USER MANUALS

Neaspec NeaSNOM

Figure 1: NeaSNOM AFM assembly with part of the Imaging module at left and the FTIR module at the right.

This guide is for performing nano-imaging and nano-FTIR microscopy with the Neaspec NeaSNOM. The nano-imaging module is on the left side of the system Figure 1, while the nano-FTIR module is on the right side. The modules should only be aligned by trained beamline staff.
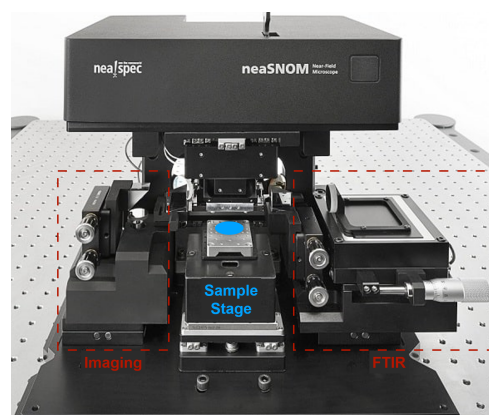
Nano-Imaging and Spectroscopy with the NeaSNOM

Sample Considerations and Suggestions Samples must be compatible with an atomic force microscope (AFM) for measurements to be successful: surface roughness, dimensions and adhesion are all factors which should be considered when preparing to take measurements. If possible, verify sample compatibility by collecting AFM topography measurements before your beamtime.
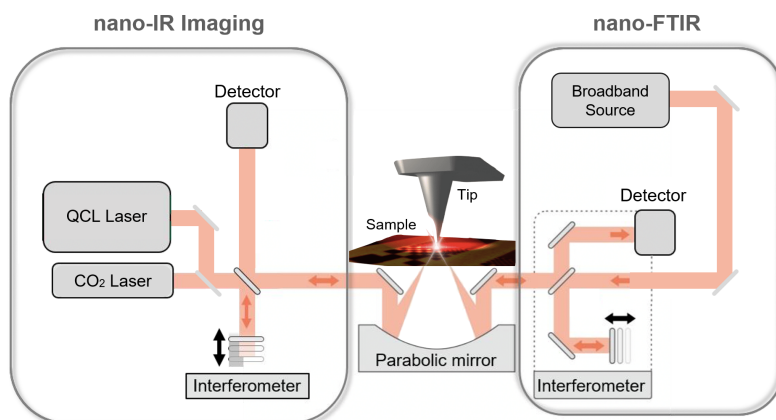

Figure 2: Schematic of the Neaspec setup. The left nano-IR imaging module collects single frequency or narrow band near field images with laser-based light sources. The right nano-FTIR module uses the IR output of the synchrotron for a light source.

In the case of sub-micron or nanoscale materials and features, it can be helpful to either pattern the sample with reference markers, mount the sample on a substrate with reference points, or locate the region of interest with a high-objective microscope prior to beamtime.

The resolution of the technique is limited by the probe tip radius ($\approx$25 nm) - therefore, trying to image features below this dimension is not recommended. Strong resonance effects from features below this dimension may be measured, but clearly resolving the source of the signal may be challenging.

Quick Start Guide:

1. Fill the detector with lN_2

2. Create project in the *Browse Projects* window

3. Check tip resonance, replacing if necessary

4. Insert reference sample (typically, Au for synchrotron light and Si for QCL)

5. Approach tip to reference

6. Optimize the IR signal by adjusting the parabolic mirror

7. Switch to sample of interest

8. Approach sample

9. Take a quick AFM scan of the surface, identifying a region of interest

10. Collect high-resolution near-field image

11. Collect infrared spectra (nano-FTIR only)

12. Save data to network User folder

13. Note in beamline log any issues with the system and the condition of the present tip for next User

14. Remove sample, clean up the workstation and tip/sample preparation bench

Data Processing Suggestions

For FTIR data, Use the 2nd derivative to locate peak positions and identify baselines for removal.

Vertical offsets may be subtracted from nano-FTIR phase data, but not the amplitude. This is because the amplitude signal is calculated by dividing the sample response by the reference response; therefore, subtraction of an offset alters the amplitude data. The phase data is already a difference measurement.

Note: if an offset is subtracted, it is no longer possible to convert between conjugate measurements (Amplitude/phase ⇔ Real/imaginary).

Nano-FTIR Spectroscopy with the NeaSNOM

Detailed Procedure for Operation of the nano-FTIR
Returning Users may proceed with the steps detailed below. New Users are asked to wait for a Beamline Scientist to provide safety instructions before proceeding.
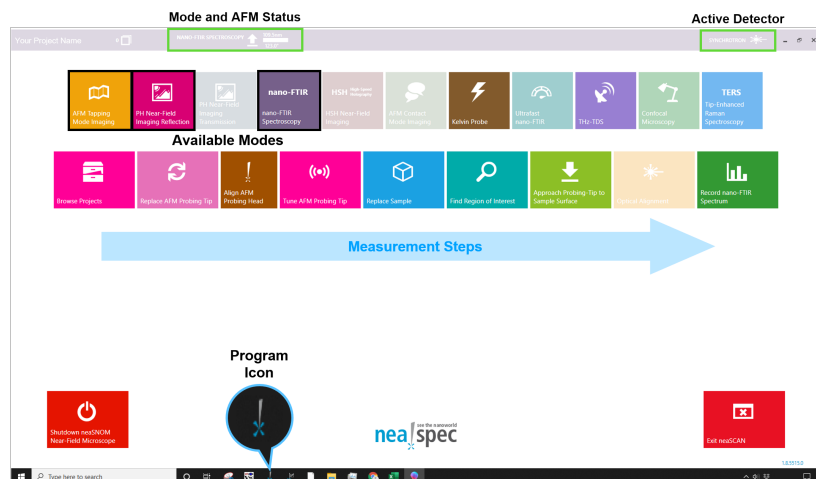

Figure 3: Neaspec home screen with available modes and measurement steps indicated. The current operational mode, AFM status, and the active detector. The detector must be manually selected after changing modes or software restarts. Note: let the beamline scientists know if the computer needs to restart as any remote connections will be disconnected.

