15. Conductive AFM

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15.1. parts list

To be completed at a future date.

15.2. Required items:

- An ORCA conductive AFM cantilever holder, such as 901.767 shown in Section 12.1.1 on page 141.
- The gas cell body (901.746) shown in Section 13.1 on page 148.
- The gas cell cable (448.137) shown in Section 13.1.2 on page 149.
- The Electrical Sample Puck Assembly (448.140) , included in the Environmental Scanner Accessory kit.
- An electrically conducting cantilever, such as the Asylum Electrilever.

15.3. Tutorial: Conductive AFM

1. This technique will work only with the Gas Cell Body, but with any sample stage. Please follow the tutorials on:
   - Sample stage and cell body assembly: Section 13.3 on page 158.
   - Mounting the sample stage onto the Environmental Scanner: Section 13.4 on page 164.
2. Install the gas cell cable. See on page 151.

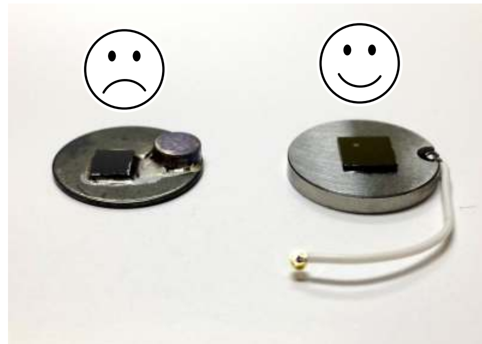
3. Prepare the cantilever holder and AFM. Follow the AC Mode Tutorial from Step 1 on page 131 THROUGH Step 17 on page 136 to prepare your AFM system and cantilever holder.

4. **Sample mounting:**

   - Mount your sample onto the Electrical Sample Puck Assembly (448.140) using silver paint or some other conductive adhesive.

5. **WARNING: Never load "non-flat" samples into the ES scanner.**

   - Unlike the S scanner, the ES scanner cannot accept "non-flat" samples.
   - *A "non-flat" sample is any sample in which the region to be scanned is not the tallest feature on the sample puck.* For example, the sample shown shown on the left has a magnet glued to the puck that is taller than the sample.
   - Given the low profile of the ES cantilever holders, it would not be possible to engage on the sample shown shown on the left since the magnet would hit the cantilever holder before the cantilever engaged on the sample.
   - For electrical measurements, use the low-profile electrical sample puck (as shown on the right) provided in the accessory kit.

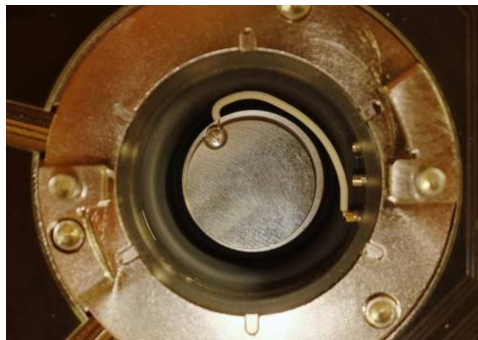


Warning: Pay attention! Attempting to starting a tip approach on a "non-flat" sample may cause serious damage to your cantilever holder and/or sample stage.

6. **Insert sample into the AFM:**

   - Remove the cantilever holder.
   - Insert the sample puck as shown.
   - Use blunt tipped tweezers to connect the wire as shown., to the front connection. This applies a sample bias.

   Note Lower the cell body to get more sample access: see Step 17 on page 136.



7. Continue the AC mode tutorial from Step 20 on page 137.
8. Once imaging, refer to *Applications Guide, Chapter: Conductive AFM*.

15.4. Testing the ORCA Amplifier

To be completed at a future date.

