

Kelvin Probe Force Microscopy (KPFM)

Standard Operating Procedure for KPFM on the Dimension Icon

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Introduction to KPFM

Kelvin Probe Force Microscopy, or KPFM, is a form of scanning probe microscopy that combines AFM with highly localized surface potential measurements, thereby permitting concurrent measurement of sample topography and work function (i.e., surface potential) at a scale of tens of nanometers. The work function is defined as the minimum amount of work required to completely remove an electron from a solid (i.e., the energy necessary to move an electron from the Fermi level to a vacuum immediately outside the solid). Different materials will exhibit different work functions based on their composition and electronic structure. Thus KPFM can be used to identify and characterize nano or microstructural phases within a sample, often in combination with complementary techniques such as SEM-EDS. It should be noted that the surface potential measured in KPFM is the difference in work function between the sample surface and the probe (i.e., the relative surface potential). However, it is possible to make absolute surface potential determinations if the probe is first calibrated by carrying out KPFM on a standard reference sample consisting of a material with a known work function such as gold.

In addition to KPFM, the related technique of EFM (electrostatic force microscopy) can be utilized to measure differences in surface potential across a sample. EFM directly measures the electrostatic forces experienced by the probe tip through tracking changes in the amplitude, frequency, or phase of the probe's oscillation as it rasters at a fixed distance above the surface. In KPFM, an alternating current (AC) voltage is applied to the probe. A difference in electrostatic potential between the probe and the sample surface will cause the probe to oscillate due to this applied AC bias. By applying an additional DC bias to the probe to null its oscillation at the AC frequency, the contact potential difference between the sample surface and the probe can be determined (i.e., $V_{CPD} = -V_{DC}$). Although KPFM is more complex to implement than EFM because it involves nulling the probe-sample surface potential difference (also known as the contact potential difference, or CPD), it is more sensitive, better for quantitative measurement of surface potentials, and less prone to topographical artifacts than EFM.

Available KPFM Modes

There are a variety of different possible implementations of KPFM, depending upon the AFM mode chosen for the topography scan and exactly how the surface potential is detected and nulled. This leads to four different KPFM modes being available on the Icon system, as summarized in Table 1.

Table 1. Available KPFM Modes

Topography/AFM Mode	Tapping	PeakForce Tapping
Amplitude Modulation (AM)	AM-KPFM	PF AM-KPFM
Frequency Modulation (FM)	FM-KPFM	PF FM-KPFM

Topography Measurement: Tapping vs. PeakForce Tapping

Traditionally, KPFM has been combined with tapping (intermittent contact or AC) mode AFM. Tapping mode requires a tune to be performed to determine the natural resonant frequency of the probe, followed by optimization of the amplitude setpoint and integral and proportional (I and P) gains. When combined with frequency modulation (FM) KPFM detection, tapping mode KPFM can be carried out in a single pass, which is convenient but susceptible to topographical artifacts since sample height and surface potential are being measured simultaneously. In the case of PeakForce Tapping, force curves are rapidly acquired at a fixed frequency (0.25-2 kHz) with a user-defined peak force to map out the sample's topography. In addition to affording direct control over tip-sample forces (useful for minimizing tip wear and/or damage to fragile samples), PeakForce Tapping allows for greater ease of use via ScanAsyst as well as acquisition of potentially useful nanomechanical data channels (e.g., adhesion, deformation, dissipation, and Young's modulus).

Surface Potential Detection: Amplitude vs. Frequency Modulation

The contact potential difference between the sample and probe can be detected by means of either amplitude modulation (AM) or frequency modulation (FM). In amplitude modulation, an AC bias is applied to the probe as it is rastered over the sample at a user-determined fixed height above the surface in lift mode after having first scanned the line to determine the sample's topography. The CPD causes the probe to oscillate at the applied AC frequency, which is typically chosen to be at or near the probe's natural resonance frequency to enhance probe responsiveness and sensitivity. An additional DC bias is then applied to null the amplitude of the probe's oscillation, at which point $V_{DC} = -V_{CPD}$.

