Chapter 7 Head, Probe and Sample Preparation

This chapter provides instructions for head, probe and sample preparation for imaging with the MultiMode SPM. It describes how to remove and install the microscope head, how to change the probe probe holder, how to mount the probe, load and position samples, and a general description of how to engage and withdraw the tip. These procedures are common for most types of MultiMode SPM imaging.

Please refer to the following sections:

- **Initial Preparation for Contact AFM Imaging:** Section 7.1
    - **Prepare the Sample:** Section 7.1.1
    - **Load the Sample:** Section 7.1.2
    - **Load Probe in Probe holder:** Section 7.1.3
    - **Install the Probe holder:** Section 7.1.4
- **Laser Alignment:** Section 7.2
    - **Method 1: OMV Method:** Section 7.2.1
    - **Method 2: The Projection Method:** Section 7.2.2
    - **Maximize the SUM Signal:** Section 7.2.3
- **Start the Microscope Program:** Section 7.3
    - **Typical startup:** Section 7.3.1
    - **Select Mode of Operation:** Section 7.3.2
    - **Loading a saved workspace:** Section 7.3.3
- **MultiMode SPM Voltage Meters:** Section 7.4

Other chapters in this manual describe how to perform specific types of imagery. The table below outlines where you will find additional information for each type of imagery. If you are new to SPM and want to practice, we suggest you begin with Contact AFM in Chapter 9.

For Specific Information Regarding:	**See Chapter:**
Contact AFM	9
Tapping Mode	10
Fluid Operation	11
Scanning Tunneling Microscopy (STM)	12
Lateral Force Microscopy (LFM)	13
Force Microscopy	14
Interleave Scanning	15
Magnetic Force Microscopy (MFM)	16
Electric Force Imaging	17

This instrument uses a semiconductor diode laser emitting a maximum 1.0mW beam at 690nm. The light is emitted downward and normally reflects back into the system's optics from the back of the cantilever probe. Note that the laser is powered when the SPM head is plugged into the microscope's support ring and the **Mode** switch is set to either **AFM** & **LFM** or **TM AFM** (tapping mode). Exceeding regulatory requirements, the MultiMode SPM head contains an internal switch which reduces laser power when the head is tilted. Operators should use care, however, to avoid staring into beams reflected from sample surfaces.

WARNING: During and prior to set up of the laser, it is especially important to avoid looking directly at the laser beam or at the laser spot. Care should be taken when highly reflective samples are inserted onto the chuck. Avoid looking at all reflected laser light. Operators should use care to avoid staring into beams that may be reflected from sample surfaces.

AVERTISSEMENT: Avant d’utiliser le laser, et durant tout le temps pendant lequel il fonctionne, il est impératif de ne pas regarder directement le faisceau. Il est impératif de faire très attention lorsque des échantillons très réfléchissants sont déposés sur la platine. Eviter toute exposition à la lumière laser. Durant l’utilisation, ne pas fixer les faisceaux laser réfléchis par les surfaces d’échantillons.

WARNUNG: Es ist sehr wichtig, vor und während der Laserjustierung nicht in den Laserstrahl oder auf den Laserpunkt zu schauen. Seien Sie bitte sehr vorsichtig, wenn stark reflektierende Proben auf dem Probenteller liegen. Vermeiden Sie unter allen Umständen, in das reflektierte Laserlicht zu schauen. Alle Bediener des Mikroskops sollten größte Vorsicht walten lassen um zu vermeiden, in den von der Probenoberfläche reflektierten Laserstrahl zu schauen.

7.1 Initial Preparation for Contact AFM Imaging

7.1.1 Prepare the Sample

Verify that your sample will fit atop the scanner tube and is less than 8mm thick. If you already have prior experience with loading samples into the MultiMode SPM system, load your sample now. Otherwise, read the next section for suggestions on how to prepare and load small samples.

If it is your first time operating the microscope, we recommend that you image the calibration sample provided with the instrument (usually a 10µm-pitch grid of 200nm step height).

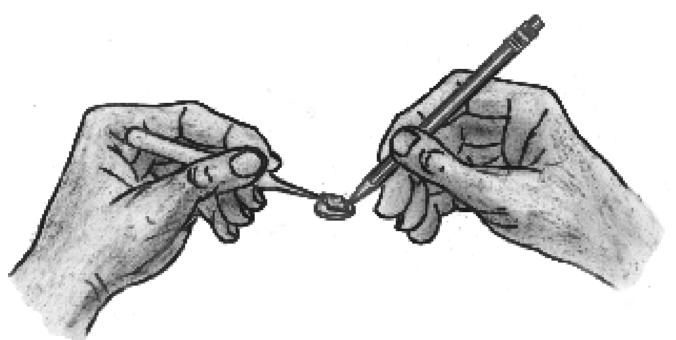
1. The calibration sample or other small sample should be placed on one of the 15mm diameter metal disks used for sample mounting. The MultiMode SPM is provided with several steel sample disks that can be attached to the magnetic sample holder, located atop the scanner tube.

2. Provided with the instrument are red and white colored “sticky tabs,” which are 2-sided adhesive patches. Peel off a “sticky tab” from the provided sheet, and place it on the steel small sample puck, then peel off the red-and-white paper. This leaves a patch of the two-sided adhesive on the steel sample disk, which will hold the sample to the disk.

3. Using tweezers, place the small sample to be imaged firmly on the “sticky tab” adhesive (see Figure 7.1a). Alternatively, a small sample can be glued down to the sample puck using cyanoacrylate glue (superglue).

4. Place the small sample disk atop the scanner.

Figure 7.1a Gently Press the Sample onto the Sticky Tab Until Secured



7.1.2 Load the Sample

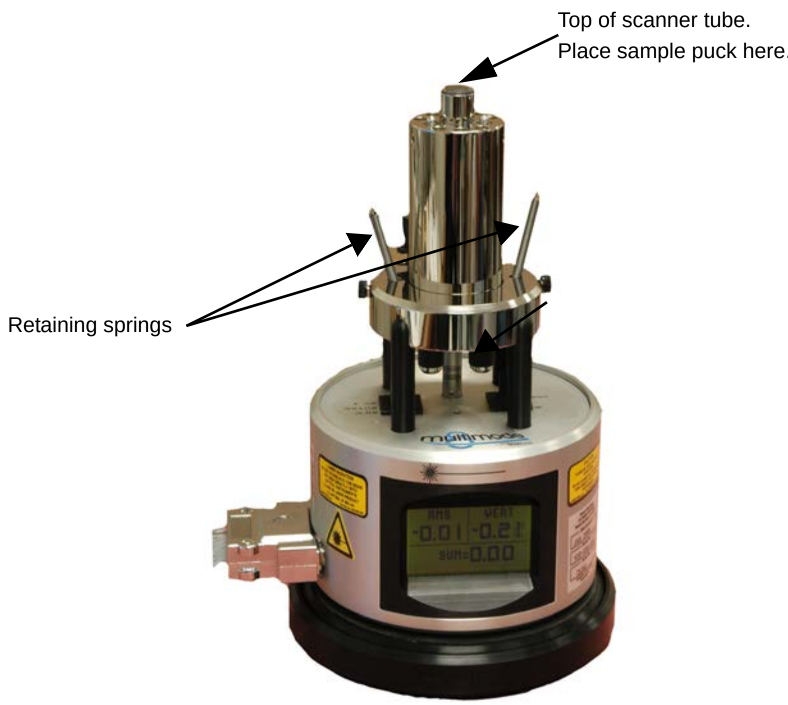
Remove Head and Load Sample

1. Remove the head as required by unfastening the retaining springs on either side and unplugging the head’s micro-D connector.

2. Gently lift the head off and set aside. This will expose the top of the scanner tube.

3. Mount the sample puck with the calibration standard on the scanner tube. An internal magnet holds the puck down.

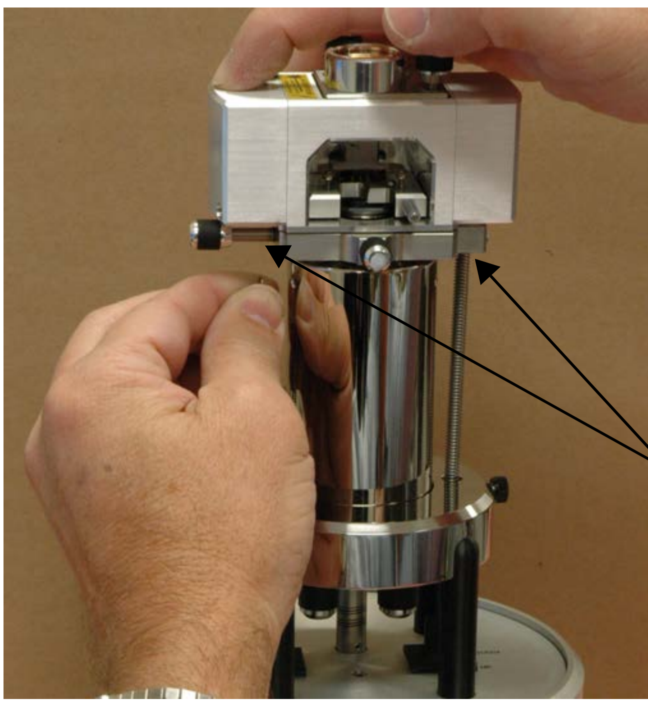
Figure 7.1b MultiMode Base with Scanner Mounted on Support Ring



Reinstall the Head

- Adjust the sample height so that it will protrude no more than 2 mm above the head's XY translation stage. If a probe holder is in the head and the sample is too high, the probe will be damaged when the head is installed. Adjust the sample height as needed by using the Tip Up/Down motor drive switch on the MultiMode base.
- Remount the head by gently lowering it over the scanner tube while checking for clearance.
- Secure both retaining springs and plug the head's connector into the support ring (see Figure 7.1c).