Software Home Screen The home screen window for the Neaspec software is shown in Fig 3. The available modes are indicated in the top row of colored boxes while the measurement steps and their associated windows are shown in the bottom row. Note that the cur-
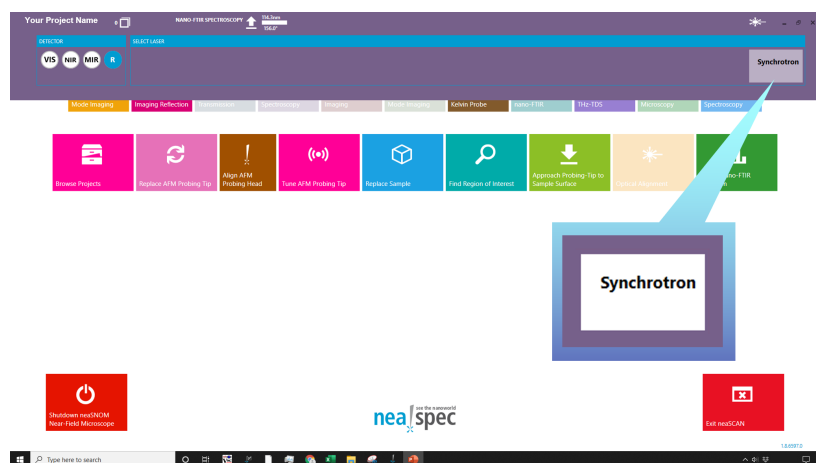

Figure 4: Verify the appropriate detector is selected for the measurement. For nano-FTIR, verify the *Synchrotron* box is selected. The box will be white when active.

rent AFM mode and the status of the AFM are shown in the banner at the top of the window. This banner changes color depending on which mode is active. Also note the active detector is indicated at the right hand side of the banner. For nano-FTIR, the active detector is *Synchrotron* as shown in Fig 4.

Browse Projects Window The *Browse Projects* window is the first window in the measurement process and allows users to create and specify the folder to which data should be saved. Start by typing in the name of the folder in the box shaded red in Fig 5. If the folder doesn't exist, create it by typing a project name. The name will then appear in the banner at the top of the window. The contents of the project are visible in the center and right panels of the *Browse Project* window. Navigate to the *Home* window and proceed with the measurements.

Figure 5: Browse projects window. Use this window to create and access user folders. Note: the software is not presently able to connect to the IR data network. Data files must be saved to the IR network after collection.
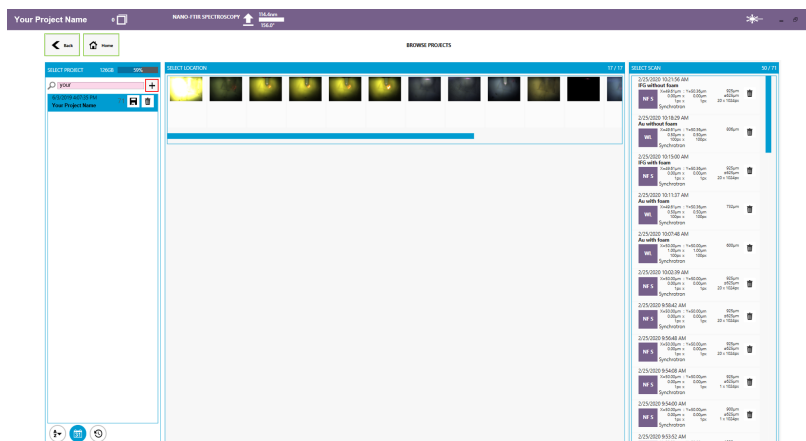
Replace sample Window This window is useful for making coarse adjustments to the sample stage, primarily when a new sample is introduce to the system. The stage can be driven horizontally with the 3D mouse to bring the sample of interest to the bright spot of the optical microscope light. Note: the relative motion controlled by the 3D mouse changes depending on the window perspective.

Figure 6: The replace sample window provides a side-view of the sample stage and parabolic mirror. The sample stage moves toward the parabolic mirror from off-screen at right if the previous sample was ejected using the software window. The 3D mouse can be used to easily bring the stage back to illuminated region of the optical microscope. The blue arrow drawn on the image indicates the direction of the sample state motion.
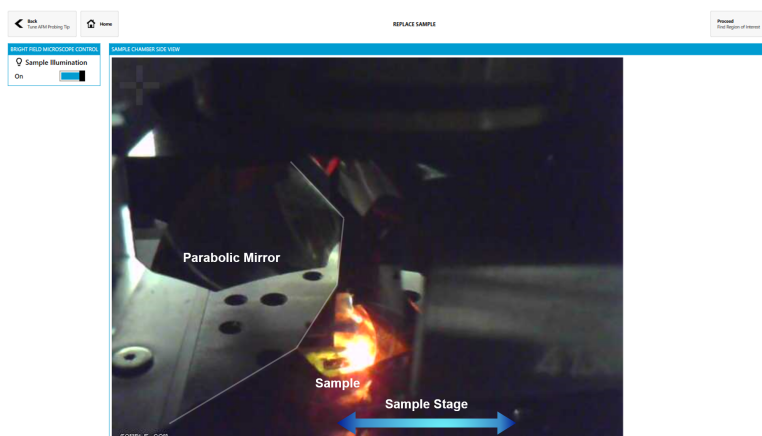
Finding a Region of Interest Go to the next screen *Find region of interest*. If the cantilever is in focus (as shown in frame 1 of the Fig 7) press *Focus Sample* in the bright field microscope control box until the