In FM-KPFM, the probe is mechanically oscillated at its resonance frequency f_0 by the dither piezo while an AC bias is applied at a different (lower) frequency (typically 2 kHz). Again a DC bias is applied to null oscillation of the probe due to the applied AC bias, although in this case the oscillations being nulled are the sidebands at $f_0 \pm f_{AC}$. In the case of tapping mode FM-KPFM, the topography and surface potential measurements can be carried out simultaneously during a single pass. In contrast, PF FM-KPFM utilizes lift mode, sequentially scanning topography and surface potential, similar to AM-KPFM. While single pass tapping FM-KPFM is thus $\sim 2x$ faster than PF FM-KPFM, it is more susceptible to topographical artifacts as noted above.

Important Considerations: Potential Artifacts, Sample Prep, & Resolution

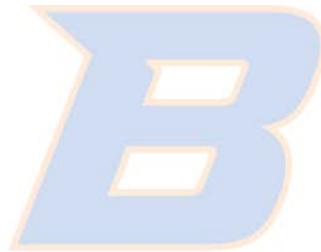
For all KPFM modes, it is essential to verify that contrast observed in the potential channel is truly due to differences in surface potential, not topography. For the three KPFM modes that employ lift mode (i.e., all but the single pass tapping mode FM-KPFM), this can be done by increasing the lift height. Since van der Waals forces drop off much more rapidly with distance than electrostatic interactions, a feature in the potential channel that disappears with a small increase in lift height is likely a topographical artifact. A good rule of thumb is to start with a lift height that is approximately equal to the height of the tallest topographical feature in the area being scanned. Note that with single pass tapping FM-KPFM, there is not an equivalent way to check for the presence of topographical artifacts, which is one reason why PF FM-KPFM is often preferable even though it requires somewhat longer acquisition times due to the dual pass nature of lift mode.

One drawback to increasing the lift height in the dual pass KPFM implementations is that spatial resolution in the surface potential channel scales with lift height. Thus one typically should choose the lowest possible lift height that does not lead to topographical artifacts in the potential data. Because of this, surface roughness of samples to be imaged with KPFM should be minimized when possible through mechanical polishing, ultramicrotomy, etc. However, care must be taken to ensure that samples are well rinsed/cleaned after polishing to remove any polishing compound residue. In addition, differences in material hardness can lead to differential polishing rates (and hence topographical features), and possible reactions between the sample and polishing compounds and/or cleaning solvents should be considered. Finally, many metals can develop a surface oxide, which may affect the measured potential. Thus, careful sample preparation is key to obtaining meaningful, reproducible KPFM data.

In addition to lift height (and to a lesser extent, probe radius of curvature), the potential detection method employed (AM vs FM) will affect the spatial resolution of KPFM measurements. The spatial resolution of FM-KPFM is significantly greater than that of AM-KPFM due to the fact that FM-KPFM detects changes in the electrostatic force gradient rather than the electrostatic force itself (as is the case in AM-KPFM). As a result, a larger fraction of the signal in FM-KPFM comes from the probe tip rather than the cantilever at a given lift height. Accordingly, AM-KPFM images will often appear “smoother” due to spatial averaging. However, although FM-KPFM images may appear noisier, the increased spatial sensitivity enables detection of small micro or nanostructural phases and more accurate determination of surface potential differences between such phases and the surrounding bulk matrix.

Suggested Default: PeakForce FM-KPFM with a PFQNE-AL

For the best combination of speed, ease of use, spatial resolution, repeatability of potential measurements, availability of additional data channels, and freedom from topographical artifacts, PF FM-KPFM is suggested as the default mode of choice. The best probe in terms of spatial resolution and reproducible surface potential measurements is Bruker's PFQNE-AL. In particular, the lack of a metal coating on the PFQNE-AL probe tip itself leads to a smaller tip radius and more consistent potential measurements since metal coatings can wear down and flake off with use. However, other modes (as described above) and probe types (e.g., SCM-PIT or MESP) can be used. Thus users should first learn about the various available modes and probes, then talk with the SSL staff and try several out to determine what best meets their project needs. The Select Experiment menu in NanoScope provides a good summary of the available modes in the Experiment Description section (see Figure 1).



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General KPFM Procedure (Dimension Icon)

- 1) Log in on the Excel sign in spreadsheet at the upper right of the computer desktop.
- 2) Open the NanoScope AFM control software.
- 3) Select the appropriate KPFM Experiment (based on the KPFM mode best suited to your sample and project needs), then select “Load Experiment” (Figure 1).

