## Document Revision History: PeakForce QNM User Guide

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<th>Ref. DCR</th>
<th>Approval</th>
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Dimension®
Dimension® Icon®
BioScope™
BioScope™ Catalyst™
Atomic Force Profiler™ (AFP™)
Dektak®

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Tapping™
TappingMode+™
LiftMode™
AutoTune™
TurboScan™
Fast HSG™
PhaselImaging™
DekMap 2™
HyperScan™
StepFinder™
SoftScan™
ScanAsyst™
Peak Force Tapping™
PeakForce™ QNM™

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TrakScan™
StiffStage™

Hardware Options:
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Signal Access Module™ and SAM™
Extender™
TipView™
Interleave™
LookAhead™
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Software Options:
NanoScript™
Navigator™
FeatureFind™

Miscellaneous:
NanoProbe®

Cover Image: Anti-bacterial film consisting of poly(methyl methacrylate) and silver nanoparticles. The sample was imaged on a Dimension Icon using PeakForce QNM at a scan size of 13.5 μm. The data shown is adhesion data overlaid on topography. The dark red spots correspond to the location of silver nanoparticles, which are difficult to identify using topography alone. (Sample courtesy of Mishae Khan and Daniel Bubb, Rutgers University.)
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Chapter 1  Introduction to PeakForce QNM Microscopy

1.1 Introduction

PeakForce QNM (Quantitative NanoMechanics), an extension of Peak Force Tapping™ mode, enables quantitative measurement of nano-scale material properties such as modulus, adhesion, deformation and dissipation. Because Peak Force Tapping Mode controls the force applied to the sample by the tip, sample deformation depths are small and the effect of the substrate on the measured modulus is decreased.

PeakForce QNM can provide compositional mapping of a complex composite material while providing equal or higher resolution than a TappingMode image (~5nm). Peak Force Tapping Mode has high spatial resolution, relatively high speed, and can detect a large range of elasticities. PeakForce Tapping mode produces similar results to HarmoniX (see the HarmoniX User Guide, Bruker p/n 004-1024-000 for details) but is much easier to use and covers a wider modulus and adhesion range.

With a calibrated cantilever, Peak Force QNM is quantitative and has high spatial resolution.

Peak Force Tapping Mode™ microscopy, the core technology behind PeakForce QNM and ScanAsyst™, is a new, Bruker-proprietary, primary Atomic Force Microscopy (AFM) mode. Other primary AFM modes include Contact, Tapping, Scanning Tunnelling Microscopy (STM) and Torsional Resonance modes.

Peak Force Tapping mode oscillates, but far below the cantilever resonant frequency, the vertical motion of the cantilever using the (main) Z piezo element and relies on peak force for feedback. Peak interaction force and nanoscale material property information is collected for each individual tap.

Because Peak Force Tapping mode does not resonate the cantilever, cantilever tuning is not required. This is particularly advantageous in fluids.

Peak Force Tapping Mode includes auto-optimization (called ScanAsyst) of scanning parameters, including gains, setpoint and scan rate. This enables users to rapidly obtain high quality images. ScanAsyst is intended to be the first choice imaging mode for NanoScope version 8.10 and later software.

Because Peak Force Tapping mode controls the applied force, tip wear is reduced.

Peak Force Tapping mode imaging increases the resolution by controlling the force that the tip applies to the sample thereby decreasing the deformation depths; this decreases the contact area
between the tip and sample. Because the deformation depths and lateral forces are small, there is minimal damage to the probe or sample.

1.2 What is in the PeakForce QNM kit?

The PeakForce QNM kit consists of the following items:

1. Software keys to enable PeakForce QNM in real-time and off-line operation.

2. A pack of ten each of the following probes:
   • ScanAsyst-Air
   • Tap150A, P/N MPP-12120-10
   • Tap300A (RTESPA), P/N MPP-11120-10
   • Tap525A, P/N MPP-13120-10

3. One pre-mounted DNISP-HS probe in the appropriate probe holder.

4. PeakForce QNM samples. Refer to PeakForce QNM Samples: Chapter 3 for details.

5. One day of PeakForce QNM applications training

1.3 Conventions and Definitions

In the interest of clarity, certain nomenclature is preferred. An SPM probe is comprised of a tip affixed to a cantilever mounted on a substrate, which is inserted in a probe holder.

Three font styles distinguish among contexts. For example:

**Window or Menu Item / Button or Parameter Name** is set to **Value**.
Chapter 2  PeakForce QNM Operation

2.1 Introduction

This chapter describes how to perform a simple PeakForce QNM experiment. Later sections will discuss PeakForce QNM parameters and their influence on the measurements.

2.2 PeakForce QNM Principles of Operation

2.2.1 Peak Force Tapping Mode

Peak Force Tapping mode, the core technology behind PeakForce QNM and ScanAsyst modes, performs a very fast force curve at every pixel in the image. The peak interaction force of each of these force curves is then used as the imaging feedback signal. Peak Force Tapping mode modulates the Z-piezo at ~2 kHz (Icon, MultiMode. Catalyst operates at ~1 kHz) with a default Peak Force Amplitude of 150 nm (0-peak). Analysis of force curve data is done on the fly, providing a map of multiple mechanical properties that has the same resolution as the height image.
2.2.2 The “Heartbeat”

The Force vs. Time display, shown in Figure 2.2a is referred to as the “heartbeat.” The initial contact of the probe with the sample (B), peak force (C) and adhesion (D) points are labelled.

![Figure 2.2a](image)

2.2.3 Force curves

Using the Z-position information, the heartbeat is transformed into a force curve, shown in Figure 2.2b. The force curve plot is analyzed, on the fly, to produce the peak interaction force as the control feedback signal and the mechanical properties of the sample (Adhesion, Modulus, Deformation, Dissipation).

![Figure 2.2b](image)
2.3 PeakForce QNM Probe Selection

It is important to choose a probe that can cause enough deformation of the sample and still retain high force sensitivity. Therefore cantilever stiffness should be selected based on the sample stiffness. Bruker’s recommendations are shown in Table 2.3a.

Table 2.3a Recommended Probes

<table>
<thead>
<tr>
<th>Sample Modulus (E)</th>
<th>Probe</th>
<th>Nominal Spring Constant (k)</th>
</tr>
</thead>
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<tr>
<td>1 MPa &lt; E &lt; 20 MPa</td>
<td>ScanAsyst-Air</td>
<td>0.5 N/m</td>
</tr>
<tr>
<td>5 MPa &lt; E &lt; 500 MPa</td>
<td>Tap150A, P/N MPP-12120-10</td>
<td>5 N/m</td>
</tr>
<tr>
<td>200 MPa &lt; E &lt; 2000 MPa</td>
<td>Tap300A (RTESPA), P/N MPP-11120-10</td>
<td>40 N/m</td>
</tr>
<tr>
<td>1 GPa &lt; E &lt; 20 GPa</td>
<td>Tap525A, P/N MPP-13120-10</td>
<td>200 N/m</td>
</tr>
<tr>
<td>10 GPa &lt; E &lt; 100 GPa</td>
<td>DNISP-HS</td>
<td>350 N/m</td>
</tr>
</tbody>
</table>

**Note:** The recommended spring constants are general guidelines that reflect a compromise between image resolution and modulus accuracy. E.g. a stiff cantilever will improve modulus accuracy at the expense of damaging the sample.

To reduce optical interference, probes should be coated on their back side.


2.4 Basic PeakForce QNM Operation

This section describes how to perform a simple PeakForce QNM experiment. Later sections will discuss PeakForce QNM parameters and their influence on the measurements.

