Asylum AFM: User Guide

At its core, the Asylum AFM will provide topographic information about a sample's surface. However, there are advanced capabilities of the tool that can also provide information about the local mechanical, electrical, and thermal properties of a sample. **Each technique requires a separate training.**

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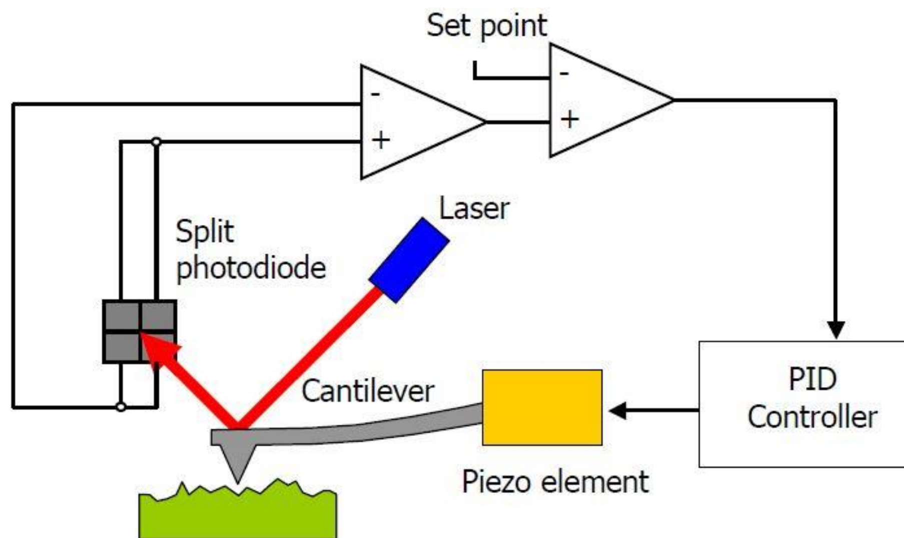
With contributions from Ricardo Iglesias, Stephanie Bohaichuk. Some sections are based on the Asylum Research Applications Guide (see https://courses.physics.illinois.edu/phys598BP/sp2014/Files/ARApplicationsGuide.pdf).

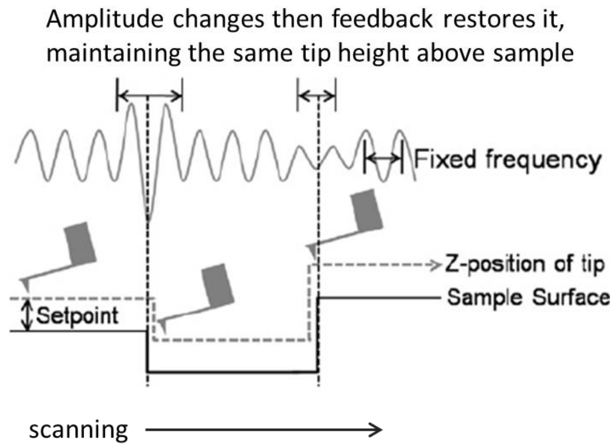
Topography (tapping and contact modes)

Principles of Technique

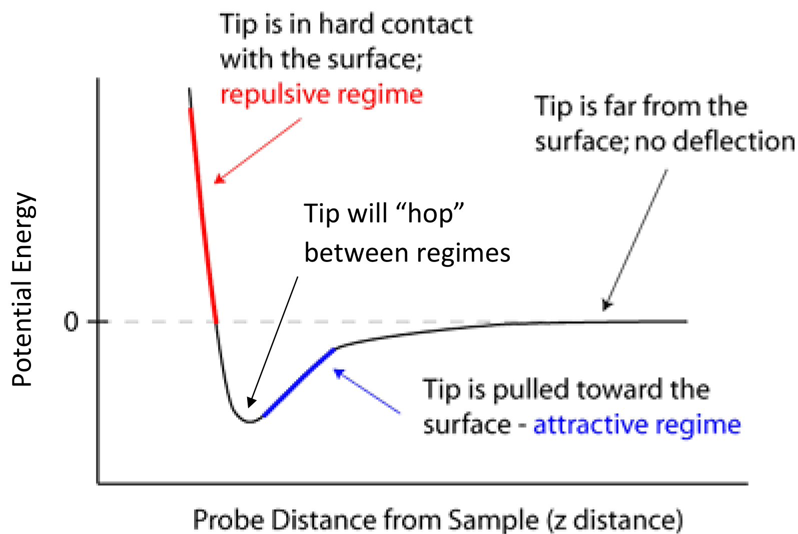
Atomic force microscopy gives information about the surface topography of a sample. It functions by moving a sharp tip at the end of a cantilever across the sample. A laser spot is reflected off the backside of the cantilever onto a photodiode. As the tip goes over features the cantilever will be deflected, altering the vertical location of the reflected spot on the detector, which can be converted to a height. Heights can be measured down to sub-nanometer and technically up to 15 µm. However, the system is really meant for nanoscale measurements and if you have features with heights above ~100 nm then it is recommended trying profilometry first.



There are two main modes for getting sample topography: tapping (or AC or noncontact) mode, and contact mode. In tapping mode, you oscillate the cantilever at its resonance frequency using a piezoelectric driver. The maximum amplitude of oscillation at the tip occurs when there is a 90° phase shift between the piezoelectric driver and the tip oscillation. When the tip comes near some topography the oscillation is damped, changing the amplitude and phase, which are measured by the motion of the reflected laser spot on the photodetector. There is a feedback loop that adjusts the tip's vertical position while scanning to keep the oscillation at a constant amplitude, and therefore keep the tip's height above the surface constant. By measuring the vertical distance that the piezo must move to do this, a height image is generated. This is referred to as amplitude modulation and is the typical mode of operation (as opposed to frequency modulation where the amplitude is fixed and the frequency changes).



Tapping mode can be done in two different regimes, attractive and repulsive. The force that the tip experiences depends on the distance from the surface, and it can track topography either by being repelled from the surface (if the tip is close) or attracted to the surface (if the tip is far away). If the phase signal is >90° then the scan is being done in the attractive regime, and if <90° then it is in the repulsive regime. The repulsive regime will generally give better images with higher resolution. The attractive regime can be gentler on both the tip and soft samples because the tip interacts less with the sample (tapping it less hard). Using a higher oscillation amplitude or a lower setpoint (moving towards the surface) will shift towards the repulsive regime.



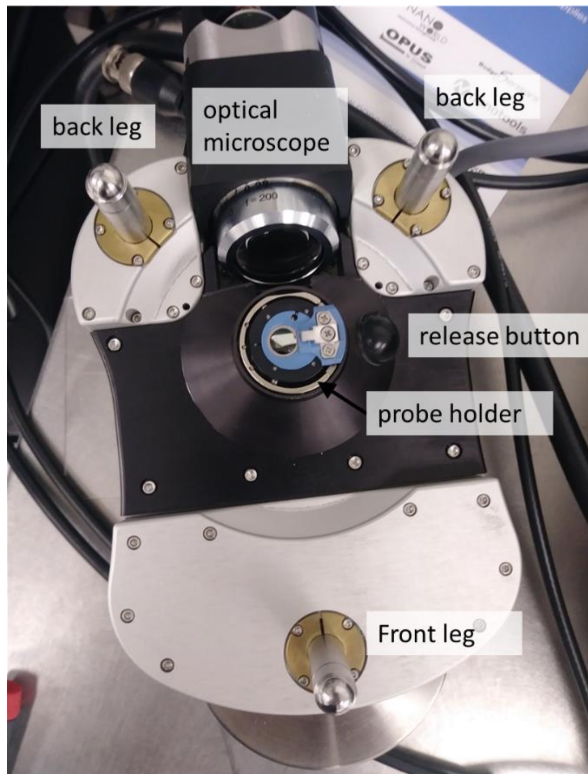
Sample topography can also be obtained in contact mode, where the tip will be in full contact with the surface instead of intermittent. In this case the tip is not oscillating, and the actual tip deflection is measured rather than a change in oscillation amplitude. Scans are done at a constant force (a constant laser deflection), with a feedback loop to hold it constant. By measuring the vertical distance that the piezo must move to maintain this, a height image is generated. Being in constant contact is harsher on softer samples, and generally requires a more flexible cantilever (with lower spring constant k and lower resonant frequency).

Basic Tool Operation (Tapping Mode)

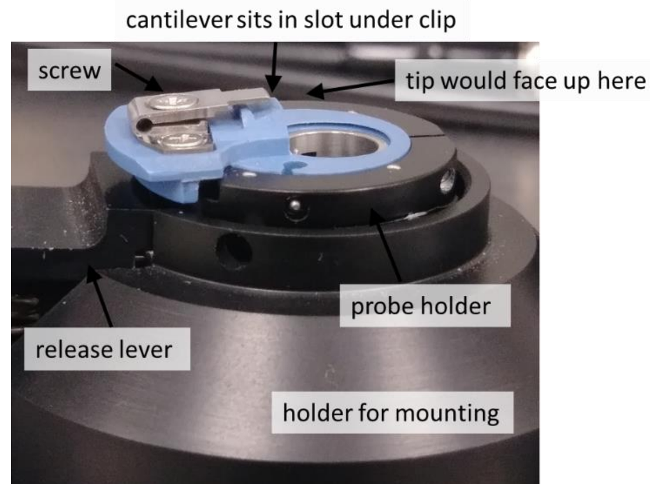
A video tutorial for how to use the AFM is here and a tutorial for tapping mode is here.

1. Mounting the AFM Probe

a. Ensure that the head is raised up, and if not raise it by turning the large front wheel to the left. Lift the AFM head up with two hands and place it with legs facing up on a table/surface. If the grey cable at the back of the head is getting twisted then flip the head over a few times to untwist.
b. If the probe holder is on the head, then press the button on the head next to the probe holder to release it. While holding this button down, slide the probe holder up and off the two ball bearings.



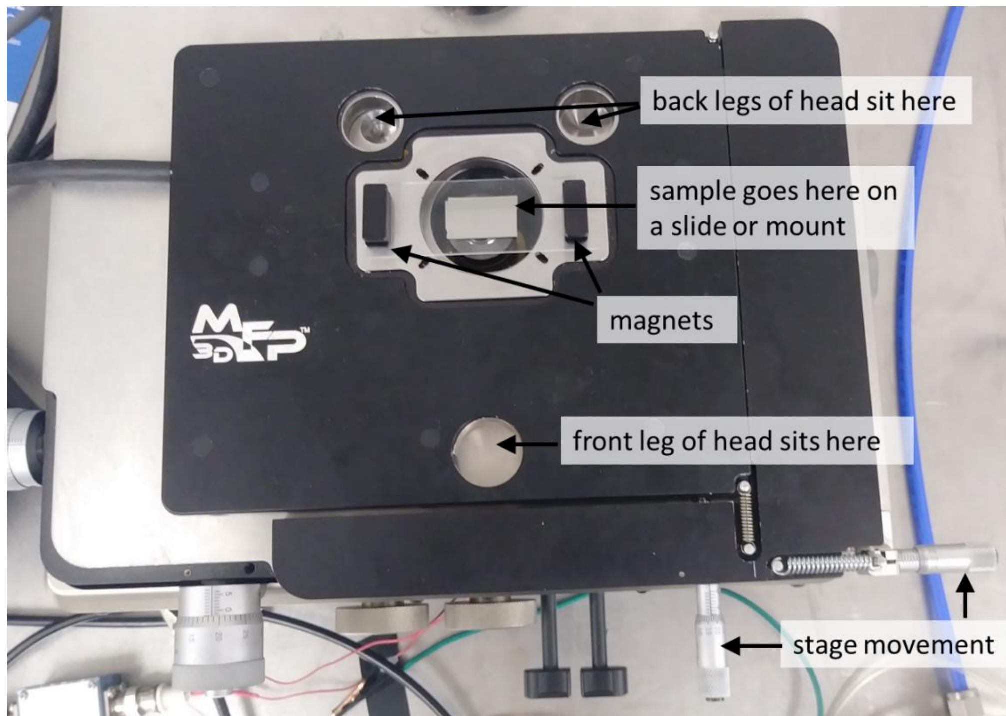
c. Place the probe holder on the black holder for mounting. Hold down the lever and insert the probe holder, with the two ball bearings sliding into the grooves in front. Release the lever to lock in place.
d. Use a Phillips screwdriver to unscrew the center screw slightly.
e. Carefully insert a cantilever under the raised clip. Look from the side to make sure that the cantilever sits in the slanted slot with the base all the way back against the end, not on top of the ridge.



f. Once the tip is in place tighten the screw so that it is finger tight, clamping down the cantilever. Remove the probe holder from the mounting holder and return to the head.

2. **Placing Sample and AFM Head**

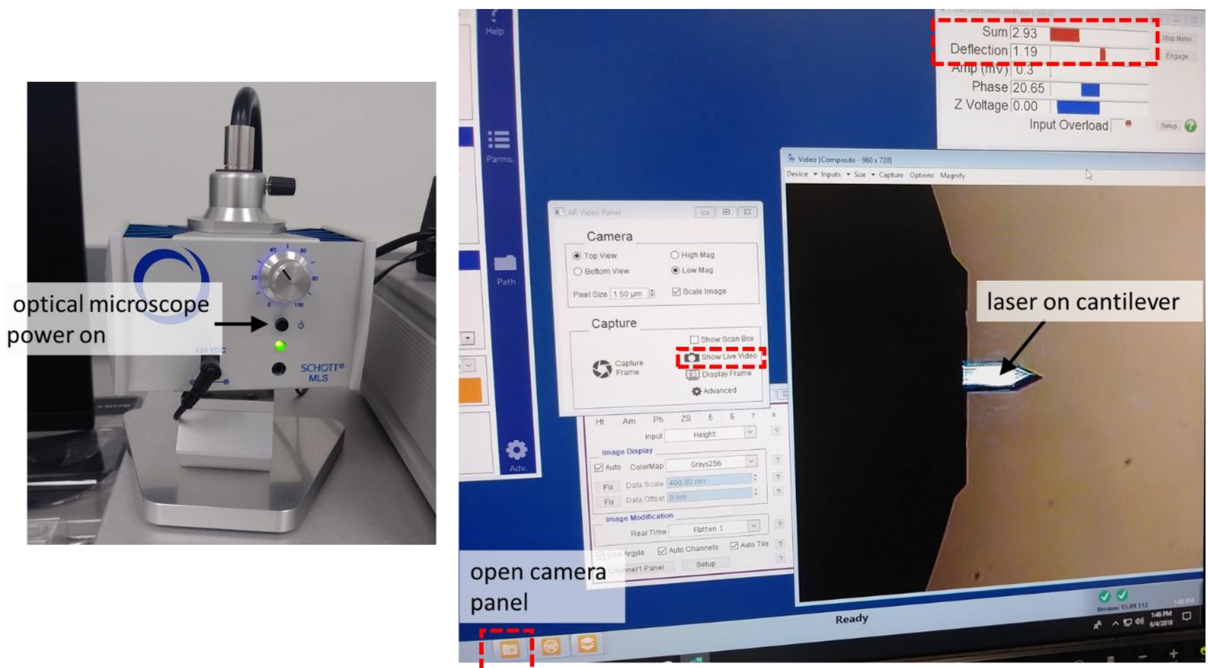
a. Load your sample. If your sample is small, fix it onto a glass slide using epoxy, Kapton tape, double-sided tape, etc. Put the glass slide onto the stage over top of the central hole and hold it in place using the magnets. If your sample is large enough to cover the hole then you can simply put it on the stage with the area of interest over the hole.