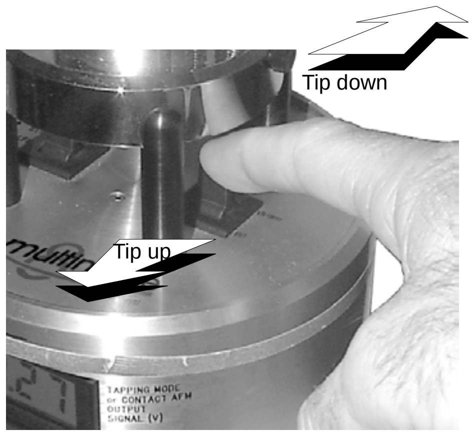
Figure 7.1c Head is Held Securely Using Retaining Springs



Check Head for Free Vertical Movement

Verify basic function of the motorized Z-axis by toggling the **Up** switch on the MultiMode base (see Figure 7.1d). This activates the leadscrew at the rear of the unit to lift the head upward. If you are using a standard, three-screw scanner, the two forward screws will also have to be rotated to keep the head level while lifting. (Single-screw, vertical scanners require only that the rear, motorized screw be rotated to raise and lower the head.) To verify rotation of the motorized screw, feel the flexible coupling on the base with your finger while you toggle the **Tip Up** / **Down** switch.

Figure 7.1d Tip Up / Down Switch on the MultiMode SPM’s Base



7.1.3 Load Probe in Probe holder

- **Contact Mode**: Install a silicon nitride probe in the AFM probe holder. Figure 7.1e shows the AFM probe holder. Ensure the gold-plated side of the substrate is placed down toward the substrate mount, with the nitride film side attached to the cantilever oriented up away from the substrate mount.

- **Tapping Mode**: The procedure for single crystal silicon probes is essentially the same as for contact AFM (see above paragraph). The substrate should be face-up, with the probe’s cantilever pointing away from the AFM probe holder. This ensures that the cantilever and tip are facing toward the sample after the probe holder is mounted in the head.

Figure 7.1e Silicon Nitride Probe Installation

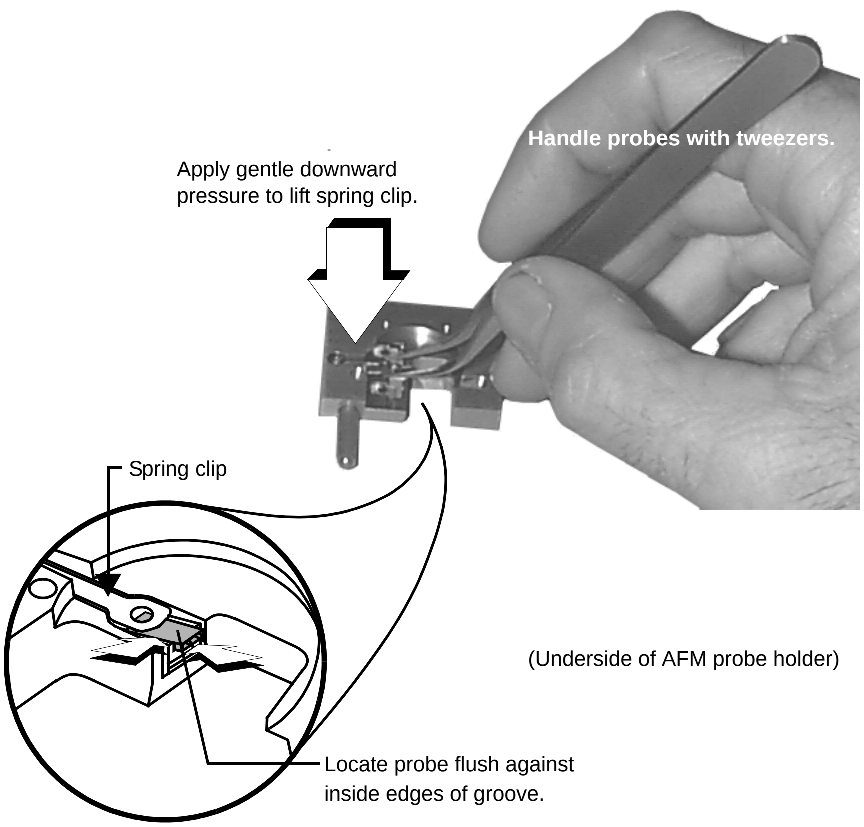
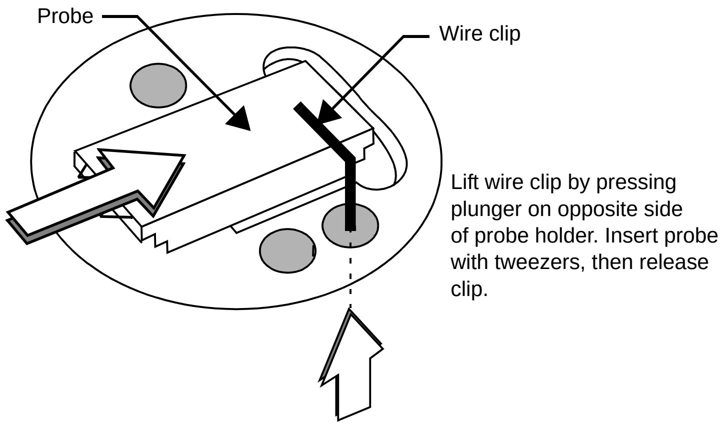


Figure 7.1f Underside Detail of Fluid Cell



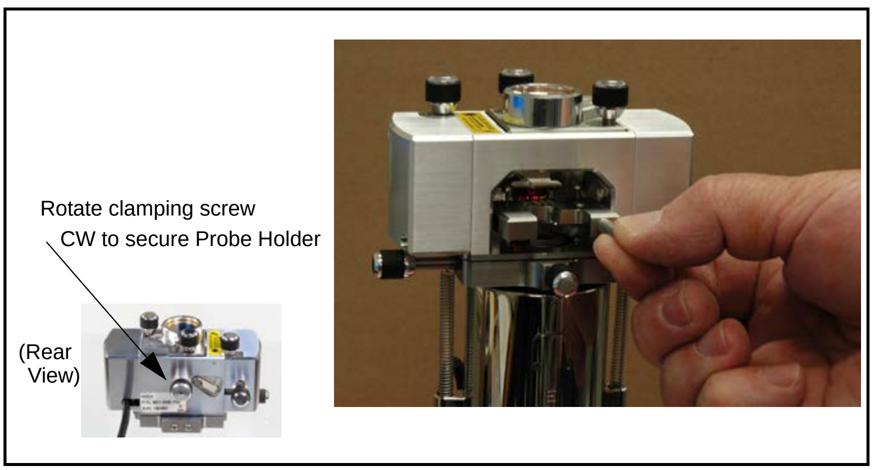
Load Probe in Probe holder

Refer to Figure 7.1e and Figure 7.1f. Turn the probe holder upside down with the groove facing up as shown. Apply gentle upward pressure against the plunger to lift the spring clip. With the spring clip lifted, carefully slide the probe into the probe holder's groove until it is located squarely against the innermost edges, then lower the spring clip by releasing pressure against the plunger. This will hold the probe securely in the probe holder’s groove. Check that the probe's substrate is flush with the back of the groove and flat against one side (this keeps the probe's cantilever oriented in the correct direction). If identical probes are loaded the same way each time, aiming the laser onto the cantilever will be much quicker and easier. Fluid cell probe installation is similar to AFM/LFM probe holders.

CAUTION:	The spring clip is extremely fragile and must be handled with great care to prevent bending.
ATTENTION:	Les embouts de ressort sont extrêmement fragiles et doivent être manipulés avec une extrême précaution.
VORSICHT:	Die Haltefeder ist sehr empfindlich und muß sehr vorsichtig behandelt werden, um ein Verbiegen zu vermeiden.

7.1.4 Install the Probe holder

Figure 7.1g Install Probe holder in Head without Touching the Sample



After the probe holder is loaded with a probe, the probe holder will be placed inside the SPM head and clamped into position using the clamping screw at rear of head.

1. Before installing the probe holder, verify that the head is sufficiently raised to clear the sample with the tip. The top of the sample should not protrude more than 1-2mm above the inside head bottom plate and is safer if set so the sample is flush with the plate. Excessive protrusion risks a crash between sample and tip when the probe holder is installed.

2. Insert the loaded AFM probe holder into the MultiMode head by placing the probe holder carefully over the sample. Do not touch the sample with the probe holder.

3. Press the probe holder forward gently and lower it into position (see Figure 7.1g).

Three precision ball mounts inside the head mate kinematically with the probe holder underside. If the scanner cap has been properly positioned, the probe holder will come to rest with the probe just above the sample surface. If the scanner cap is adjusted too high, the tip will be plunged into the sample surface and broken. If it appears the probe may crash when the probe holder is installed, remove the probe holder completely and use the **Up** switch on the MultiMode base to obtain sufficient clearance. Inexperienced users should practice with scrap probes and samples to learn proper loading procedures.

7.2 Laser Alignment

This section describes two methods for aligning the laser for all modes except STM. The first method uses an Optical Viewing Microscope (OMV). The OMV method requires that the sample be reflective. The second method is a “projection” method. In the projection method, you remove the MultiMode head and project the laser beam onto a surface (white paper or other suitable surface) to show a silhouette of the cantilever in order to align the laser. The projection method is used if you do not have an OMV or if your sample does not reflect the laser enough to be viewed in the OMV.