9. Conductive AFM (ORCA)

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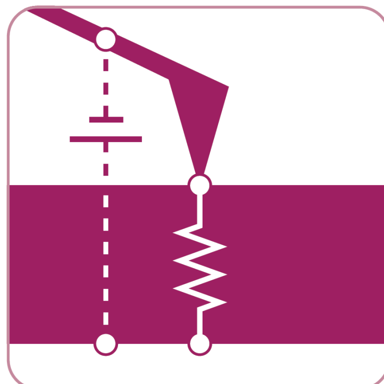

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9.1. Introduction

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

A schematic of CAFM is shown in 9.1.

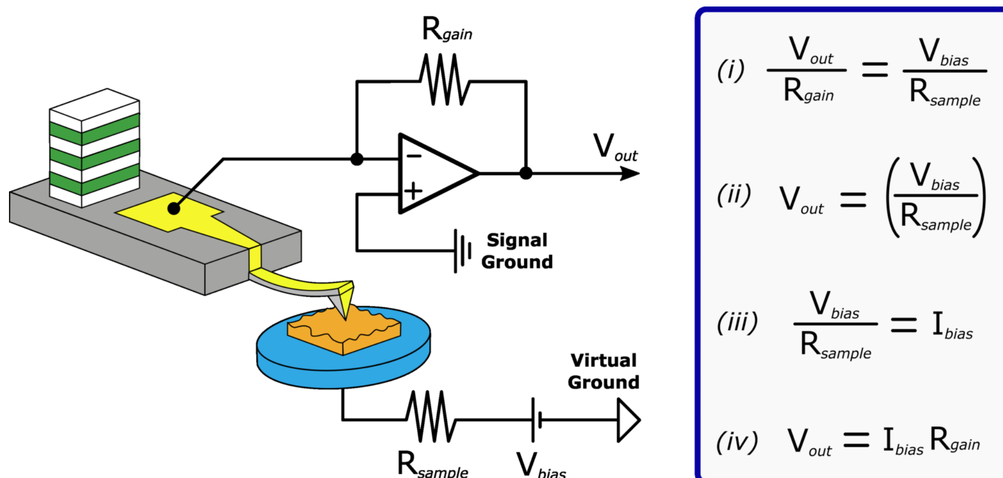


Figure 9.1.: Schematic of CAFM with relevant equations. Current is passed through the tip and into a transimpedance amplifier, converting the current to a voltage.

9.2. Video Tutorial

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

9.2.1. Terminology

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

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

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

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

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

9.2.1.1. Spectroscopy vs. Imaging

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

9.3. The Hardware

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

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

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

9.4. Probes

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

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

9.5. Repeatability in CAFM

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

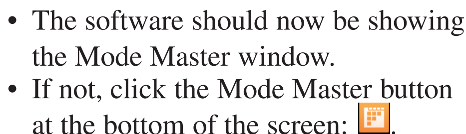
9.6. Current Imaging

9.6.1. How to Guide

1.

The Mode Master window:

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



2.

Select Mode:

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

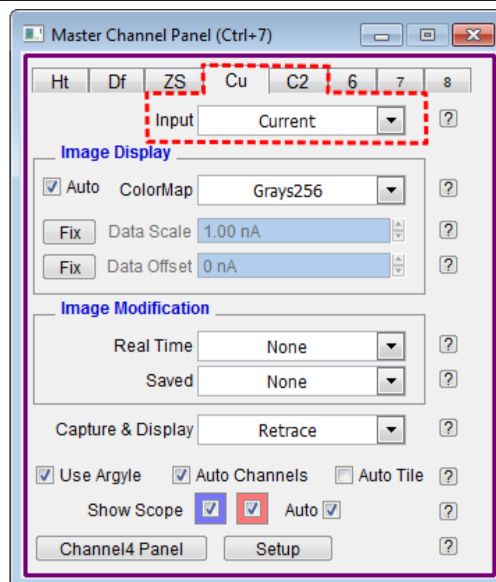
Note The ORCA cantilever holder must be first attached to the AFM to load the ORCA template.

