

Dimension Icon/FastScan SOP

PeakForce Tapping

System Startup

- 1) Turn on the AFM and associated electronics by flipping the rocker switch on the power conditioning unit located next to the computer tower underneath the control desk. If the AFM controllers are already on, proceed to Step 2).

WARNING: Never power cycle the computer with the AFM on, as this could cause severe damage to the piezos!

NOTE: Allow laser to warm up for ~30 minutes prior to imaging if very precise measurements are required for your experiment.

- 2) Log in using the Excel sign in spreadsheet located on the upper right of the computer desktop.
- 3) Open the AFM control software by double clicking the Nanoscope icon on the computer desktop next to the login spreadsheet.
 - a) Check to make sure the microscope setup shown in the picture (e.g., FastScan or Icon head and associated optional modules) is the one desired. If not, click on the “Change Scanner” or “Change Microscope Setup” link as appropriate (Figure 1). Otherwise, proceed to Step 4).
 - b) Follow the onscreen directions to install the correct AFM head. After switching to the correct head, Nanoscope will ask you to shut down the software. Click Ok and restart the software.
- 4) Select the appropriate Experiment Category, Experiment Group, and Experiment (based on the AFM mode best suited to your sample and desired data), then click “Load Experiment” (Figure 1).

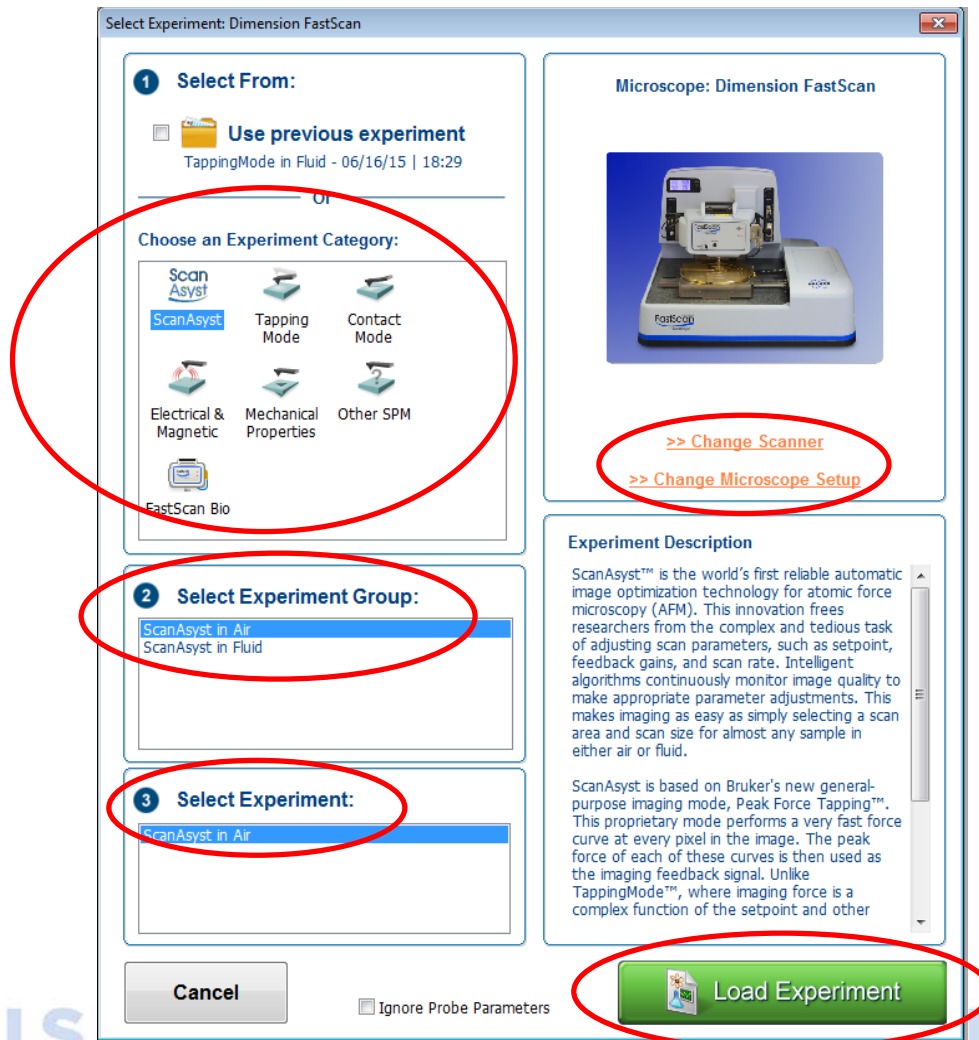


Figure 1. Experiment and microscope selection page.

NOTE: The following two sections describe the differences between setting up the Icon head versus the FastScan head for imaging.

Icon Head Setup

The Icon head is preferable for larger area scans (up to 90 μm x 90 μm) and is required when using the various optional modules for electrical imaging modes. However, the drawback to the Icon head is that it cannot image at as high a PeakForce frequency as the FastScan head (2 kHz vs 8 kHz maximum, respectively). The Icon head and associated optical assembly (video camera) can be seen set up in Figure 2. If you are using the FastScan head, skip ahead to Step 11).

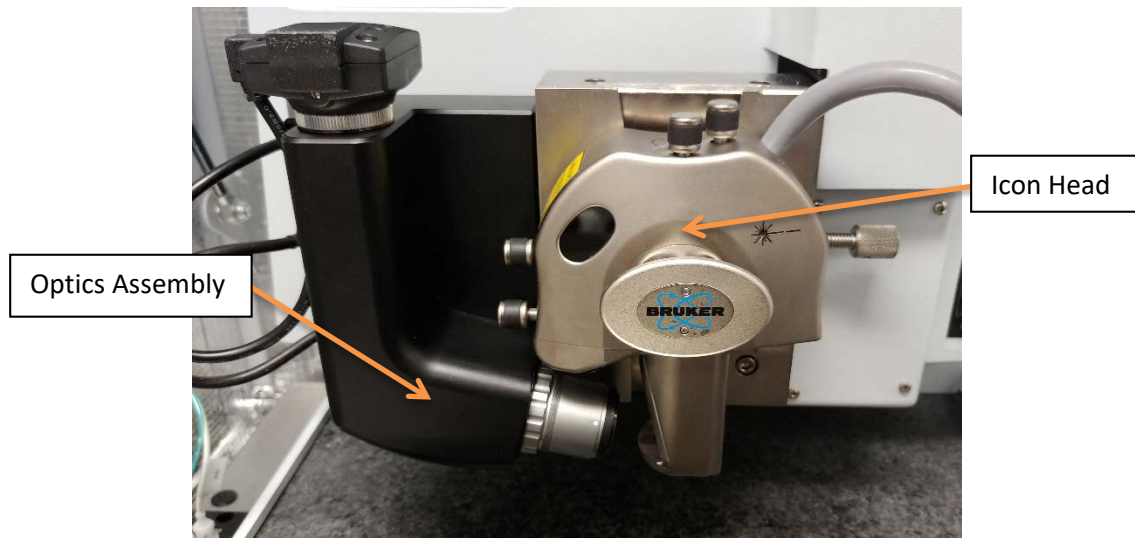


Figure 2. The Icon head and optical assembly (video camera) installed on the AFM.

