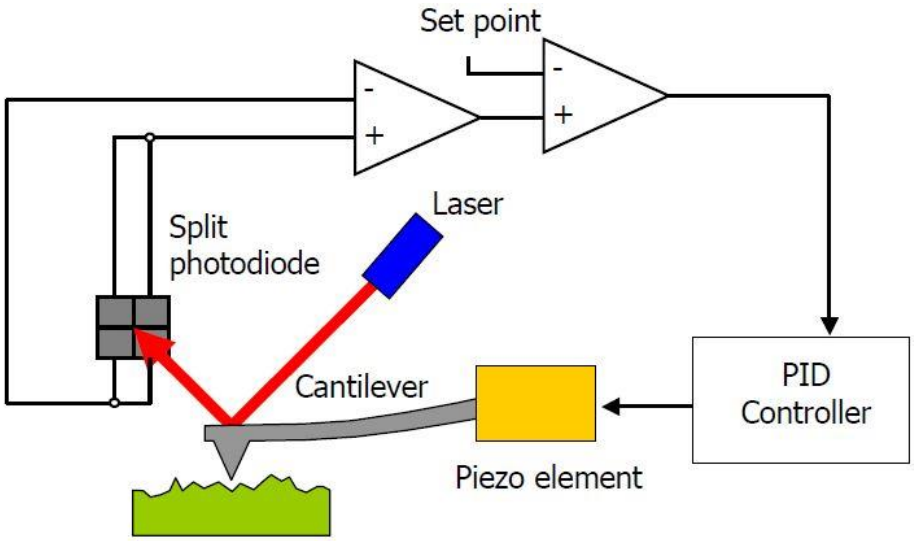
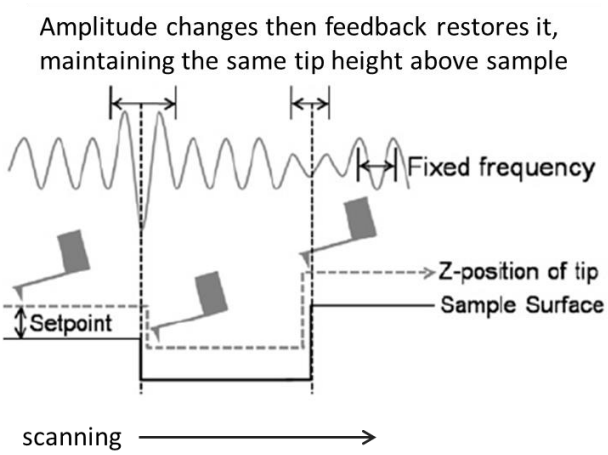
Topography (tapping and contact modes)

Principles of Technique

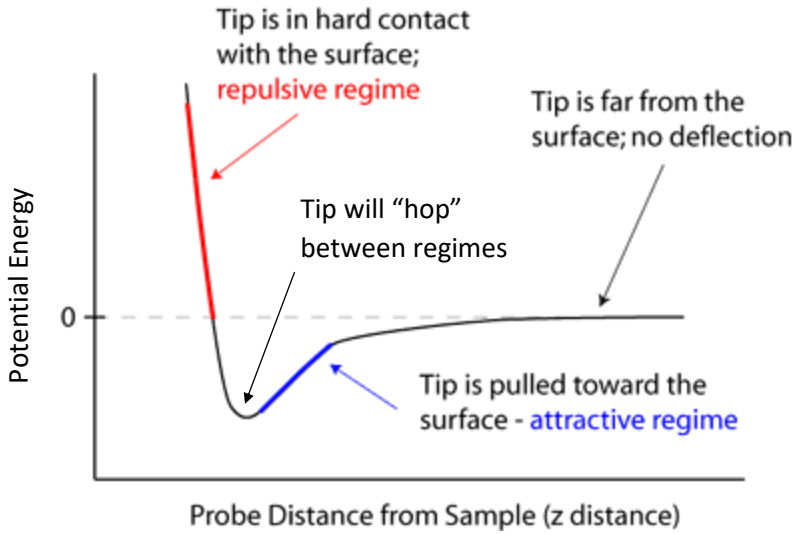
Atomic force microscopy gives information about the surface topography of a sample. It functions by moving a sharp tip at the end of a cantilever across the sample. A laser spot is reflected off the backside of the cantilever onto a photodiode. As the tip goes over features the cantilever will be deflected, 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.