2.4.1 Select the Microscope

Follow the Select Microscope procedure described in your microscope Instruction Manual.
2.4.2 Configure the Hardware

1. If you have a MultiMode, set the mode selector switch on the MultiMode base to AFM & LFM. See Figure 2.4a.

Figure 2.4a Mode selector switch
2.4.3 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the Select Experiment window, shown in Figure 2.4b.

**Figure 2.4b** The PeakForce QNM in Air Select Experiment window

2. Select MECHANICAL PROPERTIES in the Experiment Category panel.

3. Select QUANTITATIVE NANOMECHANICAL MAPPING in the Select Experiment Group panel.

4. Select PEAKFORCE QNM IN AIR in the Select Experiment panel and click LOAD EXPERIMENT.
5. This opens the **Workflow Toolbar**, the **Scan 4 Channels** (Icon) windows, the **Force Monitor** window and the **Scan Parameters List** window, shown in **Figure 2.4c**.

**Figure 2.4c** PeakForce QNM in Air (Simple Mode) configuration

---

**2.4.4 Head, Cantilever and Sample Preparation**

1. Install a suitable probe onto an AFM cantilever holder. See **PeakForce QNM Probe Selection**: **Section 2.3**.

2. Load the cantilever holder with installed tip into your microscope.
2.4.5 Align Laser

1. Align the laser using the laser control knobs.

   **Note:** Coated cantilevers are strongly recommended to increase the laser sum signal and decrease interference effects.

   **Note:** Maximize the laser sum signal to avoid optical interference.

   **Note:** Try not to change the laser spot position during the experiment. This may change the Deflection Sensitivity and therefore the property measurements.

2.4.6 Adjust Photodetector

1. Adjust the photodetector.
2.4.7 Set Initial Scan Parameters

Scan Panel

In the Scan panel of the Scan Parameters List, set the following initial scan parameters (see Figure 2.4d).

1. Set the Scan Size.
2. Set the Scan Angle.

Feedback Panel

1. Set ScanAsyst Auto Control to ON (see Figure 2.4d).

Figure 2.4d  PeakForce QNM in Air (SIMPLE MODE) Parameters Panel
Channels

1. Set the **Channel 1 Data Type** to **HEIGHT SENSOR** (see Figure 2.4e).
2. Set the **Channel 2 Data Type** to **PEAK FORCE ERROR** (see Figure 2.4e).
3. Set the **Channel 3 Data Type** to **DMT MODULUS** (see Figure 2.4e).
4. Set the **Channel 4 Data Type** to **LOGDMT MODULUS** (see Figure 2.4e).
5. Set the **Channel 5 Data Type** to **ADHESION** (see Figure 2.4e).
6. Set the **Channel 6 Data Type** to **DEFORMATION** (see Figure 2.4e).
7. Set the **Channel 7 Data Type** to **DISSIPATION** (see Figure 2.4e).
8. Set **Data Scale** to a reasonable value for the sample or click the **AUTO SCALE** icon after engaging.

   **Note:** For example, for a 200nm step height calibration sample, a reasonable **Data Scale** setting is 300nm initially.

9. Set **Line direction** to either **TRACE** or **RETRACE**.

**Figure 2.4e**  Suggested PeakForce QNM Channel Settings

<table>
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<th>Channel</th>
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<th>Man</th>
<th>Scale</th>
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<td>Height Sensor</td>
<td></td>
<td></td>
<td>50.00</td>
<td>RT Plane</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
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2.4.8 Engage

1. Select Microscope > Engage or click the ENGAGE icon on the Workflow Toolbar. A pre-engage check begins, followed by Z-stage motor motion.

2. To move to another area of the sample, execute a Withdraw command to avoid damaging the tip and scanner.

2.4.9 Image the sample

1. If needed, right-click in the Force Monitor window and click UNDOCK. See Figure 2.4f. You may DOCK the undocked Force Monitor window by right-clicking in it and clicking DOCK.

Figure 2.4f  Undock the Force Monitor window
2. Select one plot to be **FORCE VS. TIME** and the other to be **FORCE VS. Z**.

3. Once scanning, the **Force Monitor** window, shown in Figure 2.4g, should display a **Force vs. Z** plot and a “heartbeat” (**Force vs. Time**) plot.

**Figure 2.4g  Force Monitor window**

Note: The cantilever oscillation after it snaps off the sample surface, shown in Figure 2.4g, is normal. On occasion this oscillation will continue until the probe tip again contacts the sample surface. This oscillation will be heavily damped by this contact. Even if the oscillation is not fully damped, the remaining oscillation at the force peak will be small and will merely add a small amount of noise to the feedback.
4. The **HEIGHT** channel in the **Scan** window, shown in **Figure 2.4h**, will display a topographical image of your sample.

**Figure 2.4h**  Height Image of a PS + LPDE blend.
2.5 PeakForce QNM Channels

This section discusses channels that are specific to PeakForce QNM mode. Mechanical properties can be extracted from the calibrated (see Chapter 4) force curves.

2.5.1 DMT Modulus

The reduced Young’s Modulus, $E^*$, is obtained by fitting the retract curve (green line in Figure 2.5a) using the Derjaguin, Muller, Toropov (DMT) model\(^1\) given by

$$F_{\text{tip}} = \frac{4}{3} E^* \sqrt{Rd^3} + F_{\text{adh}}$$

Where $F_{\text{tip}}$ is the force on the tip, $F_{\text{adh}}$ is the adhesion force, $R$ is the tip end radius and $d$ is the tip-sample separation.

Figure 2.5a Force vs. Separation plot

---

Figure 2.5b shows a DMT Modulus map of PS+LDPE blend.

Figure 2.5b  DMT Modulus map of a PS+LDPE blend

2.5.2 Log DMT Modulus

The logarithm of the elastic modulus of the sample based on the DMT model.
2.5.3 Adhesion

The peak force below the baseline, shown in Figure 2.5c. Figure 2.5d shows an adhesion map of a PS+LPDE blend.

**Figure 2.5c**  Adhesion on a PS+LDPE blend

**Figure 2.5d**  Adhesion map of a PS+LDPE blend
2.5.4 Peak Force

This channel produces a map of the peak force (see Figure 2.5g) measured during the scan. Because the PeakForce QNM mode uses peak force as the feedback signal, this channel is essentially the Peak Force Setpoint plus the error. Figure 2.5e and Figure 2.5f illustrate the peak force location.

**Figure 2.5e** The “heartbeat,” Force vs. Time

**Figure 2.5f** Force curve, Force vs. distance
Figure 2.5g  Peak Force Error map of a PS+LDPE blend
2.5.5 Dissipation

Energy Dissipation (W) is given by the force times the velocity integrated over one period of the vibration:

$$W = \int_{0}^{T} F \cdot \dot{Z} \, dt = \int F \cdot dZ$$

$F$ is the interaction force vector and $dZ$ is the displacement vector. Because the $Z$ motion and the velocity reverse direction in a half cycle, the integral is zero if the load and unload curves coincide. The dissipation is therefore the hysteresis between the load and unload curves. Pure elastic deformation has no hysteresis which corresponds to very low dissipation. Energy dissipated is displayed in electron volts as the mechanical energy lost per tapping cycle.

The Dissipation channel plots the dissipated energy in each cycle by integrating the area between the Trace (load or extend) and Retrace (unload or retract) curves as shown in the blue area in Figure 2.5h.

**Figure 2.5h** Dissipation (shaded area) in a polystyrene (PS) and Low-density polyethylene (LPDE) blend
Figure 2.5i shows the dissipation image of a PS+LDPE blend.