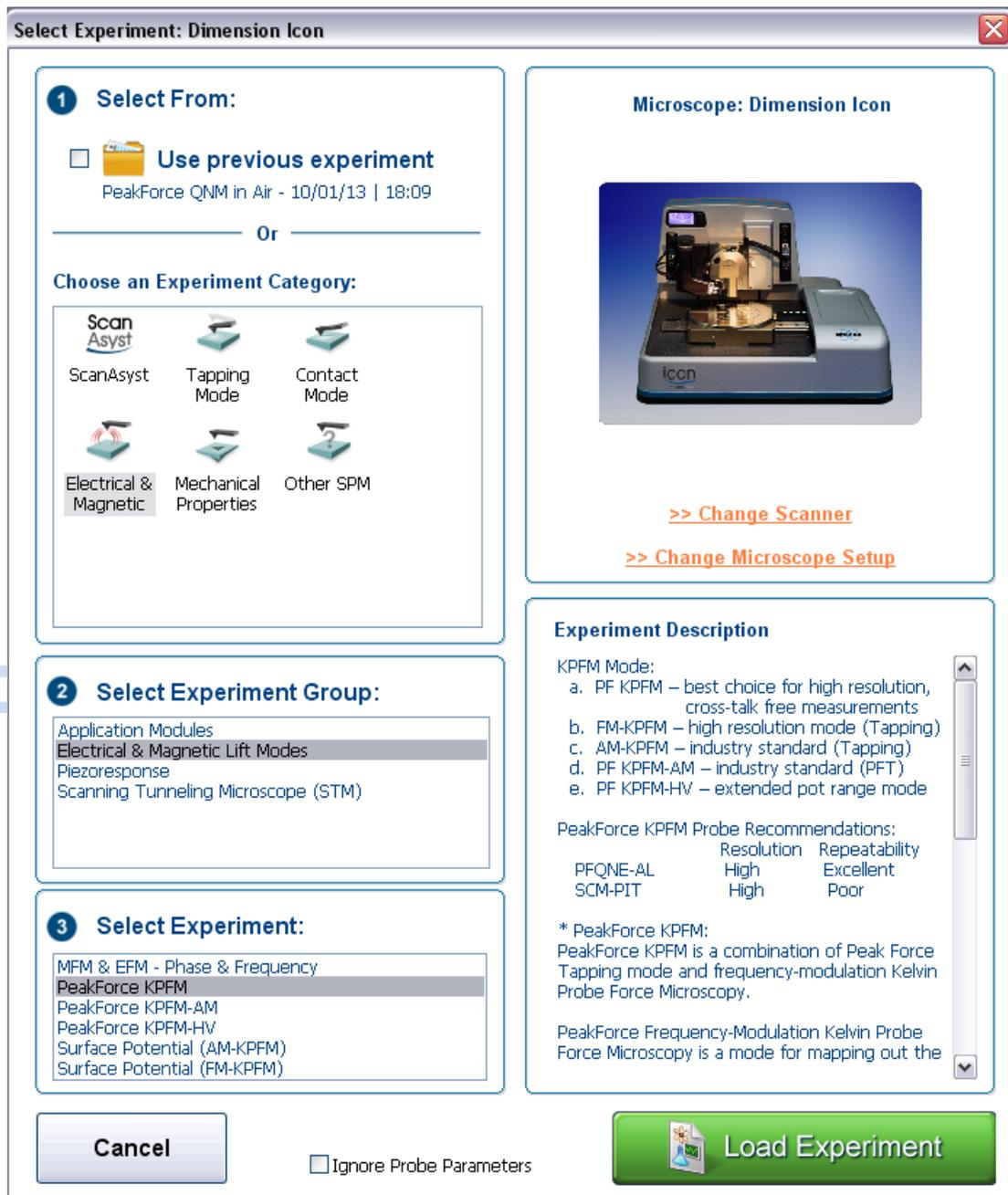


Figure 1. Select Experiment screen for choosing a KPFM mode on the Dimension Icon system with suggested default mode (PF FM-KPFM) selected.