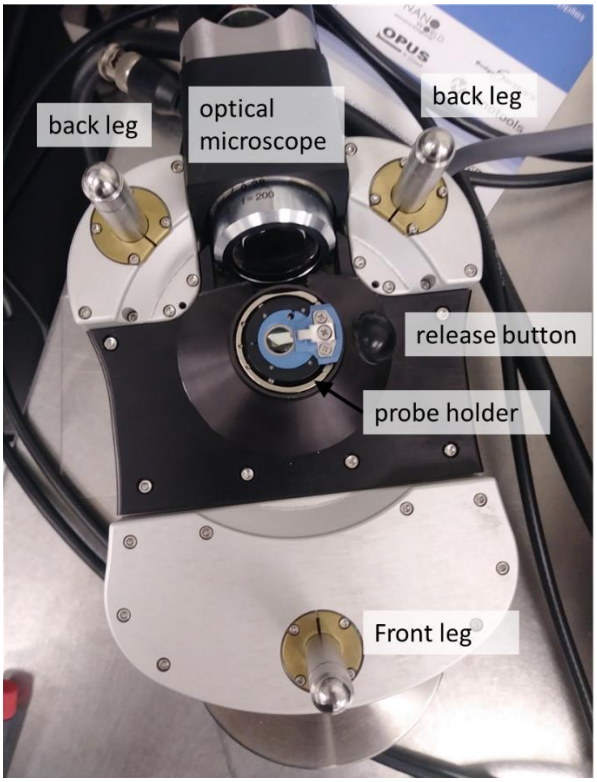
Sample topography can also be obtained in contact mode, where the tip will be in full contact with the surface instead of intermittent. In this case the tip is not oscillating, and the actual tip deflection is measured rather than a change in oscillation amplitude. Scans are done at a constant force (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).

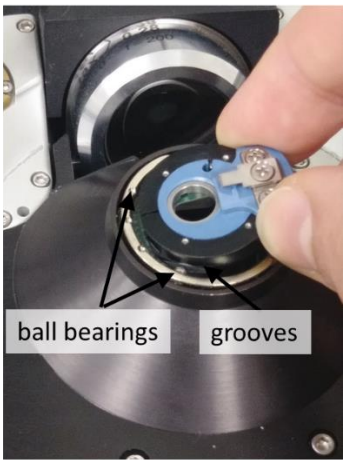
Basic Tool Operation (Tapping Mode)

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

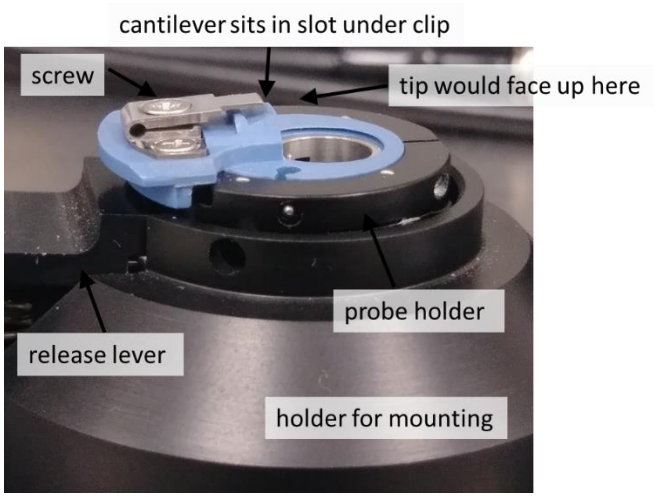
1. Mounting the AFM Probe

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.