- 5) Load a probe on the Icon head.
 - a) Before loading the probe, mount a probe holder on the Icon probe mounting block (Figure 3) to provide a stable surface while loading.
 - b) Mount a probe on the probe holder and secure it with the probe holder spring clip (Figure 3 and Figure 4).
 - i) Ensure the probe is parallel to and equidistant from all 3 sides of the channel in the probe holder by observing it under the optical microscope. Ensuring the probe is not touching the back of the holder will greatly reduce noise by eliminating coupling/transfer of any mechanical vibrations to the probe.
 - ii) Gently manipulate the probe with a pair of tweezers as necessary to align it properly. See Figure 4 for a detailed schematic of the probe holder.

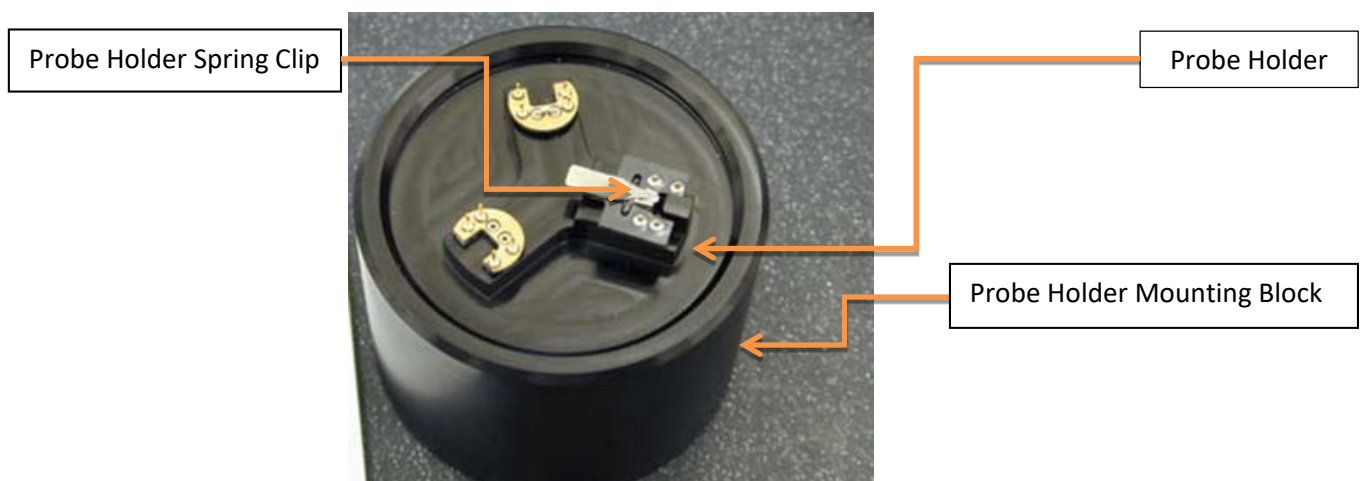


Figure 3. Probe holder mounting block with three probe mounting stations.

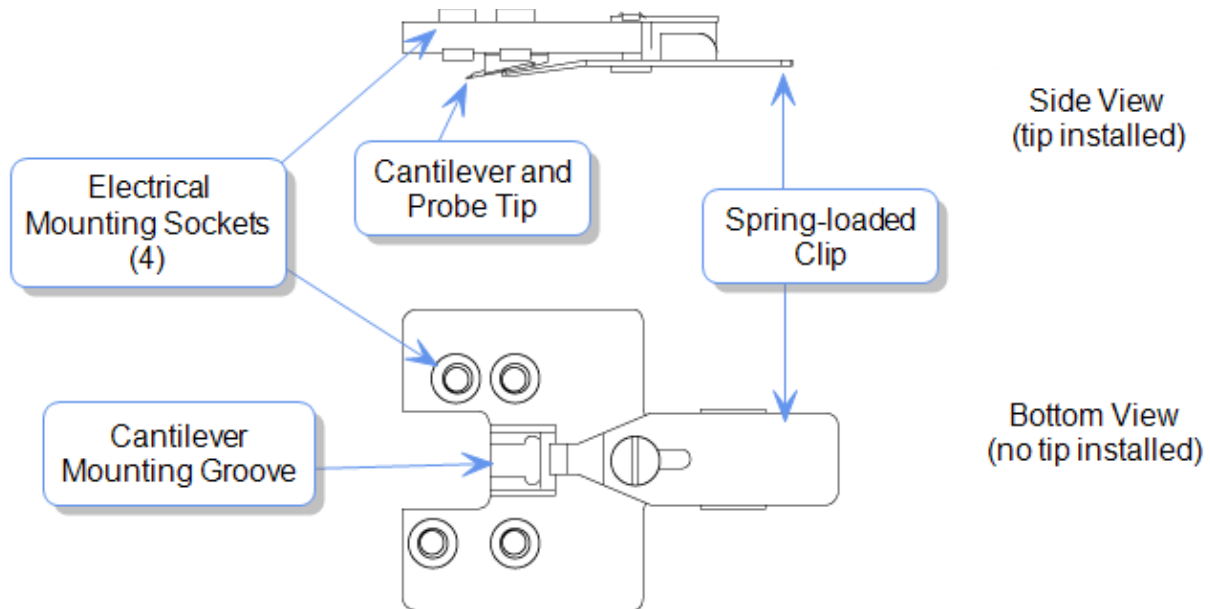


Figure 4. Schematic of the Icon head probe holder.

- 6) Unlock the AFM head from the dovetail rail by turning the set screw's knurled knob counterclockwise (Figure 5).