NOTE: Allow the laser to warm up for ~30 minutes prior to imaging if very precise measurements are required to ensure pointing stability.

WARNING: NEVER power cycle the computer with the AFM, NanoScope controller, and/or associated electronics on; voltage spikes could cause severe damage to the electronics and piezoelectric actuators.

- 4) Mount a conductive probe (e.g., PFGNE-AL, SCM-PIT, MESP, etc.) and install the Icon head.
 - a) Place the probe holder on one of the three stations atop the probe holder mounting block (Figure 2).

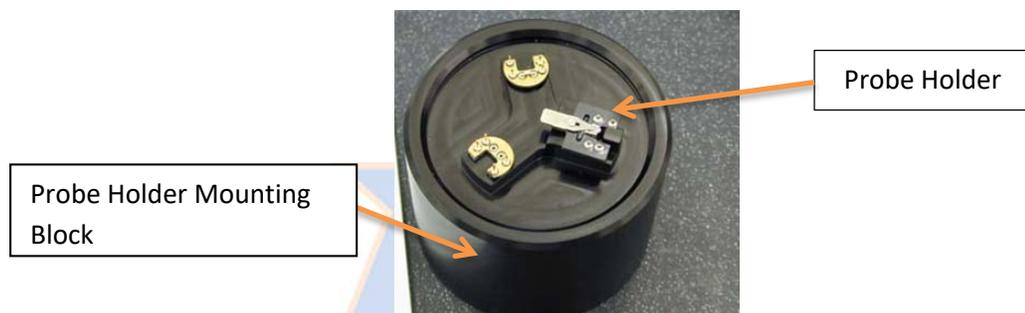


Figure 2. Probe holder mounting block with three probe mounting stations.

- b) Place a conductive probe in the cantilever mounting groove on the probe holder and secure it with the spring-loaded clip (Figure 3). Ensure the probe is parallel to and roughly equidistant from all edges. Confirm the probe is not touching the back of the groove by observing it under an optical microscope. Gently adjust the probe as necessary with a pair of tweezers.

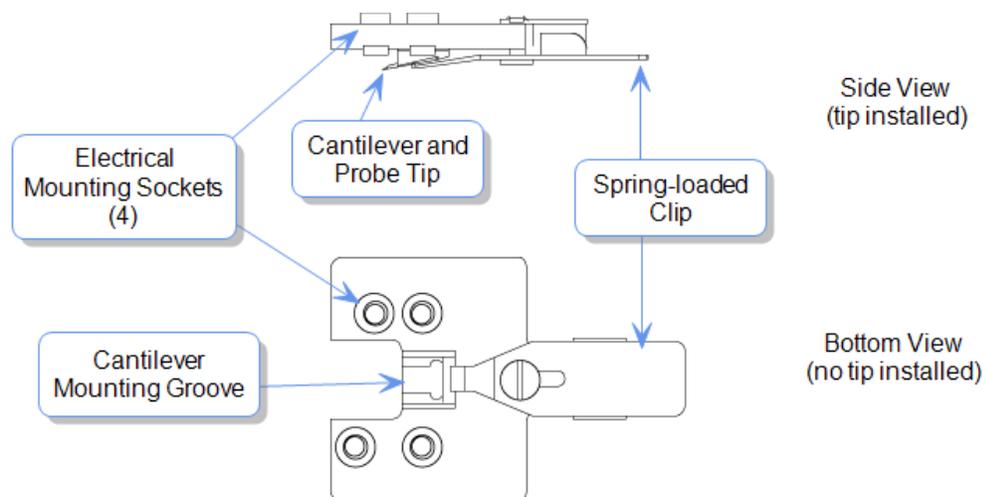


Figure 3. Schematic of the probe holder for the Dimension Icon head.

- c) Loosen the Icon head from the AFM by rotating the set screw counterclockwise (Figure 4).