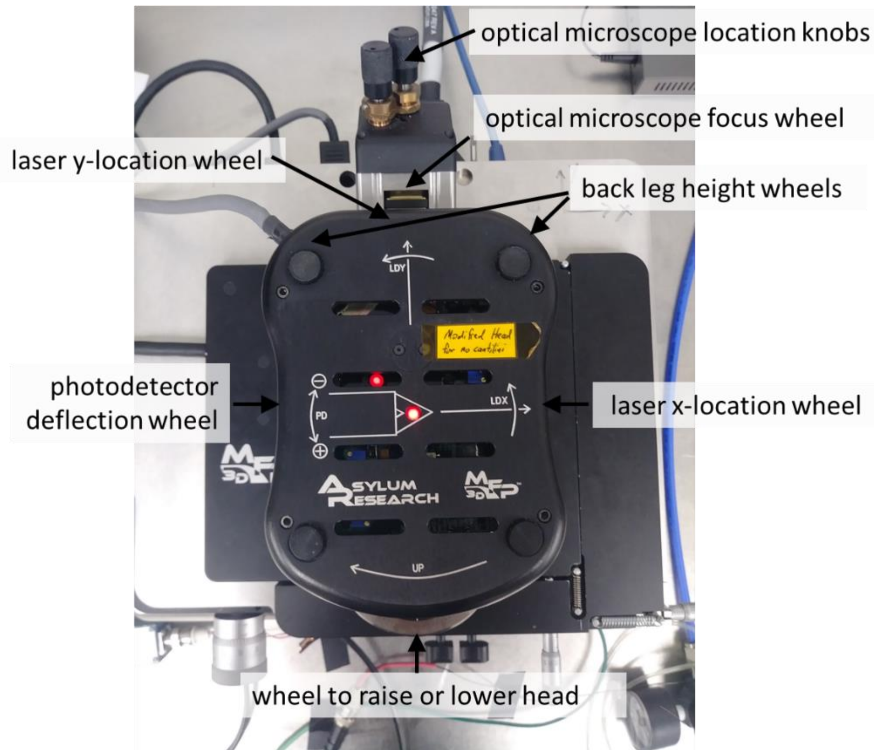
b. Lift up the head with two hands and flip it back over. Place the two hind legs of the head into the slots at the back of the stage first, then gently lower the front leg into the front slot. Make sure the probe does not collide with the sample, as this will render it unusable. If the probe looks like it will get too close to the sample, use the front wheel on the head to raise the front leg (turn left).
c. Open the Asylum Research software (version 15) from the desktop and once it says Ready at the bottom select the desired mode of operation, AC Air Topography.

3. **Setting up the Camera**

a. Press the camera button in the software's menu bar at the bottom of the screen. If a window does not automatically open with the camera's view, then press *Show Live Video* on the AR Video panel.



b. Turn on the light for the optical microscope and adjust the brightness.
c. Move the field of view with the back two knobs on the AFM head until you see the cantilever tip.
d. Make sure the tip is in focus. The focus can be changed by using the wheel at the back of the head, in front of the two field of view knobs. You can zoom in by pressing the *Magnify* button at the top of the camera view window.



4. **Setting up Laser**

a. Align the laser spot on the cantilever, using the LD X and LD Y (Laser Diode X/Y) wheels at the back and right of the head. You should be able to see the laser shining on the tip once it is aligned.
   i. If the laser spot isn't seen, try moving it inwards using the LD X wheel first. Raster in a methodical way rather than randomly (eg. move in the x-direction then sweep in the y, move more in the x-direction and sweep y again, etc.).
   ii. If the sum is very low and the laser spot dim, then you may have found a ghost reflection. Try searching nearby for the real laser spot.

b. Adjust the laser location to maximize the sum. The actual value of the sum will vary on the cantilever type and coating (3 to 5 is typical). You may get slightly better sensitivity with the spot closer to the end of the tip, but worse signal-to-noise ratio if too close.

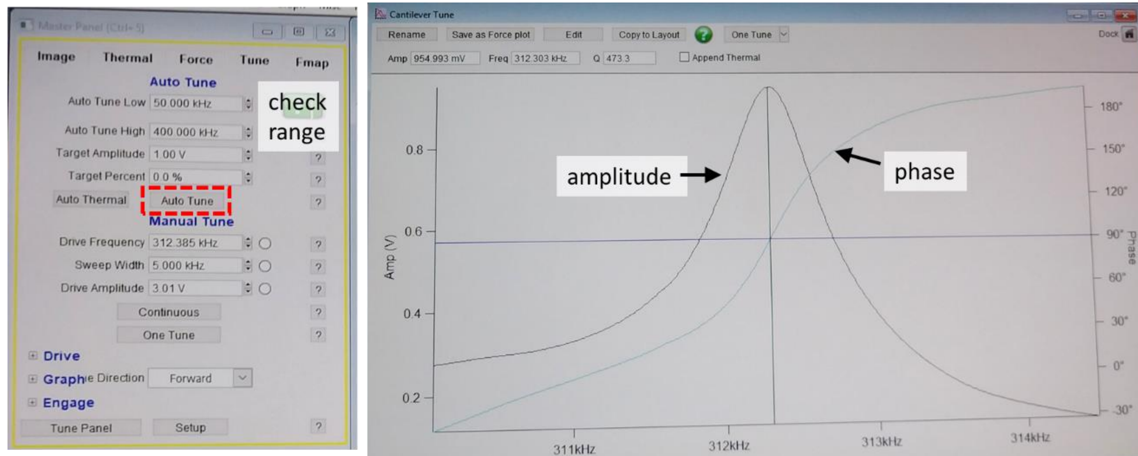
5. **Deflection and Tuning**

a. Minimize deflection using the photodetector (PD) wheel on the left of the head to get as close to 0 as you can. The setpoint values that you choose for scanning will be based on this point as a reference.
   i. Note that sign matters. If the deflection is negative, move the PD wheel in the (+) direction (towards you) and if it is positive, rotate the wheel in the (-) direction (away from you).

a. Tune the tip by going to the *Master Panel* → *Tune tab* → *Auto Tune.* It will make a sound when finished and will display a plot of amplitude and phase.

i. Make sure the auto-tune frequency range contains the tip's resonance frequency (usually found on the tip's box)

ii. The auto tune should end up with a clean single peak near the tip's resonance frequency in the center of the window. The phase should cross 90° at the resonance peak frequency. The drive amplitude depends on cantilever type and age but should be <1 V. If it is >1.5 V or the tune is poor then try reloading the tip.



6. Setpoint and Engaging

a. Check the setpoint in *Master Panel* → *Image tab* → *Setpoint.* A reasonable value is around 80% of the free air amplitude (typically around 800 mV for a free amplitude of 1V).

   i. *[advanced]* If interested, you can do a thermal tune of the cantilever (*Thermal tab* → *Collect Thermal Data*). This lets it oscillate naturally in air (not driving it with the piezo), and by zooming in and fitting the resonance peak you can get the thermal sensitivity. This is a conversion factor for amplitude [V] to deflection [nm]. You want your setpoint to be larger than your tallest feature, and can convert from nm to the required setpoint in V using this factor.

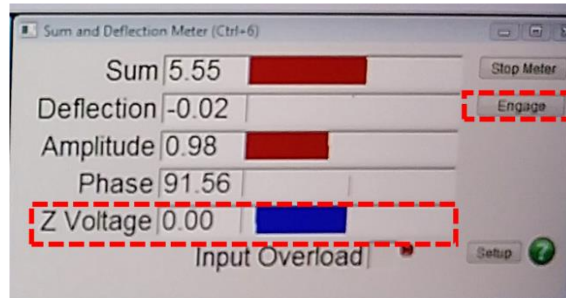
b. ***Click Engage***. If you don't click engage for the final approach you will smash the tip (engage turns the feedback on so if the tip gets too close it will lift)! You should see the Z-voltage bar fill to maximum. Lower the tip towards the sample by turning the front wheel to the right. When you are almost at the surface (it looks almost in focus) you should turn this *slowly* so as not to overshoot and smash the tip into the sample. The tip has reached the sample when the Z-voltage start to decrease (and the amplitude will decrease to the setpoint value), and the software will make a sound. Continue lowering until the Z-voltage reaches halfway (around 70), so you can utilize the full scanner range.

   i. Before the tip is contact with the surface (Z voltage decreases) you can move on the sample using the micrometers at the stage edge. Never move the stage while in contact; you need to withdraw first before you can move.

   ii. If you see the deflection suddenly change or the tip suddenly jerk, then something may have touched the tip holder before the actual tip itself. Make sure that you are

over the sample, and that nothing else tall is impeding the approach (eg. the magnets, electrical probes, etc.).

iii. The tip is lowered by shortening the front post of the head. It is okay if the head looks somewhat tilted towards you as you lower it. If it is tilted significantly (it may be after others have used the electrical probe station), then you may also want to lower the back legs using the wheels at the back of the head. Always raise or lower the back legs together so that the head does not tilt sideways.



c. Withdraw and fix the deflection back to zero if it has drifted.
d. *[optional]* Re-Tune by going to *Master Panel* → *Tune* → *Auto Tune.*
e. Click *Engage* again. You may now begin scanning.

7. Scanning

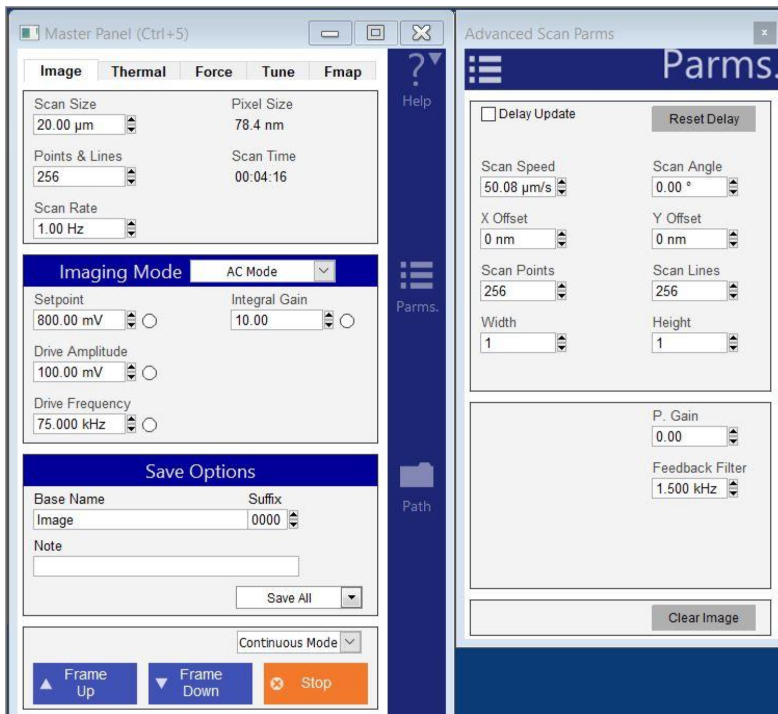
a. ***Press Frame Down or Frame Up to begin Imaging.***
b. The image will be saved once the scan is complete, with the filename and location set at the bottom of the *Master Panel*. You can also choose to have it take one scan then stop, or do continuous imaging.
   i. The file location is saved by date and is set in *Master Panel* → *Path*.
   ii. A number will automatically be appended to the filename so that you don't need to change the name each time you do a scan.
   i. If you hit stop, the scan will stop and the tip will withdraw. If you want to save the partial image, then you must click on the *Save Partial* button.
c. Adjust the scan parameters. You want the trace and retrace (blue and red) heights to match well. Parameters can be changed in the *Master Panel* by typing in a value or using the arrows. Or, it may be done using the wheel on the controller box next to the screens, turning the outer wheel to select the parameter and the inner wheel to change the value.
   i. The scan size and resolution are set in the upper section of the master panel. If you click on the *Parms* button, then a panel with additional settings will open. You can choose a non-square size and resolution here.
   ii. In the additional parameters panel you can set an x- and y-offset (up to ±45 μm). If you need to move further, withdraw the tip and use the stage micrometers. You can also change the scan angle.

iii. A scan rate that is faster means an image is taken faster. However, a slower scan rate can help you track your features better and will help the tip last longer. The optimal scan rate depends on your scan size and feature height; if you use a large scan size or tall features then you should reduce your scan rate (eg. 1 Hz could be fine for a 5 μm x 5 μm scan but is probably too fast for a 20 μm x 20 μm scan).

iv. If you don't see any features in the trace/retrace and they don't match at all when you start scanning, then you are not in contact with the sample yet and should lower the setpoint. Lowering the setpoint will effectively move the tip closer to the sample and will increase the interaction with the surface. This can improve tracking of surface features, but if the setpoint is too close to the sample then the tip can wear down (especially if you have tall features). Lowering the setpoint can switch the imaging mode from attractive (phase >90°) to repulsive (phase <90°).

v. Increasing the integral gain is recommended if the trace and retrace do not quite match (the feedback is too slow), or the surface is not being tracked well giving you poor resolution. However, too high of a gain results in wild oscillations in the feedback as well as a high-pitched ringing. Gains of 5 - 35 are common.

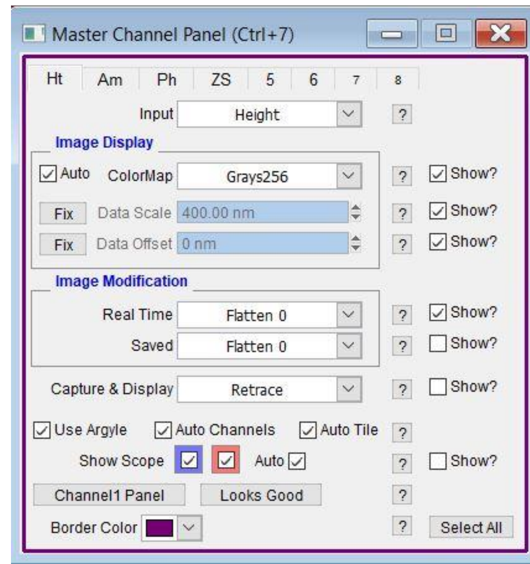
vi. Increasing the drive amplitude causes the tip to oscillate with a larger amplitude and can also improve tracking of the surface. This has a similar effect to lowering the setpoint, but puts more energy into the tip and can wear it out faster. Increasing the drive amplitude can also switch the imaging mode from attractive to repulsive.



d. Most of the parameters will be updated in real time, though not all, such as the scan rate. You can choose to "delay update" which waits until the current image is finished to execute the changes performed.

e. Right click on an image and select *Fix Scale* for auto contrast and brightness.

f. The *Master Channel Panel* lets you choose which channels are being displayed (eg. height, phase, amplitude, ZSensor, etc.). In this panel you can choose whether to display and capture the trace, retrace, or both as well as what kind of flattening to display in real-time and save (click on *Setup* to see all the options). Data will be saved with your chosen flatten, but if you later open the image using the Asylum software you can remove this flatten. You can also adjust contrast, brightness, and the colour scheme (*ColorMap*) of the image.



g. Scanning is sensitive to vibrations or loud noises. You may place the caution sign on the door to alert others entering the room.