7.2.1 Method 1: OMV Method

Figure 7.2a OMV System Components

1. Use the OMV to locate and focus on the cantilever.
2. Focus below the tip on the surface.
3. Bring the head/tip down using and the stepper motor and, if applicable, the front manual screws, until the cantilever is almost in focus.
4. While maintaining focus, use the OMV stage screws to locate the red laser reflection spot.

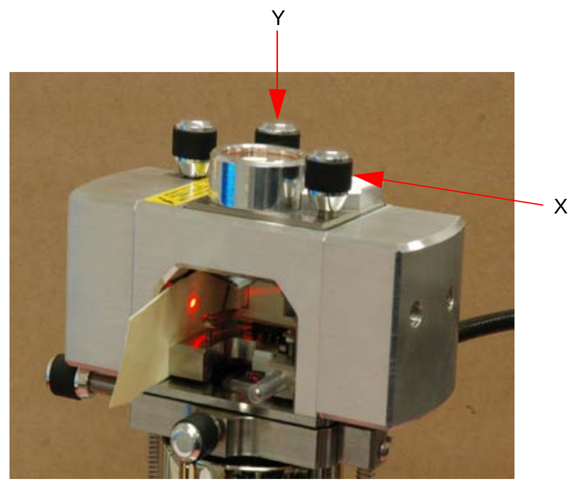
CAUTION:	Turn down the illuminator intensity before proceeding with laser alignment.

5. Use the manual laser knobs on top of the head to move the laser onto the end of the cantilever.

6. A quick way of checking laser alignment is to place a piece of paper in front of the photodetector to ensure the laser reflection is solid. (A small “post-it” note folded lengthwise works well).

   Note: Because the slip of paper prevents light from reaching the photodetector, the sum signal cannot be monitored while using this method.

Figure 7.2b Laser Alignment with Piece of Paper



7. Reposition the laser with the screws on top of the optical head, if necessary.

7.2.2 Method 2: The Projection Method

CAUTION: Turn down the illuminator intensity before proceeding with laser alignment.

You can also align the laser by moving the laser beam relative to the cantilever while observing the laser spot on a piece of white paper below the optical head. If the laser is not on the cantilever substrate, the laser appears as a bright red spot on the surface below. When the laser is aligned on the cantilever, a shadow appears on the surface below.

The X direction runs along the major axis of the substrate (parallel to the length of the cantilever). The right-front laser control knob, atop the optical head, controls the laser beam movement along the X direction. The back-left laser knob, atop the optical head moves the beam along the Y direction perpendicular to the cantilever and substrate's major axis. In the vision system display, the X direction is right-to-left across the screen, and the Y direction is top-to-bottom.

The procedure for aligning the laser is slightly different for etched silicon and for silicon nitride probes. The sections below detail the procedures for aligning the laser on the cantilever and tips for each probe type.

Etched Silicon Probes (tapping mode)

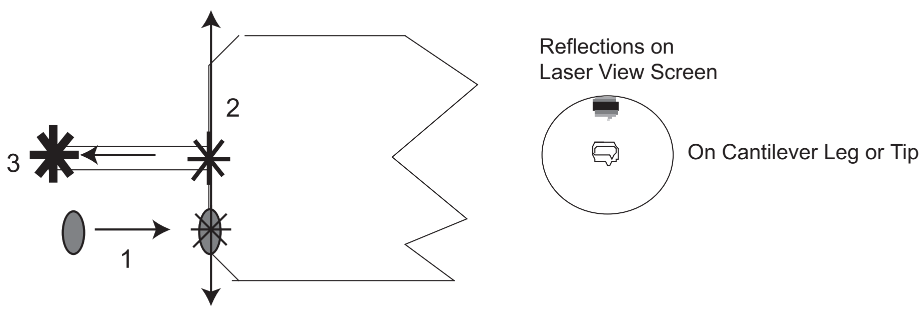
1. In this procedure, shining the laser beam on a piece of white paper serves as a guide to aligning the laser beam with the end of the cantilever.

CAUTION: Use extreme caution if you choose to remove the optical head and hold it over a piece of paper. Hold the head firmly, and be mindful of the wire between the head and base. Dropping the optical head would most likely result in damage and could result in necessary factory repairs.

2. Verify the laser beam is visible on the surface below. If it is not, turn the front-right laser control knob counter-clockwise until the laser spot appears on the surface below.

3. Turn the front-right laser control knob clockwise to move the laser in the X positive direction (right) until the laser spot disappears from the surface below. Turn the front-right laser control knob counter-clockwise until the laser spot just reappears. The laser is now positioned at the edge of the substrate (see Point 1 in Figure 7.2c).

Figure 7.2c Etched Silicon Tip Laser Alignment



4. Turn the back-left laser control knob clockwise or counter-clockwise to move the laser in the Y direction (parallel to the substrate edge and perpendicular to the cantilever) until the beam crosses the cantilever and a shadow appears over the laser spot on the surface below. Turning the back-left laser control know clockwise moves the laser beam back from the front of the MultiMode head. The laser is now positioned over the cantilever (see Point 2 in Figure 7.2c).

5. Verify that the laser is deflecting off the cantilever by moving the laser on, over, and off the cantilever by turning the back-left laser control knob less than 1/8 of a turn.

6. Turn the front-right laser control knob counter-clockwise to move the laser in the X negative direction on the cantilever until the laser crosses the tip-end of the cantilever and falls on the surface below.

7. Move the laser onto the tip-end of the cantilever by reversing the direction of the front-right laser control knob clockwise until the spot disappears from the surface below (see Point 3 in Figure 7.2c).

8. Verify the laser spot by placing a piece of paper in front of the photodetector (see Section 7.2.1, Step 6). If necessary, reposition the laser with the front-rear and back-left knobs.

Silicon Nitride Probes (Contact Mode AFM)

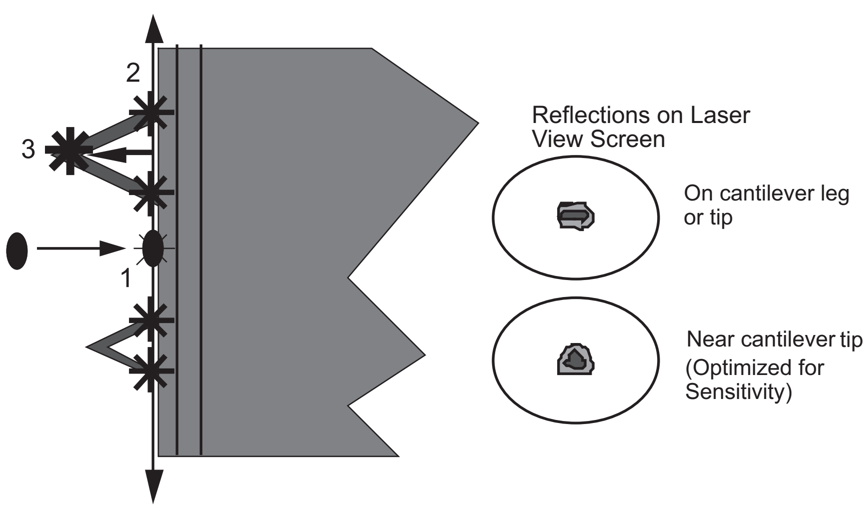
1. In this procedure, the laser is aligned by shining the laser beam on a piece of paper (or other projection surface) to show a silhouette of the cantilever.

CAUTION: Use extreme caution if you choose to remove the optical head and hold it. Hold the head firmly and avoid damaging the wire between the head and base. Dropping the head is likely to result in damage which will require factory repair.

2. If the laser beam is not visible on the projection surface, turn the front-right laser control knob counter-clockwise until the laser spot appears.

3. Turn the front-right laser control knob clockwise to move the laser in the X positive direction until the laser spot is blocked by the probe substrate and the projected beam disappears. Turn the right-rear laser control knob counter-clockwise until the laser spot just reappears. The laser is now positioned at the edge of the substrate (see Point 1 in Figure 7.2d).

Figure 7.2d Silicon Nitride Laser Alignment



4. With the laser positioned at the edge of the substrate, use the back-left laser control knob to move in the Y direction (parallel to the edge of the substrate) until the laser is positioned at the base of a desired cantilever. Ensure that the cantilever selected is appropriate for the analysis which is to be performed. In this example, we will assume that the cantilever at point 2 is the desired one (see Point 2 in Figure 7.2d).

   Note: It may be necessary to cross a maximum of four legs on the two V-shaped cantilevers to determine which is the correct cantilever.

5. Adjust the back left and front right controls to move the laser beam position to the end of the cantilever (see Point 3 in Figure 7.2d) as seen in the projected silhouette.

6. When the beam is positioned at the point of the "V" of the cantilever, remount the head onto the scanner.

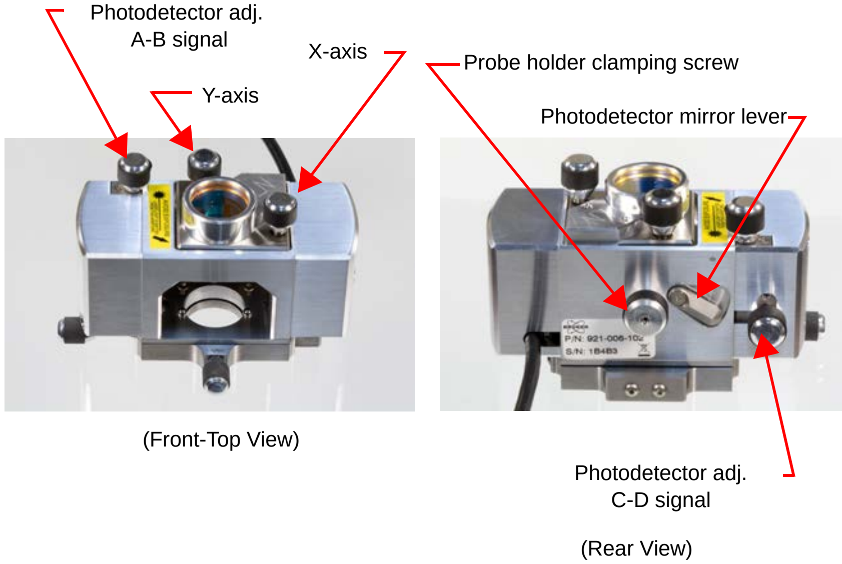
7. Verify laser alignment by placing a piece of paper (a small "post-it®" note folded lengthwise works well) into the head (ref Figure 7.2b).

