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AFM – Bruker Atomic Force Microscopy System

Standard Operating Procedure

4D LABS Confidential

Revision: 1.1 — Last Updated: April 28, 2016, Revised by Chris Balicki

Overview

This document will provide a detailed operation procedure of the AFM System. Formal Training is required for all users prior to using the system.

Revision History

#	Revised by:	Date	Modification
0	Chris Balicki	2015/05/01	Document Initial Release
1	Chris Balicki	2016/04/28	Added PeakForce QNM Procedure
2			
3			
4			

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General Information

The Bruker AFM System is a powerful all-in-one tool enabling a variety of techniques including:

- Contact Mode
- Tapping Mode
- Peak Force Tapping / QNM
- STM
- ScanAsyst (proprietary Bruker AFM technique)

The system is capable of analyzing samples in air and in fluid. The system is capable of stage heating and cooling. Various other techniques are available and will be brought online on a case by case basis according to user demand.

Basic training commences with probe tip setup and basic surface scanning in ScanAsyst in Air mode. Bruker's proprietary ScanAsyst mode, a simplified version of Peak Force Tapping, allows for automatic optimization of critical AFM parameters such as tapping/contact force, signal gain, scanning rate, and piezo limit, which allows for quick and well optimized scanning with minimal effort.

Advanced training will often require additional phases of training. Users are reminded to not operate the system beyond the scope of their training.

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ScanAsyst in Air

Startup and Experiment Selection

1. Verify that the Windows is not currently running any programs by ensuring that the Windows Taskbar is clear.
2. Double-click on the Nanoscope 9.1 icon to open the AFM Control program.
3. Select the ScanAsyst in Air Experiment
    a. Under **Choose an Experiment Category**, select “ScanAsyst”.
    b. Under **Select Experiment Group**, select “ScanAsyst in Air”.
    c. Under **Select Experiment**, select “ScanAsyst in Air”.
    d. Click “Load Experiment”.
    e. Wait for the software to complete the experiment setup. Verify in the **Workflow Toolbar** (left hand side) that the ScanAsyst in Air experiment has been loaded. The default subsection selection should be “Scan”.

Probe Tip Setup (when loading a new probe tip)

1. In the **Workflow Toolbar**, click “Navigate”.
2. Under **Section 2 – Focus Sample**, Subsection 2.2, select the high speed option (100%).
3. Move the probe head upward by clicking on the up arrow (Z Motor Up) for 3 seconds.
4. Unlock and raise the front cover using the dedicated T-Allen wrench.
5. Release the SPM head by loosening the dovetail release screw (see Figure 1 and 2).
6. Lift the SPM head out of the dovetail groove with one hand and flip it upside down to expose the cantilever holder.
7. With the other hand, remove the cantilever holder. Replace the SPM head.
8. If a <Crash Protection> status log appears, close it by clicking in the top right.

Figure 1

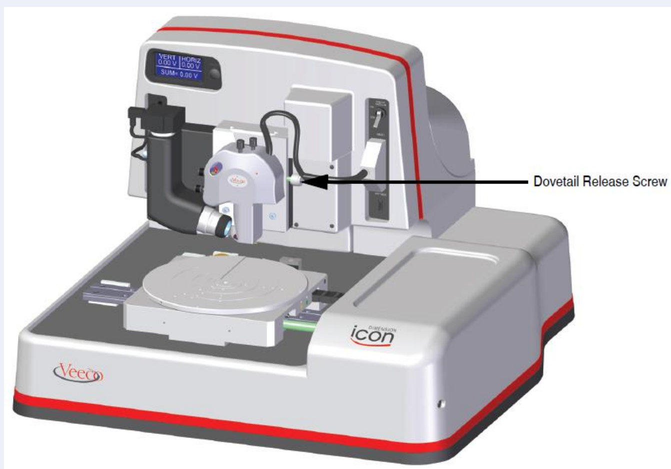

Figure 2

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9. Place the cantilever holder in the appropriate position on the cantilever holder stand (see Figure 3 and 4)
10. Press down and slide back the spring clip on the cantilever holder.

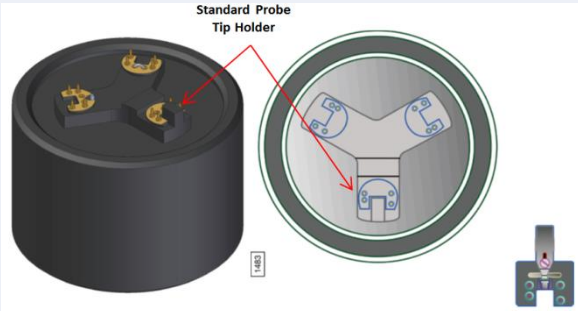

Figure 4

Figure 4

11. With a sharp set of tweezers, grasp the cantilever firmly on the sides and lift to free it from the gel-pack. Within the gel-pack boxes, the tip ends are pointing upward and do not need to be turned over before being placed in the cantilever holder.
12. Orient the cantilever intended for imaging away from the cantilever holder and place the cantilever substrate in the cantilever holder groove.
13. Carefully maneuver the cantilever substrate until it is flush against the back edge and lying flat in the cantilever holder groove (see Figure 5).
14. Press the spring-loaded probe clip down, gently push forward over the cantilever substrate, and release the spring clip to hold the cantilever substrate in place in the cantilever holder groove (see Figure 6).
15. With the same hand as before, remove the cantilever holder from the cantilever holder stand.

Figure 6

Figure 6

16. With the other hand, lift the SPM head out of the dovetail groove and flip it upside down.
17. Install the cantilever holder to the SPM head by aligning the pins and pressing down gently.

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18. Replace the SPM head gently into the dovetail groove. Ensure the SPM head is at the bottom of the dovetail groove.
19. Secure the SPM head by tightening the dovetail screw.
20. In the **Workflow Toolbar**, click "Setup".