optical focus is at its lowest point. (If the tip is not in focus, contact a Beamline Scientist or proceed to the Advanced section of this manual and follow the instructions for bringing the cantilever into focus)
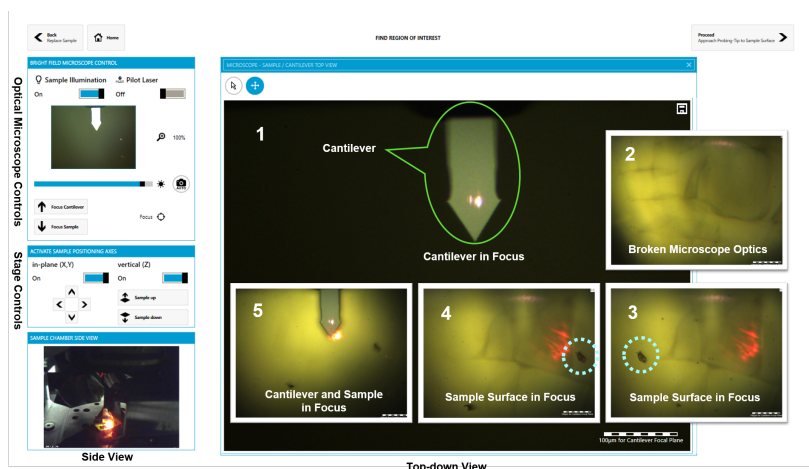

Figure 7: Sample window. The numbered insets show the progression of typical views when nearing the sample surface. Use surface defects like those indicated in the dotted blue circles in insets 3 and 4 to verify the sample is in focus (move the sample stage left and right; any surface features will likewise move).

Raise the sample by pulling up on the 3D mouse until the sample is just in focus. As you approach the sample surface, the field of view will gradually get brighter. Once the cracks in the microscope optics come into focus (panel 2 of Fig 7), proceed with raising the sample very slowly as the focal plane should be close. Verify the sample is in focus by laterally moving the 3D mouse (left-right) and observing features or debris on the surface move (panels 3 and 4 of Fig 7, dashed blue circles). Use the stage controls to navigate the surface until a region of interest is located, and place it near the cantilever apex.

Once the region of interest is in focus, raise the optical focal plane by pressing the *Focus Cantilever* button until it reaches the end of its range. Then lower the focal plane slightly by clicking the *Focus Sample* button about 15 times.

Caution: the following step greatly increases the risk of crashing the tip!

To decrease the tip-sample distance, slowly press the *Sample Up* button in the *Activate Sample Positioning Axes* box until the surface is nearly in focus. Then, proceed to the *Approach Probing Tip to Sample Surface* Screen.

Engaging the Tip Close the door to the microscope, then click on the engage tip button (down arrow) in the *Approach Tip to Sample Surface* window.

Caution: Never touch the door, instrument, or optical table when the tip is in contact with the sample to reduce possible damage to the AFM tip.
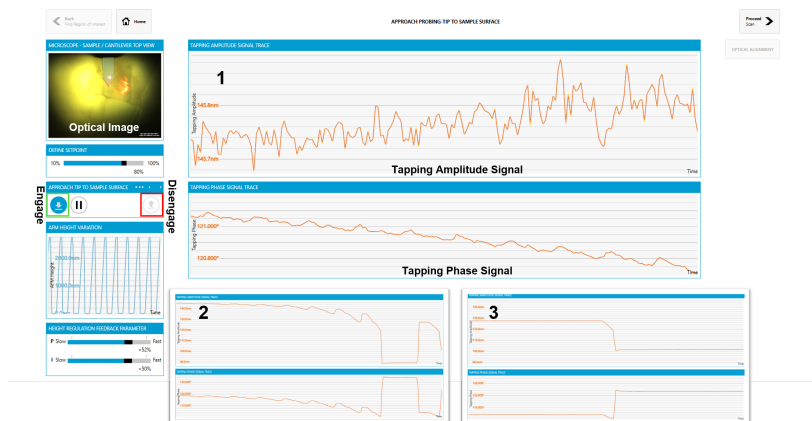
Figure 8: The approach probing tip to sample surface window is used for engaging the tip and monitoring the height and tapping amplitude during the process. Click the down arrow (boxed in green) to engage the tip. The up arrow (boxed in red) disengages the tip. The insets show the characteristic progression of the tapping amplitude and phase as contact is made with the sample.

Press the Engage/tip down button and the instrument will slowly step towards the sample surface until it is in contact, as shown in the panels of Fig 8. As the tip lowers toward the sample surface, the tapping amplitude should display a saw-tooth or zig-zag type behavior. Once the tip makes contact with the surface, a clear "snapping in" behavior should be observed in the amplitude. If the sample surface is no longer in contact, disengage the tip by pressing the up arrow and return to the previous window. Very small adjustments can be made to the stage height using the stage control arrows until the surface is in focus. Be very careful as the tip can easily crash.

Optical Alignment In the *Optical Alignment* window, the position of the parabolic mirror can be adjusted to maximize the higher harmonics of the near-field signal incident on the detector. With the tip in contact, press the *Auto* button. The system should automatically optimize the mirror position.

Figure 9: The optical alignment window shows the near-field optical amplitude, the interferogram, and the Fourier Transform of the interferogram (FTIR power spectrum) in the center panel. On the right side are the parabolic mirror alignment tools. The auto-align button is indicated by the red box. This button (or the individual direction controls) is used to maximize the overall near-field signal before data collection.
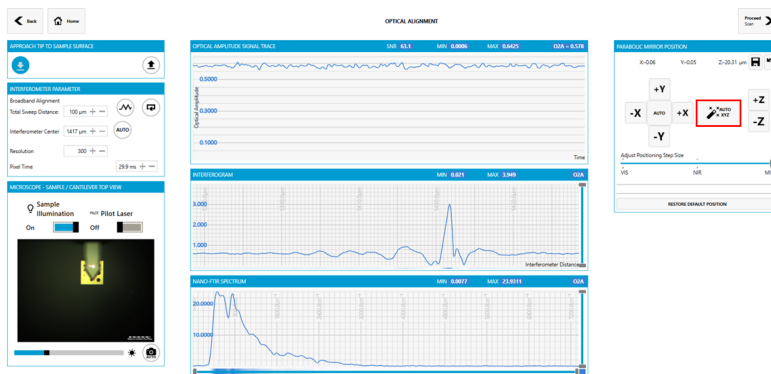
The parabolic mirror is aligned and the near-field signal maximized using a gold reference. The gold is spectrally flat and has a high reflectively, simplifying the alignment process. Once alignment

is complete, proceed to the *Scan* screen. This is where AFM images will be captured before spectra are collected.