**Figure 2.5i** Dissipation image of a PS+LDPE blend
2.5.6 Deformation

The maximum deformation of the sample (defined as the distance from the base of the Deformation Fit Region position to the peak interaction force position) caused by the probe. See Figure 2.5j. Figure 2.5k shows a deformation map of a PS+LPDE blend.

Note: The total deformation will be slightly larger than the displayed deformation because the default Deformation Fit Region is 85% of the full deformation. See Deformation Fit Region: Page 28.

**Figure 2.5j** Deformation
2.6 PeakForce QNM Parameters

2.6.1 Feedback Parameters

Peak Force Setpoint

The setpoint for peak force. If the deflection sensitivity is calibrated, the force (in Newtons) will be displayed.

When the ScanAsyst Setup is ON, Peak Force Setpoint is automatically controlled by NanoScope software. Under some conditions, you may desire to control the Peak Force Setpoint manually. A Peak Force Setpoint that is too high can either damage the sample or wear the tip. It is generally desirable to reduce the Peak Force Setpoint to as small a value as is possible. However, in order to achieve accurate Elastic modulus measurement, sufficient sample deformation is needed. If the deformation is less than 2nm, increase the Peak Force Setpoint to achieve sufficient sample deformation.

Note: When performing AUTO CONFIG operations with a small Peak Force Setpoint (less than ~20mV), the tip may drift out of contact with the surface and will be unable to return and track the surface. It is therefore recommended using a relatively large Peak Force Setpoint while performing AUTO CONFIG operations and reducing the Peak Force Setpoint later if necessary.
Feedback Gain

The gain of the Peak Force Tapping feedback control loop.

**Note:** Both Peak Force Setpoint and Feedback Gain are dynamically and automatically controlled when ScanAsyst Auto Control is set to ON.

**Note:** A Feedback Gain that is too large will cause oscillation of the system and increase noise, while too small a Feedback Gain will result in poor sample tracking.

Low Pass Deflection Bandwidth

The low pass filter is used to reduce deflection noise. Lower bandwidths will reduce noise but will distort the force curve and introduce errors in quantitative nanomechanical property measurements.

*Range and Settings:* 10 kHz - 65.56 kHz *(Default value: 40 kHz).*

ScanAsyst Setup

*Range and Settings:* **NEVER:** Does not allow ScanAsyst Auto Control.

**ALLOW:** Allows ScanAsyst Auto Control.

**Note:** **SHOW ALL,** discussed in the *NanoScope Software Version 8 User Guide,* must be enabled to view and edit this parameter.

ScanAsyst Noise Threshold

ScanAsyst Noise Threshold is linked to the Feedback Gain and is used to tune it. Larger ScanAsyst Noise Thresholds will result in better sample tracking but increased oscillation noise. Lower ScanAsyst Noise Thresholds will result in a cleaner image but the sample tracking will suffer.

*Range and Settings:* 0.5 nm is appropriate for most samples while 1 nm is appropriate for rough samples and 0.05 nm may be appropriate for very flat samples.

**Note:** When ScanAsyst Auto Z Limit control is turned ON, the ScanAsyst Noise Threshold parameter is automatically set by the program and cannot be changed.
ScanAsyst Auto Config Frames

At the end of every N frames, an AUTO CONFIG operation is performed.

Range and Settings: 0 - 100. If ScanAsyst Auto Config Frames = 0, periodic AUTO CONFIG operations are not performed.

ScanAsyst Auto Control

Range and Settings: OFF: Turns ScanAsyst Auto Control OFF.
ON: Turns ScanAsyst Auto Control ON.
INDIVIDUAL: Allows individual control of ScanAsyst Auto Gain, ScanAsyst Auto Setpoint, ScanAsyst Auto Scan Rate and ScanAsyst Auto Z Limit.

ScanAsyst Auto Gain

ScanAsyst Auto Gain allows NanoScope to dynamically control Feedback Gain.

Range and Settings: OFF: Turns ScanAsyst Auto Gain OFF.
ON: Turns ScanAsyst Auto Gain ON.

ScanAsyst Auto Setpoint

ScanAsyst Auto Setpoint allows NanoScope to dynamically control the Peak Force Setpoint.

Range and Settings: OFF: Turns ScanAsyst Auto Setpoint OFF.
ON: Turns ScanAsyst Auto Setpoint ON.

Note: This option is very useful for users who want to change the Peak Force Setpoint manually to achieve adequate deformation on the sample while leaving ScanAsyst Auto Gain ON.

ScanAsyst Scan Auto Scan Rate

ScanAsyst Scan Auto Scan Rate allows NanoScope to control the Scan Rate.

Range and Settings: OFF: Turns ScanAsyst Scan Auto Scan Rate OFF.
ON: Turns ScanAsyst Scan Auto Scan Rate ON.
ScanAsyst Auto Z Limit

ScanAsyst Auto Z Limit allows NanoScope to control the Z Limit. The ScanAsyst Auto Z Limit function will detect if the surface is sufficiently smooth to allow reduction of the Z Limit and thus avoid bit noise in the Height and Height Sensor channel. This will be effective after a whole frame of the image is scanned. If the Z Limit needs to be reduced, the ScanAsyst Noise Threshold will automatically be reduced to 0.15 times the original ScanAsyst Noise Threshold to reduce the oscillation noise for smooth samples.

Range and Settings: Off: Turns ScanAsyst Auto Z Limit Off.

On: Turns ScanAsyst Auto Z Limit On.

2.6.2 PeakForce QNM Control Parameters

Peak Force Amplitude

The zero-to-peak amplitude of the cantilever drive in the Z axis (Z modulation). Increasing Peak Force Amplitude will reduce the contact time during each tip tapping cycle on the sample and help tracking the rough and/or sticky sample by avoiding a situation where the tip is unable to pull off from the sample. Reduced Peak Force Amplitude is desired in liquid on flat samples. Less Peak Force Amplitude results in less hydrodynamic force disturbance.

Lift Height

The distance that the Z-piezo is retracted from the sample during an AUTO CONFIG operation.

Note: Changing the Lift Height will automatically start the AUTO CONFIG function (see Optimizing a ScanAsyst Image: Page 38) and retract the Z piezo to the specified Lift Height. Clicking AUTO CONFIG will automatically calculate the Lift Height and perform an AUTO CONFIG operation.
**Top Fit Region**

The **Top Fit Region** of the unload force curve, shown in Figure 2.6a, is excluded from the DMT Modulus calculations.

**Note:** A smaller **Top Fit Region** means that less region of the force curve is excluded from the DMT modulus calculations.

*Range and Settings*: 0 - 94%.

*Typical*: 10%.

**Figure 2.6a** DMT Fit regions of the Force curve

---

**Unload Fit Region**

The **Unload Fit Region** of the force curve, shown in Figure 2.6a, is included in the DMT Modulus calculations.

*Range and Settings*: 0 - 100%. 100% is defined as the force between the adhesion point and the peak force.

*Typical*: 70%.

The portion of the force curve between the **Top Fit Region** and the **Unload Fit Region** is included in the DMT Modulus calculations. For typical numbers discussed here, the region between 10% and 70% of the unload force curve will be included in the DMT modulus calculations.
Deformation Fit Region

The Deformation Fit Region of the load force curve, shown in Figure 2.6b, is excluded from the Deformation channel display. This parameter is used to reduce the effect of baseline noise.

Range and Settings: 50 - 100%. 100% is defined as the force between the zero force point and the peak force in the load curve.

Typical: 85%. The portion of the force curve above the 85% point is displayed in the Deformation channel.