Note:
At this point, the image should be focused on the cantilever. If the substrate is visible but the cantilever is not, it is likely that the cantilever is broken and that the probe tip is no longer useable.

21. Under **Section 2 – Align Laser on Probe**, click on "Move to the Alignment Station".
22. Review the warning message. Acknowledge by clicking "Yes".
23. Wait until the probe movement has completed.

Note:
At this point, the image should automatically adjust focus from the cantilever tip to the cantilever tip reflection. A laser reflection should also be visible.

24. Align the laser onto the cantilever using the dimension head laser control knobs located on top of the SPM head. Maximize the sum voltage signal (shown on the bottom of the screen). A useful video is shown by clicking "Watch Video" under Subsection 2.2 in the Setup screen. Once the signal is maximized, turn the rear-right laser control knob (x-direction) counterclockwise such that the laser is moved slightly closer to the cantilever tip and the voltage signal is reduced by 0.1V.
25. Adjust the photodetector horizontal and vertical deflection voltage signal values using the photodetector adjustment knobs located on the left side of the SPM head. Adjust the knobs until both the horizontal and vertical voltage signal values read near 0V. A useful video is shown by clicking "Watch Video under Subsection 2.3 in the Setup screen.
26. Under **Section 3 – Focus Tip**, click on "Return from the Alignment Station".
27. Review the confirmation message. Acknowledge by clicking "Yes".

Note:
At this point, the image should be focused on the cantilever and not on the cantilever tip reflection. However, if focus is poor or nonexistent, a few additional steps are required:

28. Under Subsection 3.2, use the focus controls to focus the optics on the cantilever. Adjust the speed as needed. Refine the focus until the cantilever is clear. The cantilever tip should appear light in colour and somewhat reflective if the backside of the cantilever has a backside reflective coating. This image is notably different from the tip reflection which is used for adjusting the photodetector laser.
29. Click on the tip of the cantilever to locate the probe tip. This is required for ease of tip location during sample analysis.

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Sample Loading

Note:
If working with very light samples, rotate the stage so as to locate the dedicated vacuum port.

1. In the **Workflow Toolbar**, click "Navigate".
2. Place the sample on the sample stage and activate the vacuum port if necessary.
3. Rotate the sample stage such that the sample is either directly under the probe head or as near as possible. Be careful so as avoid contact between the probe and the sample. If contact is anticipated, move the probe head upward by clicking on the up arrow (Z Motor Up) located in **Section 2 – Focus Sample**, Subsection 2.2, until there is sufficient room between the probe and the sample.
4. Verify that there is at least 3mm of clearance between the probe head and the sample.
5. Under **Section 2 – Focus Sample**, Subsection 2.1, select the "Tip Reflection" method.
6. Under Subsection 2.2, adjust the scan head downward slowly towards the surface until the tip reflection is in focused view. Provided the probe head was high enough, you may see the sample focus plane first. Continue the slow descent of the probe head until the tip reflection comes into focused view.
7. Once tip reflection is in focused view, click "Sample (default)". Verify the sample surface is in focused view. This may be difficult with featureless samples.
8. Under **Section 3 – Navigate to Scan Centerpoint**, use the XY controls to navigate to the desired measurement area. The red crosshair indicates the approximate position of the probe tip.

Measurement Setup and Collection

1. In the Workflow Toolbar, click "Check Parameters".
2. In the Ribbon Toolbar, click on the "Expand Mode" icon.
3. Adjust the scan size and aspect ratio to the desired value.

Note:
At this stage, it is important to note the change in tip velocity. Provided a constant scan rate, a larger scan size will increase the tip velocity. Tip velocities in excess of 100um/s should be avoided; otherwise, the probe tip is at greater risk of chipping and/or more severe damage. Generally speaking, the slower the tip velocity, the greater the scan resolution. However, given a large scan size, a slow tip velocity may result in prohibitively long scan times. Ultimately, as probe tips are the property of the user, it is at the user's discretion what scan parameters are most useful.

4. If the AFM front cover is open, lower and lock the front cover using the dedicated T-Allen wrench.
5. In the Workflow Toolbar, click "Engage".

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6. Monitor the Trace and Retrace lines located below the main image. In a short period of time, they should converge and coincide well with one another, indicating that the probe tip is tracking effectively and producing a clean measurement scan.
7. When the probe is tracking effectively, the user is permitted to adjust the following parameters to optimize the measurement scan:

Scan Parameters	Feedback Parameters	Limits
Scan Size	ScanAsyst Auto Control: On/Individual	Z Range
Aspect Ratio	ScanAsyst Auto Scan Rate: On/Off	
X/Y Offset	ScanAsyst Auto Z Limit: On/Off	
Scan Angle		
Scan Rate		
Samples/Line		

Note:
For large changes to Scan Size / Scan Rate, it is recommended to disengage from the sample and to reengage following parameter adjustment.

Saving a Scan

1. Confirm that your sample is tracking and imaging well. If not, adjust the measurement parameters as needed.
2. On the right hand side, click on the "Change Current Directory" icon and select your corresponding data folder.
3. Click on the "Select Capture Directory" icon and select your corresponding data folder.
4. Rename your file accordingly and press Enter. The software will automatically append ".000".
5. Click on the "Capture" icon. The icon should indicate that it has been selected.
6. Click on the "Frame Down" icon.
7. Wait for the scan to complete one full cycle. The scan image should appear in your selected capture directory for each scan cycle. The file name will increment accordingly.

Note:
Any changes to the measurement parameters will reset the scan cycle. Following any changes to the measurement parameters, it is recommended to select "Frame Down" in order to reset the scan cycle.