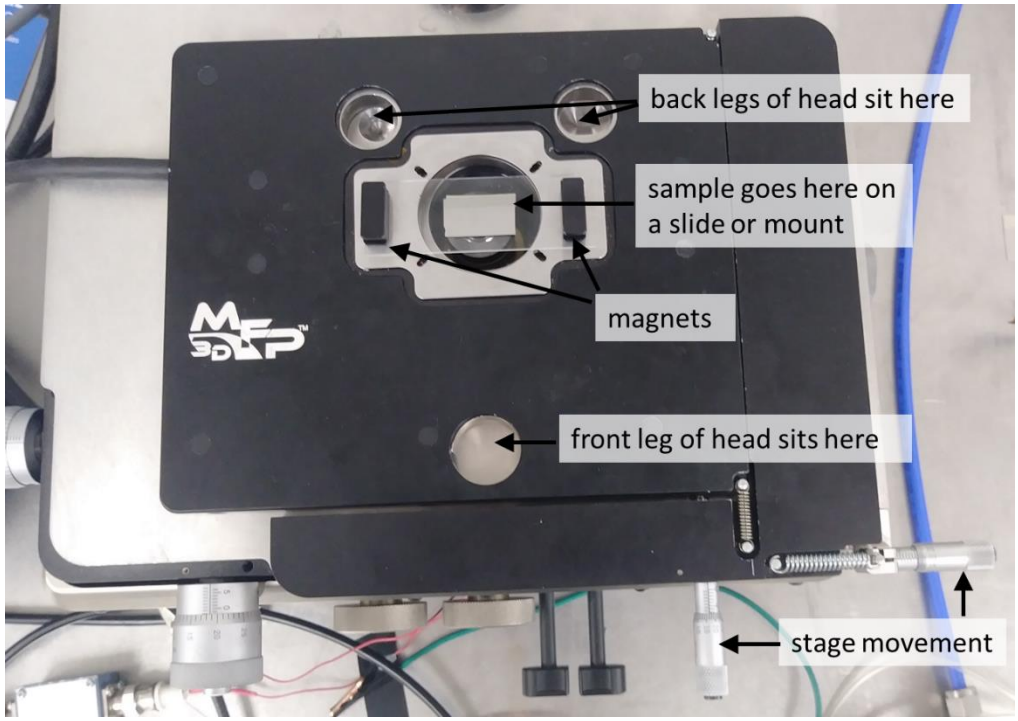
c. Place the probe holder on the black holder for mounting. Hold down the lever and insert the probe holder, with the two ball bearings sliding into the grooves in front. Release the lever to lock in place.
d. Use a Phillips screwdriver to unscrew the center screw slightly.
e. Carefully insert a cantilever 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**

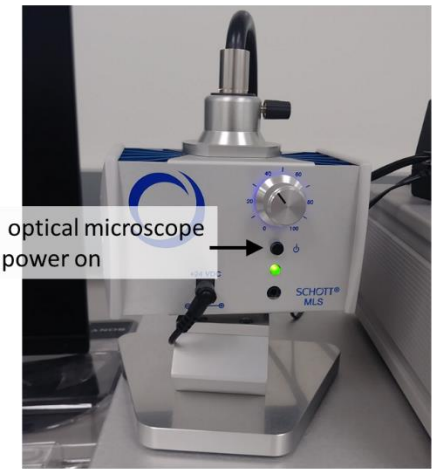
a. Load your sample. If your sample is small, fix it onto a glass slide using epoxy, Kapton tape, double-sided tape, etc. Put the glass slide onto the stage over top of the central hole and hold it in place using the magnets. If your sample is large enough to cover the hole then you can simply put it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

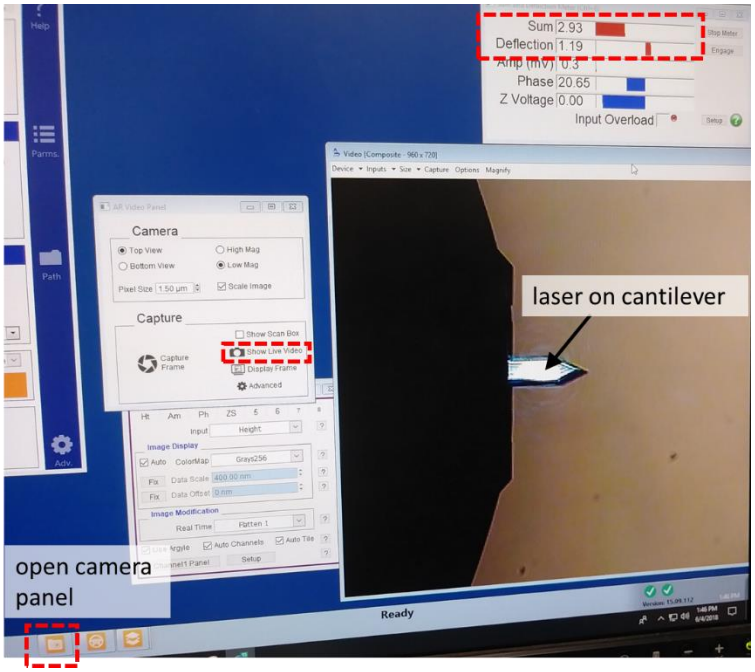


b. Lift up the head with two hands and flip it back over. Place the two hind legs of the head into the slots at the back of the stage first, then gently lower the front leg into the front slot. Make sure the probe does not collide with the sample, as this will render it unusable. If the probe looks like 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.