Figure 2.6b  DMT Fit regions of the Force curve
2.6.3 Cantilever Parameters

The following parameters are needed to calibrate PeakForce QNM.

**Spring Constant**

Measure the spring constant of the probe and input that value into this panel. Spring constant may be measured using the Thermal Tune function in NanoScope software. Refer to *Calibrate the Spring Constant Using Thermal Tuning: Section 4.4* for details.

**Tip Radius**

Measure the tip radius and input the value in this panel. Tip radius may be measured using a tip characterizer sample and the Tip Qualification function in NanoScope software. Refer to *Measure the Tip Radius: Section 4.5* for details.

**Poisson's Ratio**

Poisson’s ratio of the sample. This is used to calculate the sample modulus, $E_s$, from the measured reduced modulus, $E^*$: Refer to *Calibrate Peak Force QNM: Section 4.6* for details.
2.6.4 PeakForce QNM Limits

These parameters work like other limits in the NanoScope software. Numbers in the NanoScope software are represented using 16 bits and thus various quantities are represented as illustrated in Figure 2.6c.

Figure 2.6c Illustration of PeakForce QNM Limits

As with other limits, setting the limit too high increases the bit noise. Setting the limit too low can result in wrapped data and inverted contrast. The limits of the following parameters can be set by the user:

- Force Limit: affects the Peak Force and Adhesion channels.
- Dissipation Limit: affects the Dissipation channel.
- DMT Modulus Limit: affects the DMT Modulus channel.
- LogDMT Modulus Limit: affects the LogDMT Modulus channel.
- LogDMT Modulus Offset: affects the LogDMT Modulus channel.
2.6.5 Limits Parameters

Z Limit

Permits attenuation of maximum allowable Z voltage and vertical scan range to achieve higher resolution (smaller quantization) in the Z direction.

Range or Settings:

- Dimension Icon: 8.33 V (~0.241 μm) to 309.3 V (~9 μm).
- MultiMode 8: 11 V (~0.1375 μm) to 416 V (~5 μm).
- BioScope Catalyst: 1 V (~0.1252 μm) to 145 V (~18 μm).

Note: SHOW ALL, discussed in the NanoScope Software Version 8 User Guide, must be enabled to view and edit this parameter.

Z Range

Permits attenuation of the range of the Z piezo as measured by the Z sensor to achieve higher resolution (smaller quantization) in the Z direction.

Range or Settings:

- Dimension Icon: 0.2 nm to ~10 microns
- BioScope Catalyst: 0.2 nm to ~28 microns

Note: The Z Range parameter does not apply to the MultiMode because it does not incorporate a Z sensor.

Deflection Limit

Use this parameter to attenuate the maximum allowable deflection signal to achieve higher resolution. If this value is too small, saturation of the Deflection channel will occur.

Range or Settings: 4.096V - 24.58V.
2.6.6 Other Parameters

Peak Force Engage Setpoint

Laser interference from reflective samples can cause a “false engage” which can be avoided by using a large Peak Force Engage Setpoint. This is why a relatively large and conservative Peak Force Engage Setpoint default value is used. But large Peak Force Engage Setpoints can damage samples and probes, particularly cantilevers with high spring constants. To reduce the engage force, reduce the Peak Force Engage Setpoint. When you reduce Peak Force Engage Setpoint, you should also reduce SPM engage step, found in Microscope > Engage Settings > General, shown in Figure 2.6d.

Range or Settings:

- Dimension Icon: 0.001 V - 1.229 V (Default value: 0.15 V).
- MultiMode 8: 0.001 V - 1.229 V (Default value: 0.15 V).
- BioScope Catalyst: 0.001 V - 1.229 V (Default value: 0.3 V).

![Figure 2.6d SPM engage step](image-url)
Medium

The medium surrounding the sample and probe. This parameter is selected when you select either PeakForce QNM in Air

Range and Settings: **AIR**, **FLUID**.

**Note:** **SHOW ALL**, discussed in the *NanoScope Software Version 8 User Guide*, must be enabled to view and edit this parameter.
2.6.7 Parameter Visibility

The visibility of various parameters depends on the selected mode. Table 2.6a shows parameter visibility as a function of microscope mode.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Simple Mode</th>
<th>Expanded Mode</th>
<th>Show All</th>
<th>Other Dependencies</th>
</tr>
</thead>
<tbody>
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<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>Feedback Gain</td>
<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>Low Pass Deflection Bandwidth</td>
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<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>ScanAsyst Setup</td>
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<td>No</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>ScanAsyst Noise Threshold</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
<td></td>
</tr>
<tr>
<td>ScanAsyst Auto Config Frames</td>
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<td>No</td>
<td>Yes</td>
<td></td>
</tr>
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<td>ScanAsyst Auto Control</td>
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</tr>
<tr>
<td>ScanAsyst Auto Gain</td>
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<td>Yes</td>
<td>Yes</td>
<td>ScanAsyst Auto Control</td>
</tr>
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<td>ScanAsyst Auto Setpoint</td>
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<td>Yes</td>
<td>ScanAsyst Auto Control</td>
</tr>
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<td>Expanded Mode</td>
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<td></td>
<td>Deformation Fit Region</td>
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<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td></td>
<td>Poisson’s Ratio</td>
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<td>Yes</td>
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<td>Peak Force QNM Limits</td>
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<td></td>
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<td>Yes</td>
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<tr>
<td></td>
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<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td></td>
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<tr>
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<td>No</td>
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</tr>
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<tr>
<td></td>
<td>Deflection Limit</td>
<td>No</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>
2.7 Capture Buttons

The capture buttons in the Force Monitor window allow you to collect data for use with the NanoScope Analysis off-line analysis software.

1. Start to collect a ScanAsyst/Peak Force Tapping image.

2. When you are in a region of interest, click the CAPTURE LINE button, shown in Figure 2.7a, to capture a scan line.

Figure 2.7a CAPTURE LINE button
3. The **High Speed Data Capture** window, shown in Figure 2.7b, will open and the **Status** will change when the data has been captured. **UPLOAD DATA** to the PC when the capture is complete. When **CAPTURE LINE** is used this way, the off-line NanoScope Analysis software will correctly associate the capture line of the high speed data capture with the line in the image.

**Figure 2.7b** High speed data capture is complete. However, the data is not immediately transferred to the PC.

4. Click the **UPLOAD DATA** button to transfer the captured data to the computer. While the data transfer process takes place, the scan data will look corrupted because the DSP time is shared between PeakForce QNM properties computation and data transfer.
2.8 Optimizing a ScanAsyst Image

When the relative Z position between the probe and sample is modulated, parasitic cantilever motions occur. These motions include free-cantilever oscillation after snapping off the surface, deflection triggered by harmonics of the piezo motion or viscous forces. This parasitic deflection, defined as the deflection signal variation when the tip is NOT interacting with the sample, limits the low force range of ScanAsyst operation. Low force control is the most important factor to achieve high resolution imaging and property measurements.

During peak force tapping operation, the Auto Config operation is used to analyze the parasitic deflection signal including its data pattern by comparing the known source of parasitic excitation, namely the cantilever resonance at pulling off, modulation harmonics and other system actuation sources. The signature of the interaction, in the shape of heartbeat signal, is extracted from the parasitic deflections. The recovered heartbeat signal becomes the interaction force curve plotted in the time domain.

Figure 2.8a shows the heartbeat and force vs. Z curves of an image before and after Auto Config correction. The low frequency noise in the baseline has been removed.