Measurement Analysis and Export

1. On the right hand side, confirm your capture directory.

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2. Double-click on the desired scan image file. The file will open in a new NanoScope Analysis window. The default view will be 2D Image
3. Use any of the analysis tools to collect data as required. Common tools include:
    a. 2D Image
    b. 3D Image
    c. Roughness
    d. Section
    e. Flatten
4. Following analysis and/or image adjustment, the image and/or screen view can be exported by right-clicking and selecting export.
5. Save the image file to your corresponding data folder.
6. Files can be sent from the computer via email (flash drives are generally prohibited).

Software Shutdown

1. When finished, close all open software by clicking in the top right of each open window.
2. The computer may be left powered on.

PeakForce QNM in Air

PeakForce QNM, simply put, is the advanced mode of ScanAsyst. It too utilizes peak force tapping which is highly effective in AFM work. Thus, the commonly used ScanAsyst-Air probe tips are suitable for PeakForce QNM. Several additional measurement channels are active in PeakForce QNM, such as Modulus and Adhesion. With tip qualification and sample calibration, it is possible to measure these materials properties to a certain degree of accuracy.

For adhesion measurements, it is suitable to calculate only the deflection sensitivity and the spring constant by the thermal tune procedure. For modulus measurements, Relative Modulus Calibration is required in addition to the adhesion measurement requirements.

Users must request additional training before receiving permission to use this mode.

Startup and Experiment Selection

1. Verify that the Windows is not currently running any programs by ensuring that the Windows Taskbar is clear.
2. Double-click on the Nanoscope 9.1 icon to open the AFM Control program.

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3. Select the ScanAsyst in Air Experiment
    a. Under **Choose an Experiment Category**, select "Mechanical Properties".
    b. Under **Select Experiment Group**, select "Quantitative Nanomechanical Mapping".
    c. Under **Select Experiment**, select "PeakForce QNM in Air".
    d. Checkmark "Ignore Probe Parameters".
    e. Click "Load Experiment".
    f. Wait for the software to complete the experiment setup. Verify in the **Workflow Toolbar** (left hand side) that the PeakForce QNM in Air experiment has been loaded. The default subsection selection should be "Scan".

Probe Selection and Setup

1. Select a suitable probe tip.

    Note:
    For measuring Adhesion only, the basic ScanAsyst-Air tip will suffice. For measuring Modulus, it is important to choose a probe tip that produces sufficient sample deformation while still retaining high force sensitivity. Bruker's recommendations are as follows:

SAMPLE MODULUS (E)	PROBE TIP	NOMINAL SPRING CONSTANT (k)
0.7 < E < 20 MPa	SNL-A / ScanAsyst-Air	0.5 / 0.4 N/m
5 MPa < E < 500 MPa	Tap150A	5 N/m
200 MPa < E < 2000 MPa	RTESPA	40 N/m
1 GPa < E < 20 GPa	Tap525A	200 N/m
10 GPa < E < 100 GPa	DNISP-HS	350 N/m

Table 1

2. Perform the tip loading and setup procedure as outlined earlier in this document. The procedure is the same as for ScanAsyst in Air.

Calibrate Deflection Sensitivity

1. Load the 'Sapphire-12M' sample from the Peakforce QNM Sample Kit.
2. Approach the surface and find the correct sample focus. Use the tip reflection method but take additional precaution as the sample is transparent and the tip reflection may be easily missed.
3. Find a clean area.

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4. In the Workflow Toolbar, click "Check Parameters".
5. In the Ribbon Toolbar, click on the "Expand Mode" icon.
6. Adjust the scan size to 0nm.
7. For stiff probe tips, lower the engage setpoint to 0.05V (Microscope → Engage Settings → PeakForce Engage Setpoint)
8. If the AFM front cover is open, lower and lock the front cover using the dedicated T-Allen wrench.
9. In the Workflow Toolbar, click "Engage".
10. Once engaged on the surface, in the Workflow Toolbar click "Ramp".
11. Adjust the trigger threshold within the range of 0.1 – 0.5V. A setting of 0.1V will preserve probe tip sharpness whereas a setting of 0.5V will provide better accuracy.
12. In the Ribbon Toolbar, click on the "Start Single Ramp" icon.
13. On the resultant force curve plot on the right hand side, perform the following:
    a. Zoom in on the area on interest by pressing CTRL and dragging a box with the mouse cursor.
    b. Pull in the cursors from the right hand side.
    c. Place the cursors accordingly to capture the slope of the force curve deflection.
    d. On the Ribbon Toolbar, press the "Update Sensitivity" icon. Take note of the deflection error sensitivity and click "OK". Click "No" when prompted to continue calibrating DDS3 drive amplitude sensitivity.