AFM and Near-Field Imaging The *Scan* window contains the controls for AFM and near-field imaging. With the Au reference, set up a scan area of 10 μm x 10 μm or 15 μm x 15 μm, with a pixel area of 256 x 64. Change the integration time per pixel to change the scan speed (shown next to the integration time) to be 2 or until the font is green in color. Click in the box above the images to change the name of the images and save to your specified folder **AFTER** image collection has begun. If you change the name prior to starting an image scan, the software will overwrite your previous scan with the new name. Once imaging is complete, proceed to the next screen.
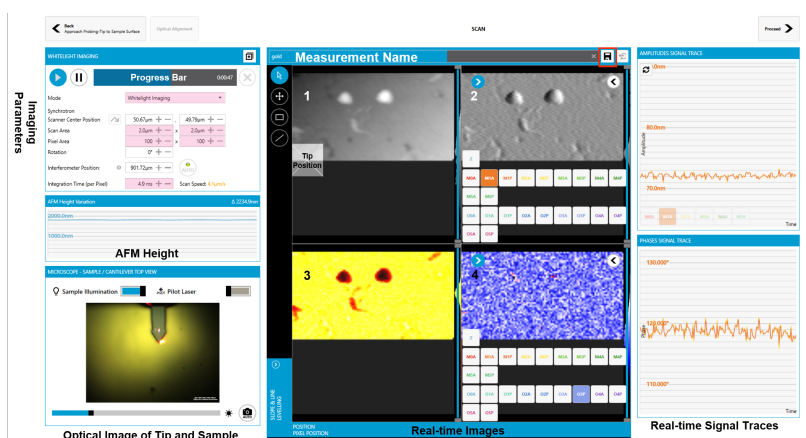

Figure 10: The Scan window is used for imaging with Neasnom. Four panels are available to show a variety of recorded signals. A typical measurement may show the Z height, first harmonic mechanical amplitude (M1A), the third harmonic amplitude (O3A) and the third harmonic phase (O3P). Signal traces are shown at the right. Note: all of the channels are saved automatically. Use the Imaging Parameters box for AFM scanning control (highlighted in red).

Collecting nano-FTIR Spectra In the *Take Spectrum* box, click in the "Choose a position from previous 2D-Scans" drop down and select the completed scan. Use the line tool and point to a position on the sample image that you want to scan. For a single spot, the scan area can be 0 x 0 microns, and a pixel area of 1 by 1.

The interferometer centerburst should be toward the right side of the scanning window. Rather than adjusting the interferometer distance and pixels/spectral bandwidth, it is suggested that you modify the spectral resolution (highlighted in red in the *Take Spectrum* box). Generally, the system noise prevents spectral resolution below 8 cm^{-1} and higher resolution measurements take significantly more time. Note: the progress bar estimates the collection time. If the sample does not have features above 2400 cm^{-1}, 650 pixels should be sufficient for accurate data collection.

Use the Selection Tools to indicate measurement positions on an AFM or near-field image. The default is point spectroscopy (drag the tip to the location of interest on the loaded sample image). To take a line of spectra, use the line tool and drag from the starting point to the end point. The scan area will update to a length and a width. The pixel area will determine how many points you take, where the first box will be the number of rows, and the second box
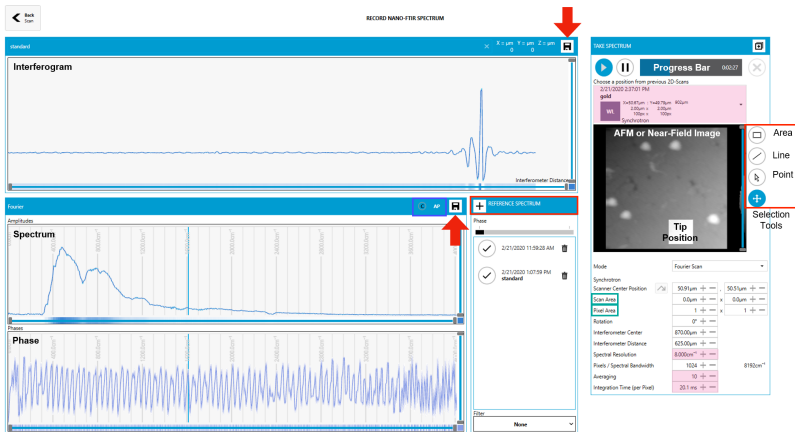
Figure 11: Nano FTIR Spectroscopy window showing an interferogram, along with the associated spectrum and phase (or real and imaginary components). The controls on the right hand side adjust the interferogram collection. Tools are present for point spectra, line and area scans. Note: a reference spectrum needs to be loaded to properly calculate the spectra from the interferogram. Ideally, from a gold reference collected during the alignment.

will be columns.

For example, a line going through the top of a 20 μm x 20 μm image, a line 20 x 0 microns can be sampled every 2 microns by setting the pixel area to 10 x 1.

Adjust the averaging and integration time according to the signal to noise and acquisition time. Note: it is suggested that multiple shorter spectra are collected from the same region or point and averaged together later rather than setting a high number of averages. This prevents significant time and data loss if an issue arises with either the light source or the system.

Saving Your Data Save the Au reference or sample spectra by changing the same in the top box and clicking the save button. Also click the Fourier save button. This will save the referenced spectrum.

Before you start collecting real data, you must select a reference spectrum by going to the reference spectrum box and clicking the + button and selecting the appropriate file.