Figure 2.8a The heartbeat and force curves of an image before (left) and after (right) Auto Config correction.
If your force vs. time curves show parasitic background noise or the force vs. height curve load and unload curves are overlapping due to background noise, click the **AUTO CONFIG** button, shown in Figure 2.8b, to invoke the real-time pattern analysis algorithm that removes parasitic deflection.

**Note:** Clicking **AUTO CONFIG** will automatically calculate the **Lift Height** and perform an **AUTO CONFIG** operation.
Note: When performing AUTO CONFIG operations with a small Peak Force Setpoint (less than ~20mV), the tip may drift out of contact with the surface and will be unable to return and track the surface. It is therefore recommended using a relatively large Peak Force Setpoint while performing AUTO CONFIG operations and reducing the Peak Force Setpoint later if necessary.

2.9 Advanced Atomic Force Operation

2.9.1 Displaying Parameters

You can adjust the number of parameters shown in the Scan Parameter List using several methods.

Simple Mode

1. The default SIMPLE MODE, intended for novice users and shown in Figure 2.9a, displays the minimum number of parameters needed to make an image.

Figure 2.9a The SIMPLE MODE view of the Scan Parameter List for PeakForce QNM in Air
Expanded Mode

1. The **Expanded Mode** view, shown in Figure 2.9b, increases the number of displayed parameters enabling expert users to fine tune an image.

**Figure 2.9b** The **Expanded Mode** view of the Scan Parameter List for ScanAsyst in Air

<table>
<thead>
<tr>
<th><strong>Scan</strong></th>
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<tbody>
<tr>
<td>Scan Size</td>
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<tr>
<td>Aspect Ratio</td>
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<td>X Offset</td>
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<td>Y Offset</td>
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<table>
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<td>Peak Force Setpoint</td>
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<td>Feedback Gain</td>
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<th><strong>PeakForce QNM Limits</strong></th>
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<tr>
<td>Force Limit</td>
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<tr>
<td>Dissipation Limit</td>
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<td>DMT-Modulus Limit</td>
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<td>Sample Bias Control</td>
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<td>Units</td>
<td>Metric</td>
<td></td>
</tr>
<tr>
<td>Minimum Engage Gain</td>
<td>10.0</td>
<td></td>
</tr>
<tr>
<td>Peak Force Engage Setpoint</td>
<td>0.1500 V</td>
<td></td>
</tr>
<tr>
<td>Bidirectional Scan</td>
<td>Enabled</td>
<td></td>
</tr>
<tr>
<td>Tip Serial Number</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Output 1 Data Type</td>
<td>Off</td>
<td></td>
</tr>
<tr>
<td>Output 2 Data Type</td>
<td>Off</td>
<td></td>
</tr>
</tbody>
</table>
1. From the Menu bar, click **EXPERIMENT > CONFIGURE EXPERIMENT**. This opens an information window, shown in **Figure 2.9c**.

*Figure 2.9c  The Configure Experiment information window*

2. Click **OK** to open the **Configure Experiment** window, shown in **Figure 2.9d**.

*Figure 2.9d  The Configure Experiment Window*

3. Check a box in the **Add Commands** panel to add that command to the **Workflow Toolbar**.

4. Click **OK** to accept your choices and close the **Configure Experiment** window.
5. Right-click in the **Scan Parameter List** and select **SHOW ALL**, shown in **Figure 2.9e**.

**Figure 2.9e  Select SHOW ALL items**

- **Scan**
  - **Scan Size**: 500 nm
  - **Aspect Ratio**: 1.00
  - **X Offset**: 0.000 nm
  - **Y Offset**: 0.000 nm
  - **Scan Angle**: 0.00°
  - **Scan Rate**: 0.977 Hz
  - **Samples/Line**: 512

- **Feedback**
  - **Peak Force Setpoint**: 0.1000 V
  - **Feedback Gain**: 5.000
  - **ScanKey Auto Control**: On

- **Cantilever Parameters**
  - **Spring Constant**: 0.3000 N/m
  - **Tip Radius**: 10.0 nm
  - **Sample Poisson’s Ratio**: 0.300

- **PeakForce QNM Limits**
  - **DMAT Modulus Limit**: 1024 A/mm

- **Limits**
  - **Z Range**: 7.42 μm

- **Other**
  - **Units**: Metric
  - **Show Parameter List 1**
  - **Show Parameter List 2**
  - **Configure Links ...**
  - **Show All**
This makes all **Scan Parameters** visible along with two check boxes, the left, green, check box for the **SIMPLE MODE** and the right, red, check box for the **EXPANDED MODE**. See **Figure 2.9f**.

**Figure 2.9f** Enable Parameters

![Enable Parameters](image)

The checked **✓** parameters display in normal Real-time mode while those parameters without a **✓** will not display in normal Real-time mode.
Check the parameters that you want displayed and right-click in the Scan Parameter List and select Show All items to hide the unchecked parameters. The panel will once again appear in normal Real-time mode.
Chapter 3  PeakForce QNM Samples

Five samples are supplied with the PeakForce QNM kit.

Note: All samples are homogeneous and glued to Bruker sample pucks.

Note: Probe or sample contamination may compromise the accuracy of the quantitative measurements.
3.1 PDMS-Soft-1

Nominal modulus: 2.5 MPa.

This 150 μm thick PDMS gel sample is formulated for long shelf life and stability.

Suggested probe: ScanAsyst-Air.

A typical force curve of this sample is shown in Figure 3.1a and an image of the modulus is shown in Figure 3.1b. The standard deviation of the modulus in this image is 0.7 MPa.

**Figure 3.1a** Typical force curve of a PDMS-Soft-1 sample

![Figure 3.1a](image)

**Figure 3.1b** Typical modulus image of a PDMS-Soft-1 sample

![Figure 3.1b](image)
3.2 PDMS-Soft-2

Nominal modulus: 3.5 MPa.

This 150 μm thick PDMS gel sample is formulated for long shelf life and stability.

Suggested probe: ScanAsyst-Air.

A typical force curve of this sample is shown in Figure 3.2a and an image of the modulus is shown in Figure 3.2b. The standard deviation of the modulus in this image is 0.5 MPa.

**Figure 3.2a** Typical force curve of a PDMS-Soft-2 sample

**Figure 3.2b** Typical modulus image of a PDMS-Soft-2 sample
3.3 Polystyrene

Nominal modulus 2.7 GPa.

This polystyrene sample is spin-cast on a silicon wafer.

Suggested probes: RTESPA/Tap300 or Tap525.

A typical force curve of this sample is shown in Figure 3.3a and an image of the modulus is shown in Figure 3.3b. The standard deviation of the modulus in this image is 0.2 MPa.

Figure 3.3a Typical force curve of a Polystyrene sample

![Figure 3.3a](image)

Figure 3.3b Typical modulus image of a Polystyrene sample

![Figure 3.3b](image)
3.4 HOPG

Highly Oriented Pyrolytic Graphite.

Nominal modulus 18 GPa.

Suggested probes: TESPA, diamond

A typical force curve of this sample is shown in Figure 3.4a and an image of the modulus is shown in Figure 3.4b. The standard deviation of the modulus in this image is 2 MPa.

**Figure 3.4a**  Typical force curve of a HOPG sample

**Figure 3.4b**  Typical modulus image of a HOPG sample
3.5 Fused Silica

Corning 7980 fused silica.

Nominal modulus 72.9 GPa.

Suggested probe; Diamond.
Chapter 4   Calibration

4.1 Introduction to Calibrating PeakForce QNM

To quantify the forces as well as other mechanical properties of your sample, it is important to understand the PeakForce QNM calibration procedure.