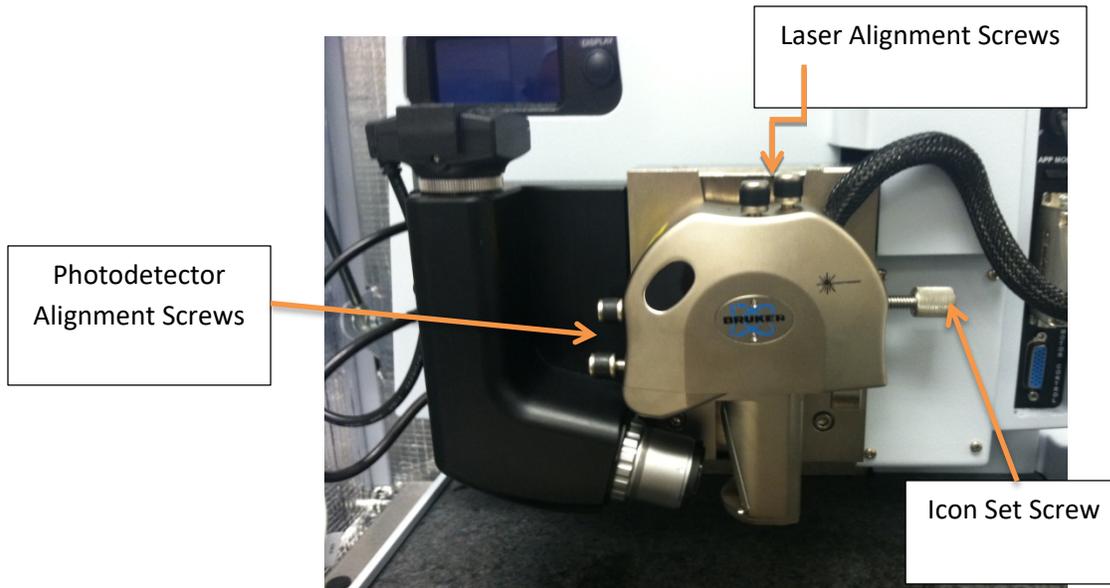


Figure 4. Dimension Icon head with alignment and set screws labeled.

- d) Carefully remove the Icon head by sliding it up the dovetail rail. Install the probe and probe holder by aligning the holes on the probe holder with the contact pins on the head (Figure 5).

WARNING: Handle the Icon head with extreme care! The piezoelectric scanner tube and associated closed loop sensors are very sensitive/fragile and expensive to repair/replace.

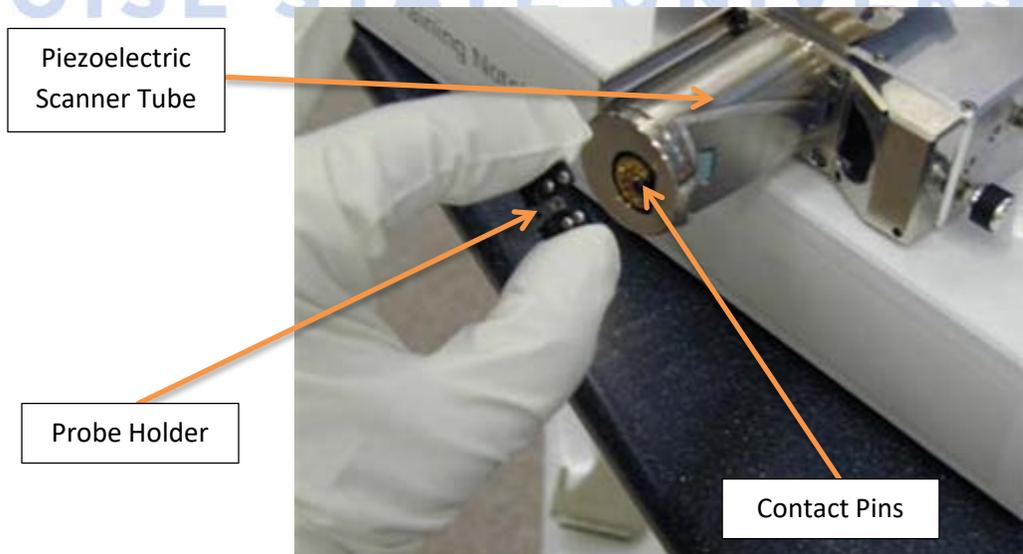


Figure 5. Installation of a probe holder onto the Dimension Icon AFM head.