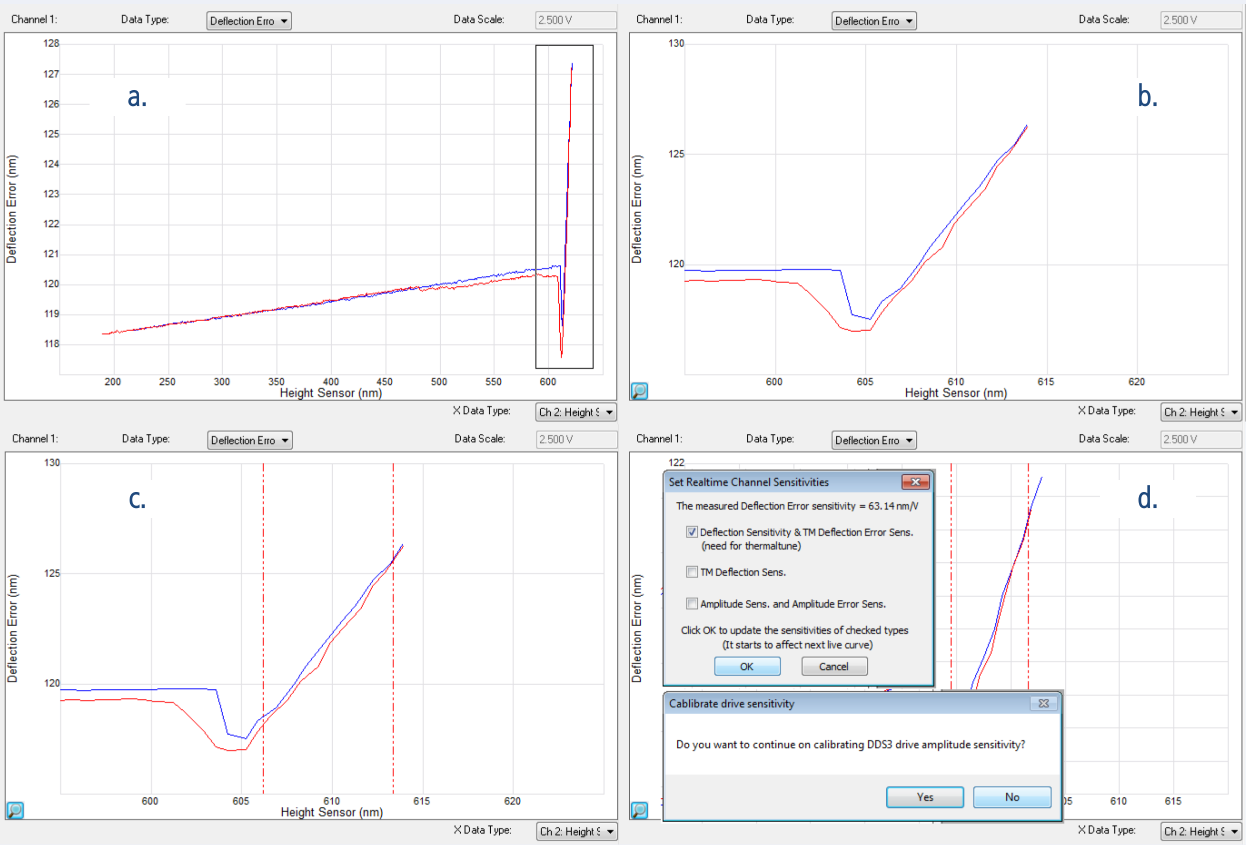

Figure 7

14. Repeat steps 12-13 up to 3-5 times and take an average for deflection error sensitivity for greater accuracy. Go to Calibrate → Detector to check the deflection sensitivity and/or enter the average value accordingly.
15. In the Workflow Toolbar, click "Withdraw".
16. Unload the 'Sapphire-12M' sample and return it to the Peakforce QNM Sample Kit.

Calculate Spring Constant by Thermal Tune

1. On the Ribbon Toolbar, click on the "Thermal Tune" icon.
2. Select the appropriate Thermal Tune Range for the installed probe tip. Check the frequency provided online, within the AFM Probes Manual, or on the probe tip packaging.
3. Click on Acquire Data and wait for the data acquisition.