Note: For best results, the calibration process will need to be performed for each probe.

4.2 Absolute vs. Relative Calibration Methods

There are two methods of obtaining calibrated, quantitative results from PeakForce QNM. The first method (the relative method) avoids accumulated errors that can cause errors in modulus measurements, but has the downside in that it requires a reference sample that can be measured by the same probe as the unknown sample. The second method (the absolute method) does not require a reference sample, but requires accurate measurement of the tip end radius (typically by scanning an artifact sample like TipCheck) and spring constant (typically with thermal tune for soft cantilevers). Both methods require measurement of the deflection sensitivity on a hard sample.

For both methods, it is important to choose a probe that can cause enough deformation in the sample and still retain high force sensitivity. Figure 4.2a shows the recommended probes and the modulus range over which they work best.
Figure 4.2a  Modulus ranges covered by various probes. The modulus of the reference sample for each range is indicated as well.

Table 4.2a  Legend

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Chemical Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>PDMS</td>
<td>Polydimethylsiloxane</td>
</tr>
<tr>
<td>LPDE</td>
<td>Low-density polyethylene</td>
</tr>
<tr>
<td>HPDE</td>
<td>High-density polyethylene</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>PS</td>
<td>Polystyrene</td>
</tr>
<tr>
<td>HOPG</td>
<td>Highly Oriented Pyrolytic Graphite</td>
</tr>
</tbody>
</table>
4.2.1 The Relative Method

The relative method of calibration uses a sample of known modulus to obtain the ratio of spring constant to the square root of tip end radius. It is still important to accurately calibrate the deflection sensitivity in order to obtain modulus results. An outline of the procedure follows:

1. Calibrate the **Deflection Sensitivity** on a clean, hard sample (Sapphire or Silicon, which can be used for samples with modulus less than 10 GPa). See **Calibrate the Deflection Sensitivity**: Section 4.3 for a procedure to measure this.

2. If quantitative **Adhesion** or **Dissipation** data is required, use the NanoScope **Thermal Tune** function to obtain the spring constant, otherwise enter the nominal value from the manufacturer. See **Calibrate the Spring Constant Using Thermal Tuning**: Section 4.4 for a procedure to measure this.

3. Image the reference sample using **PeakForce QNM** and adjust the **Tip Radius** parameter to make the measured **Modulus** equal the known value of the reference sample.

4. Image the unknown sample adjusting the **Peak Force Setpoint** to match the deformation depth used during imaging of the reference sample.

4.2.2 The Absolute Method

The absolute procedure is very similar to the relative procedure except for two important differences:

1. The spring constant calibration (Step 2) is not optional.

2. Instead of using the reference sample, the tip end radius is measured by scanning a tip calibration artifact (such as TipCheck Bruker part #RS) and analyzing the resulting image. See **Measure the Tip Radius**: Section 4.5 for a procedure to measure this.

   **Note:** The absolute procedure has the benefit that there is no concern over the accuracy of the modulus of the reference sample or whether it ages over time or becomes contaminated.
4.3 Calibrate the Deflection Sensitivity

Because Peak Force QNM mode ramps the Z piezo and acquires force curves, measuring deflection sensitivity requires fewer steps than the normal ramp procedure.

1. Click the Select Experiment icon. This opens the Select Experiment window, shown in Figure 4.3a.

![Figure 4.3a The PeakForce QNM in Air Select Experiment window](image)

2. Select Mechanical Properties in the Experiment Category panel.

3. Select Quantitative Nanomechanical Mapping in the Select Experiment Group panel.
4. Select **PEAKFORCE QNM in Air** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. Set the **Scan Size** to 0 nm.

6. **ENGAGE** the probe onto a clean sapphire (required for cantilevers with $k > 200 \text{ N/m}$) or silicon surface using the **PeakForce QNM in Air** mode.

7. Activate **RAMP**, mode by clicking the **RAMP** icon in the **Workflow Toolbar**. This causes the system to stop scanning, and the probe to position above the center of the previous image.

8. Enter the following parameter settings in the designated panels of the **Ramp Parameter List**:

   a. In the **Ramp** panel select:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ramp output</td>
<td>$Z$</td>
</tr>
<tr>
<td>Ramp size</td>
<td>100nm - 1.00(\mu\text{m})</td>
</tr>
<tr>
<td>Scan Rate</td>
<td>1.00Hz</td>
</tr>
<tr>
<td>Number of samples</td>
<td>512</td>
</tr>
</tbody>
</table>

   b. In the **Mode** panel select:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trigger mode</td>
<td>Relative</td>
</tr>
<tr>
<td>Trig threshold</td>
<td>0.2V</td>
</tr>
</tbody>
</table>

   c. In the **Channel 1** panel select:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>Data Type</td>
<td>Deflection Error</td>
</tr>
<tr>
<td>X Data Type</td>
<td>$Z$</td>
</tr>
<tr>
<td>Display Mode</td>
<td>Deflection Error vs. $Z$</td>
</tr>
</tbody>
</table>

9. Click the **RAMP SINGLE** icon on the NanoScope toolbar or select **Ramp > Ramp Single**, from the menu bar.
10. Move two cursors onto the Deflection vs. Z plot (see Figure 4.3b).

11. Arrange the cursors so that they surround the contact (steepest) portion of the graph (see Figure 4.3b).

**Figure 4.3b**  Force Curve Cursors

12. Click the **Update Sensitivity** icon in the NanoScope toolbar or select **Ramp > Update Sensitivity**. The software will automatically calculate the deflection sensitivity and open the Set Realtime Channel Sensitivities window (see Figure 4.3c).

**Figure 4.3c**  Deflection Sensitivity Dialogue Box

13. Click **OK** to accept this deflection sensitivity in the dialogue box that displays, and it will automatically be entered into the **Deflection Sensitivity** parameter.
4.4 Calibrate the Spring Constant Using Thermal Tuning

Note: Bruker recommends **Thermal Tune** included in NanoScope software for probes with spring constants less than or equal to 1 N/m. Other methods (Sader¹, added mass, vibrometer, pre-calibrated probes) are recommended for probes with higher spring constants. These techniques are reviewed in detail in Bruker Application Note 94: *Practical Advice on the Determination of Cantilever Spring Constants*.

1. Ensure that the probe is withdrawn adequately from the sample before activating **Thermal Tune**. The probe should not interact with the sample during its self excitation under ambient conditions.

2. Click **CALIBRATE > THERMAL TUNE** or the **THERMAL TUNE** icon in the NanoScope tool bar (shown).

3. Select a frequency range that includes the resonant frequency of the cantilever. See **Figure 4.4a**. Stiff cantilevers may require the 5 - 2000 kHz range.

**Figure 4.4a** Select **Thermal Tune** Frequency Range

---

¹ See http://www.ampc.ms.unimelb.edu.au/afm/calibration.html
4. Click **ACQUIRE DATA** in the **Thermal Tune** panel, shown in Figure 4.4b.

**Figure 4.4b** The **Thermal Tune** panel

5. The microscope will acquire data for about 30 seconds.

6. Zoom in on the region around the peak.
7. Click either the **LORENTZIAN (AIR)** or **SIMPLE HARMONIC OSCILLATOR (FLUID)** button to select a Lorentzian or a simple harmonic oscillator model, respectively, of the PSD to be least squares fit to the data.