- e) Reinstall the Icon head by inserting it into the dovetail rail and gently sliding it downward until it reaches the bottom. Using a ball driver, tighten the set screw until it is snug. If applicable, reconnect the cable from the Icon head to the AFM and tighten the retaining screws.

WARNING: Do not jar the Icon head! The piezoelectric scanner tube and associated closed loop sensors are very sensitive/fragile and expensive to repair/replace.

- 5) Load a sample and turn on the stage vacuum (if necessary).

NOTE: The stage can be manually rotated for ease of loading the sample.

WARNING: Always rotate the stage slowly and visually check that the AFM head and probe clear the sample to prevent damage to the tip and/or sample. Use caution when focusing and approaching the surface using the Z motor; set speed low initially to reduce the risk of damage to the head, probe, and sample. Remember, the piezoelectrics located in the AFM head are fragile and expensive to replace!

- 6) Check to make sure the sample has good continuity using a multimeter. If there is not a good electrical connection between the sample and the stage/chuck, the potential channel data obtained during KPFM imaging will be noisy and/or erroneous.
 - a) Turn the knob on the multimeter to the audible diode/continuity check setting.



- b) Attach one lead to the sample surface and the other to the stage/chuck.
 - c) If there is a good electrical connection between the sample and the stage, the multimeter should emit a continuous tone. This means current is flowing between the multimeter leads through the stage and sample. If there is no audible tone (or the multimeter beeps intermittently), there is not a good electrical connection and you must reposition or remount your sample to establish a continuous connection.

NOTE: If your sample is mounted on a metal puck, applying a thin ribbon of silver paste from the puck to the top surface of the sample is often sufficient to ensure electrical continuity.

- 7) Align the laser on the probe cantilever.
 - a) Press the display button next to the LCD screen above the Icon head and optics so that it turns on to show the SUM and vertical and horizontal deflection readings.
 - b) Use a sheet of white paper to view the laser light leakage around the edges of the cantilever (Figure 6).

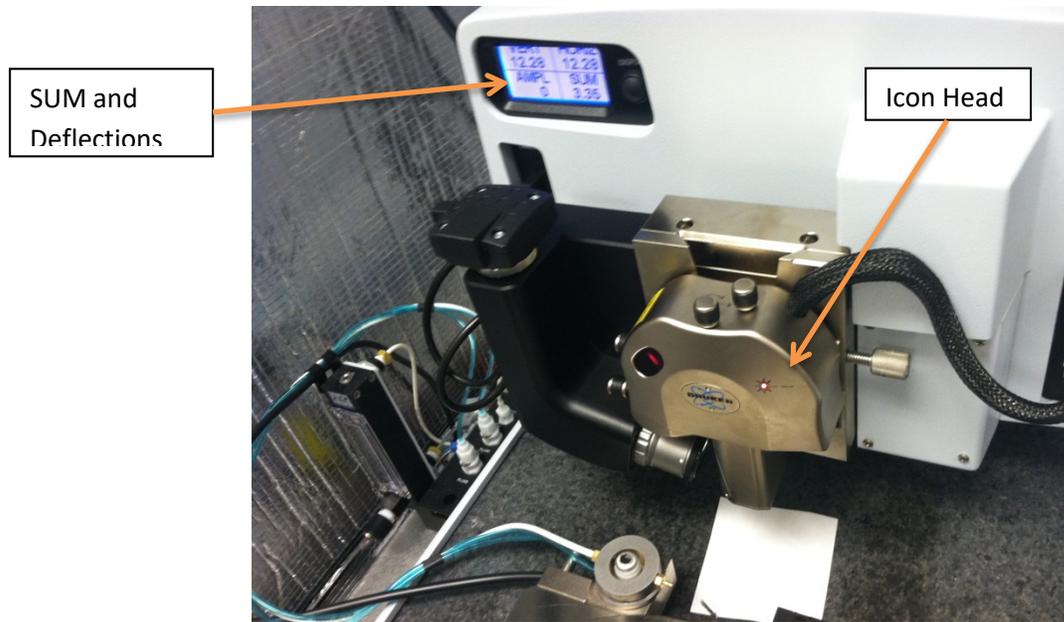


Figure 6. Alignment of the laser onto the probe cantilever.