8. Real-time Scan Manipulation using the Image

a. To place a line on an image, right-click on an image and select *Line*
   i. To change the scan angle, draw a line then right-click on the line and select either the *Scan Parallel* or *Scan Perpendicular* option (If it does not allow you to do this, then you need to go to the *Master Channel Panel* → *Setup* and check *Use Argyle*). Or, you can change the angle in the *Parms* panel.

b. To set the next scan to be zoomed into an image, right-click and select *Box*. Draw a box around the area of interest then right-click and select *Zoomzoom*.

c. To translate, right click on a spot in the image and select *XY Offset*. This moves the selected point to the center of your scan.

8. Analysis

Files are saved as with an ibw extension. These can be opened in the Asylum Research software (a free demo can be downloaded from the company, but you will not be able to save modified images), or they can be analyzed in other software such as Gwyddion. Images can also be exported as tiff or text files by opening them in the Asylum software and selecting *Tiff Export 1x* or *ASCII Export* from the *Commands* dropdown.

a. To analyze an image on the tool, open it in the Asylum software.

b. Press the *M* button at the top of the image to Modify it.

c. You can modify the image using several functions:

   i. A zeroth order flatten will do a match line correction (it will align each scan line so they have the same median values, correcting tilt in the y direction and any drift/offset from line to line).

   ii. A first order flatten will correct for tilt within each line (x direction), then align so each line has the same median value (y direction) like the zeroth order.

   iii. An xy-planefit will fit a plane to the data and then subtract if off. This corrects tilt but not drift from line to line.

   iv. In the *Mask* tab you can exclude some areas of the image in the flattening or planefit calculations. To exclude tall features, you can drag along the histogram and the corresponding mask will show up on the image. You can also *Exclude* or *Include* regions in your mask by pressing these buttons and then drawing on the image using shapes listed in a column next to the image. Your drawing may not work if a mask already exists, so *Reset* and try again. When done drawing, hit *Make Mask*. Use *Reset Mask* to remove the mask. There is also an option in the flattening to use a *Magic Mask*, where the software will try to pick a mask for you.

   v. Press *Restore Layer* to undo the last change you made, and *Ultra Restore Layer* to return to the raw data (click *Setup* if you don't see this).

d. Press the *A* button to perform analysis on the image.

   i. This will show statistics on the image in the *Roughness* tab, and if you have a mask the statistics will reflect this. Use the sliders and boxes near the top to make a quick rectangular mask to examine roughness in a local region of the image.

   ii. The *Histogram* tab will let you generate a histogram showing the distribution of heights [nm] in your image.

   iii. Pressing *Crtl-I* will bring out a bar under the image with markers that you can click and drag onto the image. This will show the distance and height difference between the two markers.

   iv. In the *Section* tab you can draw a line on your image to look at the data along it.

Troubleshooting

- If you first engage the tip and see no features, with the trace and retrace not matching at all, then you may not be fully engaged with the surface. Try lowering the setpoint.
  - You can tell you are engaged with the sample (when you have pressed *Engage* but are not scanning yet) if you lower the setpoint and the Z voltage doesn't change.
- If there are lots of "scars" on your image (abrupt steps up and down that are not real, forming lines in the image), or the phase is jumping between >90° and <90° then you may

be mode hopping between attractive and repulsive. Try adjusting the setpoint or the amplitude to push it into one of these regimes (lower setpoint will push it to repulsive).

  - If you still have trouble, then you can adjust the drive frequency to be slightly off resonance. Go to the tune curve, right click on a point slightly to the left (right) of the resonance peak, and select *Set Drive Frequency* to push it to repulsive (attractive) mode respectively.
  - Scarring or mode hopping may also occur if your sample or tip is "sticky." If there is something on the sample or the tip, then the tip can be pulled towards the sample and get stuck there temporarily causing scars. Try using a free amplitude that is very small (so it doesn't stick) or very large (it has enough energy to unstick). Or, you can try to clean your tip by doing a very fast small area (e.g. 1 µm) scan with low setpoint. You can also try performing a force curve to put the tip in full contact with the sample to try to remove any dirt stuck to the tip, or to release any charge buildup on the tip. However, sometimes "cleaning" the tip can damage it.

- If you see repeating images in your scan (always the same shape and orientation, although different locations and sizes) that you suspect are not real, then you may have an artefact from a blunt or dirty tip. When a defective tip goes over a feature smaller than it, you end up imaging the tip shape instead of the shape of the small feature. Change to a new tip.
- If your scan is very noisy but not sticky, then make sure the vibration isolation is turned on properly (grey box with lights on the floor under the AFM/glovebox) and/or lower the gain.

Operation in Contact Mode

1. **Setup**

   a. Choose and load an appropriate cantilever. Typically contact mode cantilevers have a lower spring constant, meaning they are more flexible for better tracking the surface and will not damage soft samples.
   b. Adjust the laser spot to be on the cantilever, and maximize the sum. Set the deflection to zero by adjusting the photodetector.
   c. You do not need to tune the cantilever, as you are not driving it to oscillate.
   d. Choose an appropriate setpoint and start scanning. The setpoint determines how much force the tip puts on the sample as it scans in contact. A higher value means you are pressing harder on the sample (opposite of tapping mode).
      i. The setpoint is relative to the deflection, which is why it is important to start at 0. If you start at 0V deflection and have a setpoint of 0.5V then the total applied "force" is 0.5V. If you start with a deflection of -1V and apply a setpoint of 0.5V, then you are effectively applying a 1.5V "force."
      ii. The setpoint can be converted to an actual force in units of nN by doing a thermal tune (to get the sensitivity in V/nm) and a force curve (to get the spring constant, in N/nm) to get the required conversion factors. (see Force Curves section).

Additional Tips and Troubleshooting

- A scan angle of 90° is highly recommended. The tip can catch on something if it is scanning parallel to it, looking like a height change when there isn't one. Tips are usually shaped to give a better contact mode image when scanning 90° (they aren't perfect cones/pyramids).
- If you have a very soft sample or a stiff tip, then when trying to engage the tip may dig into the sample rather than deflecting the tip (it will actually be in contact before the system registers a deflection and says it is in contact). If this is the case, you may notice that the deflection changes significantly as you approach or that the Z Voltage is unstable (it keeps lowering, or it jumps). Try imaging with a softer tip (lower k value and resonance frequency).

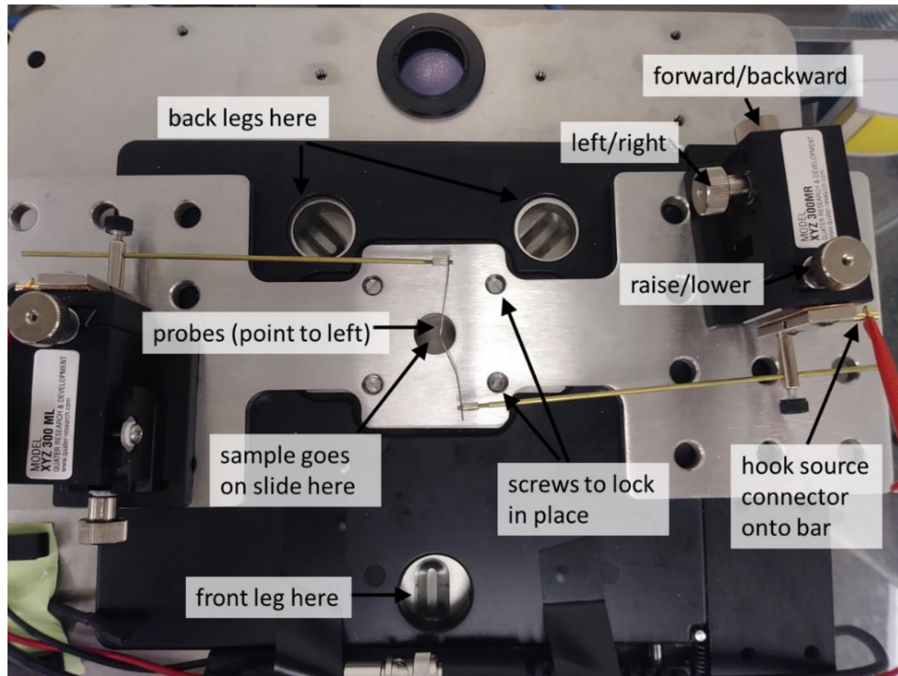
Probe Station Operation

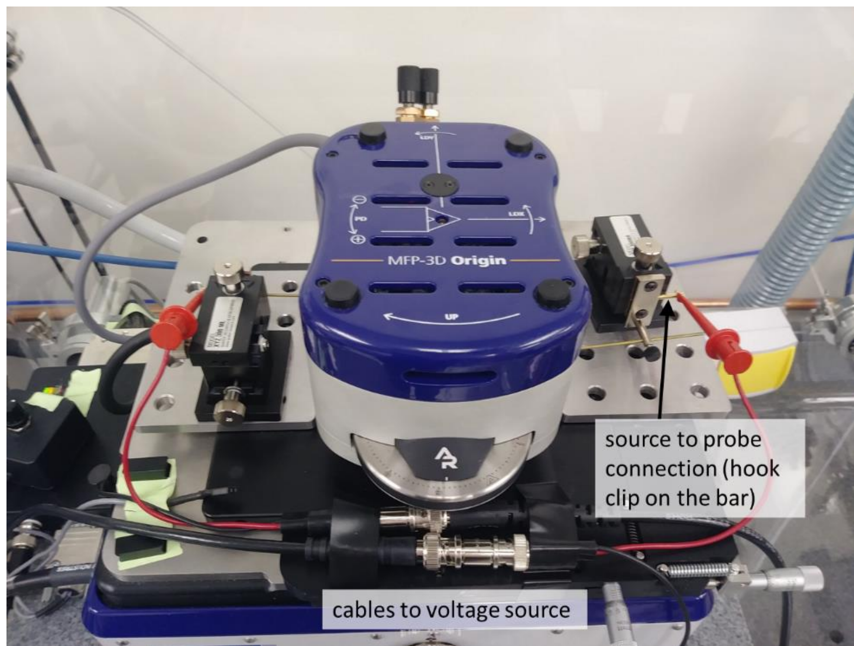
To scan devices while biased you will need to have large contact pads (or long leads to the actual device area being contacted). The tip needs space to scan and if you put the probes too close to the active region then the probes[1] might hit the cantilever holder while scanning. You should be able to put the probes >100 µm away from the scan region. An external voltage/current source can be connected to the probe station.

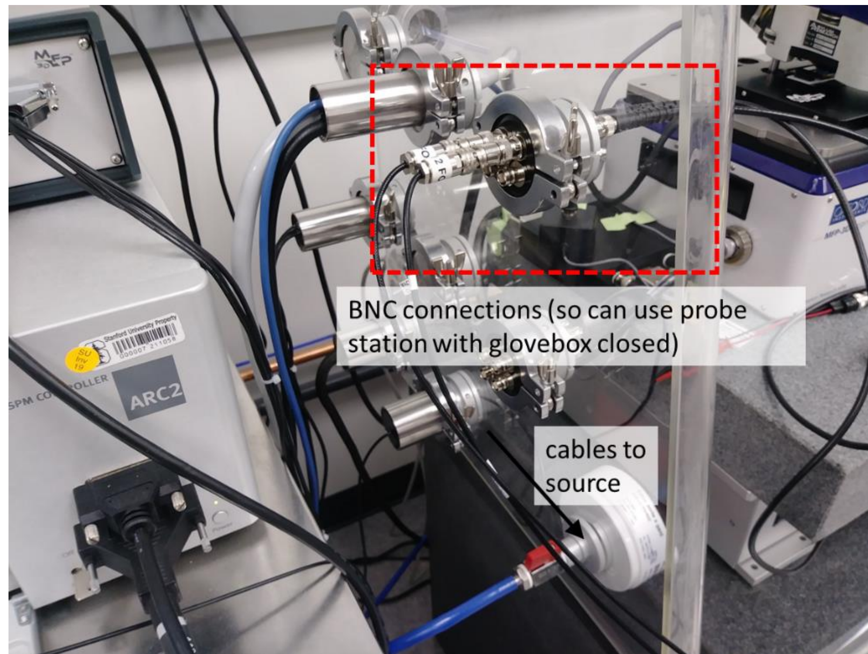
1. **Setup**

   a. The probe station is detachable from the stage. If it is not attached, then first raise the head as far as it will go (raise both the front and back legs). The probe station adds considerable height, so if you do not do this first then you may crash the tip when you put the head back on.
   b. Remove the head and place the washer over the center of the stage. Then add the probe station on top (direction matters, the probes should point to the left). It is locked in place using a flat head screwdriver to turn the screws 45°.
   c. Do not change the angle of the probes unless absolutely needed. The probes have been carefully positioned to avoid touching the AFM head when the tip is in contact.
      i. If the probes tips are dirty, you can clean them by putting IPA on a Kimwipe and gently poking the probe through it. Otherwise avoid touching the probes.
      ii. The probe arm blocks are held onto the stage by magnets, and if you touch them they will move (try to avoid).
   d. Set up the cabling to your voltage/current source (eg. a Keithley 4200).
      i. The probes station can be electrically connected to your source by using the connector with a hook on one end and a BNC connection on the other. Press the button on the end of the cone to extend the hook and clip it onto the gold bar that protrudes from the probe arm block.
      ii. You can connect the BNC end directly to your source. Or, if you plan on using the glovebox, then connect this end to the BNC connections on the inside of the glovebox wall. Connect your source to the outside of the wall.

