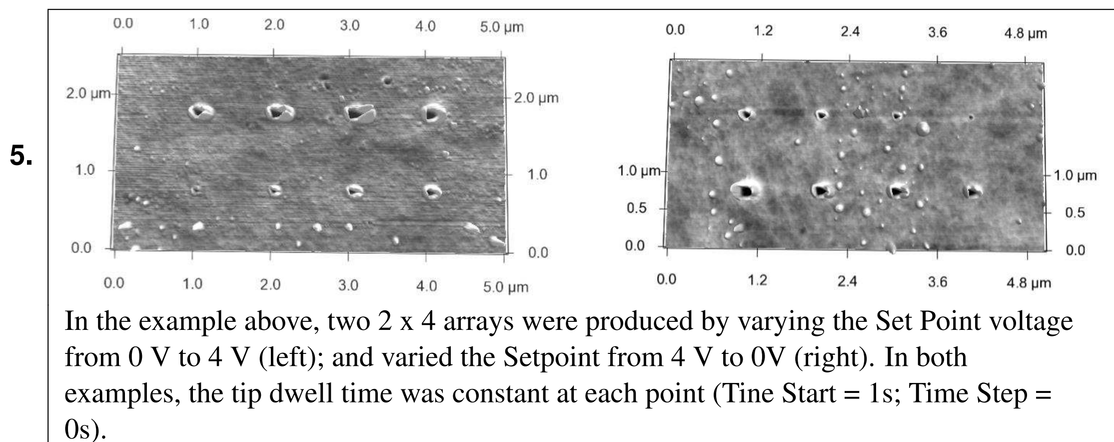


2. To systematically vary the applied force at each subsequent point in the array-
   - Define a starting Set Point voltage using 'Volt Start' setvar input.
   - Define an end Set Point voltage using 'Volt End' setvar input.
   - Click 'Update Volts' button to activate this change.



3. The individual Set Point setvar values at each point can be viewed in spreadsheet form by clicking the 'Edit Volts' button.
4. Click the 'Do It' button; the deflection at each array point can be monitored in the S&D meter.

5.



In the example above, two 2 x 4 arrays were produced by varying the Set Point voltage from 0 V to 4 V (left); and varied the Setpoint from 4 V to 0V (right). In both examples, the tip dwell time was constant at each point (Tine Start = 1s; Time Step = 0s).

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

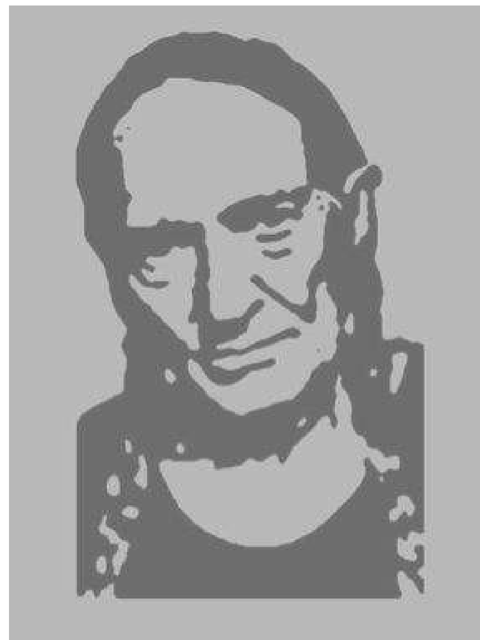
When a more custom Set Point variation is required- in this case, click the 'Edit Volts' button. This will bring up a spreadsheet that you can go into and manually change the Set Point at each respective point.

11.5. The Velocity Tab

It is best to read the help menu to learn more about the Velocity tab. Usually it is used to draw images that have shading in them. An image is loaded, the colors are converted to gray scale- dark grays are patterned using the 'Min velocity' while the bright grays are patterned at the 'Max Velocity'. All grays in between are patterned at a velocity determined by the grayscale value and the velocity range entered into the value inputs.

This feature is good for diffusion dependent direct write scanning probe lithography techniques, among others.

(a) Velocity Tab



(b) Example of image patterned through the Velocity tab.

12. Thermal Tuning

CHAPTER REV. 776, DATED 08/26/2011, 23:57. USER GUIDE REV. 1013, DATED 09/21/2012, 15:36.

Chapter Contents

A Thermal Tune is performed to determine the natural resonant frequency of the cantilever by monitoring the amplitude over a user defined frequency range. The MFP-3D uses the PD A/D converter to measure this, which can acquire frequencies up to 2.5 MHz.

Thermal Tunes are mostly used for a few specific tasks:

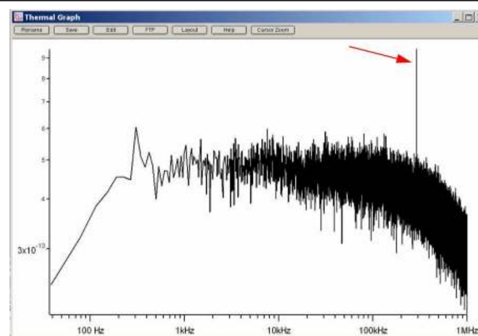
1. To determine the frequency of a cantilever for drive frequency selection for AC mode in air or fluid; OR higher resonance eigenmodes for DualAC imaging techniques.
2. As the second step in determining the spring constant.
3. Determining resonant frequency changes if the tip has picked up material (or has become broken).