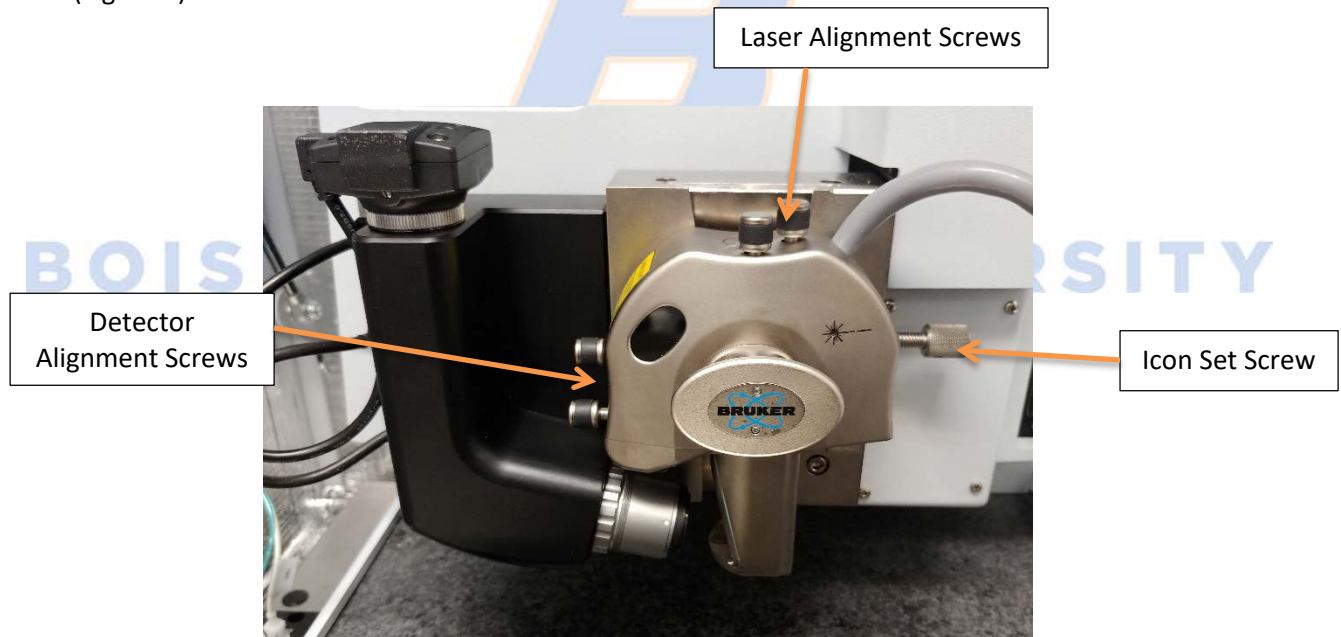


Figure 5. Icon AFM head showing the location of the different screws.

- 7) Grasp the handle on the front of the Icon head and carefully remove the AFM head by sliding it up the dovetail holder. Install the probe holder and probe by aligning the holes on the probe holder (Figure 4) with the contact pins on the AFM head (Figure 6).

WARNING: Handle the Icon head with extreme care! Any impact to the head may cause severe damage to the piezo tube, which is very expensive to repair/replace.

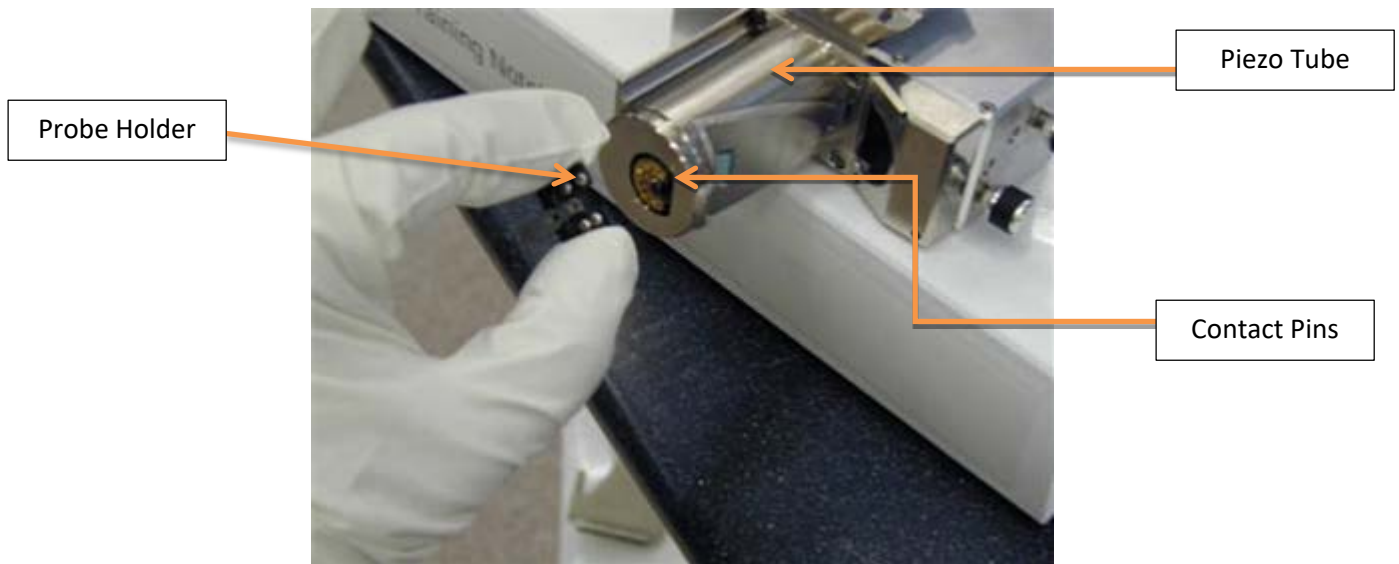


Figure 6. Installation of the probe holder on the Icon head.

- 8) After the probe holder has been installed, gently slide the Icon head back onto the dovetail holder using the attached handle and tighten the set screw. If the stage has not already been moved, use the Navigate window in the Nanoscope software to move the stage away from the Icon head (as shown in Figure 7).
- 9) Align the laser on the cantilever. You may either use the Alignment Station or place a small sheet of paper beneath the Icon head for ease of viewing the laser beam placement.
 - a) Press the display button above the Icon optics to show the SUM and vertical (VERT) and horizontal (HORIZ) deflection (Figure 7).
 - b) Use the *Laser Alignment Screws* (Figure 7) to adjust the laser until the cantilever is detected (red beam will appear split on the paper and SUM will increase drastically).
 - i) Move the laser side to side across the cantilever and watch to ensure the sum decreases as the laser drops off either side. The laser will be centered when the sum is at its maximum.
 - ii) Repeat this process moving forward off the tip of the laser and backward until the sum drops off again. The SUM should be at its maximum when the laser is properly centered (Figure 8).