7.2.3 Maximize the SUM Signal

This section describes what to do after the laser spot is aligned on the cantilever. It assumes you know how to read voltages from the meters mounted on the front of the MultiMode base. If you are unfamiliar with reading the MultiMode voltage meters, skip ahead to **MultiMode SPM Voltage Meters:** Section 7.4, then return to this section. Additional information is provided in each of the various chapters on imaging.

After the laser beam is aligned, move the mirror lever on the back of the head (Figure 7.2e) to maximize the SUM signal. Next, use the Photodetector Vertical Adjustment Knob (A-B) to set the output signal to the value required for the imaging mode (refer to specific chapters on imaging mode).

Figure 7.2e Photodetector Signals are Optimized using the Head's Adjustments



This adjustment is much less sensitive than the laser position adjustments. The maximized sum signal should be approximately **4.0** - **9.0V** for silicon nitride cantilevers. The value of this signal varies with many factors. It is important to note that it is possible to see a large response on the bar graph without having the laser beam on the cantilever, so it is important to visually verify that the laser beam is on the cantilever and not rely on the bar graph alone. Attempting to engage with the laser beam improperly aligned will usually destroy the cantilever and may damage the sample.

7.3 Start the Microscope Program

7.3.1 Typical startup

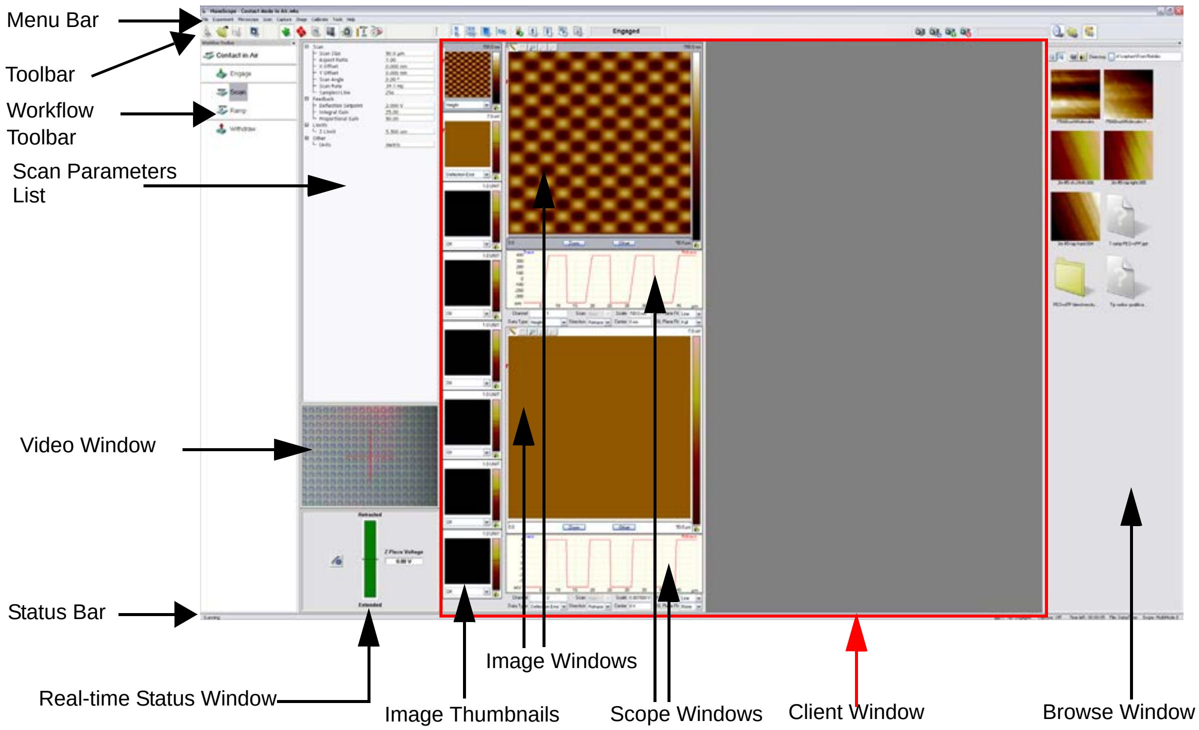
After any necessary software installation is complete, you are ready to start the NanoScope software.

> **Note:** If the system has been previously set-up and the workspace saved, the previous settings can be reloaded as described in Section 7.3.3, otherwise, complete the procedure in this section.

1. To start the NanoScope software, double-click the **NanoScope** startup icon on the computer desktop.

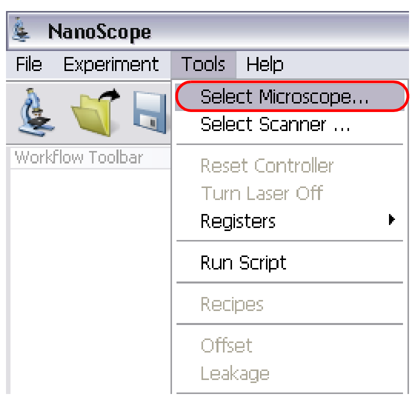
2. This opens the NanoScope software window, shown in Figure 7.3a. This large window will contain all the areas and panels you use to control the microscope and analyze your results.

Figure 7.3a NanoScope Version 8 Screen Elements



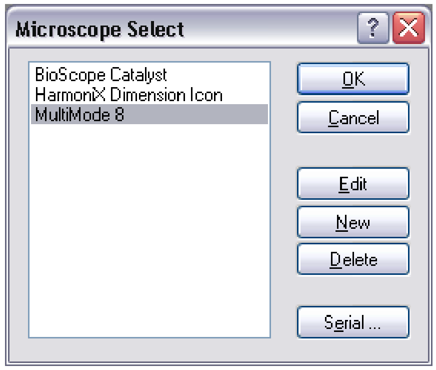
3. Set the microscope configuration by selecting **Tools** > **SELECT MICROSCOPE...** from the menu bar. See Figure 7.3b.

Figure 7.3b **SELECT MICROSCOPE** from the Tools menu



4. This opens the **Microscope Select** window, shown in Figure 7.3c.

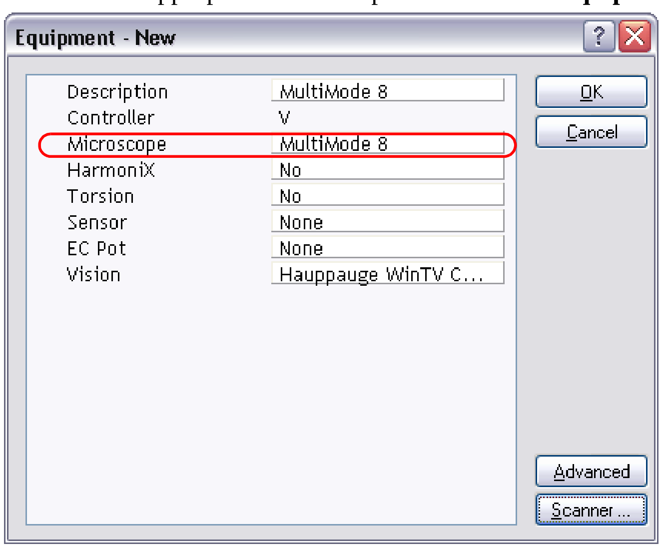
Figure 7.3c Microscope Select Dialog Box



5. Click **NEW** if this is a new microscope, **EDIT** if you wish to modify a configured microscope, **DELETE** to remove a microscope configuration or **CANCEL** to exit.

6. **NEW** and **EDIT** open the **NEW EQUIPMENT** window, shown in Figure 7.3d.

Figure 7.3d Select the appropriate microscope from the **New Equipment** window

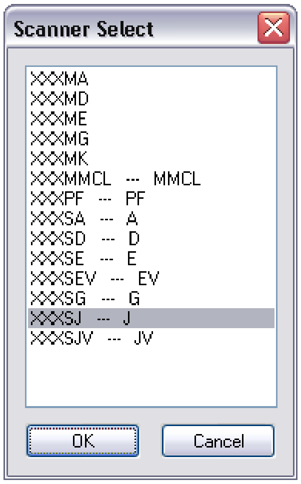


7. Select **MULTIMODE V** from the drop down **Microscope** menu. This step is not necessary if the MultiMode has not been reconfigured since the last use.

8. If the microscope has been previously set-up and the desired setting saved in a workspace, those settings can be loaded from the saved file. Refer to Section 7.3.3.

9. Click **SCANNER** to open the **Scanner Select** window shown in Figure 7.3e.

Figure 7.3e Select your scanner.