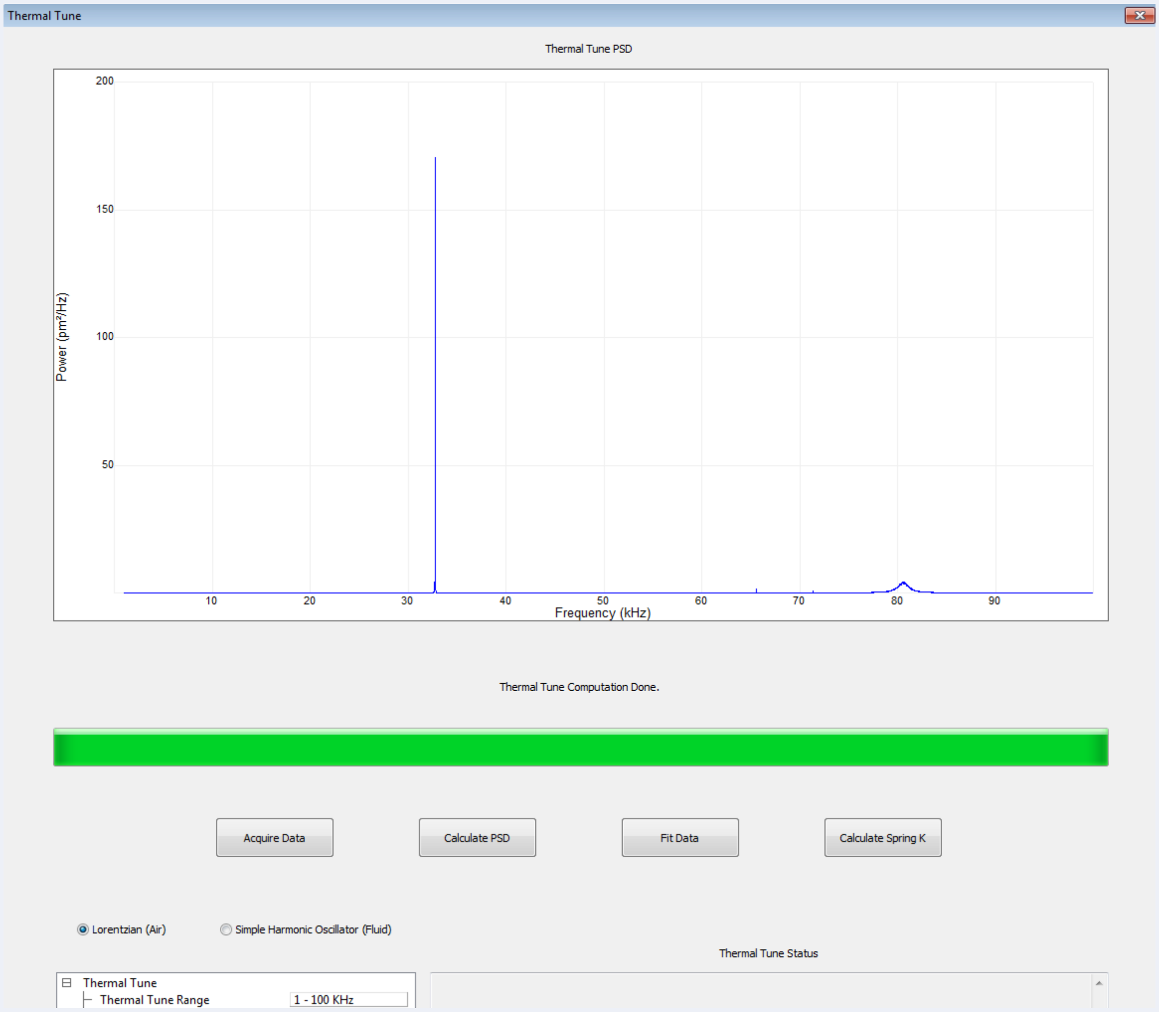

Figure 8

4. Ignore false noise peaks such as the one in Figure 8 at ~33 kHz.
5. Zoom in on the area on interest, usually a bell curve near the specified frequency of the probe tip, by pressing CTRL and dragging a box with the mouse cursor.
6. Pull in the cursors from the right hand side and place one on either side of the bell curve with ample amount of 'flat' area on either side of the bell.
7. Click on Fit Data.
8. If the fit looks good, click on Calculate Spring K.

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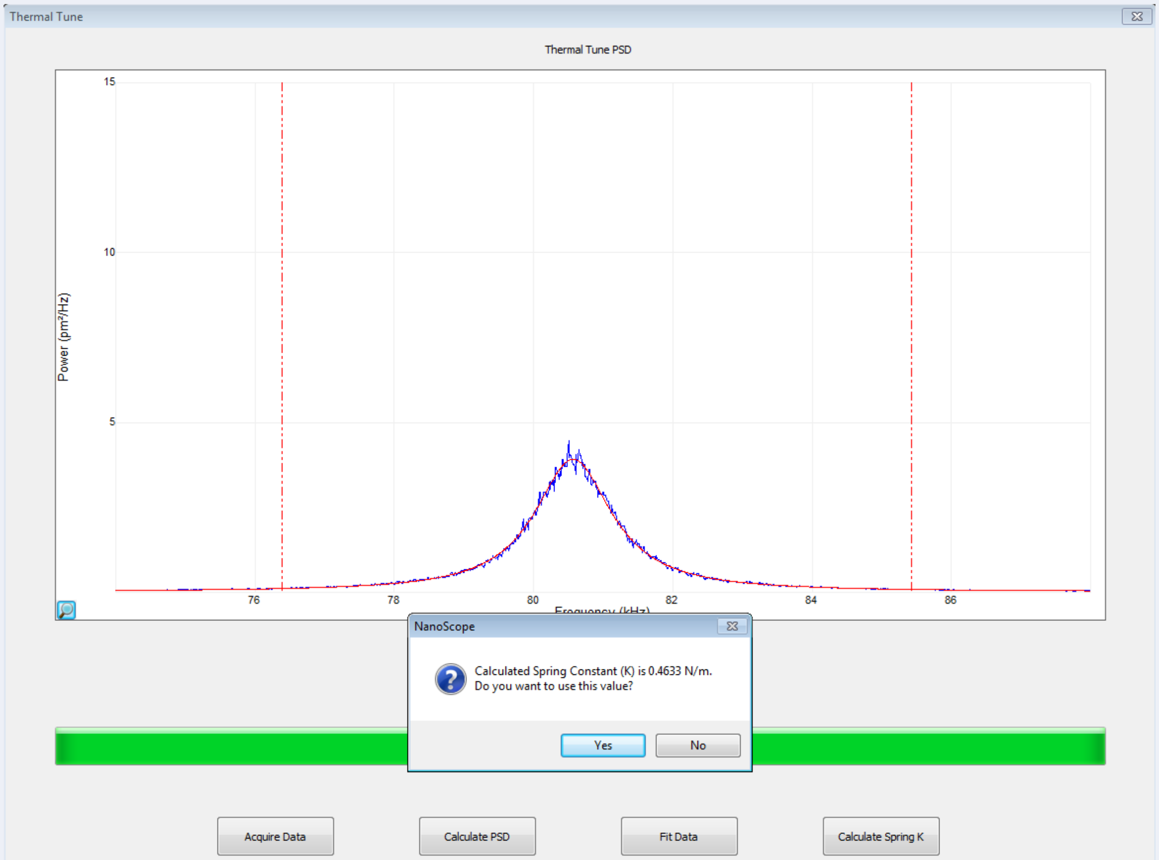

Figure 9

9. When prompted with the calculated spring constant, click "Yes". The calculated spring constant should closely match the specification of the probe tip, however, ±50% difference may occur on some tips depending on the batch quality.

   <u>Note:</u>
   For high stiffness probe tips, calculating the spring constant by way of thermal tune is usually unsuccessful. In such cases, disregard the procedure and directly enter the spring constant as specified for the probe tip into the measurement parameters under Cantilever Parameters.

Relative Calibration for Modulus Measurements

<u>Note:</u>
For calibrating modulus, it is important to choose the correct probe tip for the modulus range of interest. For the sake of this procedure, we will use the 'PDMS-SOFT-1-12M' sample, rated at 2.5GPa in conjunction with the ScanAsyst-Air probe tip. This will allow for approximate measurement of modulus on samples ranging from 0.7 - 20 MPa. Other calibration samples are available. See Table 2 below.