- c) Use the laser alignment screws (Figure 4) to adjust the laser until the cantilever is detected (red beam will appear split on the paper) and the SUM is maximized.
 - d) Use the photodetector alignment screws (Figure 4) to center the laser reflection on the position sensitive photodiode (quad detector).
- 8) Go to Setup on the Workflow Toolbar column.
- a) Ensure the probe you are using is selected in software. If necessary, click “Change Probe” and select the correct type of probe from the dropdown menu (Figure 7).

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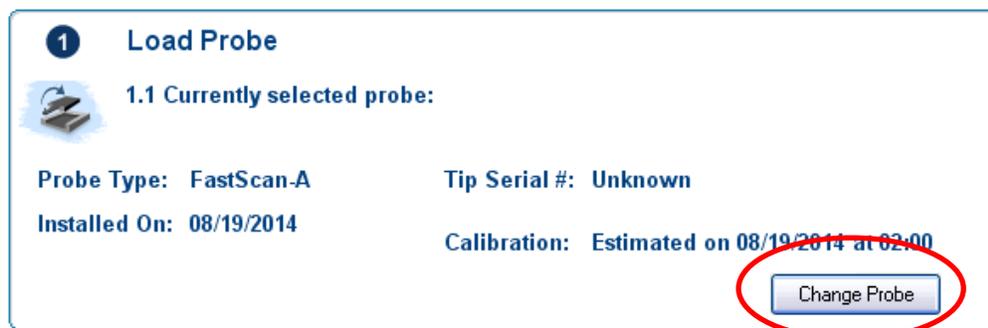


Figure 7. The “Change Probe” screen.

- b) Bring the tip into focus using the focus controls. Using slower motor speeds will prolong the life of the optics gears.

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.

- 9) Select Navigate in the Workflow Toolbar column.
 - a) The software will automatically carry out a fast thermal tune for the interleave mode at this point using the information in its probe database as a guide (which is why it is essential to select the proper probe from the Select Probe menu so that the software knows the approximate natural resonance frequency of the probe).
 - b) Cautiously approach the surface until the probe is within 1 mm of the surface (default Sample Clearance). This can be done by selecting either Sample Surface or Tip Reflection and bringing the appropriate object into focus (Figure 8).

NOTE: Use tip reflection mode when the sample surface is highly reflective and/or the sample does not have any visible defects on which to focus.



Figure 8. Z scanner head control interface for approaching/focusing on the surface.

WARNING: Use caution when focusing on the surface using the Z motor. Always move the tip 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/or sample.

NOTE: When the surface is in focus, the probe cantilever will be $\sim 1,000 \mu\text{m}$ above the surface. Although this can be changed under Engage Settings/Sample Clearance, it is NOT recommended to change this value. If however you are using a probe with an unusually long tip (e.g., pure platinum RMN probes), you may need to increase the SPM Safety Margin from its default value of $100 \mu\text{m}$ to prevent immediately crashing the tip upon commencing the engage process.

- 10) Use the stage movement controls (Figure 9) to position the desired area of the sample under the crosshairs in the optical viewing window.

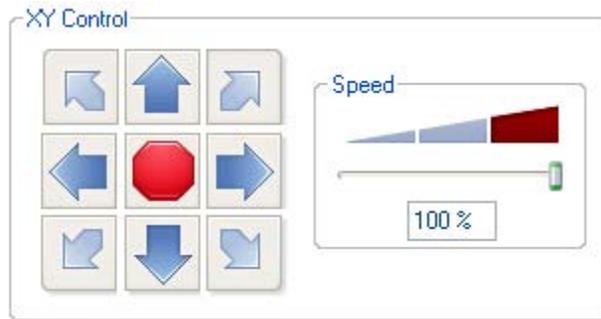


Figure 9. Stage control interface.