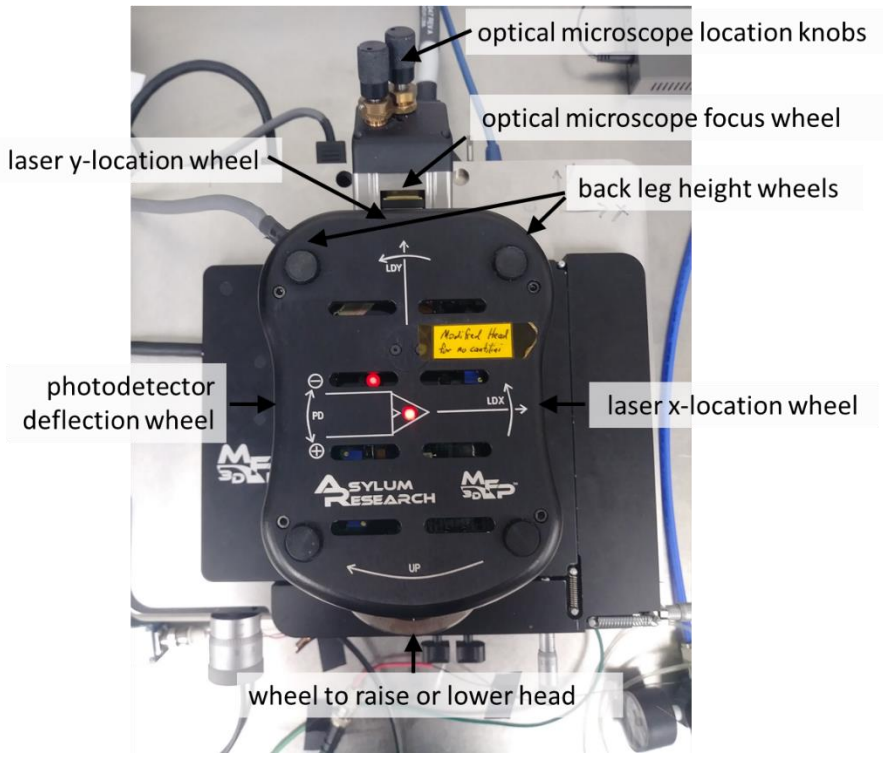
3. **Setting up the Camera**

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**

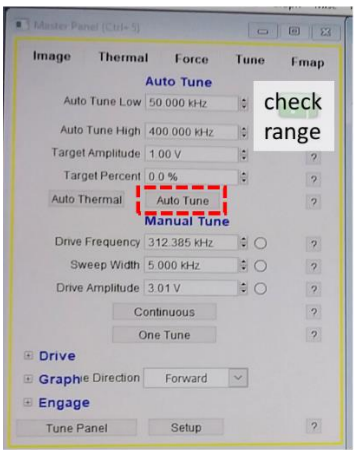
a. Align the laser spot on the cantilever, using the LD X and LD Y (Laser Diode X/Y) wheels at the back and right of the head. You should be able to see the laser shining on the tip once it is aligned.
    i. If the laser spot isn't seen, try moving it inwards using the LD X wheel first. Raster in a 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.

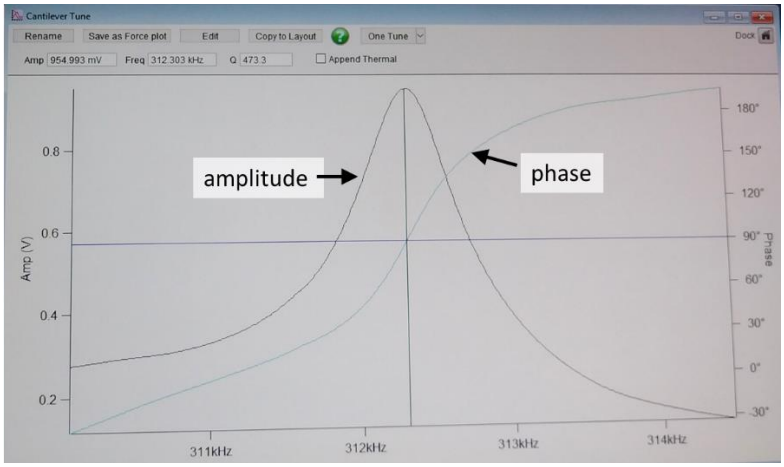
5. **Deflection and Tuning**

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.