Changing Samples Go to the *Replace Sample screen*: If the sample stage is already down (lowered), move the stage out toward the door by pushing the 3D mouse to the right. Lift the door and remove the old sample. Place a new sample on the stage and move the 3D mouse left until the sample is under the optical microscope light. Raise the sample up slightly by pulling the 3D mouse.

Backing Up Your Data to the Network Be sure to manually save a copy of your data to the network IRdata. Users can access this network from any of the IR group computers.

Pseudo-Heterodyne Imaging with the NeaSNOM

Pseudo-heterodyne imaging (PsH) is one of the operation modes available on the NeaSNOM system.

The general operation of the system can be described as follows. The atomic force microscope (AFM) cantilever oscillates around 300 kHz in tapping mode. The cantilever tip is illuminated with focused IR laser light, resulting in scattered light modulated at the tip tapping frequency Ω and higher harmonics. Back-scattered radiation interferes at an Mercury-Cadmium-Telluride (MCT) detector with a reference beam whose phase is modulated continuously with a mirror on a piezo at $\delta\Omega$ around 300 Hz. The detector signal is therefore demodulated at sideband frequencies of $n\Omega \pm m\,\delta\Omega$ for integer values of m. The signal is considered background-free and is directly attributable to the polarization of the tip for harmonics $n \geq 2$. Optical contrast obtained via higher-order harmonics in PsH mode is thus directly the result of variations in optical properties of the sample at the imaging frequency. The NeaSNOM system is able to demodulate multiple signal sidebands simultaneously and thus detect in real-time the near field amplitude A_{nf} and phase ϕ_{nf}.

Tuning Range of Available Lasers			
	(cm^{-1})	μm	meV
CO_2	900 - 1000	10 - 11	113 - 124
QCL	1590 - 1720	5.8 - 6.3	197 - 214

To change the imaging frequency, please contact the Beamline Scientist for assistance.

Data collection in Pseudo-heterodyne imaging mode on the Neaspec system follows the same general steps as the nano-FTIR process described above, with the exception that IR spectra are not able to be collected directly as single-frequency imaging is the main objective.

Detailed Procedure for nano-Imaging with PsH detection Returning Users may proceed with the steps detailed below. New Users are asked to wait for a Beamline Scientist to provide safety instructions before proceeding.
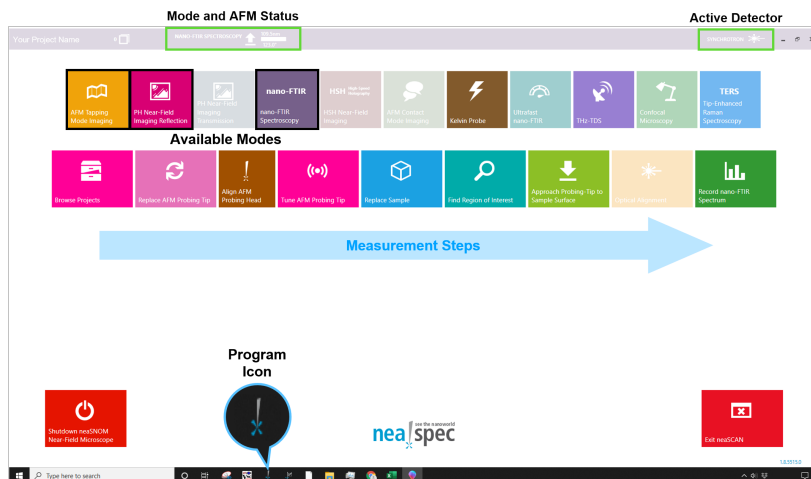

Figure 12: Neaspec home screen with available modes and measurement steps indicated. The current operational mode, AFM status, and the active detector. The detector must be manually selected after changing modes or software restarts. Note: let the beamline scientists know if the computer needs to restart as any remote connections will be disconnected.

Software Home Screen The home screen window for the Neaspec software is shown in Fig 12. The available modes are indicated in the top row of colored boxes while the measurement steps and their associated windows are shown in the bottom row.

Note that the current AFM mode and the status of the AFM are shown in the banner at the top of the window. This banner changes color depending on which mode is active. Also note the active detector is indicated at the right hand side of the banner. For PsH imaging, the active detector is *QCL* as shown in Fig 13.

Figure 13: Verify the appropriate detector is selected for the measurement. For PsH, the *QCL* box is selected for both the QCL and the CO_2 lasers. The box will be white when active.
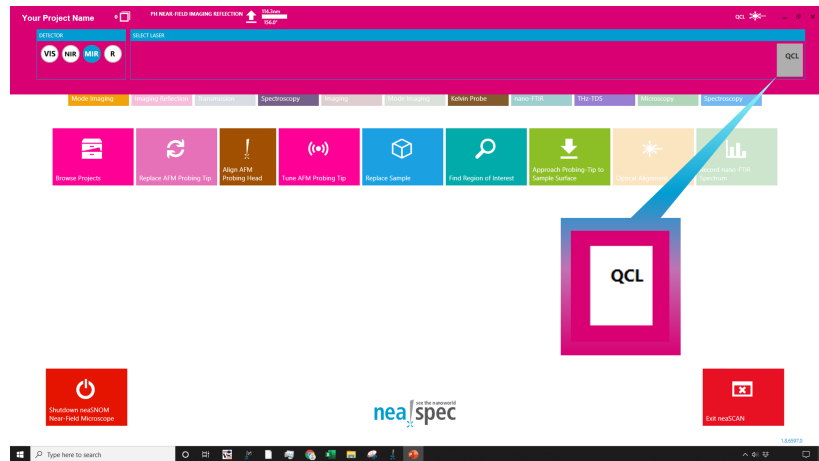
Proceed through the measurement steps outlined in Fig 12. The optical alignment in PsH mode differs from the nano-FTIR mode in that an interferogram and power spectrum will not be available. Instead, the system is aligned by decoupling the near-field amplitude and phase signal while the reference arm mirror is vibrated at a test frequency. **Insert PsH optical alignment image**