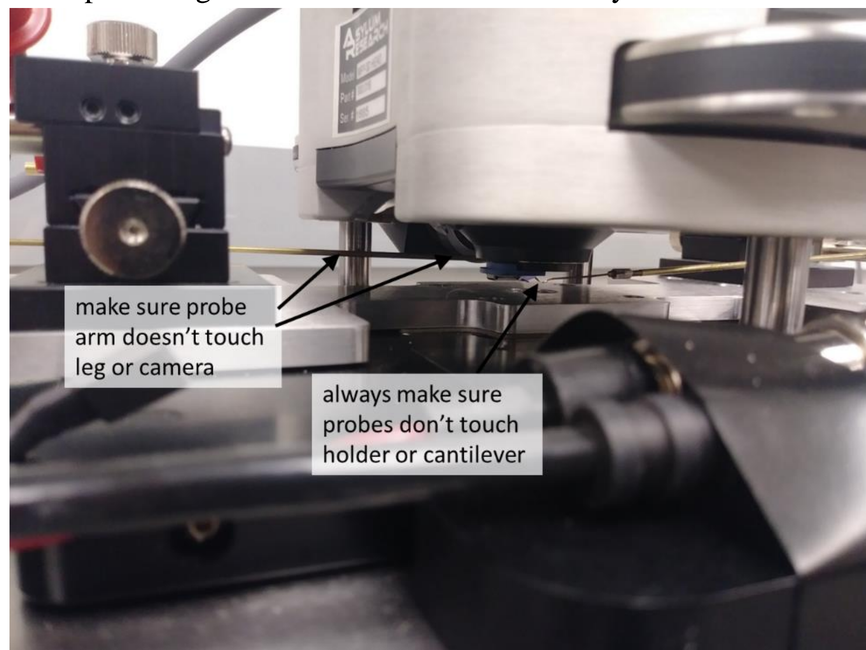
[1] In this section, "probe" refers to the electrical probe station not the cantilever or tip.


forward/backward
back legs here
left/right
raise/lower
probes (point to left)
sample goes on slide here
screws to lock in place
hook source connector onto bar
front leg here
MODEL XYZ 300 ML
MODEL XYZ 300MR


MFP-3D Origin
source to probe connection (hook clip on the bar)
cables to voltage source



e. Load your sample. First, raise the probes up (use the top knobs on the controller blocks) so you can fit your sample underneath. Mount your sample and carefully slide underneath the probes. The probes have a limited range of lateral motion and may not be able to reach everywhere on your sample so try to position your region of interest close to the probes. The probes primarily cover the bottom right quadrant of the hole. Align your pads vertically (towards the front and back of the glovebox).
f. Lower the probes a bit so they are close to the surface but not quite touching. Very carefully replace the head, putting the back legs down first then lowering the front. If you see that the probes will contact any part of the head, then remove the head and readjust the probe angles/locations and make sure they are lowered sufficiently.



2. **Contacting your Device**

   a. Focus on the cantilever, align the laser, adjust deflection, and tune (if applicable).
      i. If you are using an expensive tip (e.g. for SThM), then it is recommended that you load an old tip first. Follow the instructions below to set up the probes and your scan. Make sure you can run a topographic scan without bias on the device of interest without the probes touching anything. Once you have verified this, load the real tip.
   b. Try to locate the probes by moving the optical microscope view and focus. Ensure that the tip is far to the left of the probes.
   c. Move the probes to your device pads (use the knobs on the controller block for each probe), all the while making sure the tip is far to the left of the probes. Gently lower the probes onto your pads and test the electrical connection. Put the probes on your pads as far from the active channel as possible to give the tip space to scan.
      i. *The probes are delicate.* Lower them gently and do NOT use them for scratching surface oxide. Do not put excessive force on them – lower them until they are just contacting the pad and not digging into it. Do not bump into the system while the probes are down.
      ii. If you are not close enough to the sample yet to focus on it (and you've maxed out the optical microscope focus knob), then lower the head very slowly until you can see your devices.
      iii. If you max out the lateral range of motion on the probes, then you will need to remove the head and readjust the probes and your sample location.
      iv. Electrostatic discharge has occurred before and damaged a device and/or tip. It is recommended you ground yourself before making connections (e.g. using the bracelet on the table).
   d. Lower the head very gently and move the tip towards your device. Always watch and make sure the probes do not contact the tip or the head. If the tip makes any sudden motion, then the probe arms have likely contacted the probe holder.
      i. If you do hit the probe holder or anything else, raise the head and remove it. First try moving your device towards the bottom right part of the hole (the probes are at a better angle in this area). If that does not work, readjust the angle/location of the probes themselves. If the probes have been bent or misaligned by a previous user, contact the superuser.
   e. Engage the tip and scan to locate your area of interest. Do a scan with no bias and ensure that again the probes do not hit the holder at any point in the scan.
   f. Bias your device, and scan in the desired mode. Make sure that you bias your device at a constant voltage or current for a sufficiently long time (remember that each scan takes at least several minutes). On the Keithley, try 4000 sampling points with 0.25 s timing.
      i. If you want to bias your device while it is in an inert environment (Ar in the glovebox), then withdraw the tip once you have aligned everything and done a test scan. Raise the head a bit, and lift the probes very slightly (if you are focused optically on the tip, then try not to raise the probes so much that they come in focus at the tip height). Set up the glovebox (see glovebox section). When the

oxygen content is low enough for you, then gently put the gloves on and lower the probes onto the device pads. Engage the tip and run your experiment.

3. **Unloading**

a. Turn off the device bias and withdraw the tip. Raise the head up, and move the tip far away from the probes. Then raise the probes up slightly. Raise the head up some more.
b. Remove the head and unload your tip. Raise the probes a little more, and unload your sample.
c. Remove the probe station from the stage by turning the four central screws. Gently lift it up and put back in the black cabinet. Be very careful not to move or bend the probes.

Glovebox

Although normally open to air, the Asylum AFM is housed in a glovebox that can be sealed for a controlled environment. It comes with a front panel that can easily be attached and sealed as needed, then later removed. The glovebox can be filled with Argon (Ar) for an inert measurement environment, and is capable of reaching down to ~10 ppm of O_2. To reach O_2 levels this low takes at least 1.5 hours, and less time if you don't need very low oxygen levels. The glovebox has a load lock so that an oxygen-sensitive sample can be brought to the room in a carrier with an inert environment then put into the load lock without ever being exposed to air. Operating in a low humidity environment can reduce the layer of water normally present on all surfaces, which can be beneficial for certain scanning modes such as Kelvin probe microscopy. The electrical probe station can be used simultaneously with the glovebox, as there are BNC connections on the sidewall of the glovebox. For doing oxidation lithography, the glovebox can be sealed with a humidifier inside to increase and stabilize the humidity.

Operation

1. **Closing the glovebox lid**

    a. **SAFETY:** Ar itself is not toxic, but if there is too much Ar and not enough O_2 then you can pass out and suffocate. The amount of Ar normally used is not enough to do this, but if there is a leak or open valve for several hours the room can slowly fill with Ar. If you intend to use the Ar, contact Mahnaz to open the safety interlock. **For safety, always work with someone else**. **Open the door to the room so there is additional ventilation. If the oxygen sensor drops below 21% or alarms (goes below 19%) then leave the room.** Do not put your head next to the Ar output, or into the box if it has just been full of Ar. Ar is a heavy gas and it will sink, so there can be a danger even if the oxygen sensor on the wall doesn't read any changes. Avoid working or sitting low to the ground near the glovebox. **If at any point you feel slightly light headed or short of breath, leave the room.**

    b. It is highly recommended that you set up everything so it is ready for the final approach before you close the glovebox. It will be very difficult to load cantilevers or do precision tasks with the glovebox gloves on.

        i. If you are using the electrical probe station, then make sure the probe station is set up and the cable connections inside the glovebox are ready.

        ii. Load a cantilever into the probe holder and put the probe holder on the head.

        iii. Load your sample (if it is air sensitive you can bring it through the load lock later).

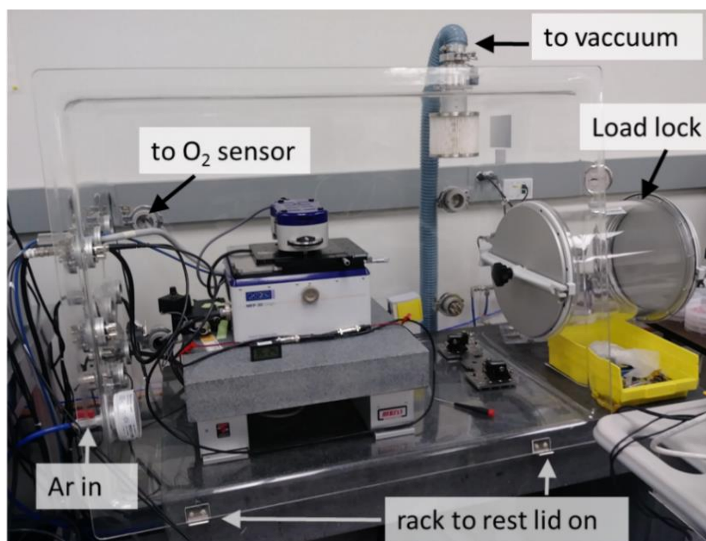
        iv. Adjust the laser, deflection, and tune if applicable. Bring the tip close to your region of interest, ready for the final approach. It is highly recommended that you do a test scan to make sure the tip is good and that the scan will not hit the electrical

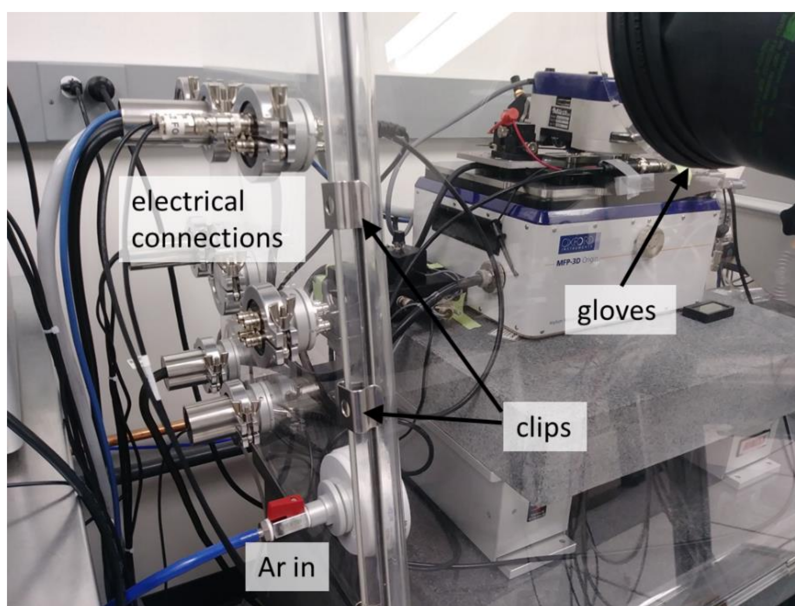
probe arms (if used). If using the probe station and your device can tolerate a very small bias in air, then it is recommended you test to make sure there is a good connection. When done, lift the probes and head up slightly (it can shake a bit when closing the glovebox).

c. Make sure both of the load lock doors are closed. Tighten the black knob to close all the way, and turn the silver bar 90 degrees to lock in place (so it is vertical). Make sure nothing blocks the door inside the glovebox from opening if you intend to use it.

d. The front lid is generally kept to the left of the AFM computer table. Gently put it on the front of the glovebox, resting it on the rack. Put the side with the gloves on the outside, and the side with the black seal on the inside. The gloves should be on the top side.

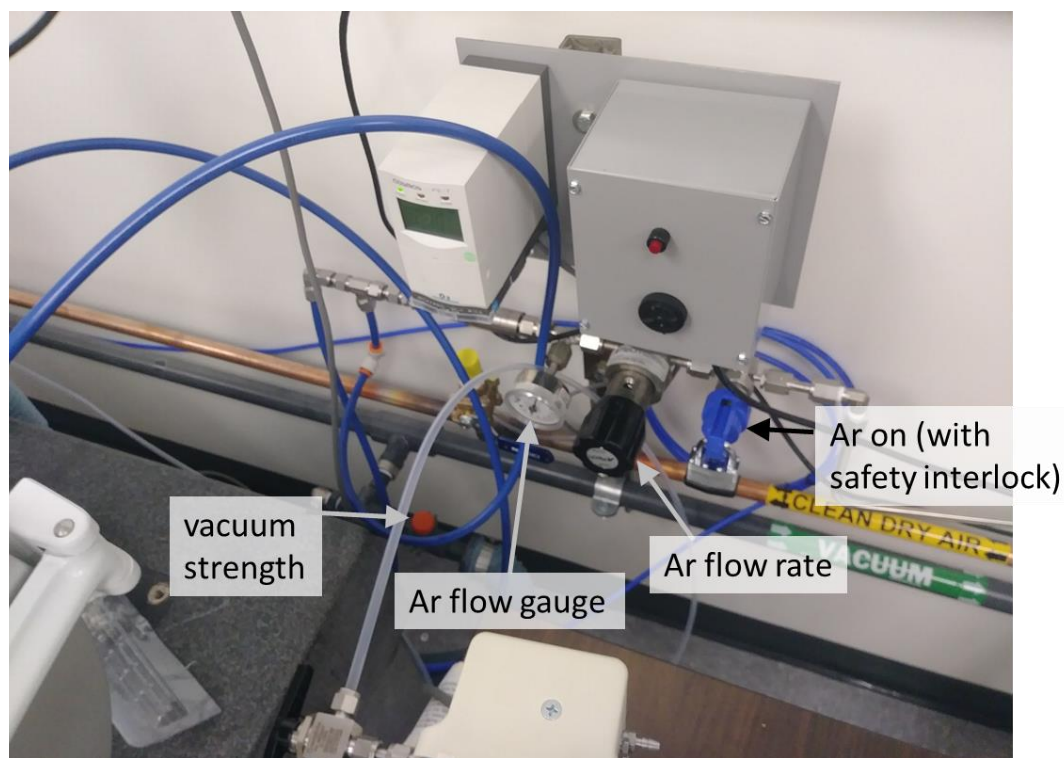
e. Clamp the rim of the lid closed with silver clips. They are in the black cabinet to the right of the glovebox. You should use all of them for a good seal, putting them along the entire perimeter with more near the corners.