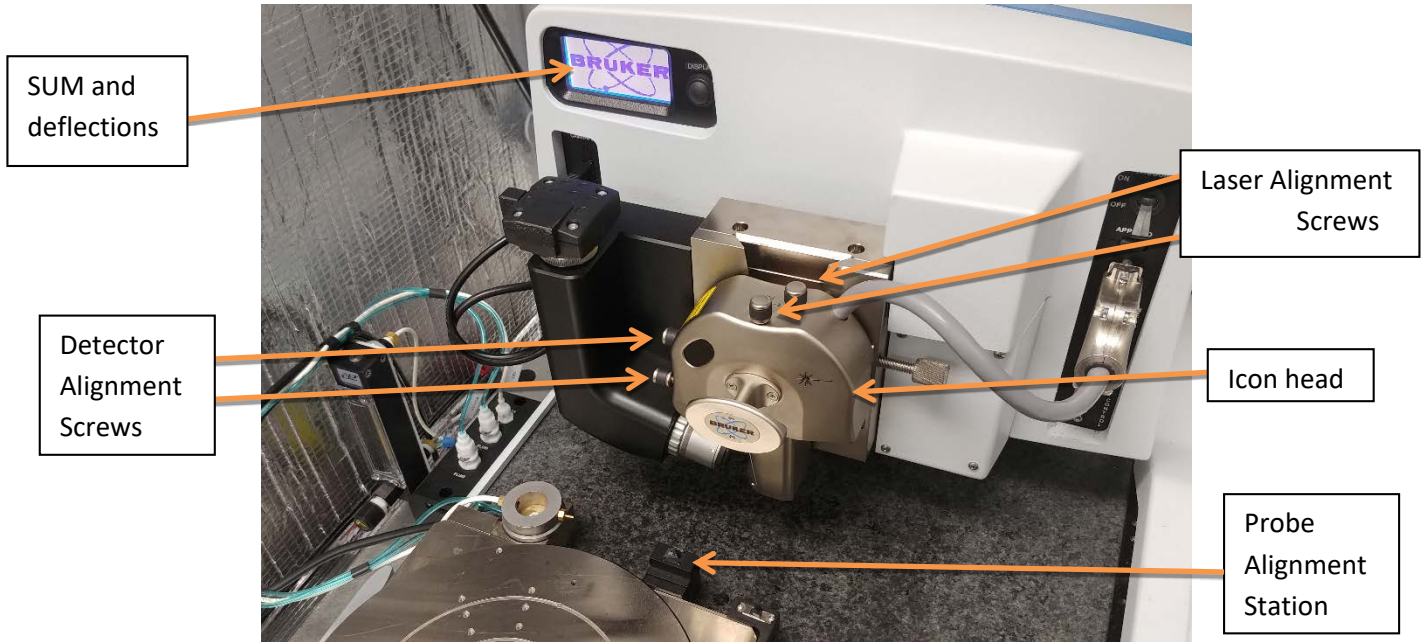


Figure 7. Alignment of the laser onto the probe cantilever.

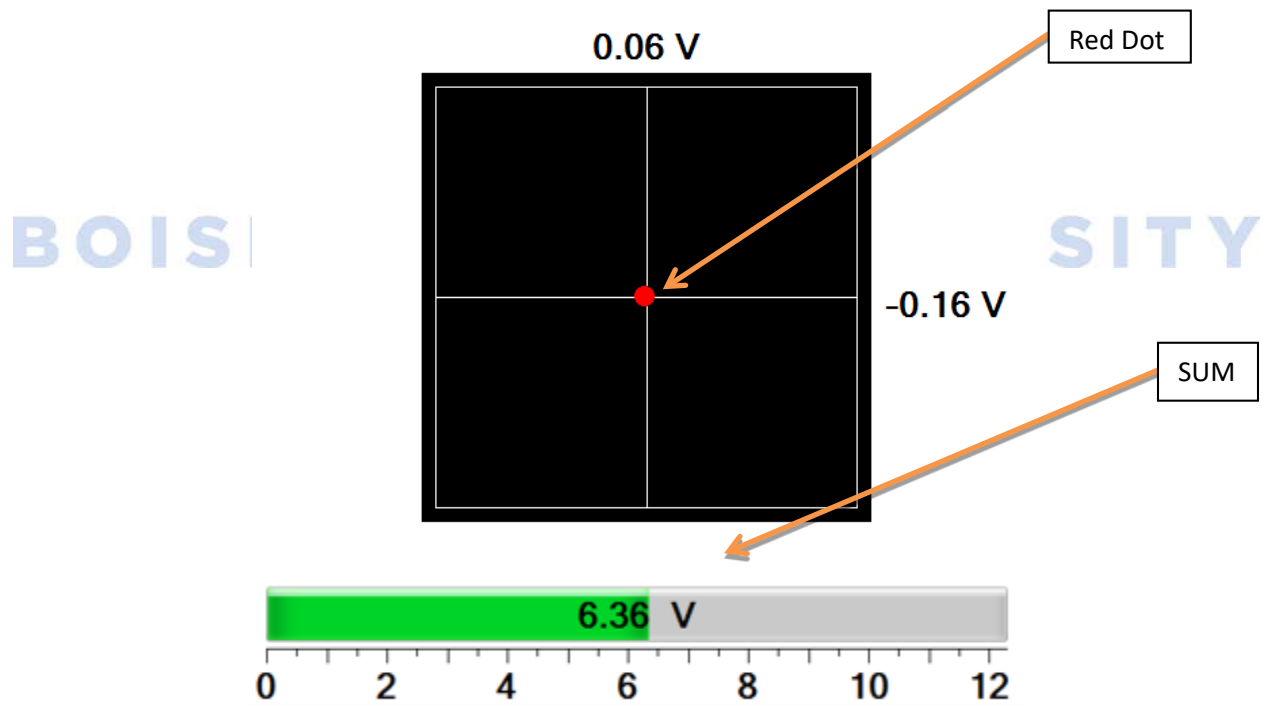


Figure 8. Position sensitive photodiode display window showing beam location and signal strength (SUM and Displacement).

- 10) Adjust the red dot (Figure 8) using the *Detector Alignment Screws* (Figure 5 and Figure 7) until the vertical and horizontal deflections are approximately zero (± 0.05 V).
 - a) If the SUM drops off, re-optimize the *Laser Alignment Screws*.