3. Load a conductive cantilever and sample as standard for Contact Mode imaging. This includes any necessary bias wires and/or resistors. See appropriate manual for set up instructions.
4. Set the AFM to operate in **Contact mode** .
5. Set *Scan Angle* to 90º.

6.

Open the Channels

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

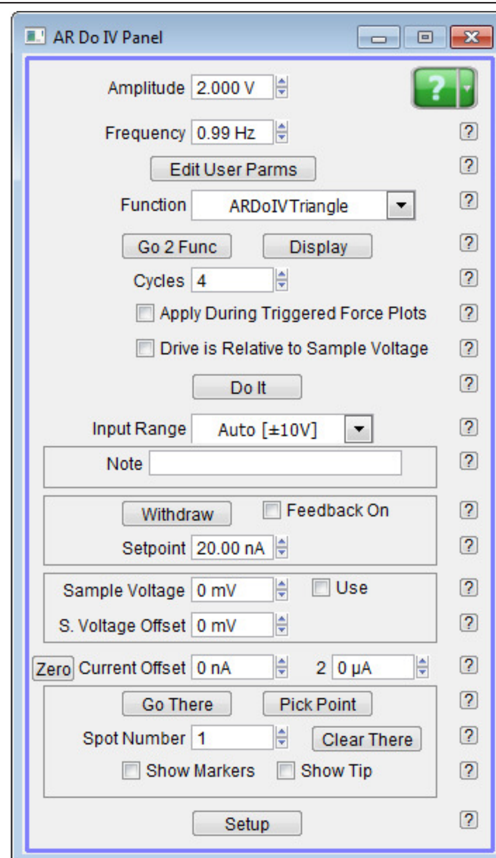


7. Select **Current** as one of the channel *Inputs* under the Master Channel Panel (*AFM Controls* ▷ *Master Channel Panel (Ctrl+7)*). For the Dual-gain ORCA holder you will have two channels instead, **Current 1** and **Current 2**.

8.

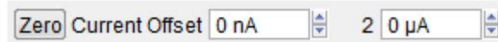
Do IV Panel

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



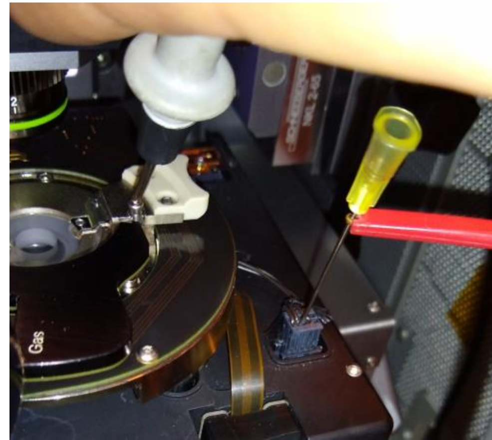
9. **Current Offset**

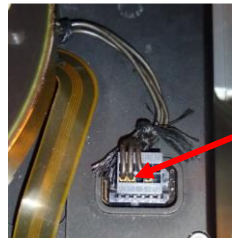
- There are often small, persistent current offsets associated with the AFM electronics that must be subtracted. Zero the *Current Offset* by pressing the Zero button in the **AR Do IV Panel**.



10. **Applying Voltage**

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





Measure pin on bottom row second from left.

Black probe on metal portion of cantilever holder.