10. Select the **SCANNER** and click OK. This returns you to the **Microscope Select** window, shown in Figure 7.3c.

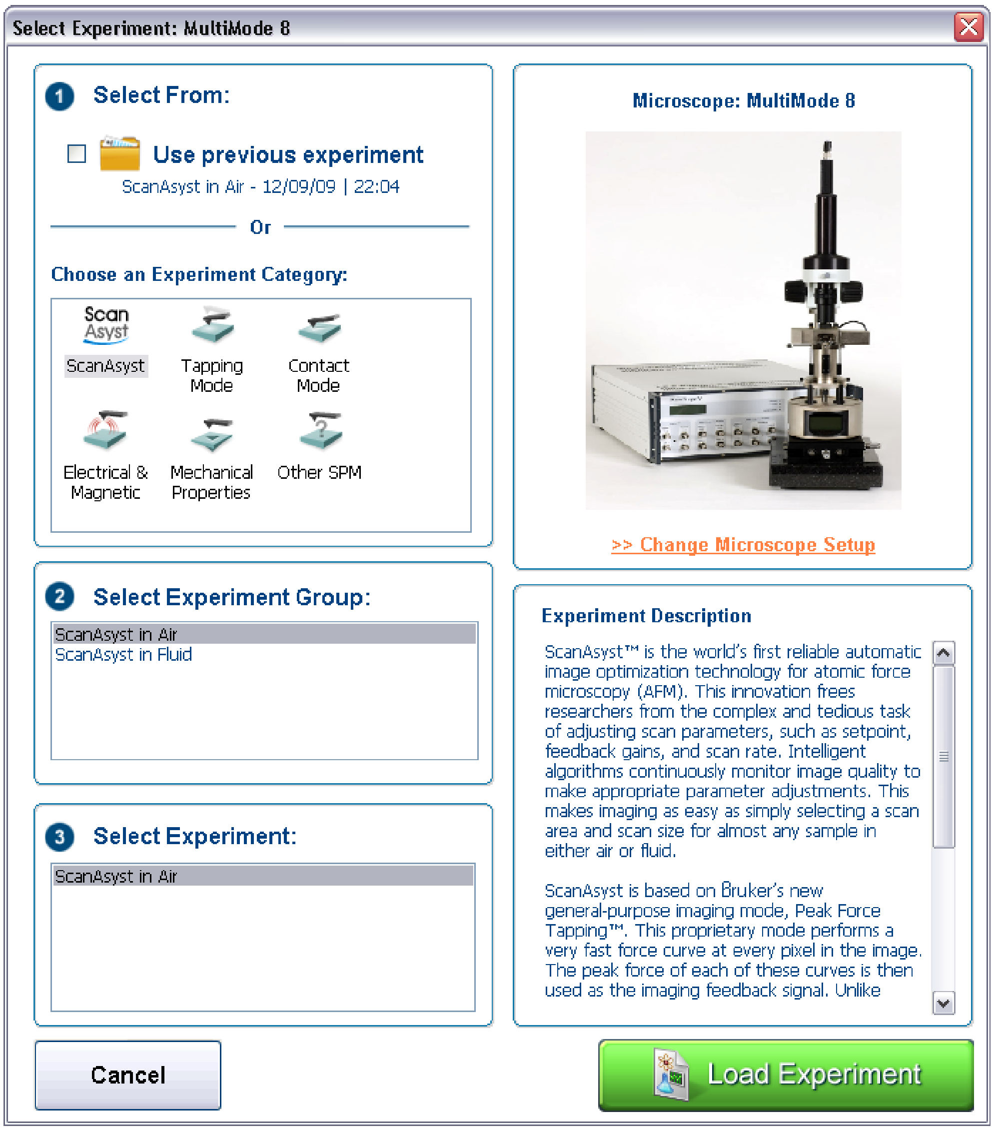
11. Click **OK** to close the **Equipment** window.

12. Click **OK** to close the **Microscope Select** dialog box.

7.3.2 Select Mode of Operation

1. Click **EXPERIMENT** > **SELECT EXPERIMENT** or the **SELECT EXPERIMENT** icon in the top left of the NanoScope software window. This opens the **Select Experiment** window, shown in Figure 7.3f.

Figure 7.3f The **Select Experiment**, Contact Mode, window



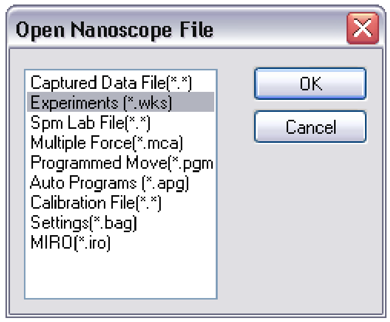
2. Select the **Experiment Category** and **Experiment**.

3. Click **START EXPERIMENT**.

7.3.3 Loading a saved workspace

1. Previously saved settings may be loaded by: **File > Open** and selecting the previously saved Experiment (*.wks) file.

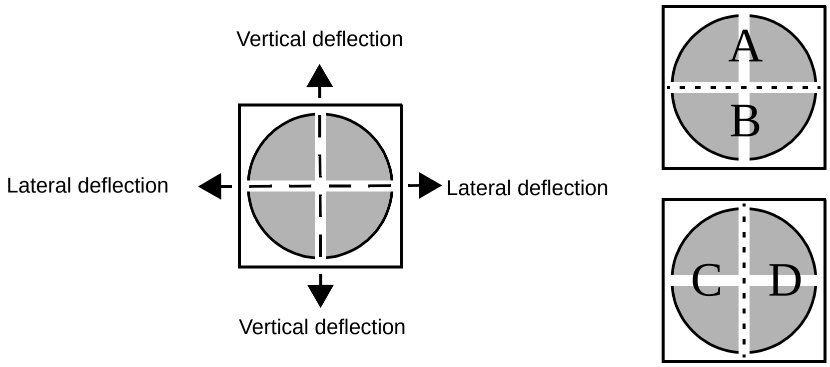
Figure 7.3g Open Workspace



7.4 MultiMode SPM Voltage Meters

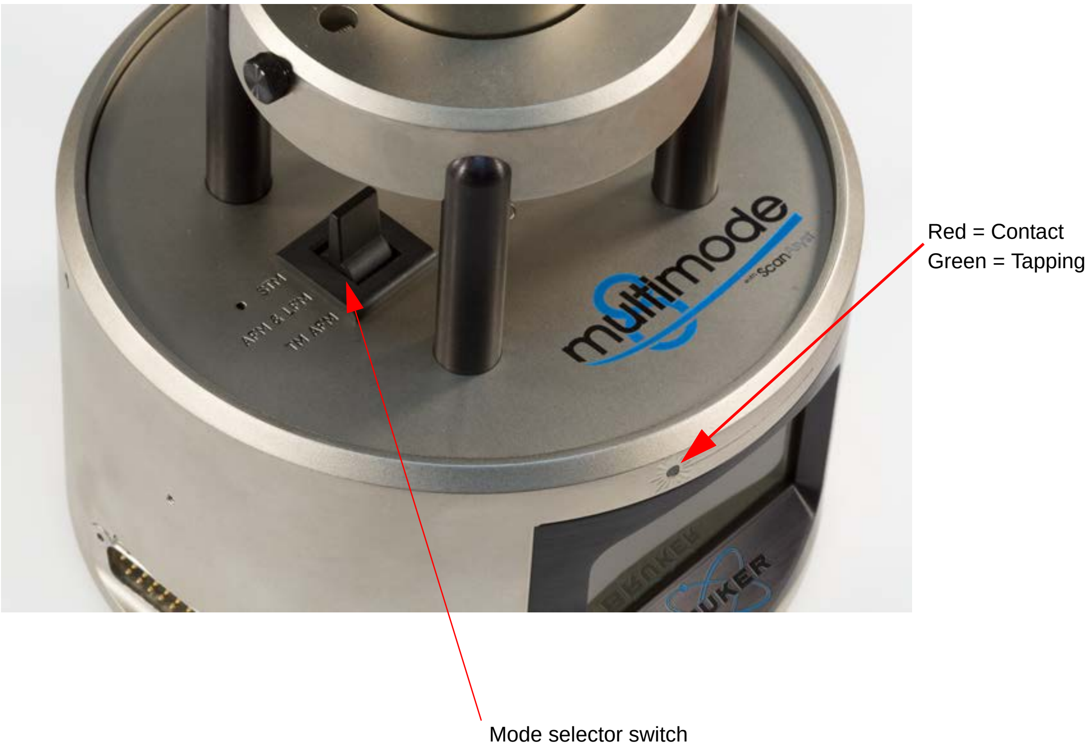
- A complete description of the signals coming into and out of the MultiMode SPM is available in Support Note 424, *Signal Access Module V.* The SAM is normally used for accessing these signals directly. A brief description of the SPM's voltages and their interpretation using the meters on the front of the base is provided here for quick reference.
- The MultiMode SPM base is equipped with meters which indicate voltage coming from the four-segment photodetector. The photodetector array is represented in Figure 7.4a.

Figure 7.4a Photodetector layout



- The meters will display differently depending on which operating mode (see Figure 7.4b) has been selected using the switch on the base. In addition, an LED indicator light will change color to red in contact mode and green in tapping mode.

Figure 7.4b Mode selector switch



- In AFM & LFM (Contact) mode, the display will show values for VERT, HORZ and SUM. See Figure 7.4c.

Figure 7.4c AFM & LFM mode meter display

- In TM AFM (Tapping) mode, the display will show values for RMS, VERT and SUM. See Figure 7.4d.

Figure 7.4d TM AFM (Tapping) mode meter display.

- The MultiMode SPM's **SUM** meter indicates the *total voltage generated by the photodetector* -- the combined voltage of photodetector segments. This is displayed during all modes (except STM when all meters are off).

- **VERT** is the *difference in voltages* (A-B) between sections vertical halves of the photodetector. (In Tapping Mode it is the average difference).

- **HORZ** is the *difference in voltages* (C-D) between sections horizontal halves of the photodetector.

- **RMS** is the *AC voltage* resulting from the oscillation of the cantilever which causes the reflected beam to oscillate in the vertical direction.