- b) Iterate adjusting the *Laser Alignment Screws* and *Detector Alignment Screws* until the SUM is maximized and the deflections are both approximately zero. This will ensure that the laser is centered on both the back of the cantilever and the detector, delivering accurate deflection signals and improving the quality and resolution of the scan. Skip ahead to Step 17).

WARNING: It is extremely important to make sure that the laser is aligned on the back of the cantilever, not the probe substrate. If the laser is aligned on the probe substrate, you will still get a good sum, but the sum won't drop off rapidly in all directions and the probe will crash into the surface when engaging.

FastScan Head Setup

The FastScan head is preferable for higher rate scans (PeakForce frequency up to 8 kHz). However, the maximum scan size when using the FastScan head is approximately $35\ \mu\text{m} \times 35\ \mu\text{m}$ (versus $90\ \mu\text{m} \times 90\ \mu\text{m}$ for the Icon head). If you are using the Icon head and have already installed a probe, proceed to Step 17); otherwise, follow the directions below to mount a probe on the Z-scanner and install it on the FastScan head.

- 11) Secure the Z-scanner (FastScan probe holder) to the FastScan probe mounting block (Figure 9).



Figure 9. FastScan Z-scanner (probe holder) and mounting block.

- 12) Using tweezers, gently lift the probe holder clip and swing it out to the probe loading position (Figure 10).
- 13) Install a probe in the probe mounting groove and bring the clip to the locked position. Ensure the probe is parallel to and equidistant from all 3 edges of the mounting channel by observing under the optical microscope. Gently manipulate the probe as necessary with a pair of tweezers.

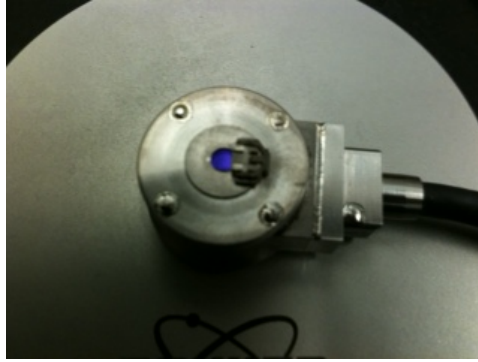


Figure 10. FastScan Z-scanner (probe holder) with clip in locked position.

- 14) Install the Z-scanner and probe on the FastScan head.
 - a) Ensure the Z-scanner fits snugly onto the head and seals by gently manipulating with two fingers until you feel it 'click' into place.
 - b) Flip the switch labeled Z-scanner Vacuum on the FastScan head to the On position. Gently wiggle the Z-scanner to ensure it is snug and level.

NOTE: If the Z-scanner is not installed correctly, issues will arise later during imaging.

- 15) After loading the Z-scanner, go to the Setup workflow window in Nanoscope (Figure 11).

WARNING: Ensure the High Voltage light is not illuminated prior to installing the probe holder. Failure to do so could result in electrical shock.

- a) Select "Setup" in the workflow column, then click "Change Probe" (Figure 11).
- b) The "Change Probe" dialog window will appear; select the "Move to probe loading position" icon (Figure 12). This will move the stage away from the head so that the probe holder can be loaded.
- c) Select the type of probe loaded for use during the experiment from the drop down menu under the "New Probe of type:" heading (Figure 12).
- d) Adjust the laser spot to the recommended size (Small/Medium/Large) using the laser adjustment switch located on the right side of the FastScan head.
- e) Click OK when finished.

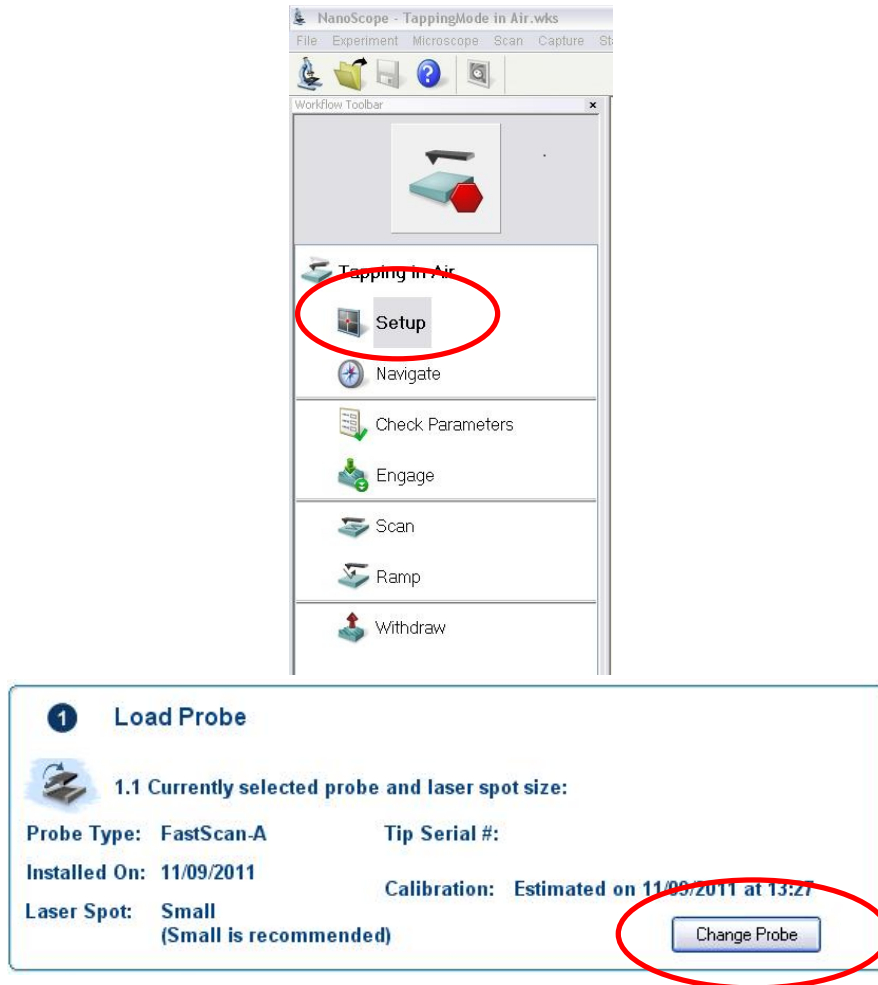


Figure 11. Workflow Toolbar with Setup circled at top. The "Change Probe" option is circled at bottom.