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

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

9.7. Current-Voltage (I-V) Spectroscopy

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

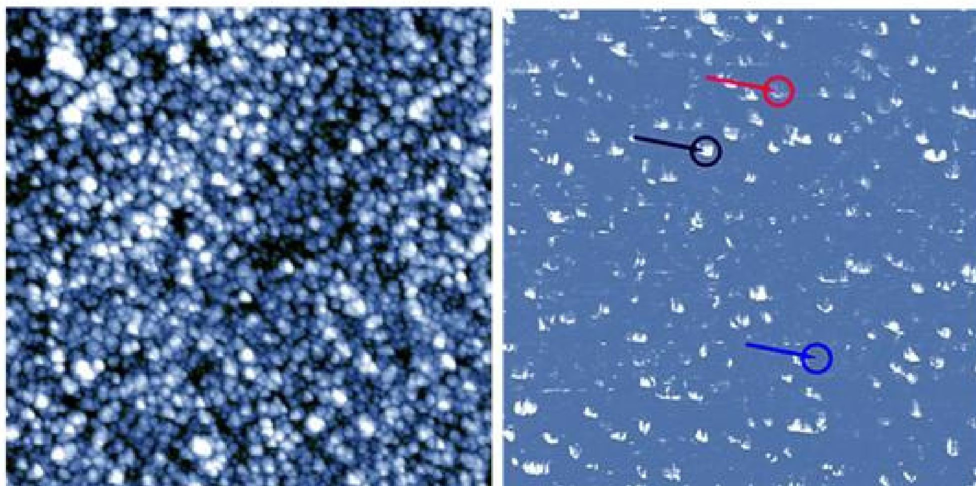


Figure 9.2.: This image is fairly typical of a thin film CAFM image. Note that tip shape artifacts are visible on the current image (left), but not on the topgraphic image (right). As the metal coating on the probe tip wears, or the tip is contaminated, these artifacts will be more prominent. Rocky Mountain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

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

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

9.7.1. How to Guide

1.

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

2. Before attempting to do I-V curves on a sample, it is also typically recommended that you first run I-V curves on the included test resistor to confirm everything is working properly, see[1].

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

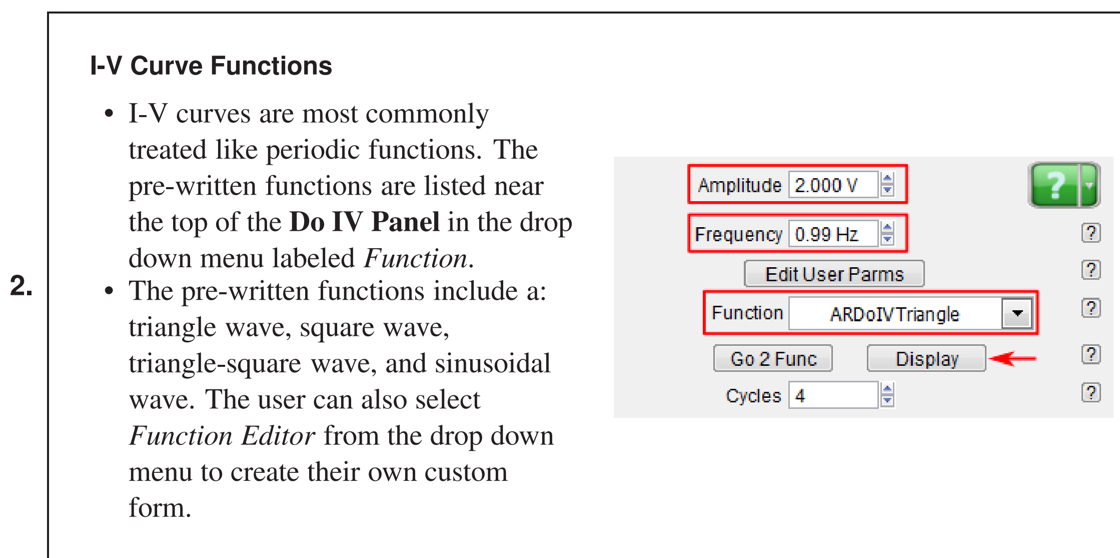
9.7.1.1. Setting up the I-V Curve

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

2.

I-V Curve Functions

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

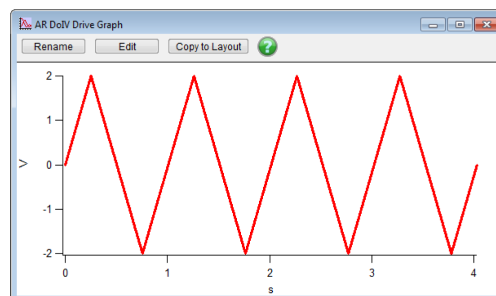


[1] *MFP-3D User Guide, Chapter: Conductive AFM (ORCA) Hardware.*.