Chapter 8 ScanAsyst

This chapter covers procedures for operating the MultiMode Scanning Probe Microscope (SPM) using ScanAsyst^{TM} mode. It is assumed that the operator has previously prepared a ScanAsyst probe and aligned the laser per instructions provided in Chapter 7 of this manual. Specific information regarding tip preparation is also provided in Chapter 6.

- **Introduction:** Section 8.1
- **ScanAsyst Principles of Operation:** Section 8.2
  - **The "Heartbeat":** Section 8.2.1
  - **Force curves:** Section 8.2.2
- **Probe Selection:** Section 8.3
- **Basic ScanAsyst AFM Operation:** Section 8.4
  - **Configure the Hardware:** Section 8.4.1
  - **Adjust the Detector Offsets:** Section 8.4.2
  - **Signal Settings:** Section 8.4.3
  - **Adjust tip height above sample surface:** Section 8.4.4
  - **Select the Microscope:** Section 8.4.5
  - **Select Mode of Operation:** Section 8.4.6
  - **Position Tip with OMV:** Section 8.4.7
  - **Set Initial Scan Parameters:** Section 8.4.8
  - **Image the sample:** Section 8.4.9
- **ScanAsyst and Peak Force Tapping Mode Parameters:** Section 8.5
  - **Feedback Parameters:** Section 8.5.1
  - **Peak Force Tapping Control Parameters:** Section 8.5.2

- **Limits Parameters:** Section 8.5.3
- **Capture buttons:** Section 8.6
- **Optimizing a ScanAsyst image:** Section 8.7
- **Advanced Atomic Force Operation:** Section 8.8
  - **Displaying Parameters:** Section 8.8.1

8.1 Introduction

ScanAsyst™ is the world's first imaging mode with automatic image optimization technology for atomic force microscopy (AFM). This patent-pending innovation frees researchers from the task of adjusting scan parameters, such as setpoint, feedback gains, and scan rate. Intelligent algorithms continuously monitor image quality to make appropriate parameter adjustments. This makes imaging as easy as simply selecting a scan area and scan size for almost any sample in either air or fluid.

ScanAsyst is based on Bruker's patent-pending, new general-purpose imaging mode, Peak Force Tapping™. This proprietary mode performs a very fast force curve at every pixel in the image. The peak force of each of these curves is then used as the imaging feedback signal. Unlike TappingMode™, where imaging force is a complex function of the setpoint and other variables, Peak Force Tapping provides direct force control. This allows it to operate at even lower forces than TappingMode, which helps protect delicate samples and tips. Together, these capabilities make ScanAsyst the most powerful and productive way to use AFM.

Peak Force Tapping mode modulates the MultiMode Z-piezo at ~2 kHz with a default **Peak Force Amplitude** of 150 nm (0-peak).

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.

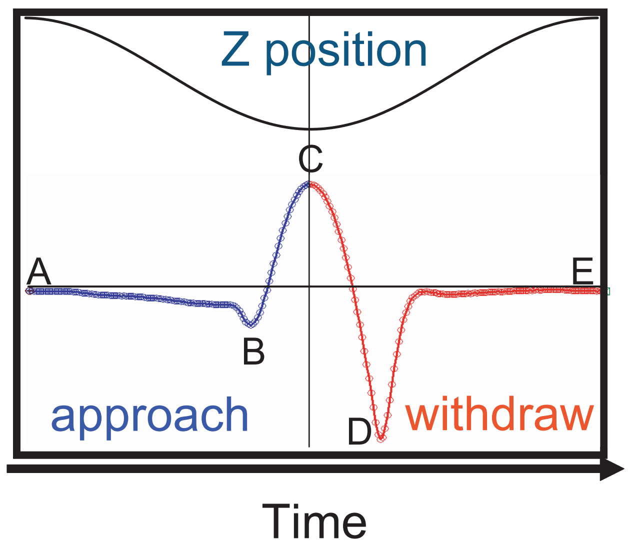
8.2 ScanAsyst Principles of Operation

Peak Force Tapping mode, the core technology behind the ScanAsyst mode, performs a very fast force curve at every pixel in the image. The peak of each of these force curves is then used as the imaging feedback signal. Peak Force Tapping mode modulates the MultiMode Z-piezo at ~2 kHz with a default **Peak Force Amplitude** of 150 nm (0-peak).

8.2.1 The "Heartbeat"

The **Force vs. Time** display, shown in Figure 8.2a 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.

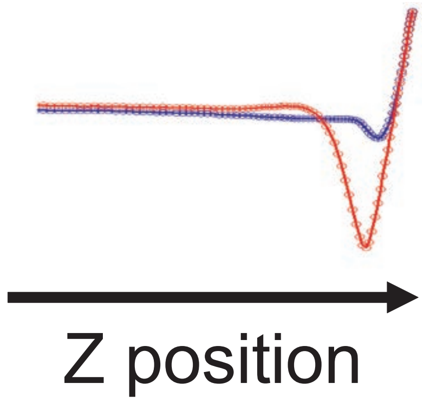
Figure 8.2a The "heartbeat." Blue indicates approach while red indicates retract.



8.2.2 Force curves

Using the Z-position information, the heartbeat is transformed into a force curve, shown in Figure 8.2b. 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 8.2b Force curve



8.3 Probe Selection

Bruker recommends ScanAsyst probes for the ScanAsyst/Peak Force Tapping mode:

- ScanAsyst-Air (k ~ 0.4 N/m, tip radius < 10 nm)
- ScanAsyst-Fluid (k ~ 0.7 N/m, tip radius < 10 nm nominal, max. = 15 nm)
- ScanAsyst-Fluid+ (k ~ 0.7 N/m, tip radius < 20 nm nominal, max. = 60 nm) These probes have a SiNx coated tip.

You may purchase these probes from http://www.brukerafmprobes.com.

8.4 Basic ScanAsyst AFM Operation

This section shows you how to perform a simple ScanAsyst experiment. Later sections will discuss Peak Force Tapping parameters and their influence on the measurements.

8.4.1 Configure the Hardware

1. Set the mode selector switch on the MultiMode base to **AFM & LFM**. See Figure 8.4a.

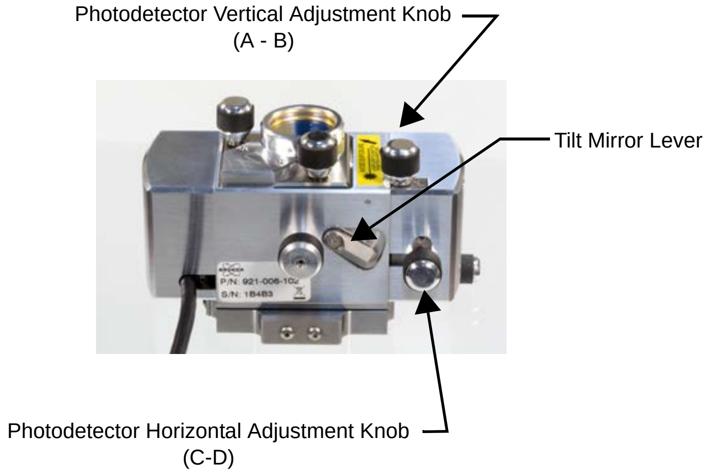
Figure 8.4a Mode selector switch

8.4.2 Adjust the Detector Offsets

Verify that the MultiMode head has been fitted with a ScanAsyst probe tip per instructions provided in Chapter 7 of this manual. The laser beam should already be positioned on the back of the cantilever. This will provide a starting point for adjusting the laser sum value.

To adjust the detector setting, first review the operation of the various adjustment screws (see Figure 8.4b). The detector adjustment screws are at the left side of the head.

Figure 8.4b Photodetector Mirror Adjustments—Rear View

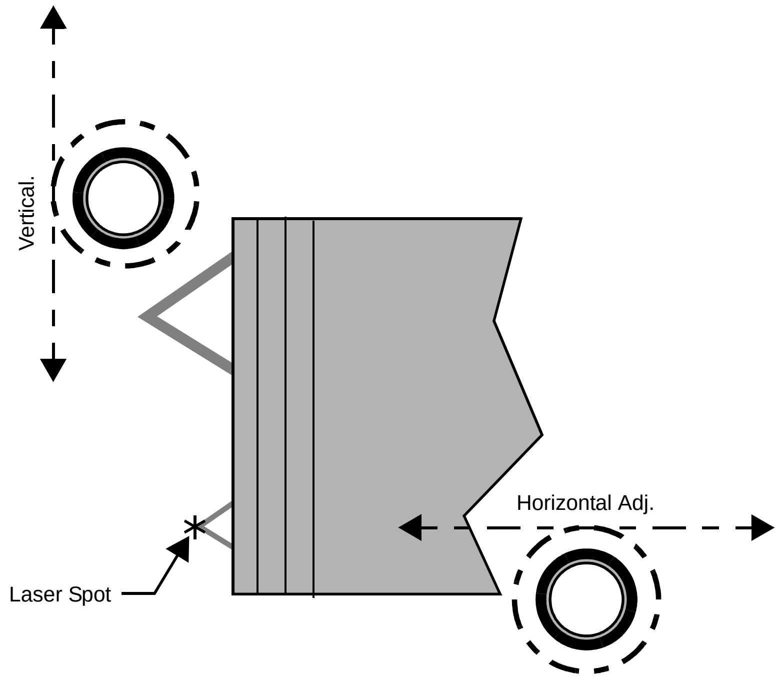


Refer to Figure 8.4c. For laser aligning screws atop the SPM head, the right-front screw moves the laser spot left-to-right (horizontally or along the X-axis). Turning this screw clockwise moves the laser spot to the right. The left-rear laser aligning screw moves the laser spot top-to-bottom (vertically along the Y-axis). Turning this screw clockwise moves the laser spot rearward.