2. **Purge with Ar (if only using for increasing humidity to do oxidation, skip this)**

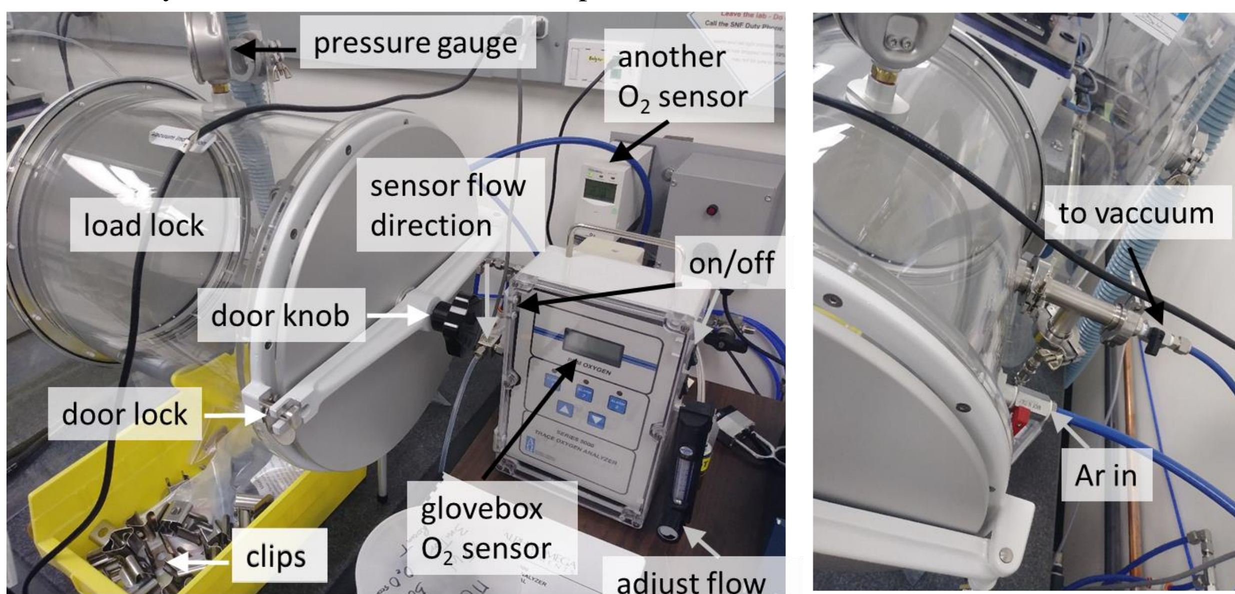
   a. Ensure that you have a good seal. Turn on the Ar by turning the blue knob on the wall CCW to the open position (after it has been unlocked) and adjust the flow rate slowly with the black knob to the left of it. There is a sensor next to the black knob to give you an idea of the flow. Don't turn on the Ar unless the glovebox is clamped shut and ready to be used so Ar doesn't flow into the room. Open the red Ar valve to the glovebox (turn it parallel to the line), found at the bottom left corner of the glovebox. The gloves should start inflating. If not, adjust the clip locations.
      i. Feel for leaks along the outer rim of the glovebox while the Ar is stronger than the vacuum. Usually there is one in the bottom left corner. Adjust the clips to cover leak areas.
      ii. Again, open the door, work with a buddy, and **if the O_2 level in the room drops below 19% there will be an alarm. Leave the room.**
   b. Turn on the vacuum to compensate. Turn the black lever to the open position on the blue hose at the top of the glovebox. Turn on and adjust the strength of the vacuum by turning the orange knob near the wall in a counter-clockwise direction.
      i. Do not have only Ar or only vacuum on, as this can push or pull on the gloves and eventually damage them. Find a balance between the vacuum and Ar so that the gloves are not inflating or deflating. Unfortunately there is no pressure sensor, so you will have to balance just by looking at the arms.



c. Set up the oxygen sensor for the glovebox. Turn on all three buttons (one on the left side, and two on the right). Turn the black arrow on the tubing to the left of the sensor to face towards you. It points in the direction that it is taking air from, so if it points to the open end of the tubing (away from you) then it is sampling room air instead. Turn the black knob in front to turn on the flow and adjust the rate. The silver ball should be floating.

   i. The screen displays the O_2 level in ppm. It will take a while for the O_2 content to reach readable levels and until then just displays OL (overload). There's a small black sensor in the glovebox that reads humidity down to 5%, which can give you an idea of how fast it is being purged until the O_2 sensor starts reading.

d. Wait until the desired O_2 level is reached. This takes at least an hour, and you may leave IF the gloves are correctly balanced (they do not move at all over the span of 5-10 min), and you leave the door to the room open.



3. Introduce sample with load lock (if applicable)

a. Make sure that the inner door is closed. Tighten the black knob to close the door seal. Make sure the silver clamp is pointed perpendicular to the bar to hold the door in place.

b. Open the outer door and put your sample into the load lock. Put it as close to the inner door as possible because the gloves can't reach very far into the load lock. Close the outer door. If the door won't open, then open the Ar valve to refill the chamber (the last person may have left the load lock under vacuum). Don't put your head right next to the door when you open it (so not to get a face full of Ar).

c. Purge the load lock several times, with both doors closed. Turn the load lock vacuum on and Ar off until the pressure drops as low as the gauge can read. Then turn off the vacuum and refill with Ar. Repeat at least twice. You will have to turn the Ar flow up while doing this, since flowing Ar to the load lock will reduce flow to the main chamber and cause the arms to deflate a lot.

d. After the final refill with Ar, you may put the gloves on and open the inner door to take your sample inside. Wear some latex or nitrile gloves when using the glovebox gloves. Do not wear rings or anything that can tear the gloves.

4. Set up scan

a. Put the glovebox gloves on. Wear some latex or nitrile gloves when using the glovebox gloves. Do not wear rings or anything that can tear the gloves.
    i. It is easier if you put all your fingers in the glove fingers first before pushing your hand into the glovebox. Be careful not to hit the AFM head when you push your arms in the glovebox.
b. Operate the tool as you normally would. Readjust the deflection. If applicable, retune the cantilever since the resonance frequency depends on the atmosphere.
c. Move to the feature of interest, and lower the electrical probes (if used) then the tip itself. Scan as usual. You may have to re-optimize some of your scan parameters.

5. Disassembling

a. When finished, raise the head and probes. If your samples are air sensitive, then you should reseal them in your carrier.
b. Turn off the vacuum and the Ar flow. Close all the valves to the glovebox and load lock, as well as the valves along the wall. Turn off the glovebox O_2 sensor.
c. **Make sure the room door is open.** Take off the clips on the glovebox rim and put them back in the black cabinet, then take off the front panel and put it back next to the table. **Wait several minutes before doing any operations inside the glovebox or on the floor near it**. It takes a while for the Ar to dissipate, and if you bend over or put your head in the glovebox right away then you may feel light-headed. There is enough air flow in the room that the Ar will quickly dissipate, and the oxygen sensor shouldn't measure any change in the room but if it does and it alarms *then leave immediately*.
d. Disassemble the rest of the AFM setup like usual.

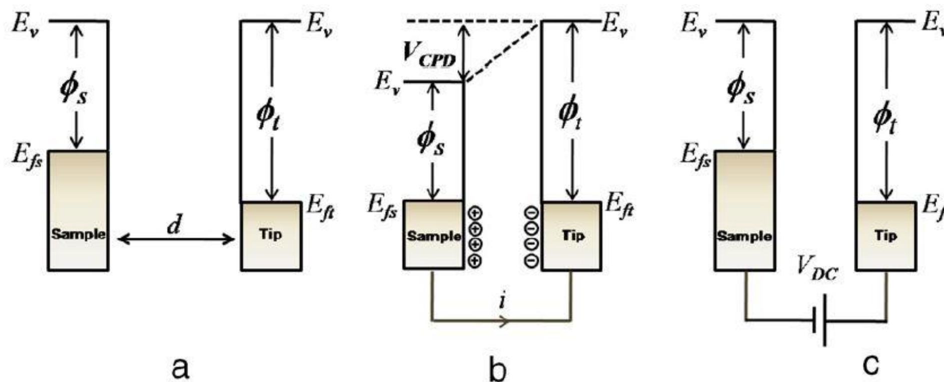
Kelvin Probe Microscopy

Principles of Technique

Scanning kelvin probe microscopy (KPM or SKPM) is a non-contact mode that images the local surface potential using a conductive tip. On an unbiased sample, contrast is generated primarily from local differences in workfunction. If carefully calibrated then the actual values of workfunction can be determined. If the sample is insulating, then it may image trapped charge. If KPM is performed on a biased electrical device, then it can be used to map the local electric fields in the device.

The Fermi levels of the tip and sample will align if they are electrically connected or close enough for tunneling to occur. If the two have different workfunctions (ϕ_s, ϕ_t) then this will result in a potential difference $V_{CPD} = \phi_s - \phi_t$ between tip and sample as well as a slight accumulation of charge. Generally, if an AC voltage is applied to the tip while near the surface, then the force on the tip will oscillate as changes in charging occur. However, if a DC bias with equal magnitude and opposite polarity to V_{CPD} is applied between the tip and sample, then the potential difference and charge accumulation will be nullified. Applying an AC bias while that same DC bias is applied results in no oscillatory force on the tip. Thus to determine V_{CPD} the tip scans across the sample at a constant height finding the DC bias required to stop an AC oscillation. The frequency ω of the AC signal is chosen to be the electrical resonant frequency of the tip for the best signal, which is very close to the thermal and mechanical resonance of the tip.

V_{CPD} is an indication of the workfunction difference between the tip and the sample. Calibration is done by scanning a reference sample with known workfunction, allowing the extraction of the tip workfunction. Then a numerical value of workfunction can be determined on new samples. If the sample is biased in an electrical device, then the measured voltage is primarily a map of the surface potential (V_{sp}) in the device (though it can have a little bit of contrast from differences in workfunction still). The DC bias on the tip required to stop the oscillations in this case is V_{CPD} + V_{sp}. The gradient of the surface potential gives the electric field.



(a) The tip and sample have different workfunctions, ϕ_t and ϕ_s respectively. (b) When electrically connected electrons will flow until the Fermi levels align. This accumulates charge and a voltage V_{CPD} is generated between the two. (c) A DC bias of -V_{CPD} applied between tip and sample can nullify this.

Mathematically, the force the tip experiences with both a DC (V_{DC}) and AC biases ($V_{AC}\sin(\omega t)$) applied to the tip, as well as a DC surface potential V_{sp} (eg. by an applied voltage in a device) is given by:

$$F = F_{DC} + F_{\omega} + F_{2\omega}$$

$$F_{DC} = -\frac{dC(z)}{dz}\left[\frac{1}{2}\left(V_{DC} \pm V_{CPD} \pm V_{sp}\right)^2\right]$$

$$F_{\omega} = -\frac{dC(z)}{dz}\left(V_{DC} \pm V_{CPD} \pm V_{sp}\right)\sin(\omega t)$$

$$F_{2\omega} = -\frac{dC(z)}{dz}V_{AC}^2[\cos(\omega t) - 1]$$

where C(z) is the capacitance between the sample and the tip, as a function of tip-sample distance z. There is a DC component to the force along with AC components with frequency ω, 2ω, and higher order terms not shown here. Note that the ω component vanishes when $V_{DC} = -V_{CPD}$ (or if the sample is biased, $V_{DC} = -V_{CPD} - V_{sp}$). The sign of V_{CPD} depends on whether the tip or the sample has a higher workfunction, and the sign of V_{sp} depends on whether the applied sample bias is negative or positive.

There are two modes that KPM can be run in: single-pass and dual-pass mode. The Asylum system by default uses the dual pass mode, in which a line is scanned first for topography and then rescanned at a constant height measuring V_{CPD}. This second scan is called the NAP mode. It is possible to measure both the topography and potential in a single scan (single-pass mode). This generally gives better resolution and is faster, but gives worse topographical cross-talk. Topography can in general affect the KPM images, causing artifacts especially at steps. Differences in potential near steps may be real if they are not affected by the NAP setpoint.

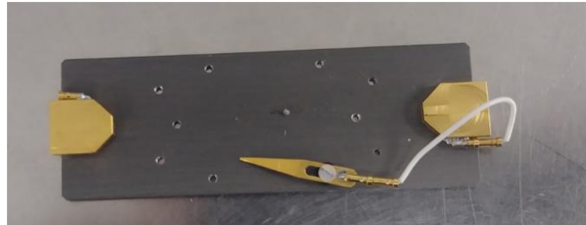
Since electrostatics generate long range forces the tip feels the surface potential of a large area around the tip. This means that the surface potential measured is an average across an area larger than the tip, limiting the resolution of the image.

One final note is that the measured workfunction of a sample depends a lot on the surface quality and anything adsorbed on it (eg. surface oxide, water vapour). Images will be cleaner and more accurate if the sample is clean and baked or scanned in the Ar glovebox environment (to remove the layer of water present on all samples in air).

For further reference see: W. Melitz, J. Shen, A. Kummel, S. Lee, "Kelvin probe force microscopy and its applications," *Surface Science Reports*, vol. 66, pp. 1 – 27 (2011).

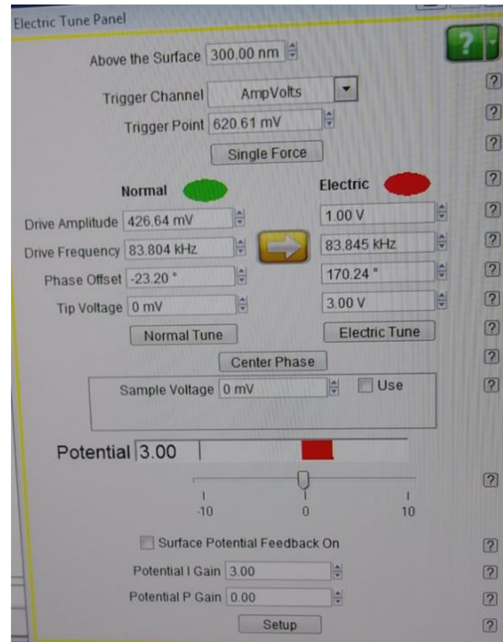
Operation

a. Select and load a conductive tip.
b. When you open the software, select the mode in *Electrical Techniques* → *SKPM*
c. Load the sample. If using the probe station to apply a bias or ground the sample follow the probe station instructions. You should ground your sample unless you are imaging built-up charge on a less conductive substrate.
    i. You can put your sample on the grey slide under the clip. The slide itself is not conductive (aluminum oxide). The best grounding option is to attach a small white wire to the cantilever holder. Such a wire is found in the ORCA or PEEK accessory kits in the cabinet (the wire has an exposed end on one side, a little half-circular ring on the other end, and has white insulation). On the back of the cantilever holder there is a small screw that should be loosened, then the half-ring of the wire should be slid under the washer and screwed back down to hold it in place. The wire will hang out from under the holder and will magnetically stick to the gold pad once the cantilever holder and AFM head are installed for scanning. This ensures that the ground of the sample and tip holder are the same.
    ii. The alternative easy way (but not a very reliable ground) is to use the wire attached to the clip and tape it to the metal part of the stage (pull out the end connected to the gold pad), which is grounded by the system.



    iii. If your sample has a native oxide on it, then you can scratch the bottom surface and attach to a conductive puck with a drop of silver paste, then ground the puck to the stage.
d. Adjust the laser spot and deflection, tune the cantilever, and approach as usual. Remember to click *Engage* before approaching.

e. Withdraw and perform an electrical tune. This is found in the *Electric Tune Panel* and will determine what frequency to use for the AC signal.



i. Click on *Single Force* at the top, which will perform a force curve. This will find your sample surface and then raise the tip up a bit so it can perform the electrical tune. A starting point is 300 nm above the surface with the trigger point equal to or smaller than your tapping mode setpoint (800 mV if you used the default setpoint). If you do not see the tip come in contact with the sample (if the force curve looks like a straight line, with no kink in it) then lower the trigger point so that the tip will be brought closer to the sample.

ii. The left hand (*Normal*) side of the panel will show the values from your tapping mode auto-tune. As a starting point populate these into the right side (*Electric*) using the yellow arrow. Set the drive amplitude to 1V for now. This corresponds to the electrical AC signal amplitude input to the tip, both for the tune and for scanning.

iii. Choose a non-zero tip voltage for the electrical tune. This is the DC voltage applied to the tip for the tune only. A good value is typically 3V. This needs to be different than $-V_{CPD}$, otherwise the tune curve will look noisy and the peak will be unclear (remember that if the DC voltage applied to the tip is equal to $-V_{CPD}$ then the tip won't oscillate anymore, so we won't be able to tune it).

iv. Press the *Electric Tune* to run the tune. Similar to a tapping mode auto-tune, a window should open showing a resonance peak. If it is noisy, adjust the tip voltage and run again. Right click on the peak location and click *Set as Drive Frequency*. This will be a similar value your tapping mode resonant frequency, but slightly different. Then, click on *Center Phase* in the *Electric Tune Panel*, which sets the phase to 90° at the resonance peak.