**Note:** The equations used to fit the filtered data are:

Lorentzian, for use in air

\[
A(v) = A_0 + \frac{C_1}{(v - v_0)^2 + C_2}
\]

**Note:** where:  
- \(A(v)\) is the amplitude as a function of frequency, \(v\)  
- \(A_0\) is the baseline amplitude  
- \(v_0\) is the center frequency of the resonant peak  
- \(C_1\) is a Lorentzian fit parameter  
- \(C_2\) is a Lorentzian fit parameter

Simple Harmonic Oscillator, for use in fluid

\[
A(v) = A_0 + A_{DC} \cdot \frac{v_0^2}{\sqrt{(v_0^2 - v^2)^2 + \frac{v_0^2 v^2}{Q^2}}}
\]

where:  
- \(A(v)\) is the amplitude as a function of frequency, \(v\)  
- \(A_0\) is the baseline amplitude  
- \(A_{DC}\) is the amplitude at DC (zero frequency)  
- \(v_0\) is the center frequency of the resonant peak  
- \(Q\) is the quality factor

8. Adjust the **Median Filter Width**, shown in Figure 4.4c, to remove individual (narrow) spikes. This replaces a data point with the median of the surrounding \(n = 3, 5, 7\) data points.

**Figure 4.4c Median Filter Width**

9. Adjust the **PSD Bin Width** to reduce the noise by averaging.
10. Drag markers in from the left and/or right plot edges to bracket the bandwidth over which the fit is to be performed. See Figure 4.4b.

11. Click **FIT DATA**. The curve fit, in red, is displayed along with the acquired data. If necessary, adjust the marker positions and fit the data again to obtain the best fit at the thermal peak.

12. Enter the cantilever **Temperature**.

13. Click **CALCULATE SPRING K**.

14. You will be asked whether you want to accept the calculated value of the spring constant, \( k \) (see Figure 4.4d). Clicking **OK** copies the calculated spring constant to the **Spring Constant** window in the **Cantilever Parameters** window.

![Figure 4.4d  Spring Constant Calculation Result](image)

**Figure 4.4d**  Spring Constant Calculation Result

### 4.5 Measure the Tip Radius

Tip radius may be measured using a tip characterizer sample and the **Tip Qualification** function in NanoScope Analysis software (NanoScope software does not include the Estimated End Radius function).

1. **Scan** (**PEAK FORCE QNM IN AIR** mode) the characterizer sample. Set the **Scan Size** to approximately 1.5\( \mu m \). Characterizer image size is important because, along with **Tip Image Size** and feature density, it determines how many peaks are used for the tip estimation.

2. Set the **Samples/Line** and **Lines** to **512**.

3. Set the **Aspect Ratio** to **2.0**

4. Set the **Scan Rate** to **0.5 Hz** or less.

5. Because this is an intentionally rough sample that can damage the probe tip, set **ScanAsyst Noise Threshold** to **1.0 nm**.

6. **CAPTURE** the characterizer image.

7. Open the **Height** channel of the saved image in the **NANOSCOPE ANALYSIS** package.
8. Flatten the image by clicking the **PLANE FIT** icon.

9. Select **XY** as the **Plane Fit Mode**.

10. Select **1ST AS** as the **Plane Fit Order**.

11. Click **EXECUTE** to plane fit the image. See Figure 4.5a.

**Figure 4.5a** Plane Fit of the Characterizer Sample
12. Click the **Tip Qualification** icon in the **NanoScope Analysis** toolbar to open the Tip Qualification window.

13. Enter the measured average **Deformation** (see **Deformation: Section 2.5.6**) into the **Height from Apex** field.

   **Note:** The **Height from Apex** parameter should equal the average penetration depth or the indentation in the force curve. The indentation can be measured as the separation from the minimum force to the peak force in the loading curve. For most samples, the indentation is very close to the sample deformation. Therefore, the average deformation can be used as **Height from Apex** parameter to estimate tip radius. But for very soft samples (<20MPa), the adhesion is very large and the difference between indentation and deformation is large so that the indentation must be measured from the force curves in the **Force Monitor** window shown in **Figure 4.5b**.

**Figure 4.5b** Typical force curve of a PDMS-Soft-1 sample. Nominal modulus: 2.5 MPa.

14. Click **Estimate Tip**.

15. Click **Qualify Tip**. **Figure 4.5c** displays the **Estimated Tip End Radius**.
4.6 Calibrate Peak Force QNM

Three parameters are needed to fully calibrate PeakForce QNM:

1. **Deflection Sensitivity.** See *Calibrate the Deflection Sensitivity:* Section 4.3 for a procedure to measure this.

2. **Spring Constant.** See *Calibrate the Spring Constant Using Thermal Tuning:* Section 4.4 for a procedure to measure this.

3. **Tip Radius.** See *Measure the Tip Radius:* Section 4.5 for a procedure to measure this.

A fourth parameter, the **Sample’s Poisson’s Ratio**, is needed to convert the measured reduced modulus, $E^*$, to the sample modulus, $E_s$. The reduced modulus is related to the sample modulus by the following equation:

$$E^* = \left[ \frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu_s^2}{E_s} \right]^{-1}$$
where $\nu_t$ and $E_t$ are the Poisson’s ratio and Young’s modulus of the tip and $\nu_s$ and $E_s$ are the Poisson’s ratio and Young’s modulus of the sample. We assume that the tip modulus, $E_t$, is much larger than the sample modulus, $E_s$, and can be approximated as infinite and calculate the sample modulus using the sample Poisson’s Ratio.

Poisson's ratio generally ranges between about 0.2 and 0.5 (perfectly incompressible) giving a difference between the reduced modulus and the sample modulus between 4% and 25%. Because the sample’s Poisson's ratio is not generally known, many publications report only the reduced modulus. Entering zero for this parameter will cause the system to return the reduced modulus. Recommended values for the sample’s Poisson ratio, $\nu_s$, are shown in Table 4.6a.

**Table 4.6a** Recommended values of the sample Poisson’s ratio, $\nu_s$, as a function of the sample stiffness, $E_s$.

<table>
<thead>
<tr>
<th>$E_s$</th>
<th>$\nu_s$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$E_s &lt; 100$ MPa</td>
<td>0.5</td>
</tr>
<tr>
<td>$0.1 &lt; E_s &lt; 1$ GPa</td>
<td>0.4</td>
</tr>
<tr>
<td>$1$ GPa $&lt; E_s &lt; 10$ GPa</td>
<td>0.3</td>
</tr>
</tbody>
</table>

**4.6.1 Cantilever Parameters**

After you have measures the cantilever Spring Constant and the Tip Radius, enter them into the Cantilever Parameters panel in the Scan Parameters window of the NanoScope software window, shown in Figure 4.6a. If available, enter the Sample Poisson’s Ratio.

**Figure 4.6a** The Cantilever Parameters panel

**4.6.2 Feedback Parameters**

**Peak Force Setpoint**

A Peak Force Setpoint that is too high can either damage the sample or wear the tip. It is generally desirable to reduce the Peak Force Setpoint to as small a value as is possible. However, in order to achieve accurate Elastic modulus measurement, sufficient sample deformation is needed. If the deformation is less than 2nm, increase the Peak Force Setpoint to achieve sufficient sample deformation.
For the relative method, you should adjust the Peak Force Setpoint to keep the Deformation the same for both the reference and measurement samples.

**Feedback Gain**

Reduce the Feedback Gain to lower the noise in the property channels.

If ScanAsyst Auto Gain is On, set the ScanAsyst Noise Threshold to 0.5nm or less.
Calibration

Calibrate Peak Force QNM
Chapter 5   Offline Analysis

5.1 Introduction

As discussed in Chapter 2, real-time PeakForce QNM saves images of processed data like modulus and adhesion. There are times when one would like to compare these processed data images with the associated force curves. For this reason, a PeakForce QNM mode off-line analysis function is included in the NanoScope Analysis package. The main intention of this function is to allow you to view and analyze force curves from areas where material properties are most likely to change. You are given options to export raw force curves that can then be analyzed in the NanoScope Analysis or third party analysis programs.