3.

Function Controls

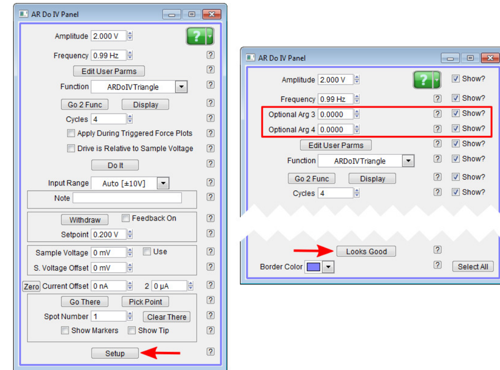
- The most convenient way to design your I-V curve is to display that function on a graph before you execute it. To do this, click the *Display* button below the *Function* drop down menu. This will bring up a graph of the voltage that will be applied to the sample as a function of time.
- You can adjust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



4.

More Function Controls

- If you need to adjust other parameters, such as including a DC offset in the curve or shifting the phase so that the bias is ramped from positive to negative or so that the ramp is only half of a curve, these can be adjusted using the parameters *Optional Arg 3* and *Optional Arg 4*.
- These parameters are hidden by default, but can be revealed by clicking on the *Setup* button at the bottom of the **Do IV Panel**. The "Arg" stands for "argument" and is will augment different things for the different pre-written functions.
- *Optional Arg 3*, generally speaking, controls the function phase. Phase is a ratio, where 1 is a 360° phase shift, 0.5 is a 180°, etc.
- *Optional Arg 4*, generally speaking, controls the offset. The offset is in units of Volts. Typing in 1 will produce an offset of 1 Volt, etc.



5.

Function Averaging

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

Cycles 4 ?
Average ?
Apply During Triggered Force Plots ?

6.

Function Frequencies

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

Points per Sec 2.000 kHz ?
Low Pass Filter 1.000 kHz ?
Input Range Auto [±10V] ?

7.

Function Filtering

- The filter placed on the analog-to-digital converter (ADC) that collects the current signal from the ORCA cantilever holder can also be controlled. To do this, change the value in the paramter labeled *Low Pass Filter* in the **Do IV Panel**. Values from 50 Hz to 25kHz are possible.
- For I-V curves, the system uses the 5MHz ADC in the system.
- For normal ORCA imaging, one of the 50kHz ADCs is used.
- **Note:** you can set the digital filter to as low as needed, however below 50Hz artifacts may appear. Ensure your filter value is set to at least twice the frequency of your function wave. More complex wave forms (non-sinusoidal) require much higher bandwidths to drive and acquire.

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

9.7.1.2. Picking Points for the I-V Curve

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

2.

Setting Points

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

Once you have set all the points you want to take I-V curves at, select the spot number you want your first I-V curve using the *Spot Number* field. Then click the *Go There* button to move the scanner so that point is under the tip. If you would like to see that the tip is there, check the box that reads Show Tip Location . This will put a red dot at the present location of the tip.

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

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

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

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

9.7.2. Dithering

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

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

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

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

The amplitude is the size of the spiral that will be traced with the tip. Cycles is the number of times the tip will turn within the circle to be dithered. The fraction is the ratio of how many turns the tip will take on the way out before spiraling back in to do the IV curve.

9.7.3. Custom drive waves using the Function Generator

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

To begin, select the function editor as the function on the DoIV panel. This will bring up a Function Editor window with a graph and some controls at the top. A default segment will appear. It can be modified using the controls at the top of the graph. The time for the segment, the start and end

amplitude, and modulation can all be added. The static controls drive the overall shape of the wave, and the dynamic can be used to add an AC dither to the segment.