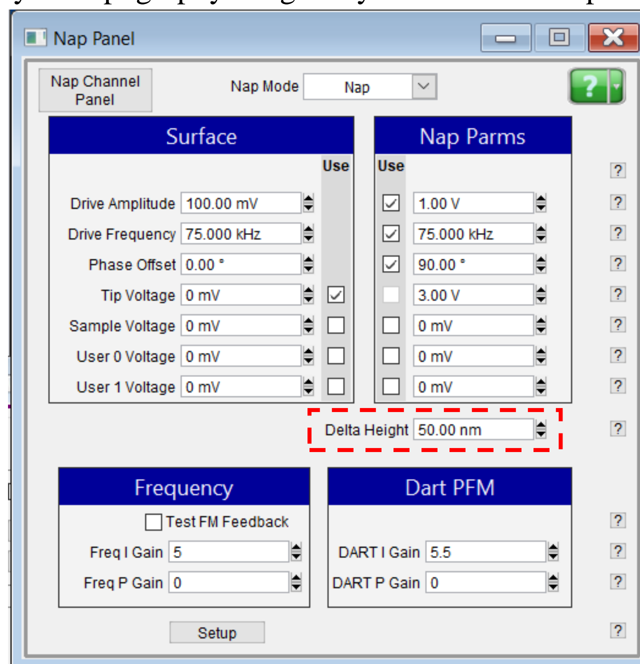
v. Pay attention to the amplitude of the resonance peak. If the amplitude is very low (especially 0.1V or less) then you can increase it by using a higher *Drive Amplitude*. This can improve your KPM signal and resolution.

f. Begin scanning. The default mode is two-pass, so it will scan a line for topography then scan the same line again in NAP mode for surface potential.

i. If you are using a bias on your sample, note that the system is only capable of measuring a surface potential within -10V to +10V (beyond that it will saturate).

g. Adjust the topography scan parameters (e.g. scan rate, setpoint) as needed to get a good topography image.

i. A high resolution KPM image depends on how good your topography image is. The NAP pass relies on the topography information to adjust the height of the cantilever as it rescans the line to measure the potential. If there are artefacts, scars, etc. in your topography image they will also show up in your KPM image.



h. Adjust the NAP parameters to get a better KPM image.

i. A better image can be obtained by lowering the delta height in the *NAP Panel*. The delta height is how far the tip moves up to do the NAP scan relative to the height of the topography scan. Lowering the delta height (it can be negative) will put the tip closer to the sample, so it will feel the electrostatics stronger and pick up a better signal. If it is too low, you may see topographical crosstalk (features that aren't actually from KPM but are from topography). If you are very close to the surface or have very tall features, then this could damage the tip.

ii. A better image can be obtained by increasing the *Drive Amplitude*. Withdraw the tip, change the drive amplitude in the *Electric Tune Panel*, retune, and start scanning again.

iii. If needed you can also adjust the *Potential I Gain* in the *Electric Tune Panel*, which is similar to the integral gain in tapping mode. Don't worry about the *Potential P-Gain.*

Conductive AFM

Principles of Technique

Conductive AFM (CAFM) is a technique that measures local differences in sample conductivity. The conductive probe is in direct contact with the sample, measuring the current flowing between the tip and sample at a given tip bias. The typical setup is to measure the current flowing vertically through a moderately resistive film on a conductive film/substrate that can be grounded. It is possible to measure current flowing laterally in a moderately conductive film on an insulating substrate, if an appropriate top contact is made. Note that CAFM is not meant for analysis of highly conductive materials since high currents will result in the metallic tip coating burning off. Areas with a directly exposed metallic film should be avoided. Typical currents in conductive AFM should be pA-nA through the tip at 1V bias (not µA-mA).

Operation

1. Setup

a. Load a conductive tip in the special ORCA holder. The back of this holder has circuity that includes a transimpedance amplifier. This allows the standard holder to measure currents between ~1 pA to 20 nA, and the dual gain ORCA holder to measure ~1 pA to 10 µA. The holder should have a white wire coming off the back of it, held by a screw. If there is a screw but no wire then remove the screw, add the C-shaped end of a wire in the ORCA kit, and screw back in place.

b. Load your sample on the special mount. Unscrew to lift the clip, put your conductive sample underneath, then screw back down.

    i. If you have a sample with a back surface oxide, then you will need to scratch a corner and immediately add a drop of silver paint (in the ORCA kit). You may stick the sample + paint onto a conductive puck and contact the puck with the clip (don't put the paint on the sample holder).

c. If not connected, connect a wire between the clip and one of the gold pads.
d. Put the head back on the AFM. The dangling wire from the ORCA holder is magnetic, and you should adjust it so that it sticks to same gold pad the clip is connected to. This completes the circuit between your sample and conductive tip.

2. Scan to get a conductivity map or do an IV Curve

a. When you open the software, select the mode via: *Electrical Techniques* → *ORCA*

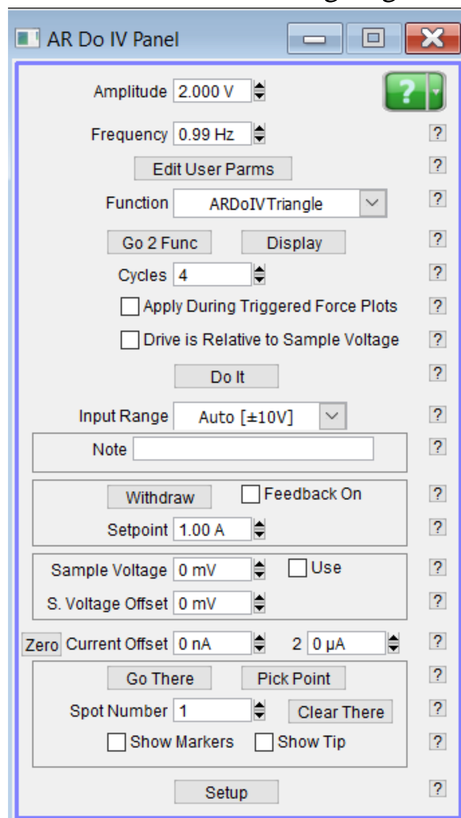
b. Choose an appropriate setpoint, with a lighter force than contact mode. Start with a light force, say 0.1V or 0.2V

i. Once you have approached and start scanning at 0V bias, you can use the *Do IV Panel* to zero-offset the current. There will be some non-zero current value read even when a bias is not applied, so zero-offsetting just subtracts this from all future scans.

ii. You can then choose a sample voltage in the *AR Do IV Panel*, and check the *Use* box to turn the sample bias on. Increase until you register current form your sample. If you don't see anything, you may need to use a negative voltage instead, especially for semiconducting samples.

iii. The head is a dual gain ORCA holder, meaning it has two current ranges it can measure. You may see contrast in one but not the other.

iv. Increasing the setpoint puts the tip in better contact with the sample and can improve signal and resolution. The integral gain also improves resolution.



c. Rather than doing a scan at a particular bias voltage, you can select specific points to run an I-V curve on. This can be useful in determining the appropriate bias to use for a scan.
    i. You can select the bias function that will be applied from among presets in the Function dropdown menu and can adjust the frequency and amplitude. Clicking *Display* will plot the function so you can see what it looks like. Or, you can use the Function Editor from the dropdown menu to customize.
    i. You can select points on your previous image using *Pick Point* in the *AR Do IV Panel*. A circle with a crosshair in it will appear on your image. Drag it to the desired location then click *That's it*. Add more until you are satisfied (you can see all of them at once by checking *Show Markers*).
    ii. To move to a point, you can enter the spot number and click *Go There* (check Show Tip to see a red dot where the tip is).
    iii. Make sure the setpoint is appropriate in the *AR Do IV Panel* and then check the *Feedback On* box to engage the tip.
    iv. Click *Do it* to actually run the IV curve.

Notes

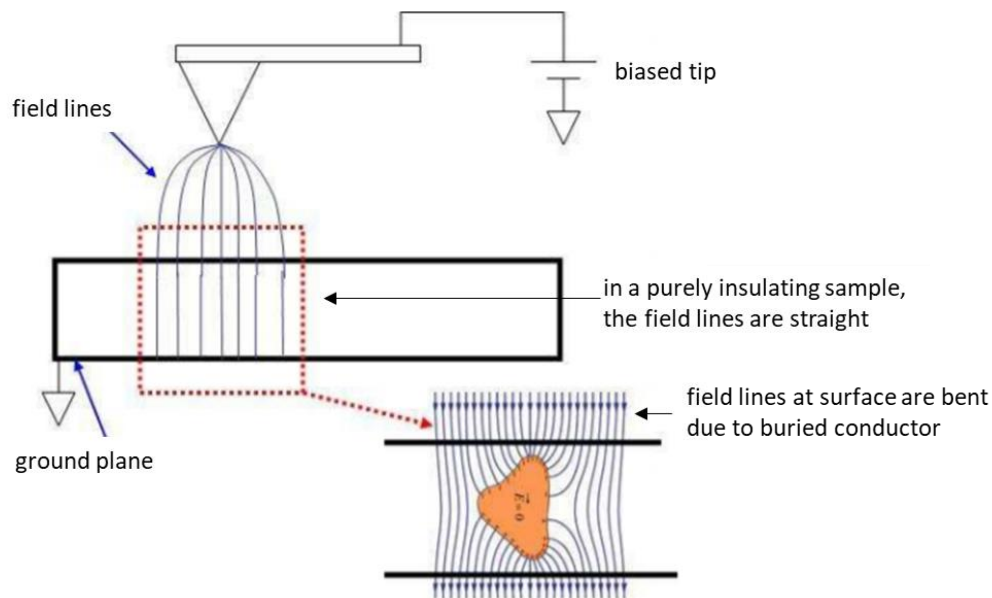
- The current is sensitive to the tip shape and wear. If you see sudden changes in the current it could be attributed to the tip chipping, picking up a particle, etc. If you have a metal-coated tip, then the metal can wear off and you will lose signal. If you see repeating artifacts in the current image, then the metal coating may have partly worn out.
- There is a contact resistance between your tip and sample, and if the tip is not in consistent good contact with the sample then this contact resistance can vary (meaning the current in your image varies, but not because of the sample itself). If this is the case, you may need to apply more force.
- Be aware that for some samples, biasing may lead to surface modification (eg. oxidation).

Electric Force Microscopy (EFM)

Principles of Technique

Electric Force Microscopy (EFM) is a non-contact technique that maps variations in the electric field across the sample. The resonance frequency and phase of a biased oscillating cantilever will be shifted due to electrostatic force gradients. These force gradients arise from changes in tip-sample capacitive coupling due to changes in sample conductivity or surface charges. This technique is sensitive to both surface and near-surface material variations (for example, a buried conductor can alter the field lines seen at the surface). Thus, it can be used to image mixtures of insulating and conducting materials. It can also be used to look at biased electrical devices.

EFM is a dual-pass technique which scans each line twice (four times if retrace and trace are collected). The first pass is normal AC topography. The second is in NAP mode, which scans at a fixed height above the surface to best image the long-range electrostatics and minimize topographic effects.



Operation

a. Load a conductive tip.

b. Load your sample, and ground if possible.

    i. If your sample is conductive or has a top conductive coating, then you can put your sample on the conductive slide under the clip. The wire attached to the clip should be taped to the metal part of the stage, which is grounded by the system.

    ii. If your sample has a native oxide on it (eg. Si), then you can scratch the bottom surface and attach to a conductive puck with a drop of silver paste, then ground the puck to the stage.

iii. If you have devices with contact pads, you may use the probe station to ground your device (see probe station section). You may put the probe on your pad, and then connect a wire from the bar on the probe arm controller block to the metal part of the stage (which is grounded).

c. In the software choose *Electrical Techniques* → *EFM*.

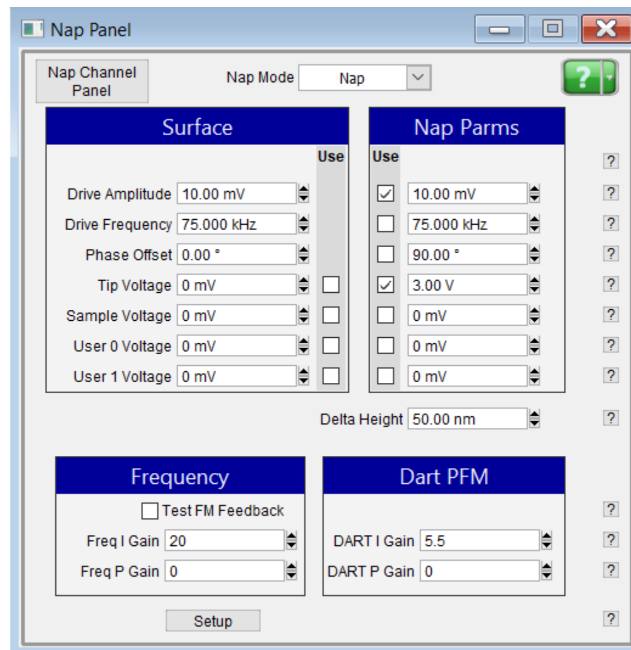
d. Setup, tune, and approach as usual.

e. Choose appropriate parameters for the NAP scan (it does a dual-pass scan, where the first is topography and the second is NAP mode to collect EFM data where the probe is raised to a fixed height) and start scanning.

   a. A reasonable *Delta Height* is 150 nm. The larger your free amplitude is, the larger your *Delta Height* should be. This sets how much the probe is elevated/lowered during the NAP phase. Decreasing this may result in a sharper image. However, lowering too much may mean the tip is making contact with the sample, which disrupts the EFM measurement.

   b. The *NAP Drive Amplitude* dictates the amplitude of tip oscillation during the NAP measurement.

   c. Changing the *NAP Tip Voltage* may make the image clearer.



Scanning Thermal Microscopy

Principles of Technique

Scanning thermal microscopy (SThM) is an excellent technique to map the heating of a sample, such the Joule heating of an electrical device during operation, or to qualitatively map local differences in thermal conductivity. With careful calibration, one can extract actual values for temperature but this is *by no means trivial.*

Probes for SThM have a small palladium resistor embedded at the end of the tip (with a very thin oxide coating, around 1nm or so). As it heats up, the resistance of the tip changes, determined by the temperature coefficient of resistance (TCR) of the palladium. This change in resistance due to heating is detected using a wheatstone bridge, generating a map of heating but in units of mV.