As discussed in Section 2.7, you should collect an image and then, at a region of interest during the capture, click the CAPTURE LINE button. This ensures capture of the raw high speed data capture (HSDC) in the DSP buffers. Remember that to transfer the data to the computer and into a file, you must click the UPLOAD DATA button in the High Speed Data Capture interface.

5.2 Procedure

1. Start the NanoScope Analysis package by double clicking the offline icon on the Windows desktop.

2. Open the PeakForce QNM HSDC file.
3. Click the QNM-Hsdc Force Curve-Image icon to open the **QNM Hsdc Force Curve-Image** window, shown in Figure 5.2a.

![Figure 5.2a](image)

**Figure 5.2a** The QNM Hsdc Force Curve-Image window

4. Click the **LOAD IMAGE** button, circled in Figure 5.2a, and select the image file associated with your high speed data capture file.
5. The solid blue horizontal line, shown in Figure 5.2b, displays the captured line.

**Figure 5.2b** The *HEIGHT* channel of the image file
6. Two vertical dashed blue cursors, shown in Figure 5.2c, display the X position of the displayed force curves when **PAIR** is checked in the **Force Curve Selection** box. The associated number boxes display the Z piezo tap number. You may move the force position by either dragging the dashed blue cursors in the image, the dashed red cursors (see Step 7) in the time display or entering the numbers in the **Force Curve Selection** panel. You may select the channel of the captured image by right-clicking in the image as shown in Figure 5.2c.

**Figure 5.2c** Vertical cursors display X position
7. The position of the force curves is also represented by the red dashed cursors in the **Deflection Error vs. Time** display, shown in **Figure 5.2d**. To Zoom in on an area of interest in the graphs, hold down the Control key and draw a box in the preferred area. To Zoom back out, double-click the image or click the magnifying glass icon in the lower left corner of the plot.

**Figure 5.2d**   The QNM Hsdc Force Curve-Image window
5.3 Controls and Settings

5.3.1 Image Line Selection

Range and Settings: **ALL**: Displays all captured lines.

**ONE LINE**: Displays one captured scan line of taps. The arrow in the **Image Line Selection** panel and the solid blue line, shown in Figure 5.2b, display the scan direction. The counters are displayed in the **Force Curve Selection** panel.

**Figure 5.3a** PeakForce QNM Controls

<table>
<thead>
<tr>
<th>Image Line Selection</th>
<th>Force Curve Selection</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>All</td>
<td>Bar</td>
<td>Force Curve Type</td>
</tr>
<tr>
<td>One Line</td>
<td>Multiple</td>
<td>Deformation Sens</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Deflection Sens</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Spring Constant</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Static Display Channel</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Retrace Image from:</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Force Curve Type</td>
<td>Both</td>
</tr>
<tr>
<td>Deformation Sens</td>
<td>172 nm/V</td>
</tr>
<tr>
<td>Deflection Sens</td>
<td>45.0 nm/V</td>
</tr>
<tr>
<td>Spring Constant</td>
<td>33.33 N/m</td>
</tr>
<tr>
<td>Static Display Channel</td>
<td>1</td>
</tr>
<tr>
<td>Retrace Image from</td>
<td>tesp-ps-lpesp.001</td>
</tr>
</tbody>
</table>
5.3.2 Force Curve Selection

Range and Settings: **PAIR**: Displays a pair of force curves. See Figure 5.2d for an example. The location of the pair of force curves can be controlled by moving either the dashed blue cursors in the image, the dashed red cursors (see Step 7 on Page 73) in the time display or entering the numbers in the Force Curve Selection panel.

**MULTIPLE**: Displays all the force curves between the vertical cursors. The location of the force curves can be controlled by dragging either the dashed blue cursors in the image, the dashed red cursors (see Step 7 on Page 73) in the time display or entering the numbers in the Force Curve Selection panel. See Figure 5.3b.

![Figure 5.3b Multiple Force Curve Selection](image-url)
5.3.3 Force Curve Type

*Range and Settings:* **BOTH:** Display both the extend and retract portions of the Z piezo ramp.

**TRACE:** Displays the extend portion of the Z piezo ramp.

**RETRACE:** Displays the retract portion of the Z piezo ramp.

5.3.4 Exporting Force Curves

Click the **EXPORT CURVES** button to export either a **PAIR** of force curves or **MULTIPLE** curves in a directory FrcExport. One file will be created for each force curve. This binary file (header is ASCII) can be opened by the NanoScope Analysis package.

Because off-line plots (either NanoScope or NanoScope Analysis) feature more display options for exported curves (FrcExport) than HSDC files, you may wish to export your curves and then open the FrcExport files. Details can be found in the Line Plot and Multiple Line Plot page of the Force Curve and Ramping Analysis in the help pages of the NanoScope Analysis package.

5.4 PeakForce QNM Input Parameters

The parameters appearing in the **PeakForce QNM Input Parameters** window have been collected in real-time but may be modified for off-line analysis here.

- Deformation Sensitivity
- Deflection Sensitivity
- Spring Constant
- Hsdc Display Channel

5.5 Exported Force Curves

Exported force curves (file names begin with FrcExport) can be viewed in NanoScope software or NanoScope Analysis software. Off-line plots feature more display options for exported curves than are available for HSDC files. These additional options are listed below.
5.5.1 Time Domain Plots

Change the X Data Type from Z HEIGHT to TIME to transform the original force curve, shown in Figure 5.5a, to a **Force vs. Time** plot (a.k.a. heartbeat), shown in Figure 5.5b.

**Figure 5.5a** Exported force curve
5.5.2 Plot Units

The Y axis can be displayed as VOLTS (Deflection), METRIC (distance in nano meters) or FORCE (nano Newtons).
5.5.3 Display Mode

Change the Display Mode to PEAKF DEFLECTION VS. SEP, shown in Figure 5.5c, to plot your data vs. separation.

![Figure 5.5c  Force vs. separation](image)

5.6 Image Math

You can use the IMAGE MATH functions in NanoScope Analysis software to re-evaluate your results if you wish to change a parameter.

For instance, you can re-compute Young’s modulus if you wish to compensate for a changed tip radius or a different spring constant.

Because

$$E \propto \frac{k}{\sqrt{R}}$$

you can scale Young’s modulus by to arrive at corrected results. Figure 5.6a shows an image of Young’s modulus in a multilayer polymer optical film before correction. Figure 5.6b shows an image of Young’s modulus in a multilayer polymer optical film after multiplying it by \((1/\sqrt{2})\) to compensate for a tip radius that has increased by a factor of 2 (e.g. from 10 nm to 20 nm).
**Figure 5.6a** Young’s modulus in a multilayer polymer optical film before correction

**Figure 5.6b** Young’s modulus in a multilayer polymer optical film after **Image Math** correction
The **Image Math** interface is shown in Figure 5.6c and the corresponding equation appears in Figure 5.6d.

**Figure 5.6c** The **Image Math** interface

- **Image A**
  - Image A
  - Channel A: 3
  - Scaling Factor A: 0.707
  - Unit: MPa

- **Image B**
  - Image B
  - Channel B: 1
  - Scaling Factor B: 0
  - Operator: +

- **Output**
  - Error: No Error

**Figure 5.6d** The **Image Math** equation

- Equation: $0.71 \text{ Image A + 0.00 Unit Matrix in MPa}$
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