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

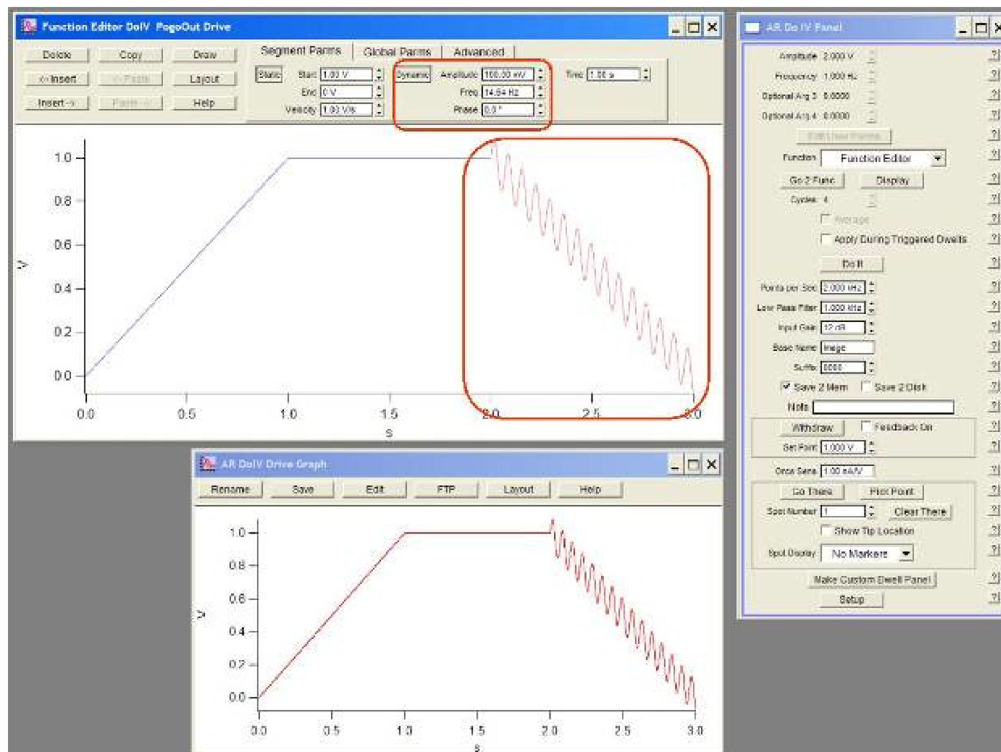


Figure 9.4.: [The Function Editor] The function editor can be used to make a variety of drive waves for current measurements. Insert new segments using the Insert button. Select different segments by clicking them. Each can be modified independently.

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

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

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

9.7.4. Viewing I-V curves

I-V curves are treated like force curves for viewing. The actual data is treated as a dwell on a force curve. See force curve review panel.