Thermal Tuning a Cantilever

1. The Thermal tab is located in the Master Panel.

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

3. Click the “Do Thermal” button; 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.



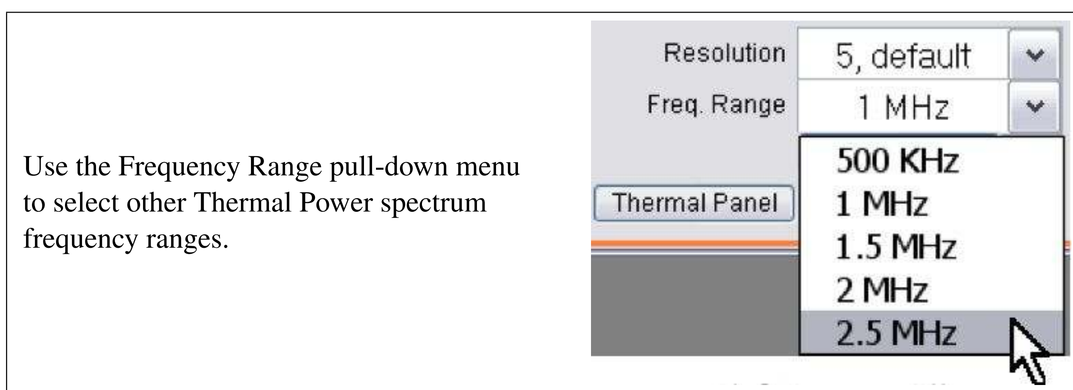
4. If the deflection is not zeroed, a dialogue will come up. Do not be alarmed: either click No and zero the PD, or if precision is not your top priority, click Yes and continue. The reason to zero them is that PDs have more linear response in their center relative to their center. It is typically recommended to zero the PD.

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 in the application. Notice the baseline is mostly noise, but the one sharp line around 300kHz is the AC160s resonant frequency (red arrow in Step 3 figure, above).

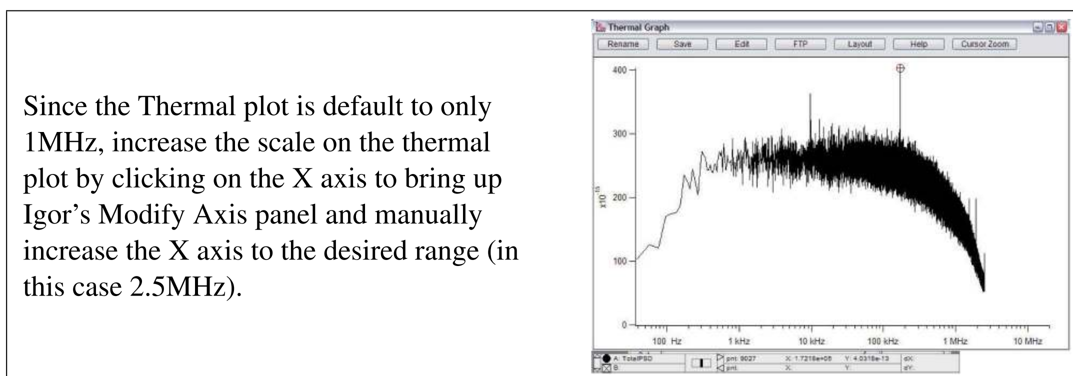
6. Click the Stop button to terminate the collection of power spectrums.

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 1MHz. 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.

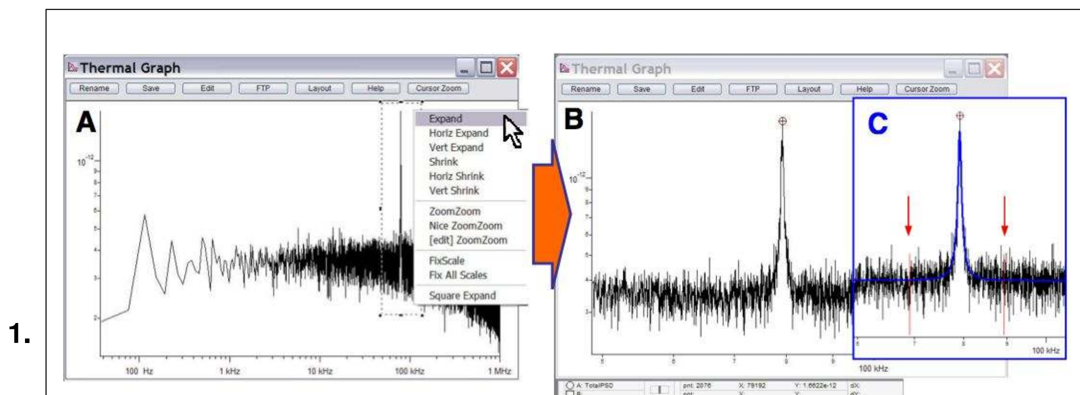


Since the Thermal plot is default to only 1MHz, increase the scale on the thermal plot by clicking on the X axis to bring up Igor's Modify Axis panel and manually increase the X axis to the desired range (in this case 2.5MHz).