- 11) Close and lock the acoustic hood enclosing the AFM.
- 12) Prior to engaging the surface, check the relevant parameters listed below depending upon which KPFM topography mode is being employed.
 - a) For both tapping and PeakForce tapping:
 - i) Zero the scan size and offsets.
 - ii) Ensure the *Interleave Mode* parameter is set to *Disabled*.
 - b) For PeakForce tapping only, set the *Engage Setpoint* to *0.1V* and the *Engage Type* to *Standard*.
 - c) For tapping mode only: Set the *Engage Setpoint* to between *0.9* and *1.1*.
- 13) Click Engage on the Workflow Toolbar column.
 - a) Ensure the tip engages the surface.
 - b) If both the probe and surface appears in focus and the Z piezo meter is not overly extended or retracted (i.e., outside the green region), the tip engaged properly.
- 14) Optimize the topography imaging parameters.
 - a) Doublecheck that Interleave Mode is set to Disabled.
 - b) Open the Height Sensor and Error channels. Optimize tracking of the sample topography just as you would in regular Tapping or PeakForce Tapping mode (refer to the corresponding SOP for more detail).

NOTE: For all imaging modes, decreasing the scan rate (and thus the tip velocity) will improve image quality. This is particularly important in KPFM to minimize the introduction of topographical artifacts in the Potential channel. Be sure the scan rate is slow enough (often 0.1-0.2 Hz) to track the largest features/topography changes present on your sample. This will pay off in the long run by preventing phase jumps (which cause the active image to reset and prevent it from capturing) and improving tracking of the surface for artifact-free surface potential images in Lift Mode.

- 15) Optimize the KPFM parameters.
 - a) Change Interleave Mode from Disabled to Lift. Allow the AFM to scan several lines in lift mode.
 - b) Click the Set Phase button a few times in quick succession to set/optimize the lock-in phase and ensure it is stable (i.e., it only changes a few tenths of a degree each time you click Set Phase).



- c) Open and view both the Potential and Phase 2 channels. If the phase was properly set in the previous step, the value for the Phase 2 channel will be close to zero. The phase should not exhibit large jumps/discontinuities and should show minimal “drift” over the course of the image.
- d) Optimize the Potential image by determining a sufficiently high Lift Height, a slow enough Scan Rate, and Lift Mode I and P gains that are neither too high nor too low ($I \approx 1-3$ and $P \approx 3-6$ are typical). A high quality Potential image should exhibit minimal streaking and few to no saturation spots due to phase “jumps”. Although lateral resolution in the Potential channel decreases with increased Lift Height, too low a Lift Height will result in topographical artifacts and/or saturation of the Potential channel. A good starting Lift Height to try is 100 nm.

NOTE: Phase jumps (i.e., discontinuities in the Phase 2 channel) will appear as black or white skips in the Potential channel. Physically this occurs when the probe is unable to properly track a large feature (peak or pit). To prevent phase jumps, increase the Lift Height and/or decrease the Scan Rate.

16) Capture Images.

- a) Select the proper folder in which to save the data.
- b) Give the image files a default nomenclature. Be sure to include any important parameters (e.g., Lift Height) and sample name.
- c) Under the Capture drop down bar there are different options for capturing an image.
 - i) Capture: Saves the next complete, uninterrupted frame to the labeled file.



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



- iii) Capture Last: Saves the last complete frame prior to the current one.



- iv) Capture Continuous: Continuously saves frame after frame until the probe is withdrawn or capture is aborted.



- v) Capture Withdraw: Captures the next complete, uninterrupted frame and then withdraws the probe from the surface.
- vi) Abort Capture: Cancels the current capture status.



17) Shut down the AFM.

- a) Once imaging is complete and images have been captured and saved, select Withdraw on the Workflow Toolbar column.
- b) Go to Navigate in the Workflow Toolbar column and move the probe away from the sample surface by selecting “Move to Sample Loading Position”.
- c) Open the AFM acoustic chamber.
- d) Slowly rotate the stage/sample chuck to allow access to the sample.
- e) Turn the vacuum off and remove the sample from the stage.
- f) Unlock the Icon head by turning the set screw counterclockwise (Figure 4).
- g) Carefully lift the head from the dovetail rail and remove the probe holder.

WARNING: Handle the Icon head with extreme care! The piezoelectric scanner tube and associated closed loop sensors are very sensitive/fragile and expensive to repair/replace.

- h) Place the probe holder on mounting block (Figure 2) and remove the probe. Place the probe back in the box and location it came from. Ensure the probe is turned perpendicular to unused probes to indicate that it has been used. Detail the condition of the probe on the box label.
- i) Close the NanoScope software.
- j) Indicate the number of hours and new probes used on the log in sheet.

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