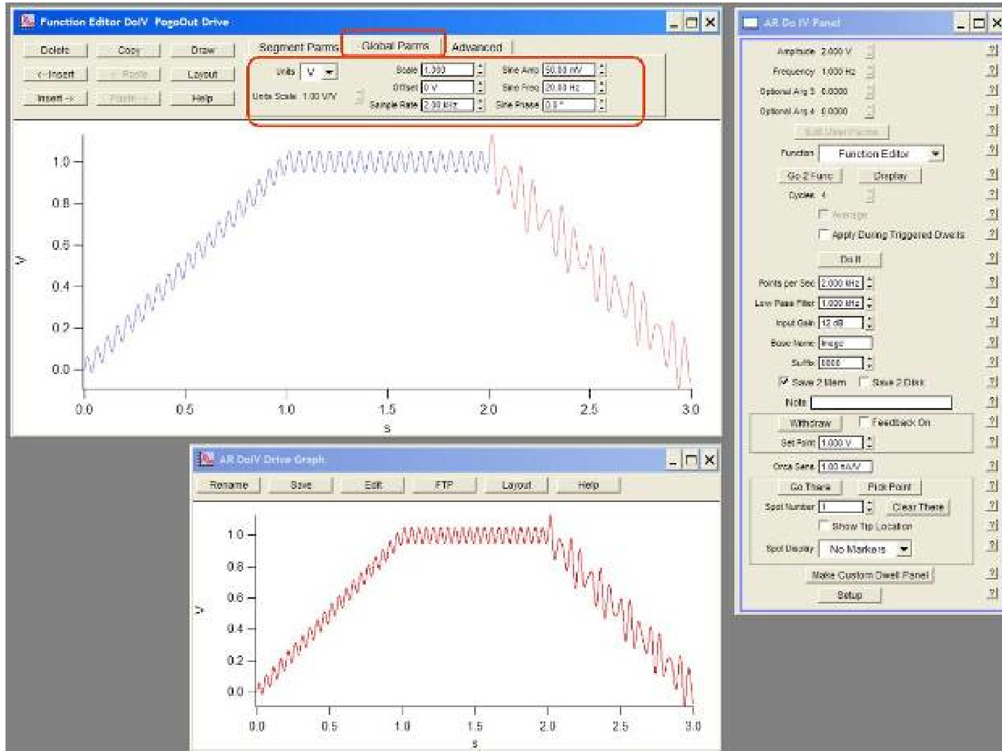


Figure 9.5.: [The Function Editor 2] Use the Global tab to modify all the individual segments at one time. Of particular use in this section is the Scale field. This allows you to scale up or down the entire wave function you have created. So if you had a custom wave from 0-1V with a dwel, scaling it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

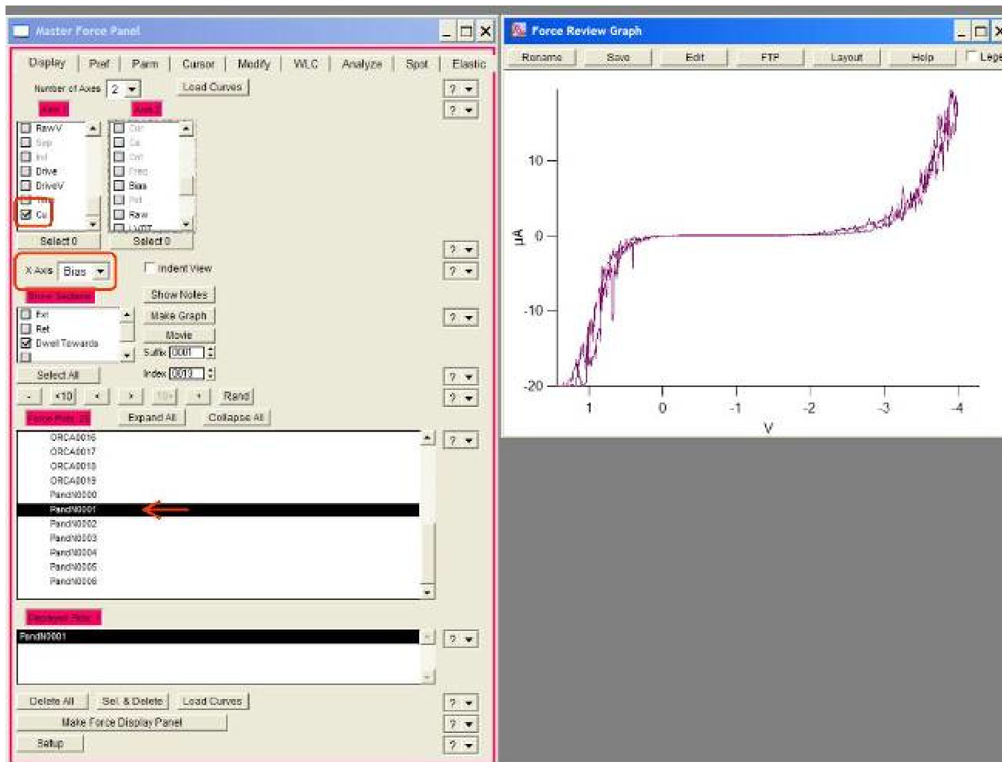


Figure 9.6.: [Current vs. Bias] To display IV curves, open the force review panel, view current (and current2 if you have a Dual Gain ORCA), Then set the x axis as bias, then select the dwell towards under the sections window. Click 'make graph' if the graph does not appear.