Figure 14: Scan window for PsH mode.
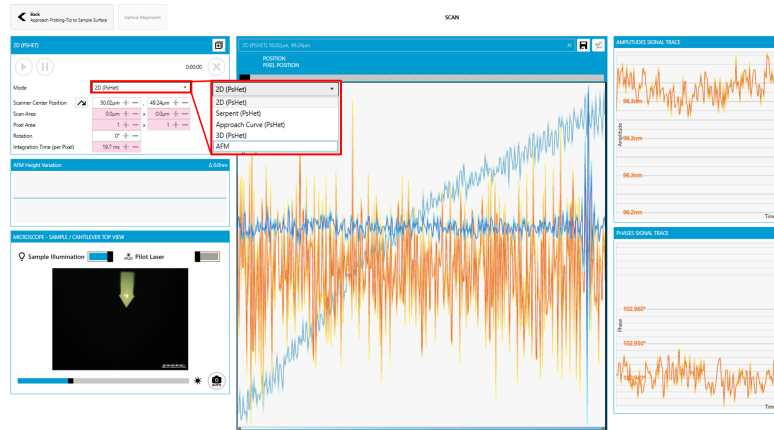
Remote Operations

The procedure for remote operation of the NeaSNOM is generally the same as in-person, with a few notable exceptions.

TeamViewer and Zoom will likely both be utilized for your remote beamtime. The beamline staff will send you TeamViewer credentials to access and control the beamline computer. A Zoom meeting may also be scheduled to locate regions of interest and discuss measurement plans.

Beamline staff will load your samples Please provide optical images and fiducial markers (when possible) to assist beamline staff in finding the region of interest on your sample.
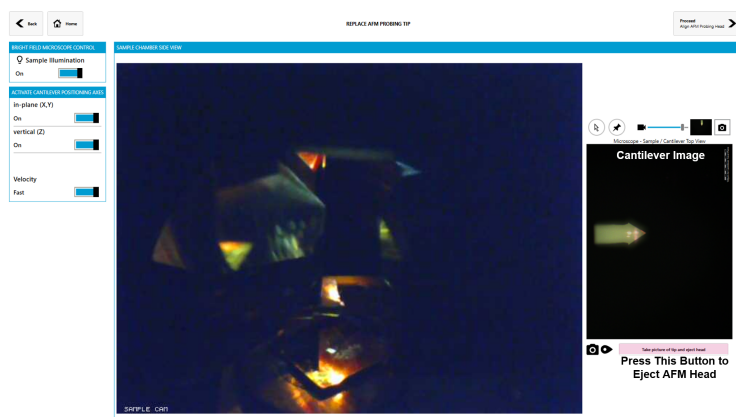
Beamline staff will be available during regular hours Beamline staff will be onsite for approximately 8 hour shifts during the day (PST). Please plan accordingly to coordinate for sample changes and detector fills while beamline staff are present. Staff are generally not able to return to the ALS after scheduled shifts, and will only be able to assist via TeamViewer.

Advanced Procedures

The following advanced procedures should only be performed with the approval of the Beamline Scientists. When in doubt, ask!

Use extreme caution when handling the AFM head and set screw. We do not have replacements for either!

Abbreviated Tip Change Procedure Extra chip carriers have been prepared with new tips by the Beamline Staff and are located on the workbench.

Disengage the tip, navigate to the *Replace AFM Probing Tip* window, and lower the sample stage by pressing down in the center of the 3D mouse. Eject the AFM head from the software by pressing the button as in Fig 15.


Figure 15: Eject AFM Head. To replace a tip, you must first drop the sample stage (press the center of the 3D mouse) and then eject the AFM head by pressing the button highlighted in red in the figure. The system will automatically move the head to the back of the AFM compartment for easy removal. In addition, a picture of the old tip location is saved for reference during this process.