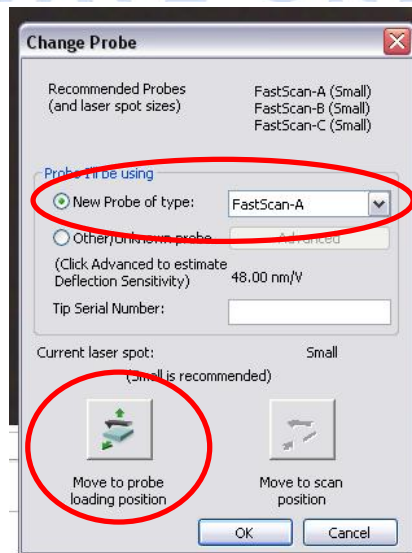


Figure 12. The Change Probe window.

16) Back in the Setup workflow window:

- a) Bring the tip into focus using the Up/Down focus controls (Figure 13). It's best to zoom in on the tip (2x or 3x magnification setting) before attempting to bring the edges of the tip into sharp focus.

NOTE: Adjustments can be made to optimize the view by zooming in or out on the video control, as well as increasing or decreasing the illumination.

- b) Align the laser towards the end of the cantilever where the tip should be by pointing and clicking on the video camera image or using the Align Laser controls (Figure 13). The precise location of the tip (i.e., tip setback from the end of the cantilever) depends on the type of probe used and can be found in the probe catalog description.
- c) Align the photodetector by pressing the "Auto-Align Detector" icon or manually moving the location of the laser's reflection with the arrows (Figure 13).

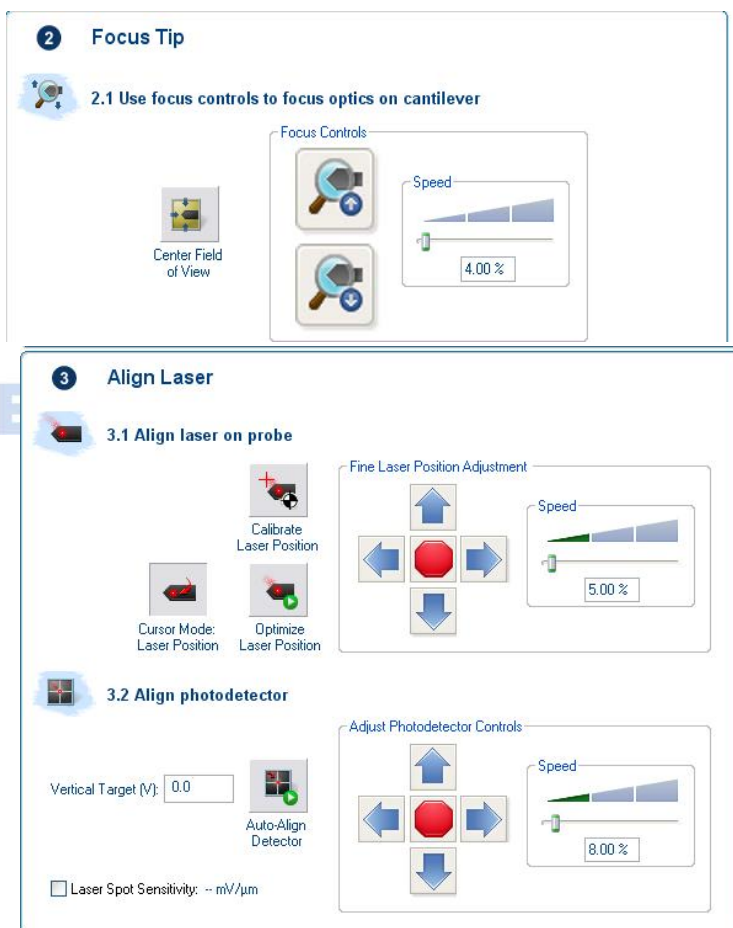


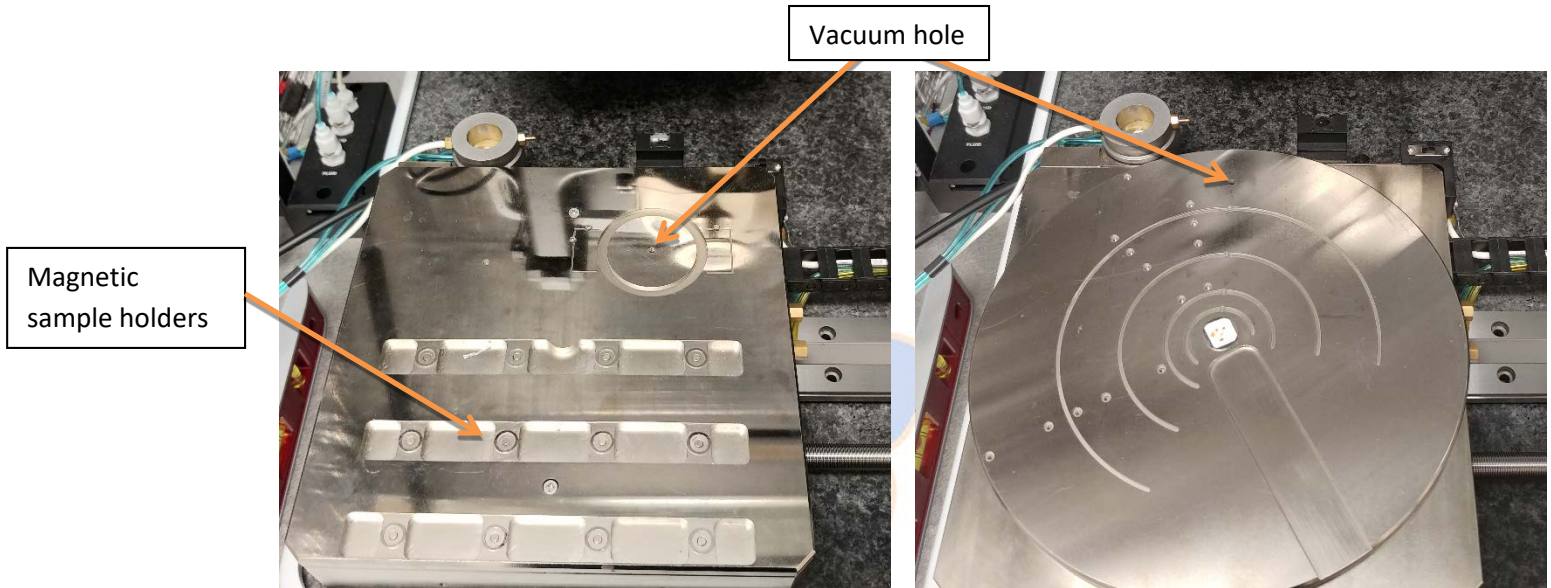
Figure 13. Focus Tip and Align Laser controls.