It is often useful to display the current and bias data as a function of time, especially for multiple curves taken consecutively, and those taken at frequencies higher than 20 Hz. To do this, under Axis 1 choose 'current' as the data type and under Axis 2 choose 'bias' (see Axes Data Types). Then choose time as the X axis. (see Force X axis).

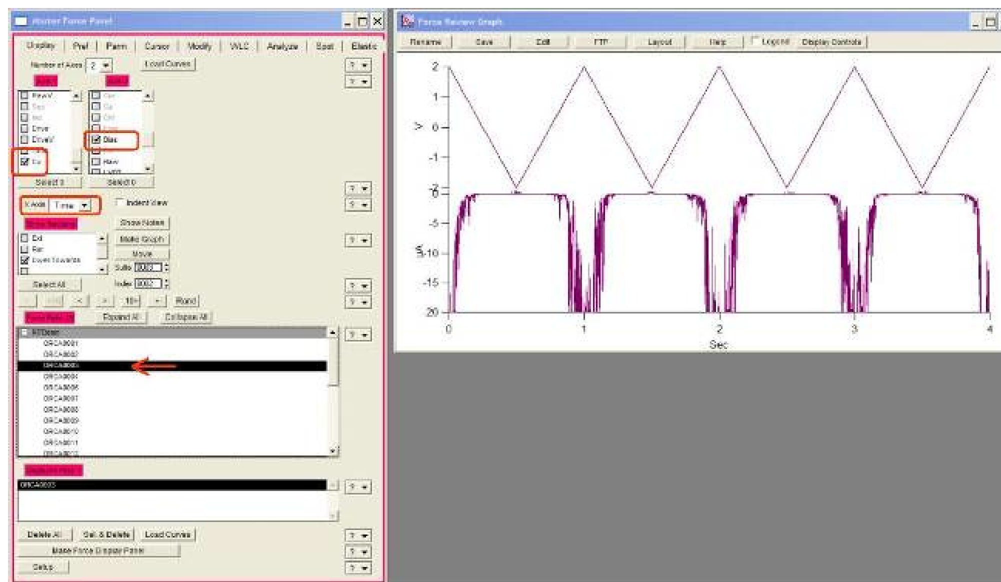


Figure 9.7.: [Current vs time] It is often useful to display the current vs time, particularly for samples that degrade or improve in conductivity with time. Si will oxidize as more current is passed through it if there is moist air near the surface.

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

9.7.5. Correcting Offsets on I-V curves

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

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

Then click on the *zero* button, which will adjust the current offset to get zero current when there is no bias on the tip.

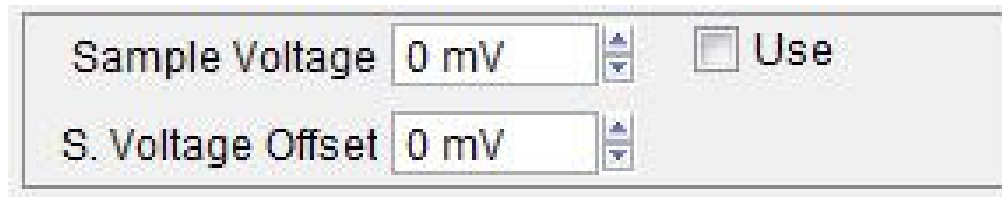


Figure 9.8.: [Current offsets]Bias and current offsets from the DoIV panel. These can be used to correct for small variations in the outputs of our DACs and the transimpedance current to voltage converter we use to measure current passing through the sample.