Remove the AFM cover, unplug the electrical connection, and remove the knurled screw holding the head in place as shown in Fig
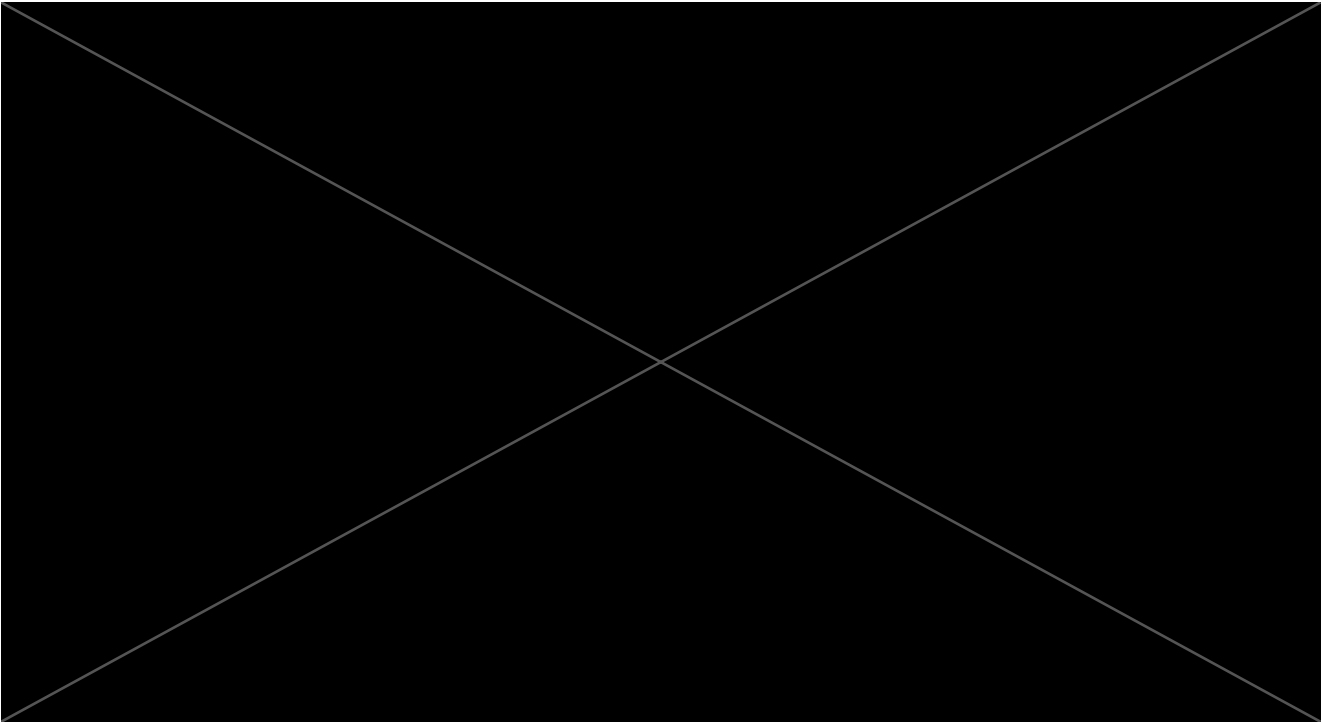
16. Carefully lift the AFM head up and away from you (there is a ledge within the microscope which prevents lifting the head straight up). Take the AFM head to the workbench and place it upside-down. From the bottom of the head, the chip holder, set screw, pin, and cantilever are all visible as in Fig 16. Locate the prepared chip carriers, tweezers, and small screw driver as shown in Fig 17.
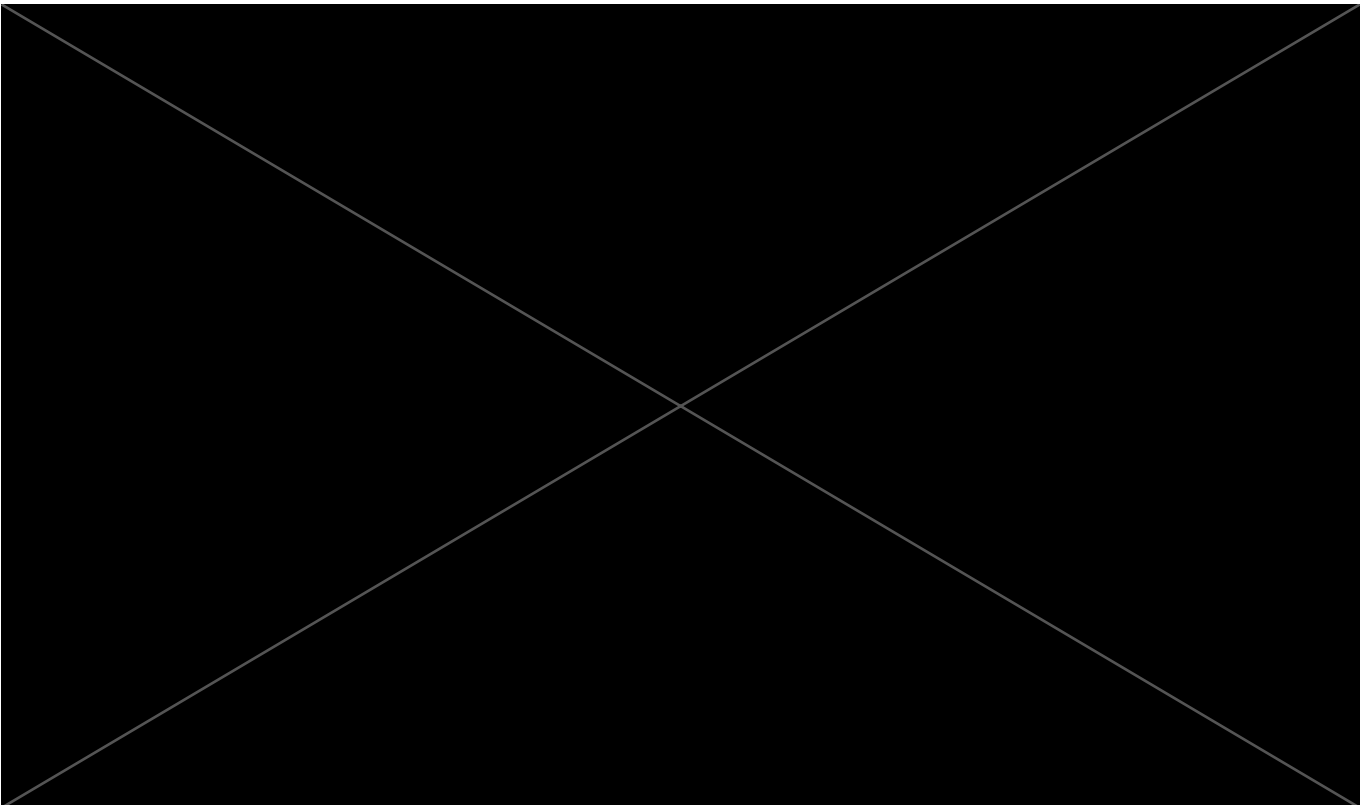
Pick up the head and, being careful not to bump the new cantilever, turn it right-side up and slowly place it back in the microscope. Recall the internal ledge prevents direct lowering of the head

into the microscope; instead, keeping the head a few inches behind the mounting track, lower the head first and then bring the head forward into place. Attach the knurled screw and then the electrical connection. Finally, replace the cover.
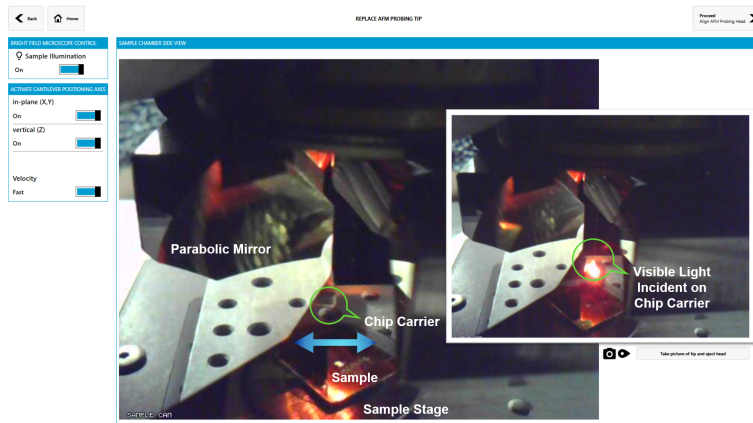