Note: Use of the laser aligning screws to adjust the laser sum signal is NOT advised; users should adjust the laser sum signal from the photodetector adjustments only.

For initial adjustment, center the spot on the cantilever as described in Chapter 7.

Figure 8.4c Laser Adjustment Knobs—Top View



8.4.3 Signal Settings

After aligning the laser, adjust the photodetector so the the Vertical Deflection and Horizontal Deflection are near 0V and the Sum is greater than approximately 5V.

8.4.4 Adjust tip height above sample surface

Next, use the adjustment screws to adjust the tip height to be just above the sample surface. The coarse adjustment screws on the scanner (if so equipped) are located in front and may be used to make gross adjustments. The tip should be positioned just high enough to reach the surface when engaged, but not so low as to risk crashing into it. Use the motorized screw to ensure the head is reasonably level. (This is not a problem on single-screw scanners.)

One method employed to adjust the height of silicon nitride tips on noncritical samples is to very slowly lower the tip using the adjustment screws until a sudden change is noted on the sum display of the MultiMode base. Most silicon nitride cantilevers are flexible and may be gently touched to the surface without damage to either the tip or the surface if the tip is lowered slowly. Watch for the change on the sum signal display! When the sum signal change is noted, stop lowering immediately. The **Tip Up** switch may then be toggled briefly to lift the tip just above the surface (the sum signal should resume its normal value). This method works well on samples which are not delicate and which can be imaged without concern for damage.

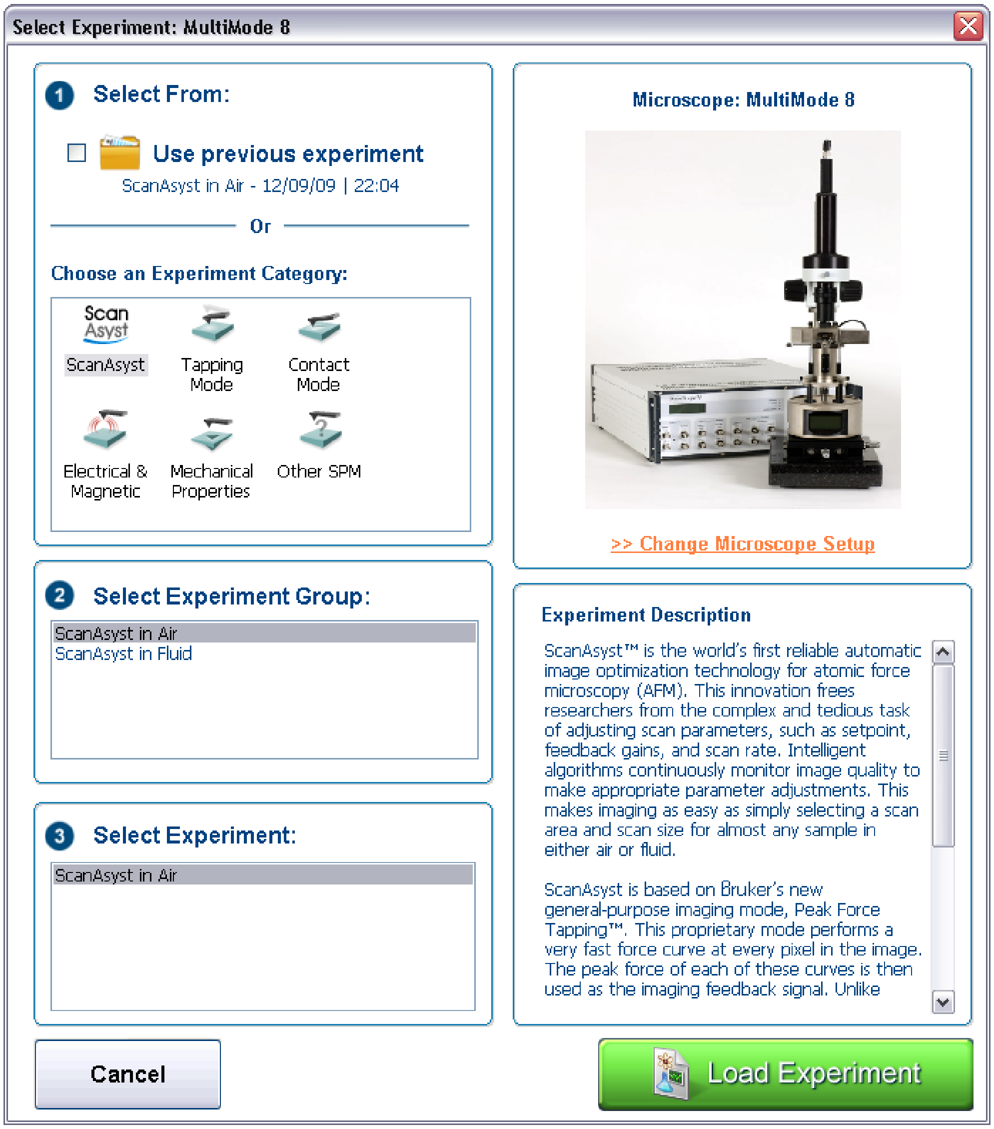
8.4.5 Select the Microscope

Follow the procedure described in **Start the Microscope Program:** Section 7.3.

8.4.6 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 8.4d.

Figure 8.4d The ScanAsyst **Select Experiment** window



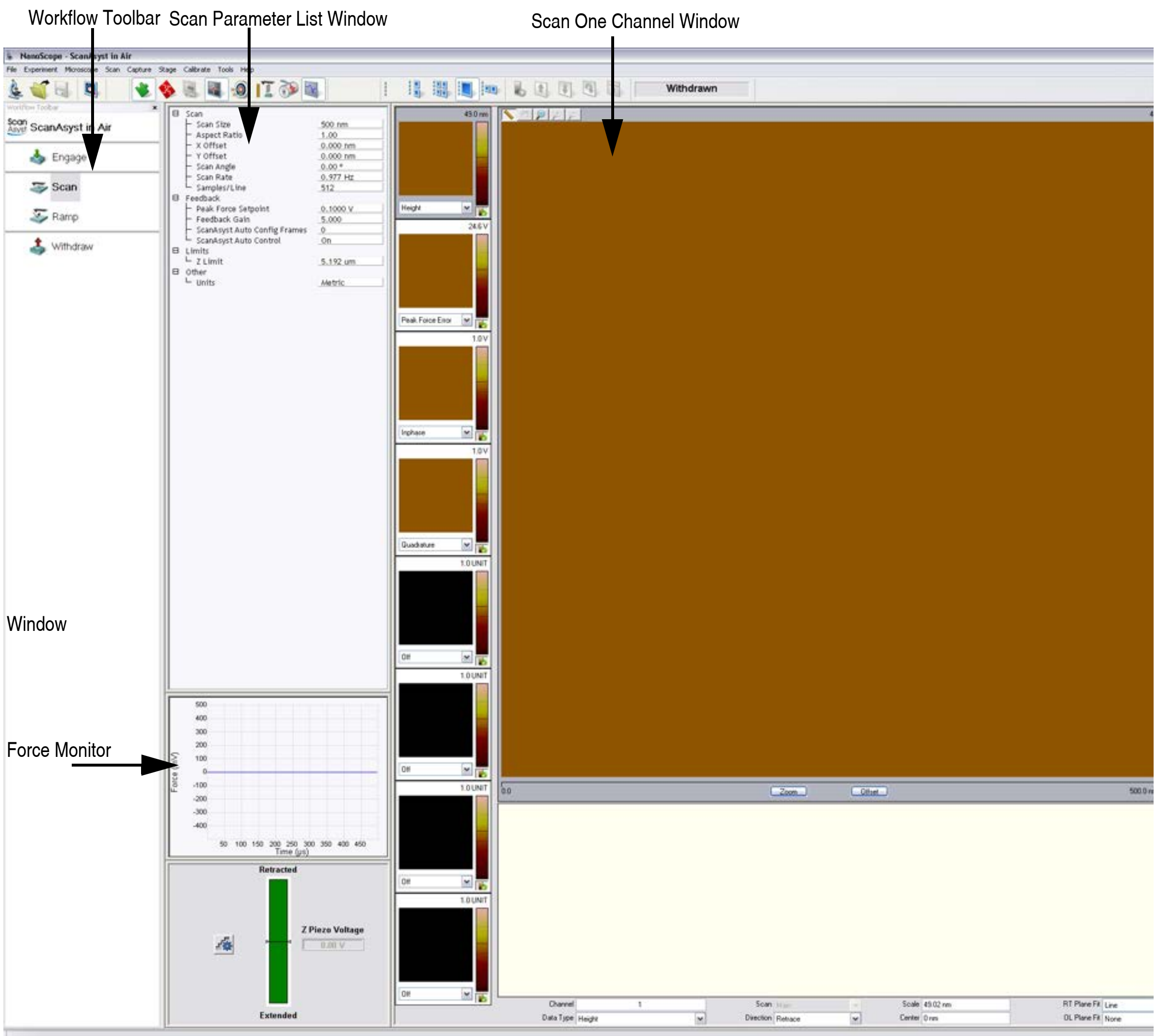
2. Select SCANASYST in the **Choose an Experiment Catagory** panel.

3. Select either SCANASYST IN AIR or SCANASYST IN FLUID in the **Select Experiment Group** panel.

4. Select either SCANASYST IN AIR or SCANASYST IN FLUID in the **Select Experiment** panel and click LOAD EXPERIMENT.

5. This opens the **Workflow Toolbar**, the **Scan One Channel** window, the **Force Monitor** window and the **Scan Parameters List** window, shown in Figure 8.4e.