Approaching and Engaging the Surface

17) Load the sample and secure it using the stage vacuum (Figure 14), ensuring the Stage Vacuum switch is in the On position and the center hole (Figure 14, right) is covered.

- a) Alternatively, the magnetic sample mount, cross-sectional sample holder, or rectangular multi-sample chuck can be used depending upon your sample.

NOTE: The circular wafer sample chuck can be manually rotated for ease of loading the sample.



BOISE STATE UNIVERSITY Figure 14. Wafer sample chucks. Multi-sample (left) and Rotating (right)

- b) Now that the sample is secured, rotate the sample chuck to position the sample beneath the AFM head and probe.

WARNING: Rotate the chuck slowly and visually check that the probe and head are clear of the sample to ensure the tip and/or sample is/are not damaged. Use caution when adjusting tip height using the Z motor; set speed low initially to reduce the risk of damage to the head, probe, and sample. Remember, the piezos located in the AFM head are fragile and expensive to replace!

18) Go to the Navigate window in the Nanoscope Workflow Toolbar (Figure 11).

- a) To lower the probe near the sample surface without crashing the tip, you can choose to focus on either the surface or the tip reflection.
 - i) In either mode, bring the surface into focus by moving the scan head down (Figure 15).
 - ii) If employing tip reflection mode, after getting the sample surface in focus, continue moving the scan head down until the probe is visible again and in focus.

NOTE: Use Tip Reflection mode to give yourself an extra millimeter of safety when focusing if the surface of the sample is reflective and/or does not have any defects to focus on. When the system is properly focused, the probe will be ~1 mm from the surface. When in Tip Reflection mode and the surface is in focus, the probe will be ~2mm from the surface.

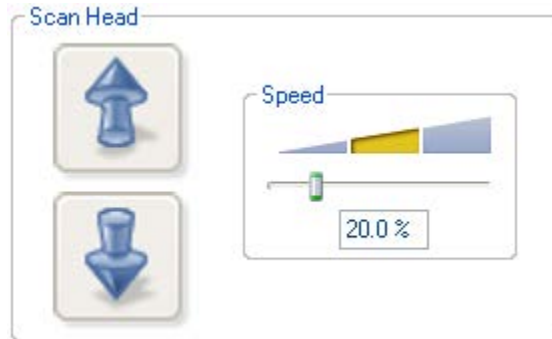


Figure 15. Z-scanner head movement.

WARNING: Use caution when focusing on the surface using the Z motor. Always move the probe away from the surface initially and check the speed. Set speed to a low speed initially to reduce the risk of damage to the head, probe, and sample.

- b) Use the stage direction controls (Figure 16) to move the sample around until the desired imaging area is centered under the crosshairs.

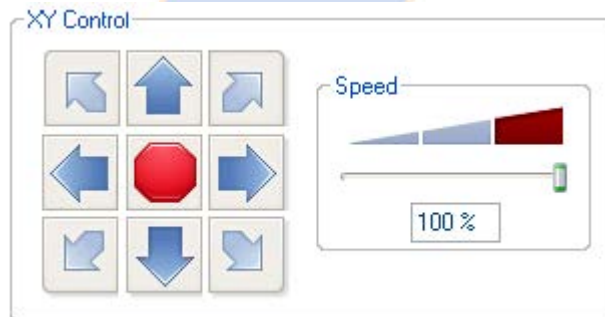


Figure 16. Stage movement direction and speed controls.

- 19) Close and lock the acoustical isolation chamber (hood) enclosing the AFM.
- 20) Prior to engaging the surface, go to the Check Parameters window in the Workflow Toolbar (Figure 11).
 - a) Zero the scan size and offsets so as to not damage the tip during the engage.
 - b) Set the Engage Setpoint to 0.1 V by selecting Microscope from the toolbar and then opening Engage Settings from the drop down box.
- 21) Click Engage in the Workflow Toolbar (Figure 11).
- 22) Ensure the probe engaged the surface (i.e., Trace and Retrace lines track each other).
 - a) Improve the force curve by right-clicking the graph (Figure 17) and selecting Autoconfig.

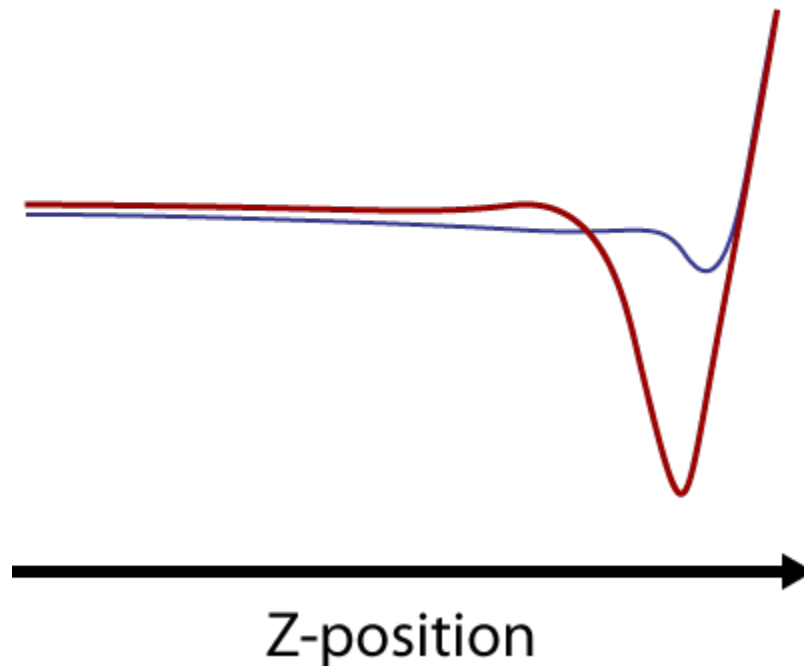


Figure 17. Ideal force curve shape for PeakForce Tapping mode.

Imaging

Optimizing Image Quality

- **Feedback Gain**

- The feedback gain controls how much the probe responds to a given change in sample height.
 - The higher the feedback gain, the greater the adjustment to a given change in topography.
- As the feedback gain is increased, the noise also grows. Eventually the additional noise outweighs the improvement in tracking. Noise can be examined graphically on the Amplitude Error channel.
- Increase the feedback gain incrementally (in increments of 5-10) until the noise grows substantially. Once this occurs, back off the feedback gain by 1-2 increment (e.g., if the noise increases greatly at a feedback gain of 50, then back the feedback gain off to 40-45).
- Typical feedback gain range for the Icon/FastScan is between 20 and 55 (although extreme cases outside these values can occur).