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SAMPLE MODULUS (E)	PROBE TIP	NOMINAL SPRING CONSTANT (k)	CALIBRATION SAMPLE	CALIBRATION SAMPLE MODULUS (E)
0.7 - 20 MPa	SNL-A / ScanAsyst-Air	0.5 / 0.4 N/m	PDMS-Soft-1-12M PDMS-Soft-2-12M	2.5 MPa 3.5 MPa
5 - 500 MPa	Tap150A	5 N/m	PS-LDPE-12M (LDPE region)	100 MPa
200 - 2000 MPa	RTESPA	40 N/m	PSFILM-12M	2.7 GPa
1 - 20 GPa	Tap525A	200 N/m	PSFILM-12M HOPG-12M	2.7 GPa 18 GPa
10 - 100 GPa	DNISP-HS	350 N/m	FSilica-12M	72.9 GPa

Table 1

1. Load the sample of known or calibrated modulus, e.g. the "PDMS-Soft-1-12M" sample, rated at 2.5GPa, from the Peakforce QNM Sample Kit.
2. Approach the surface and find the correct sample focus. Use the tip reflection method but take additional precaution as the sample is semi-transparent and the tip reflection may be easily missed.
3. Find a clean area.
4. In the Workflow Toolbar, click "Check Parameters".
5. In the Ribbon Toolbar, click on the "Expand Mode" icon.
6. Adjust the scan size to 500nm.
7. For stiff probe tips, lower the engage setpoint to 0.05V (Microscope → Engage Settings → PeakForce Engage Setpoint)
8. If the AFM front cover is open, lower and lock the front cover using the dedicated T-Allen wrench.
9. In the Workflow Toolbar, click "Engage".
10. Monitor the Trace and Retrace lines located below the Height Sensor image. In a short period of time, they should converge and coincide well with one another, indicating that the probe tip is tracking effectively and producing a clean measurement scan.
11. Within the scan parameters, perform the following adjustments:
    a. Under the Feedback group, switch ScanAsyst Auto Control to "Individual".
    b. Under the Feedback group, switch ScanAsyst Auto Setpoint to "Off".
    c. Under the Feedback group, switch ScanAsyst Auto Z Limit to "Off".
    d. Under the Cantilever Parameters, adjust Sample Poisson's Ratio per the following recommendations in Table 3:

MODULUS (E)	POISSON'S RATION (v)
E < 100 MPa	0.5
0.1 GPa < E < 1 GPa	0.4
1 GPa < E < 10 GPa	0.3

Table 3

12. Observe the force curve plots on the right hand side. The force curve trace/retrace lines should be properly synched. An example of a properly synched plot will have the following general appearance:

Figure 10

13. If necessary, click "Auto Config" several times to improve the synchronization of the trace/retrace.
14. Observe the DMTModulus channel. The trace/retrace plots are an indication of the sample modulus and should be relatively close to the known modulus of the sample.
15. Under Cantilever Parameters, adjust Tip Radius until the DMTModulus channel trace/retrace lines closely match the known modulus of the calibration sample. Typical range is between 15 – 60 nm. Allow the scan to stabilize over the course of a few minutes.

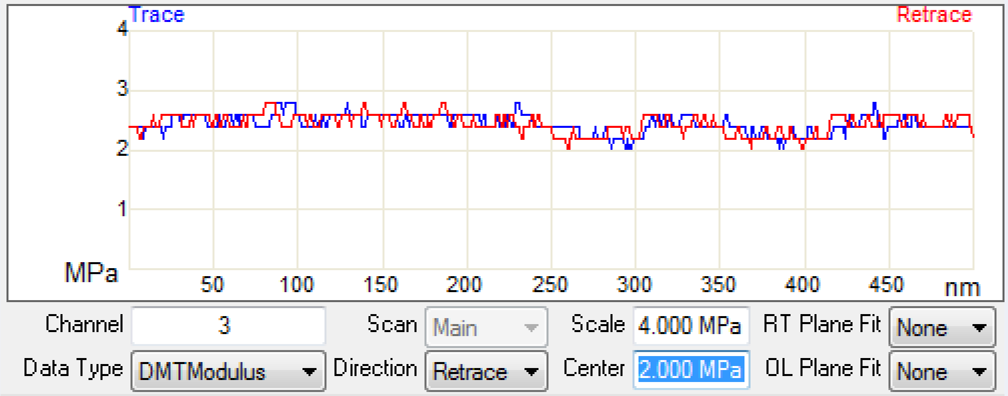

Figure 11

Note:
In Figure 11, the tip radius is adjusted to result in a modulus of roughly 2.5 MPa, as per the specification of the 'PDMS-SOFT-1-12M' sample.

16. Zoom in on the deformation portion of the force curve. The point from where the blue curve bottoms out and slopes upwards to the right is the approximate indentation. For stiff samples/probe tips, the indentation distance is sufficiently large and it is acceptable to approximate this distance visually. If the approximation is sufficient, proceed to step 17. If not, or if greater accuracy is desired, proceed to step 16a.

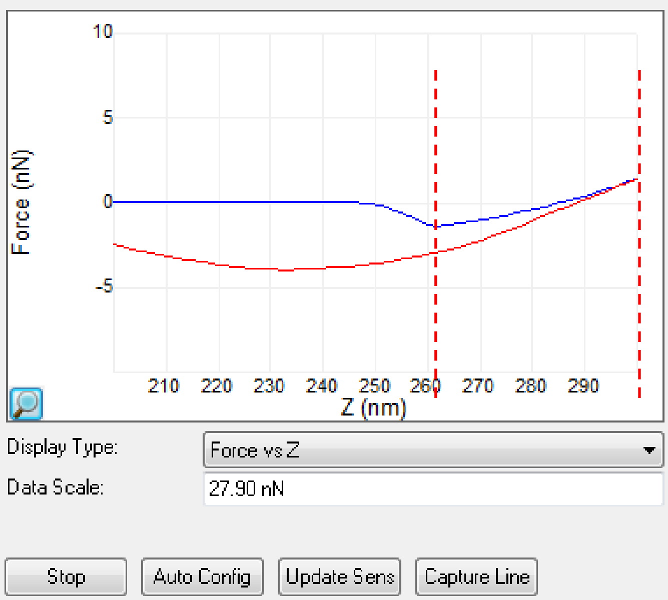

Figure 12

Note:
Figure 12, a snap shot of a dynamic force curve that can be approximated to 40nm indentation.

a. Within the force curve window, click ‘Capture Line’.
b. When prompted, click “OK”, and in the High Speed Data Capture window, click “Upload Data”
c. Close the High Speed Data Capture window.
d. Open the captured file in Nanoscope Analysis.
e. In the Nanoscope Analysis ribbon toolbar, click on the “QNM Hsdc-ForceCurve-Image” icon.
f. Click on “Export Curves…” and save the data.