Figure 18: Using the 3D mouse, bring the AFM head into the microscope field of view by driving the mouse to the right. Roughly center the visible light on the chip carrier, as shown in the inset.
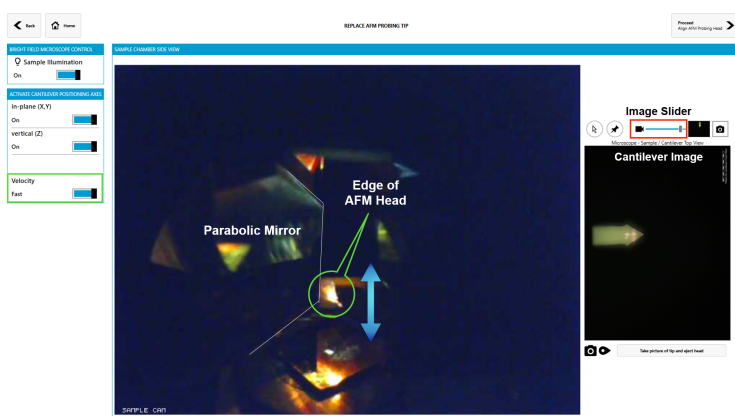

Figure 19: Bringing cantilever into Focus. Use the 3D mouse (lift or push, as needed) to resolve and align the new cantilever. The green box on the left side of the image contains a toggle switch for moving the AFM head fast or slow - it is useful to slow the movement speed when the cantilever image starts to come into focus.The image slider (boxed in red) allows you to overlay the current live tip location with the picture of the old tip location for ease of alignment.

In the *Replace AFM Probing Tip* window, use the 3D mouse to bring the edge of the AFM into view, as illustrated in Fig18 by driving the AFM head to the right. Once visible light is incident on the chip carrier, use the 3D mouse to raise the head until the cantilever is in focus (Fig 19). It is easiest to focus on the back of the chip carrier, and then slowly move to the cantilever, as shown in the series of images in Fig 20. Once the new tip is in focus, use the image slider to overlay the previous tip position and align the red reflection laser to approximately the same location on the new tip.

Proceed to the *Align AFM Probing Head* window and once again use the image slider to align the new tip to the location of the old tip with respect to the deflection laser, as shown in Fig 21.

After the AFM head alignment is complete, tune the new tip. Start a full frequency sweep by pressing the button indicated by the green box at the top left of Fig 22. A series of frequency sweeps will be performed, narrowing in on the tip resonance. After the first sweep is
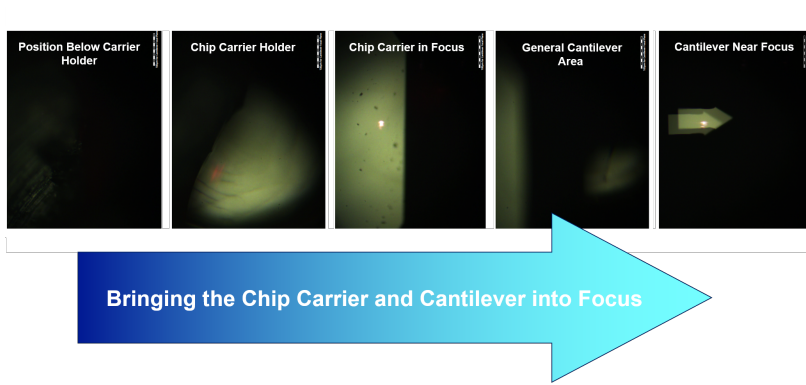
Figure 20: Bringing the chip carrier and cantilever into focus. A progression of views is shown with the chip carrier and then the cantilever coming into focus. In the right-most image, the location of the previous tip overlay is used to align the deflection laser (red light) in the same relative location of the new and old tips.
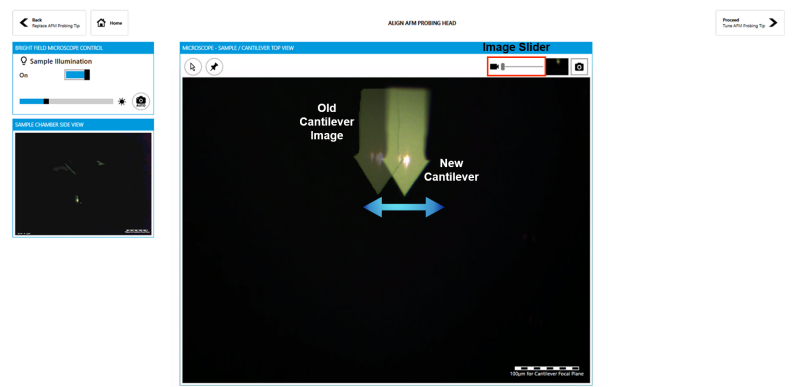

Figure 21: Align the new cantilever with the superimposed image of the previous cantilever. The transparency of image of the old cantilever can be adjusted with the slider boxed in red at the upper right side of the image. The 3D mouse is used to drive the cantilever to the correct position.

completed, highlight the peak region, as shown in the image labeled (1). With the successive sweeps (2) and (3), draw smaller and smaller boxes around the peak. After 3 iterations, the software will calculate the tip resonance and fit. In the lower left part of Fig 22, click the Auto button (boxed in green) to optimize the AFM alignment. Verify the drive voltage (highlighted in red) is less than 30 V. If the tip amplitude is low (typical free-oscillation amplitude is 100-120 nm), the drive voltage can be increased incrementally.

Figure 22: Tuning the tip. This image shows the successive zooming in on the tip resonance during a full range sweep (button boxed in green in the upper left). Once the tip frequency is identified, auto AFM alignment is performed by selecting the "Auto" button in the lower left corner of the window. The tapping voltage adjustment is highlighted in red.