- **Peak Force Setpoint**

- The Peak Force Setpoint sets the maximum amount of force the tip exerts on the sample surface during each force curve before retracting.
- The force applied can be determined through calibration of the deflection voltage signal on the photodetector.

- The conversion uses the deflection sensitivity (signal in Volts on the photodetector for a given deflection in nm) and Hooke's Law, $F=-kx$, where k is the spring constant of the cantilever (found on the probe box), and x is the distance the cantilever flexes.
 - A good starting value is ~ 0.02 V (~ 1 nN of force) for a ScanAsyst type probe.
 - The Peak Force Setpoint can be increased if the tip is not tracking well, but should be used as one of the last resorts to cleaning up the image, as it will lead to increased tip and sample wear.
- **LP Deflection BW**
 - The LP Deflection BW minimizes the noise on the force curve.
 - The lower the LP Deflection BW value, the lower the noise will become. This noise can be observed at the beginning of the force curve, prior to the drop off in force (Figure 17).
 - A good value to start with is ~ 40 kHz. This value can be decreased in increments of 5 kHz until the noise on the force curve is minimized without affecting the nanomechanical property measurements (i.e., distorting the shape of the adhesion portion of the force curve).
- **Peak Force Amplitude**
 - Peak Force Amplitude measures the height the tip lifts off the surface during each force curve along the surface of the sample.
 - The optimal Peak Force Amplitude will vary based on the general roughness of the surface and amount of tip-sample adhesion.
 - If the surface is very smooth (e.g., DNA on mica) and there is minimal adhesion (low humidity and a new probe), the Peak Force Amplitude can be as low as ~ 10 nm (20-30 nm is typical).
 - Conversely, if the surface is rough (e.g., unpolished metal) and/or there is a lot of adhesion, the Peak Force Amplitude should be greater (50-150 nm).
 - A good value to start with for a known rough surface would be ~ 100 nm. A good value to start with for a known smooth surface would be ~ 30 nm.
 - A general rule is that the Peak Force Amplitude should be greater than the lowest valleys or the highest peaks so that the tip won't crash into a large feature on the sample.
- **Peak Force Frequency**
 - Peak Force Frequency is the number of force curves recorded per second.
 - A higher Peak Force Frequency allows faster scanning and/or better S:N via more averaging.
 - Too high a Peak Force Frequency can lead to oscillations in the force curve, degrading quantitative nanomechanical (PeakForce QNM) measurements.
 - The choices available on the FastScan head are 1, 2, and 8 kHz, while the Icon has a maximum Peak Force Frequency of 2 kHz.
- **Z limit**
 - The Z limit limits the range of movement of the Z piezo. It is used to control the height resolution and inherent noise in the Z height measurement.
 - The lower the Z limit, the lower the noise and higher the height resolution.

- Be sure the Z limit is large enough so that all features on the sample can be imaged without damaging the Z piezo.
- Parameters that can affect the Z limit include:
 - Surface Roughness
 - The smoother the surface, the lower the Z limit can be. The rougher the surface, the higher the Z limit needs to be.
 - Sample Tip/tilt
 - The sample is never perfectly perpendicular to the AFM probe.
 - The tip/tilt of the sample can be observed by changing the *RT Plane Fit* to *None* on the height channel. The change in height across the sample can be observed now.
 - Peak Force Amplitude
 - The lower the PF Amplitude, the lower the Z limit can be.
 - Thermal Drift
 - The more the temperature around the sample and tip changes, the more the Z limit needs to be increased.
 - If the door on the AFM acoustical enclosure is closed, the AFM electronics will heat up more, leading to thermal drift over time. However, if the door is open, acoustical noise may appear in the AFM images. Thus deciding whether thermal drift or acoustical noise will affect the data more should be thought over prior to running the scan.
- A good value to start with is the maximum allowed by the AFM head. This value can be minimized to 1 μm . A Z limit below 1 μm is not suggested except for very small scan sizes on extremely small samples, in which case a Z limit of 500 nm is acceptable.

Capturing Images

23) Select the proper folder from the directory tree in which to save the image.

24) Give the image a logical filename (e.g., include the date, scan size, and sample).

25) Under the Capture drop down bar there are different options for capturing an image.

- a) **Capture**: Saves the next complete, uninterrupted (i.e., no changes made to scan parameters) frame with the chosen filename to the selected directory.



- b) **Capture Now**: Immediately saves the current image before completing the frame.



- c) **Capture Last**: Saves the last complete, uninterrupted frame prior to the current one.



- d) **Capture Continuous:** Continuously scans and saves frame after frame until the tip is withdrawn or capture is aborted.



- e) **Capture Withdraw:** Saves the next complete, uninterrupted frame and then withdraws the tip from the surface.

- f) **Abort Capture:** Cancels the current capture status.



System Shutdown

- 26) Once imaging is complete and all images are captured and saved, select Withdraw from the Workflow Toolbar column (Figure 11).
- 27) Move the tip further away from the sample and sample chuck by opening the “Setup” workflow window and clicking the “Change Probe” icon (Figure 11).
- 28) Open the AFM acoustic enclosure.
- 29) Slowly rotate the sample chuck to allow access to the sample.
- 30) Flip the Chuck Vacuum switch to the Off position and remove the sample.
- 31) Remove the probe holder and probe.
 - a) For the Icon head, unlock the AFM head by turning the locking screw to the right, then carefully slide the head up the dovetail rail using the handle so that the probe holder can be removed from the bottom of the Icon head.
 - b) For the Icon FastScan head, remove the Z-scanner by turning off the Z-Scanner Vacuum switch and pressing the Z-Scanner Release button while simultaneously gently wiggling the Z-Scanner downward, carefully holding it with two fingers.

WARNING: Handle the AFM head with extreme care! Any impact to the head may cause severe damage to the piezos, which are very expensive to repair/replace.

- 32) Place the probe holder on its corresponding mounting block.
 - a) Gently remove the probe using a pair of tweezers.
 - b) Place the probe back in the box and location it was removed from, ensuring the probe is turned perpendicular to unused probes (to indicate that it has been used).
 - c) Detail the condition of the probe on the box label.
- 33) Close the Nanoscope software.
- 34) Complete the Excel log in spreadsheet on the computer desktop with the number of hours and new probes used. Be sure to note any problems encountered and the solutions to those issues.