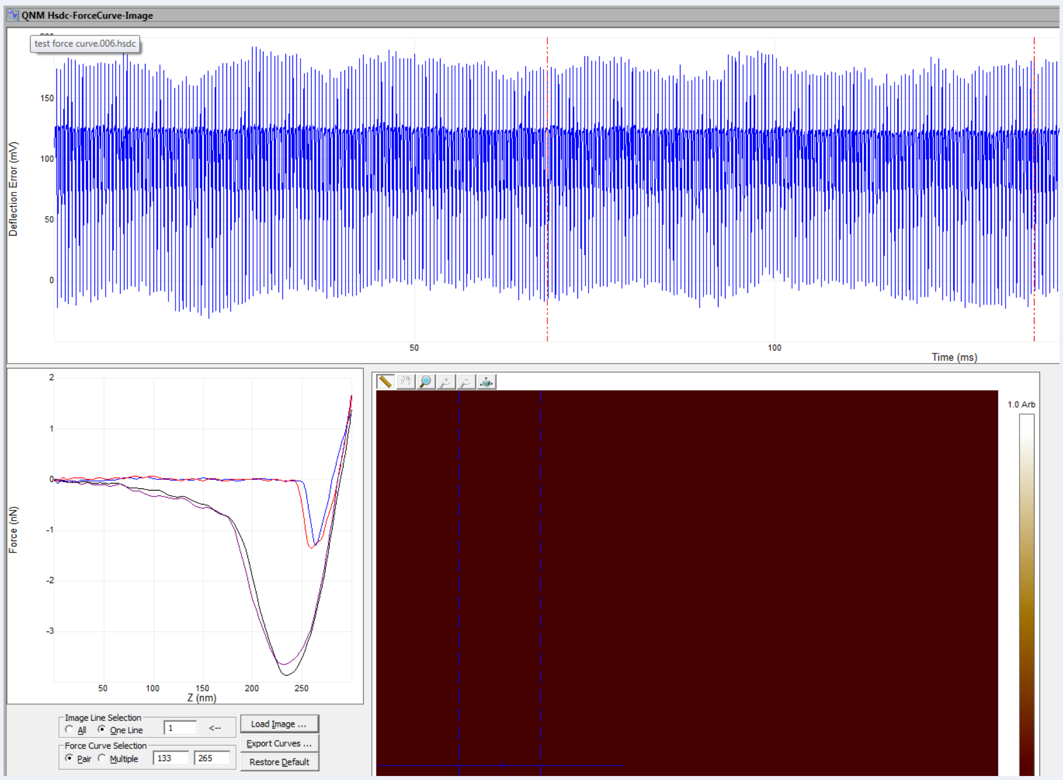

Figure 13

g. Open one of the exported force curves in a new Nanoscope Analysis window
h. Under Display Mode, change the setting to “PeakF Deflection vs Sep”.

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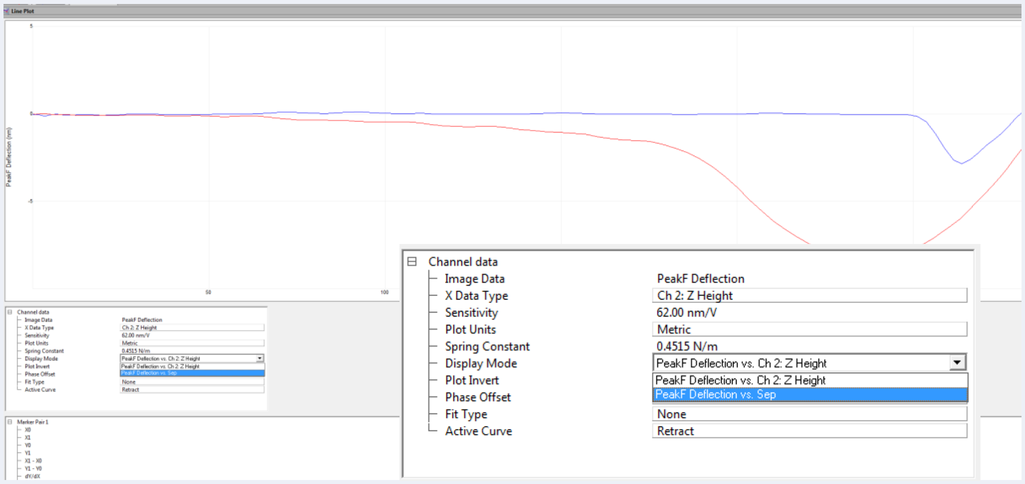

Figure 14

i. Pull in the cursors from the left, placing one at the start of the blue curve and one at the bottom, as per Figure 15.