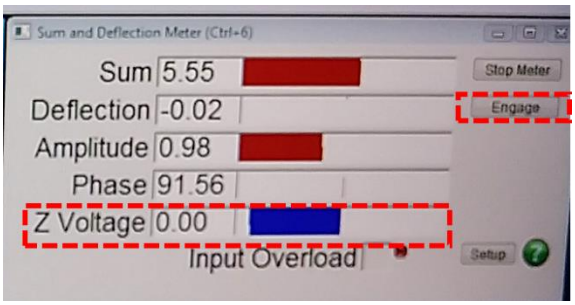
b. ***Click Engage***. If you don't click engage for the final approach you will smash the tip (engage turns the feedback on so if the tip gets too close it will lift)! You should see the Z-voltage bar fill to maximum. Lower the tip towards the sample by turning the front wheel to the right. When you 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.

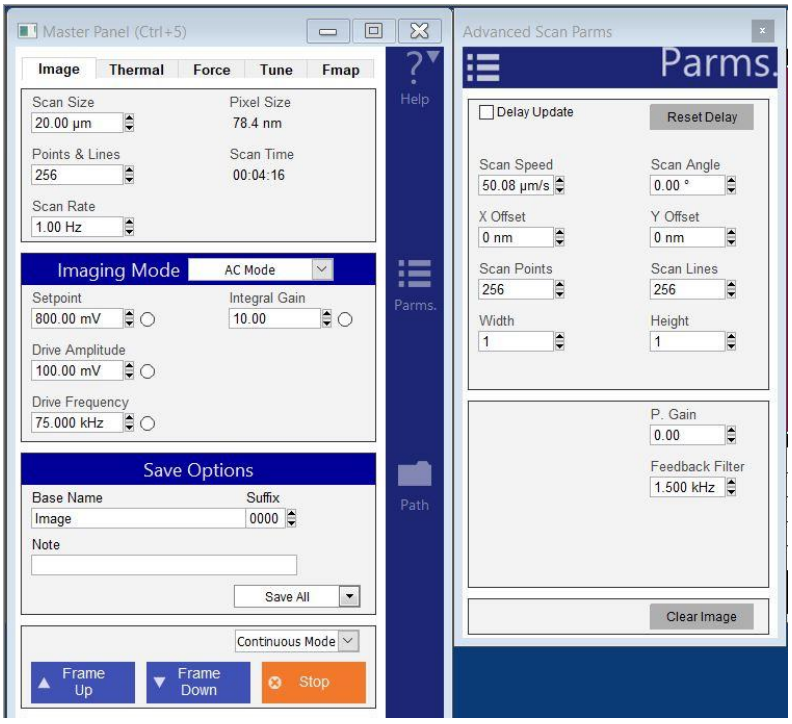
   ii. In the additional parameters panel you can set an x- and y-offset (up to ±45 μm). If you need to move further, withdraw the tip and use the stage micrometers. You can also change the scan angle.

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.

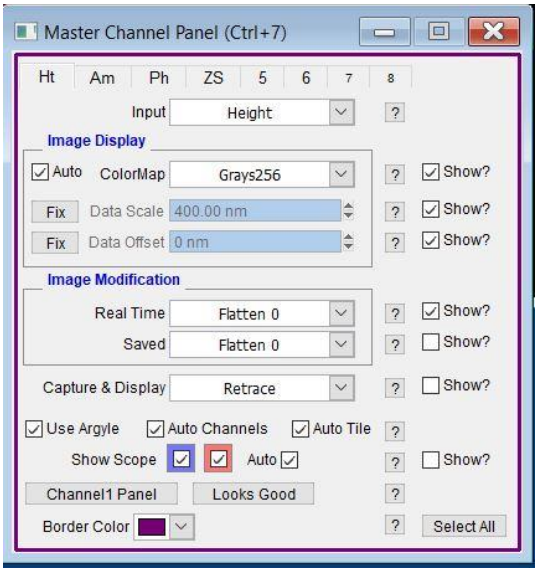



Hamster wheel to change parameters

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 resolution. 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).