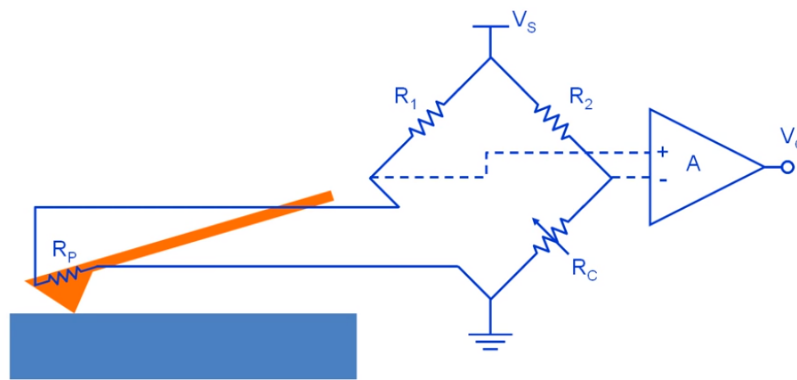


Image from PicoCAL video https://www.youtube.com/watch?v=TZb5D_T3xLo

The SThM probe is connected to one branch of a wheatstone bridge. Another branch contains a tunable resistor (potentiometer) R_c, which is initially tuned to match the probe resistance. When the sides of the wheatstone bridge are matched (so that $R_2/R_1 = R_c/R_p$) there is no voltage difference between the left and right half. No current flows in the transimpedance amplifier (A) and no voltage difference is read out. When the probe heats and R_p changes, then the bridge is no longer balanced. A current flows into the amplifier and a voltage difference is read out (this is the signal that is imaged).

The tip has a thermal exchange radius that can be over 100 nm. This means that even if the tip is small, it still collects thermal information from a larger area (the raw temperature map is essentially being Gaussian blurred by this exchange radius). This means that there can be artefacts in the scans from topographic features or surface roughness, which changes the thermal exchange radius by changing the surface area near the tip.

Also if mapping thermal conductivity differences, note that the thermal boundary resistance between the tip and sample can vary if there are material changes so the signal you get may not solely be due to different thermal conductivities (it can depend on how good the tip is in contact).

There are two modes for SThM: passive mode and active mode. In passive mode the tip is heated by the sample (such as a device Joule heating). A very low voltage (V_s) is applied to the circuit, high enough so that a voltage reading can be done without too much noise, but low enough so that

the tip is minimally heating (1°C or less). In active mode, the tip is being forced to heat by using a high voltage V_s across the circuit. Local differences in thermal properties can be mapped, since higher thermal conductivity regions will dissipate heat better and cool the tip.

For more information, see T. Borca-Tasciuc, "Scanning Probe Methods for Thermal and Thermoelectric Property Measurements," *Annual Review of Heat Transfer*, vol. 16, pp. 211 - 258 (2013).

Operation

Users need to bring their own tips for training. It costs $1700 for a box of 5 from Anasys.

The tip is in contact mode and there should not be current flowing between the sample and tip. If there is it will not give you a correct image of heating, and it will damage the tip (the tip only has a ~1nm oxide coating the metal resistor in the tip, and this will easily break down). You will need to electrically isolate your sample from the tip, using a thin capping layer like PMMA or Al_2O_3. A thinner layer is better, to give you a better thermal map. It is also recommended that you not scan over contact edges or tall features if you suspect they may not be coated well by your insulator.

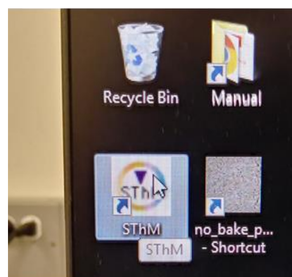
1. Setup

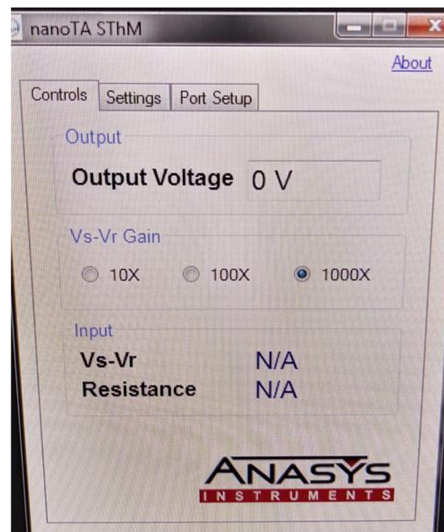
a. Load your sample.
b. If you are using the probe station, select and load a dummy cantilever. Make sure that you can approach and scan the device of interest without the probes getting in the way (see probe station section). The SThM tips are expensive, and you do not want to break one because the probes arms are in the way.
c. Load an SThM cantilever. You will need the special tip holder for it (it has a hemispherical slot, with two magnets to hold the tip).

i. Ground yourself first (the tips are sensitive to ESD). You can touch the stage or the coax leads on the controller box to the right of the screens or the metal part of the ESD mat bracelet.

ii. Place the cantilever on the tip holder. Make sure it is all the way back and straight in the groove. Then transfer from the mounting holder to the actual AFM head.
iii. Use a small piece of tape to hold down the cable on the head. Be very careful when placing or removing the head so that the cable does not catch on anything (such as the probe station parts) because it can rip off the cantilever and destroy it.
iv. Replace the head, being very careful as you lower it to ensure the tip will not hit anything.

d. Open the SThM experiment file saved on the desktop.
e. Turn on the black Anasys box underneath the AFM table (red lights should be on), and open the Anasys software for the tip controller (icon has SThM in a circle with a triangle). Connect to the black boxes by going to *Port Setup* → *Connect*. It should now say Connected in green font at the bottom of the window. Always do this before connecting the cantilever cable to the potentiometer.
f. Go to the *Controls* tab and make sure that the tip voltage is set to 0V. Always set this to 0V when connecting or disconnecting the probe (otherwise ESD will kill it), or when the probe is not in use to protect the tip.





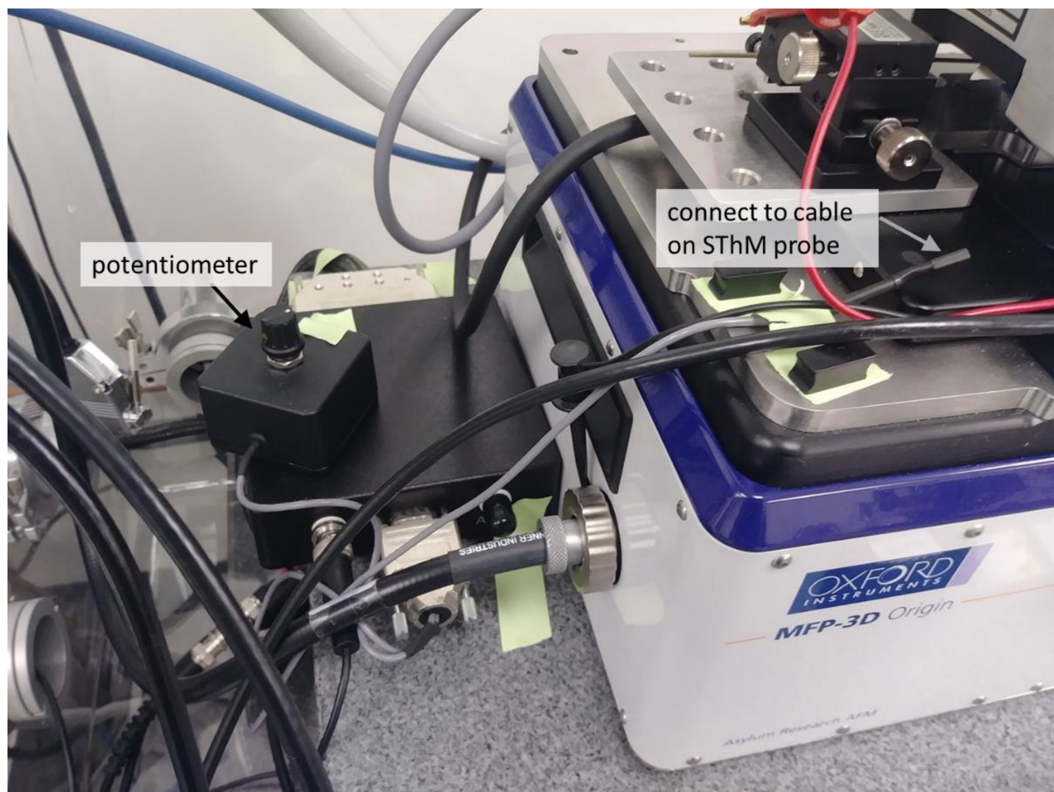
g. Place the laser spot on the cantilever. There is a transparent stripe running down the center of the cantilever, so position the laser spot on one side of this stripe and maximize the sum.

h. Set the deflection to be -1V with a setpoint value of -0.5V, giving a net setpoint of 0.5V. The image will depend on the choice of setpoint (as it alters the thermal exchange radius and the pressure on the sample from the tip), but 0.5V is a good choice for passive mode as it gives high resolution but also good signal.

   i. The photodetector has a linear response to deflection/height within the range -0.8V to +0.8V. A sample that heats and expands will cause the deflection to drift to positive values (it deflects the cantilever up more). It is suggested that you set the deflection to -1 and the setpoint to -0.5V (rather than deflection 0 and setpoint 0.5V) so that if there is this drift then you remain in the linear regime of the detector. If you have a sample that is heating considerably you may have to stop and check the deflection drift once in a while, so you don't apply too much force or too little.

   ii. You will have to readjust the deflection once you approach the sample.

i. Connect the cable on the probe to the cable from the potentiometer. Make sure the tip voltage is set to 0V first and that the Anasys system is connected. Ground yourself (you can put one hand on the coax connectors from the controller box), and slide the cover off the two prongs on the probe cable. *Do not touch the prongs*, or you may destroy the tip. Carefully slide them into the connector coming from the potentiometer.



j. Approach the sample carefully. If you are using the probe station, then carefully move and approach the probes onto your device, while grounding yourself. Test the device electrically to ensure good contact. Move the tip to the device and slowly approach, making sure there is visibly enough space between the holder and the probe arms.

k. After approaching, withdraw and set the tip voltage to 0.5V to scan in passive mode. Readjust the deflection. If you want to scan in active mode (where the tip is heating), use a higher voltage. Tips under high bias won't last as long and will degrade over time, so you should turn it back to 0V when not in use.

l. Set the amplification on the Anasys window to the highest value (1000X) to get the best signal, especially for measuring small temperature changes.

m. Engage the probe then adjust the potentiometer to match the wheatstone bridge to the tip resistance. Look at the voltage difference (Vs-Vr) on the Anasys software panel and try to get as close to 0V as possible while turning the dial on the black potentiometer box near the head. Be careful not to bump the head while it is in contact. Up to around ±0.2V is acceptable (the reading is after amplification so the raw value is actually very small).

2. **Scanning**

   a. Begin scanning. Topography and an SThM signal will be taken at the same time.
      i. If scanning a device, always do a scan at 0V bias first. This will determine if there is an SThM signal due to topography and not sample heating. If there is, likely from surface roughness or steps in topography, then be aware of artefacts from topography in the rest of your SThM scans. If you have a very rough surface with a topography signal, then local differences in thermal conductivity or temperature may be obscured, but large area information would still be valid.
      ii. A slow scan rate is better, to give the tip more time to heat and interact with the sample. You should always scan at 0.8 Hz or less. However, scans will take longer and if you are looking at devices they will be biased/stressed for longer. Be consistent with your scan rates, as the images will depend slightly on the scan rate.
      iii. If looking at devices under several bias voltages, it is better to increase the voltage monotonically due to drift from heating, rather than jumping between different values.

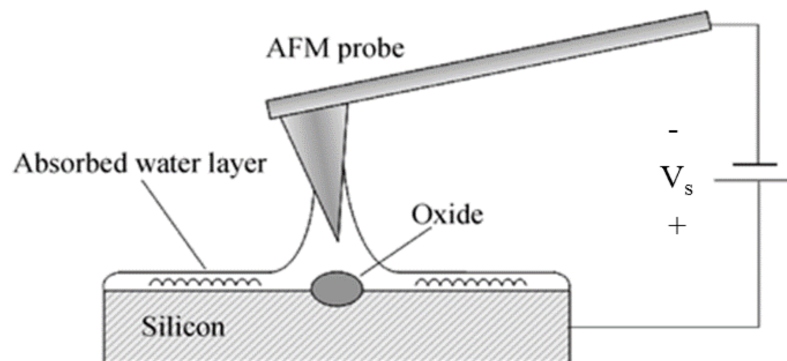
3. **Disassembling**

   a. Once finished, withdraw the cantilever and move it up and away from the probe arms (if applicable). Raise the probes, but not too high or they will hit the holder.
   b. ***Set the tip voltage to 0V***. If you unplug the connector without setting the voltage to 0V first, then the probe will die.
   c. Unplug the probe from the potentiometer, and put the cover back on the prongs. Ground yourself first and be careful not to directly touch the prongs.
   d. Remove the head, being careful not to snag the cable. Unload the tip and your sample.

Lithography and Local Oxidation

Principles of Technique

Because of small tip radii, scanning probe microscopy can be useful for patterning nanoscale features over a small area (although doing a large area is time consuming and impractical). Lithography can be done in several different ways. One is to use a stiff probe in contact mode to indent/stab/scratch the sample in a desired shape. This can be done while applying a bias to the tip. Another way to pattern samples is using local oxidation lithography (or anodic oxidation lithography), which can draw oxide on samples.



Oxidation lithography relies on the fact that samples in air have a thin adsorbed layer of water on the surface. If a negative bias is applied to the tip, then water molecules can be broken down to generate OH^- ions. These move towards the surface and react with the sample surface. Continued oxide growth may occur from either the OH^- moving down into the sample or sample ions moving upward from the highly localized field (the exact kinetics are not fully understood).

This can be done for most materials that oxidize (Si, Al, and many other metals), though the oxidation parameters will vary between materials. The sample must be capable of being grounded (it should be electrically conductive). The process generates a raised bump of oxide generally 0.5 – 15 nm tall, depending on the conditions, and there will also be some oxidation depth into the sample.

There are many parameters that can affect the width and height of the oxide formed. Lowering scan speed, increasing bias voltage, and reducing tip-sample distance or setpoint can all result in more oxidation. Generally oxidation done in contact mode, though you may be able to also do it in tapping mode with different conditions. Another variable to adjust is the humidity of the air; increasing the humidity increases the water layer on the surface and makes oxidation easier. This can be done by putting a humidifier near the AFM stage and closing the glovebox.