Save Thermal tune plots by

- Clicking the Save button at the top of the thermal plot; saves as Igor graph (.pxp)
- From File ▷ Save ▷ Graph ▷ Copy in the Igor main menu; saves as Igor graph (.pxp)
- Sent to a Layout as a graphic.

1.



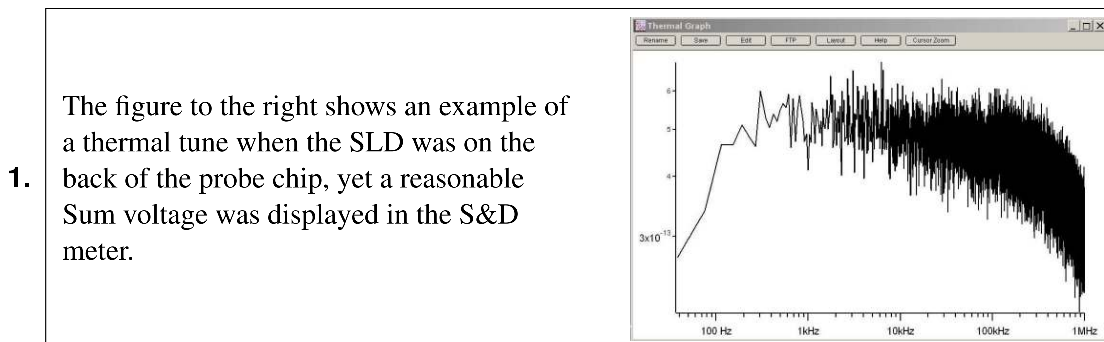
- Zoom into the area of the peak by right mouse clicking around the peak, then right or left click to see the menu (Full Spectrum thermal tune).
- Choose Expand; the result would look something like part B (Zoom of resonant peak).
- C shows the fit peak, red arrows show fit width cursors on power spectrum.

2. Next, click Ctrl + I to activate the Igor cursors.
3. Drag one of the cursors to the peak.
4. Type the X: value (~79 kHz is sufficient) in the Igor cursor tray into the Zoom Center parameter of the Thermal Tab. Note the units in the tray are in Hz, while the units in the parameter box are in kHz.
5. Click the Show Fit checkbox, a blue fit line should appear on the plot.
6. Click the Fit Guess button; a blue Fit function curve appears somewhere in the vicinity of the resonant peak. If it does not, confirm that the Zoom center setvar value is in a similar range as the resonant frequency peak.
7. Click the Try Fit button. The blue fit function curve should be fit to the resonant peak. Meanwhile the resonant frequency value and Q parameters in the Thermal Tab are updated.

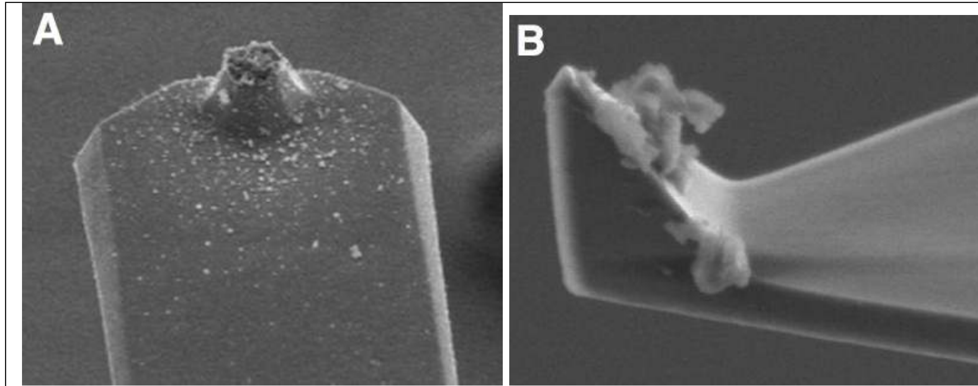
- Thermal tunes work very well for determining the resonant frequencies (and ultimately drive frequencies) of cantilevers in fluid.

The thermal tune is also a very convenient way to check whether the SLD spot is on the cantilever (when aligning SLD via IR card, sometimes a nice sum is given when the spot is on the probe substrate).

1. The figure to the right shows an example of a thermal tune when the SLD was on the back of the probe chip, yet a reasonable Sum voltage was displayed in the S&D meter.



2.



The figures above show two cases in which a thermal tune is useful at determining what happened when the resonant frequency changes throughout an imaging experiment. If the tip breaks (lowering mass, increasing f_o), or if there is an increase in mass on the cantilever/ tip from adhered debris (lowering f_o).

Part III

Bibliography, Glossary, and Index

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Glossary

Glossary Test: This is a test of the glossary feature