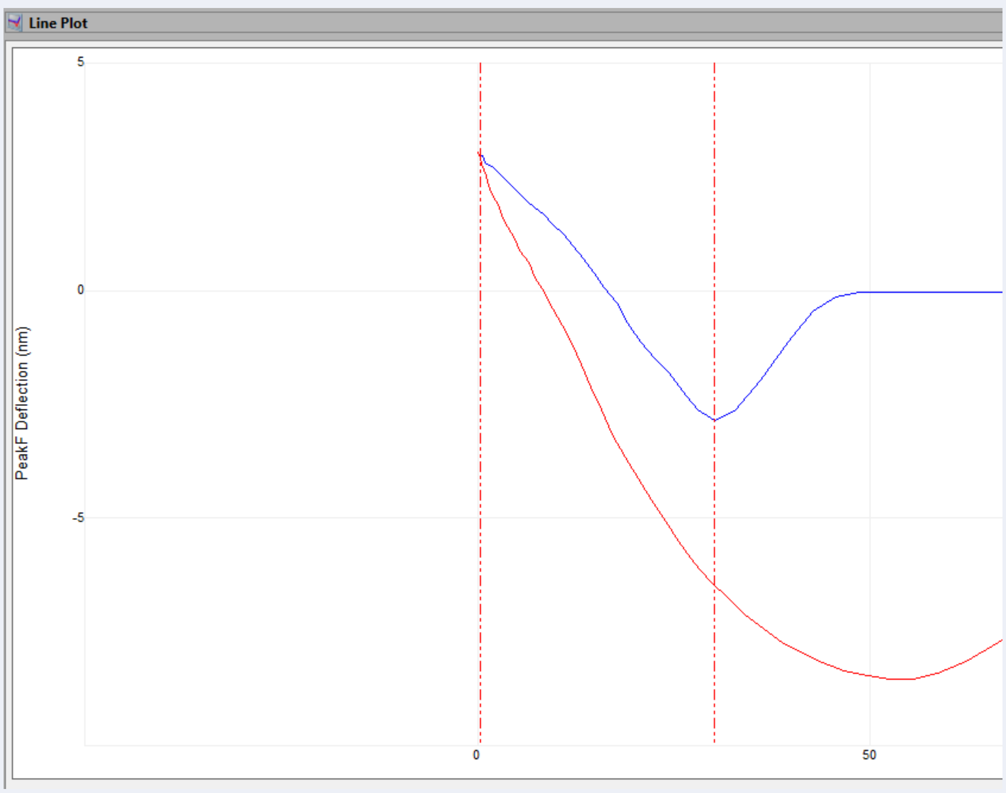

Figure 15

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   j. Measure X1 – X0. This value is one instance of the indentation distance.
   k. Repeat steps a. – j. up to 3-5 times and take an average measure of indentation distance.

17. In the Workflow Toolbar, click "Withdraw".
18. Unload the calibrated modulus sample.

Measuring Sample Modulus and Adhesion

Note:

The following procedure assumes that the prior calibration steps have been completed. To reiterate, for adhesion measurements, it is suitable to calculate only the deflection sensitivity and the spring constant by the thermal tune procedure. For modulus measurements, Relative Modulus Calibration is required in addition to the adhesion measurement requirements.

1. Load the sample to be measured.
2. Approach the surface and find the correct sample focus. Use the tip reflection method but take additional precaution as the sample is semi-transparent and the tip reflection may be easily missed.
3. Find a clean area.
4. In the Workflow Toolbar, click "Check Parameters".
5. In the Ribbon Toolbar, click on the "Expand Mode" icon.
6. Adjust the scan size to 500nm.
7. For stiff probe tips, lower the engage setpoint to 0.05V (Microscope → Engage Settings → PeakForce Engage Setpoint)
8. If the AFM front cover is open, lower and lock the front cover using the dedicated T-Allen wrench.
9. In the Workflow Toolbar, click "Engage".
10. Monitor the Trace and Retrace lines located below the Height Sensor image. In a short period of time, they should converge and coincide well with one another, indicating that the probe tip is tracking effectively and producing a clean measurement scan.

Note:
At this stage, the adhesion channel may be monitored and approximate adhesion data is provided by the trace/retrace lines. The user can take steps to capture a scan and analyze the adhesion data in Nanoscope Analysis. For measuring modulus, continue to step 11. Otherwise, proceed to conclude work at step 15.

11. Within the scan parameters, perform the following adjustments:
   a. Under the Feedback group, switch ScanAsyst Auto Control to "Individual".
   b. Under the Feedback group, switch ScanAsyst Auto Setpoint to "Off".
   c. Under the Feedback group, switch ScanAsyst Auto Z Limit to "Off".

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12. Observe the force curve plots on the right hand side. The force curve trace/retrace lines should be properly synched.
13. Zoom in on the deformation portion of the force curve. The point from where the blue curve bottoms out and slopes upwards to the right is the approximate indentation. For stiff samples/probe tips, the indentation distance is sufficiently large and it is acceptable to approximate this distance visually. If the approximation is sufficient, proceed to step 12. If not, or if greater accuracy is desired, repeat the same procedure for force curve collection and offline measurement as detailed in the Relative Calibration for Modulus procedure.
14. Adjust the Peak Force Setpoint such that the indentation distance is the same as observed during the Relative Calibration for Modulus procedure. Allow sufficient time between adjustments for the force curves to settle. Continue to adjust the Peak Force Setpoint and collect force curves to verify the indentation distance until satisfactory results are achieved.

    Note:
    At this stage, the DMTModulus channel may be monitored and approximate modulus data is provided by the trace/retrace lines. The user can take steps to capture a scan and analyze the modulus data in Nanoscope Analysis.

15. Upon completion of work, in the Workflow Toolbar, click "Withdraw".
16. Unload the sample.

System Shutdown / Startup

Note:
This procedure should only be undertaken by 4D LABS Staff in the event of a planned power outage or extended facility shutdown.

1. Close all open software by clicking in the top right of each open window.
2. Turn off the computer by clicking 'Start' followed by 'Shutdown'.
3. Beneath the table top and behind the left hand door, turn off the AFM hardware via the green power switch.
4. Turn off the UPS power supply located on the far left.
5. Follow the procedure in reverse, followed by normal computer startup to return the AFM to normal operation.

References and Files

Bruker ICON User Manual

Contact Information

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