Operation

1. **Setup**

   a. Select and load the cantilever. Contact mode lithography done by pressure on the sample can be performed with any tip that is not too soft (has a very low spring constant). Local oxidation lithography must be done with a conductive tip, and a sample that can be grounded. Note that it is possible to image in tapping mode, then do the actual lithography in contact mode.

   b. When you open the software, select the *Lithography Mode*. Or, open the *Litho Panel* in the *AFM Controls* dropdown menu while in the regular Contact or AC mode.

   c. Load the sample. If you want to do local oxidation or lithography with a bias, then you should ground your sample.

      i. If your sample is conductive or has a top conductive coating, then you can put your sample on the conductive slide under the clip. The wire attached to the clip should be taped to the metal part of the stage, which is grounded by the system.

      ii. If your sample has a native oxide on it (eg. Si), then you can scratch the bottom surface and attach to a conductive puck with a drop of silver paste, then ground the puck to the stage.

      iii. If you have devices with contact pads, you may use the probe station to ground your device (see probe station section). You may put the probe on your pad, and then connect a wire from the bar on the probe arm controller block to the metal part of the stage (which is grounded).

   d. Adjust the laser and deflection, tune the cantilever (if applicable), and approach as usual. If using the probe station, follow the probe station instructions.

2. **Lithography**

   a. Collect an image of your sample either in tapping or contact mode.

   b. If you are not in contact mode already, then withdraw the tip and change to contact mode. Choose the lithography parameters.

      i. Choose a setpoint for lithography. This is how much force the tip applies to the sample. A larger setpoint will create deeper indents (for contact litho) or wider/taller oxidation lines.

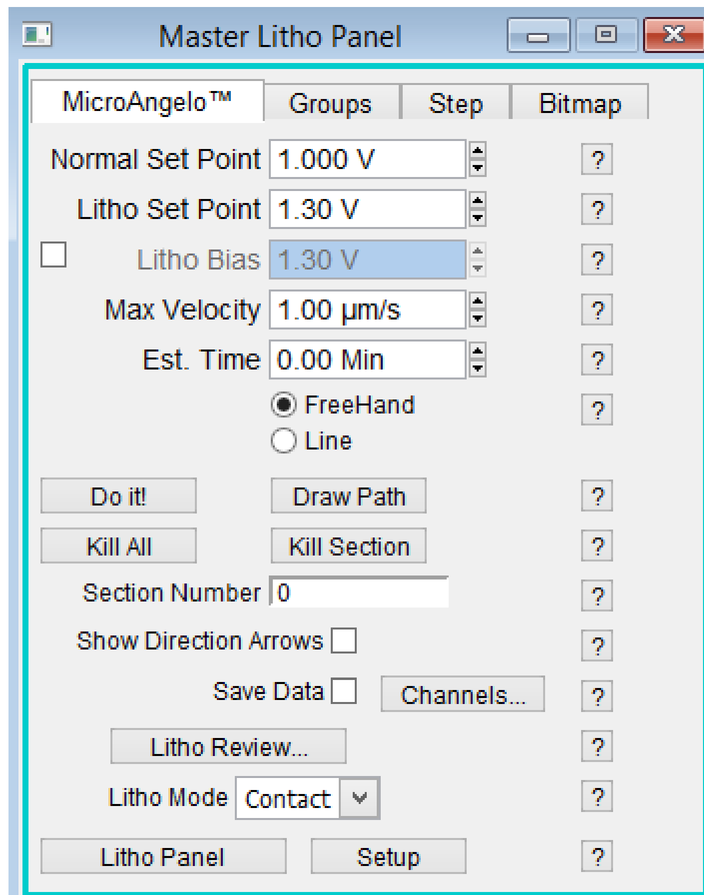
      ii. Choose a normal setpoint. This will be the background setpoint, for when the system is moving to or between your lithography locations. If you do not want to touch the sample at all then you can make this less than your deflection (if your deflection was 0, then negative). If your litho setpoint is more positive than your normal setpoint then it will move towards the sample for lithography, and if more negative then it will move further away for lithography.

      iii. If doing oxidation lithography or biased lithography, then choose a litho bias to apply. For local oxidation, you will need to apply a negative bias. A more negative bias will increase the oxidation height/width.

iv. Adjust the velocity for lithography. If your velocity is too high, then you won't see oxidation because there is not enough time for the reaction to occur. If you want thin lines of oxide, then you can make the velocity a little higher.

v. Select either *FreeHand* or *Line* then click *Draw Path* and draw on the topographic image that you just took. These will be the regions where lithography will be done. Click *Stop Draw* when finished. To delete lines you may use either *Kill All* or enter a section number and use *Kill Section*.

vi. You can also do pressure lithography at single points by doing a Force Curve (see the Force Curve section).



c. When you are ready engage the tip and click *Do It!* You can watch a red dot on the image to see where the tip is as it draws. Note that there can be a slight drift if you wait a long time between taking your image and doing lithography, so you may not quite be oxidizing the spot you think.

d. When finished, you can withdraw and switch back to tapping mode for imaging if you like.

e. You may find that in ambient atmosphere that you cannot get your sample to oxidize. If this is the case you may need to increase the bias, increase the setpoint, or reduce the lithography velocity. Alternatively, you may need to increase the humidity. There is a small humidity sensor near the AFM that can give you an estimate for the humidity. If you need

to increase the humidity in the chamber, then there is a small humidifier you can fill with DI water and use (contact the superuser).

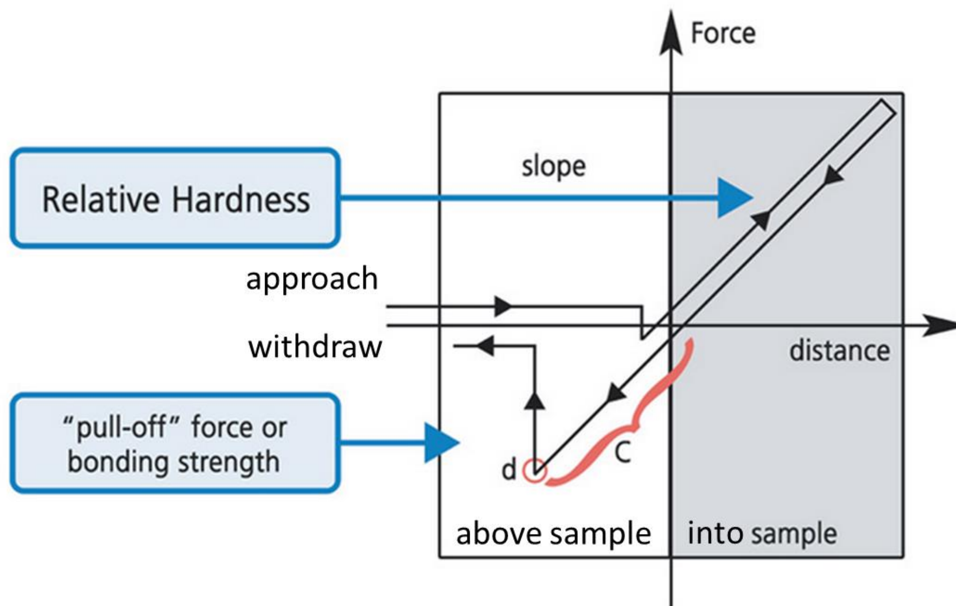
i. Place the humidifier to the right of the AFM and plug into the socket at the back of the glovebox.
ii. With the tip withdrawn, assemble the glovebox (put the lid on and attach the clips around the outside). This will help keep the humidity constant, though the seal does not have to be perfect. (see Glovebox section).
iii. Put on some latex/nitrile gloves and then put your hands in the glovebox gloves. Turn on the humidifier until you see the humidity on the sensor go up sufficiently. Wait a while for it to stabilize. Then approach the tip, and continue oxidizing.

Force Curves

Principles of Technique

In contact mode the intent is to apply a light force to a sample to measure the topography, using a soft cantilever (low spring constant) that easily deflects to track the surface. If instead a larger force is applied with a stiff cantilever then elastic and adhesive properties of a sample can be measured.

A force curve measures the tip deflection (the force the tip sees) as a function of distance from the sample surface. As the tip approaches a surface, it initially doesn't interact with the sample and the force curve looks flat. When it gets close enough to the sample to see attractive forces (van der Waals), the tip is suddenly pulled towards the surface and there is a kink in the force curve. As a larger force is applied, the cantilever presses on and indents the sample. If the cantilever is stiffer than the sample the slope of this linear part of the force curve identifies the stiffness of the sample (if the cantilever is too soft, this slope reflects the stiffness of the cantilever more than the sample).



As you then retract the tip from the surface, the cantilever is still pulled towards the sample until it suddenly breaks free of the attractive forces. This snap-back point (d) location and shape is a function of the adhesion of the sample, the water layer on the surface (via capillary forces), and any other surface contaminants.

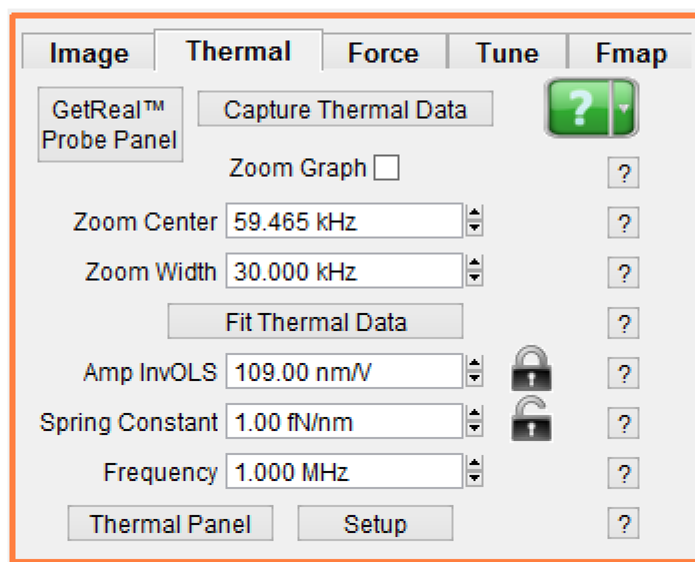
The raw units of a force curve is Volts (deflection of the tip), but this can be converted to a force if the spring constant of the cantilever is measured. Knowing this one can extract values for the elastic properties of the sample off of the force curve.

Operation

1. Calibrating the tip spring constant (if desired)

a. Load your tip and a very hard sample (eg. a glass slide).

b. Determine the thermal resonance frequency of the tip. Go to the *Thermal* tab of the *Master Panel*, and click *Capture Thermal Data*. Wait until you can see a peak, then stop collecting. Zoom into the fit and *Fit Thermal Data*.



c. Determine the virtual deflection offset (Defl InVOLS). This levels the flat part of the force curve (when it is in free air, far above the sample).

i. Carefully approach. In the *Force* tab of the *Master Panel* set up and run a force curve (see step 2 for more details). Slide the red bar on the left of the panel all the way to the top so that it gets a large region of the curve in to be in air. Example parameters are a Force Distance of 0.5 – 1 µm, a relative trigger of 0.4 - 0.6 V, a velocity of 1µm/s.

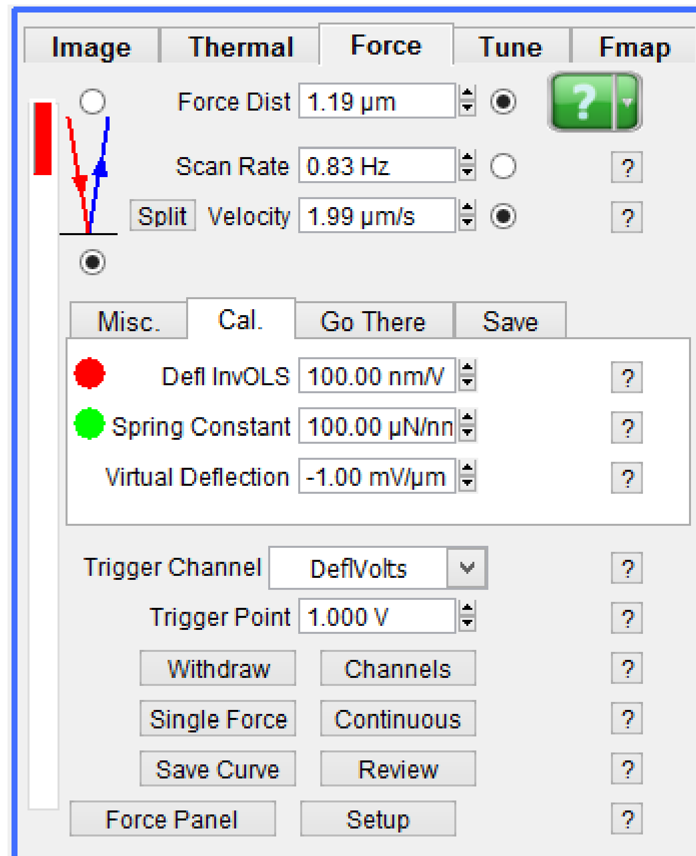
ii. Fit the slope of the long linear region on the right of the force curve. Press Crtl+I to bring up cursors and place them on the line. Click *Setup* and in the *Cal* tab

select *Virtual Defl Line* from the *Set Sensitivity* menu. Remove the fit by right clicking on the line and choosing *Remove fit_DeflVolts*.

d. Determine the InVOLS (inverse optical lever sensitivity), which relates the deflection to the height.
    i. Run another force curve. Fit the linear part on the left side of the force curve (the right side should be flat now). Choose *Defl InVOLS* from the *Set Sensitivity* dropdown menu this time.
e. Once you have values for these three things, the software will calculate your cantilever's spring constant.

2. Running a force curve

a. Load a tip and sample as in contact mode, but use a tip with a higher spring constant than for contact mode imaging.
b. Go to the *Force* tab to run a force curve. Adjust the parameters and click *Single Force* to run one curve (or *Continuous* to keep running them).
    i. The veolcity is the speed at which the tip approaches the surface.
    ii. The *Force Distance* and the *Trigger Point* determine the depth that the force curve will go (how much penetration into the sample). The curve will go until it reaches the distance or the trigger, whichever comes first.
    iii. There is a white bar with a red rectangle in it on the left hand side of the panel. This is a visual representation of the force curve distances, with the size of the red rectangle representing the depth of the curve (the Force Distance), and the location representing the start height (the top of the white bar is far away from the sample). You can click and drag to change. After the first force curve, the software knows the sample height and will automatically adjust the start height to have half the curve above the sample and half in contact.
    iv. Normally you set the *Trigger Channel* to *DeflVolts* to use the deflection of the cantilever as the stopping point. If you did calibration, you can use *Force*.



c. If you want you can do force curves on a specific part of your sample (if you have a heterogeneous material, or want to do lithography using a single point force curve).

  i. Take an image first (in contact or tapping mode).

  ii. Go to the *Go There* tab. Choose *Pick Point* and move the crosshairs that appear on your scan image to the location you want to do a force curve. Click *That's It* when you have it where you want it.

  iii. Repeat to add more if desired. You can check *Show Markers* to see all of them.

  iv. Each one is labelled by a number. To go to a particular one, enter the *Spot Number* and click *Go There*. You can check *Show Tip* to see where you are (a red dot will show up on the image where the tip is). Click *Single Curve* to run a curve at that spot.

d. You can also run a map of force curves on your sample under the *Fmap* tab in the *Master Panel*.

Contacts

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