Figure 8.4e ScanAsyst Mode in Air (Simple Mode) configuration



8.4.7 Position Tip with OMV

Focus the OMV on the sample, then raise the OMV focus until the sample is just out of focus. Lower the tip until it comes into focus. Refocus onto the sample surface and select **Engage**. Alternatively, focus on the sample surface and lower the tip until it begins to come into (but not quite) focus and then select **ENGAGE**.

8.4.8 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 8.4f).

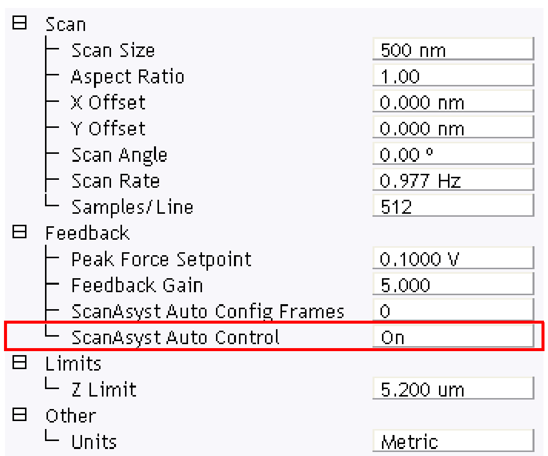
1. Set the **Scan Size**.

2. Set the **Scan Angle**.

Feedback Panel

1. Set **ScanAsyst Auto Control** to **ON** (see Figure 8.4f).

Figure 8.4f ScanAsyst in Air (Simple Mode) Parameters Panel



Channels 1, 2, 3 and 4

The default **ScanAsyst** channel settings are listed below:

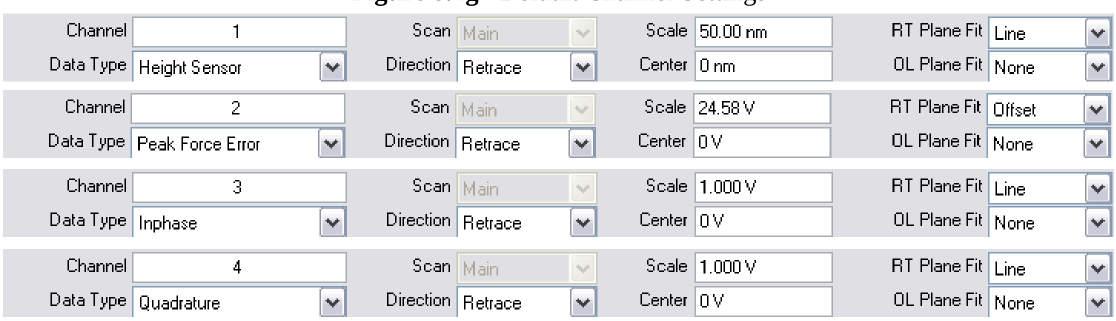
1. **Channel 1 Data Type** is **HEIGHT SENSOR** (see Figure 8.4g).
2. **Channel 2 Data Type** is **PEAK FORCE ERROR**.
3. **Channel 3 Data Type** is **INPHASE**.
4. **Channel 4 Data Type** is **QUADRATURE**.

5. Set **Data Scale** to a reasonable value for the sample or click the **AUTOSCALE** icon after engaging.

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

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

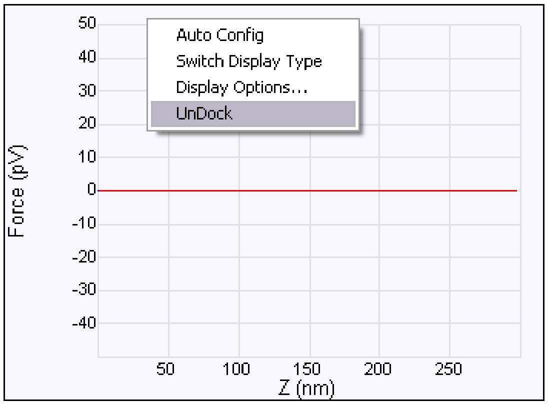
Figure 8.4g Default Channel Settings



8.4.9 Image the sample

1. If needed, right-click in the **Force Monitor** window and click **UNDOCK**. See Figure 8.4h. You may **DOCK** the undocked **Force Monitor** window by right-clicking in it and clicking **DOCK**.

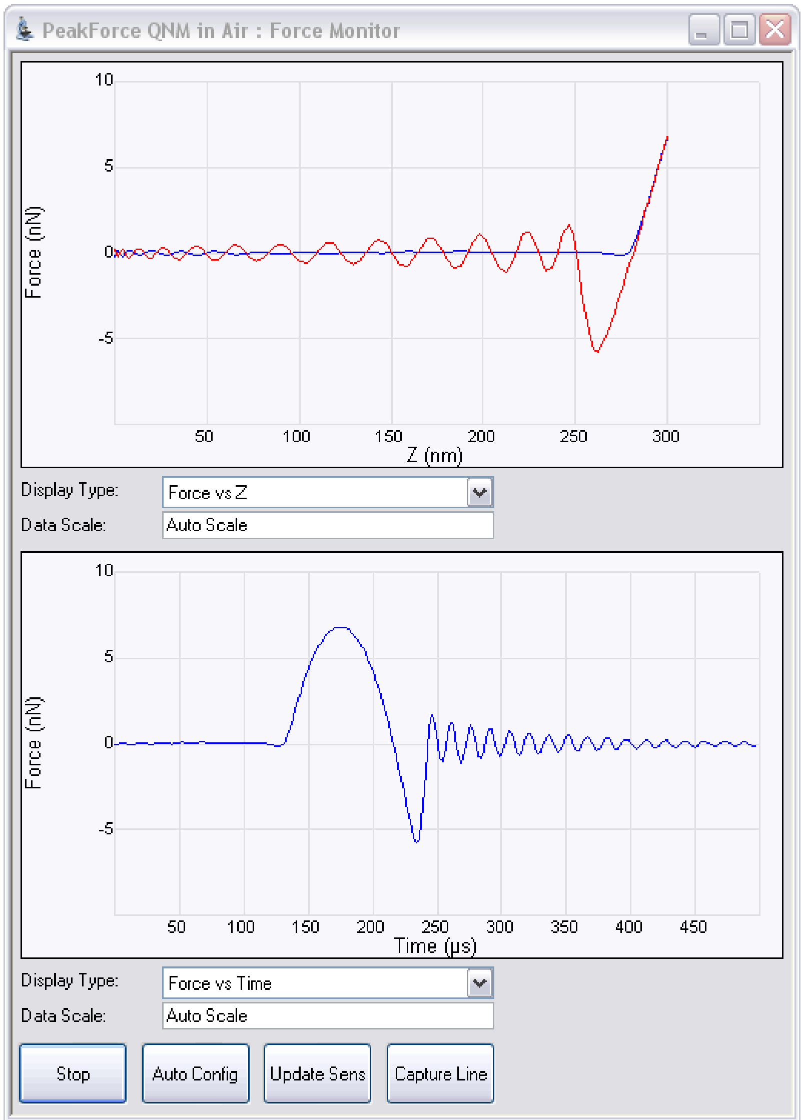
Figure 8.4h Undock the Force Monitor window



2. Select one plot to be **FORCE VS. TIME** and the other to be **FORCE VS. Z**.

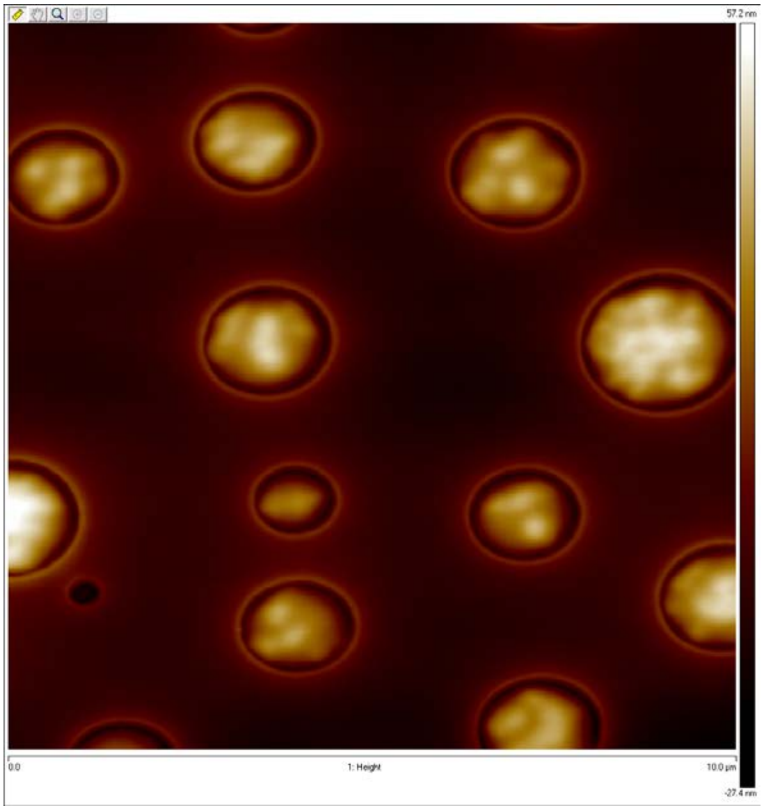
3. Once scanning, the **Force Monitor** window, shown in Figure 8.4i, should display a **Force vs. Z** plot and a “heartbeat” (**Force vs. Time**) plot.

Figure 8.4i The **Force Monitor** window



4. The **HEIGHT** channel in the **Scan** window, shown in Figure 8.4j, will display a topographical image of your sample.

Figure 8.4j Height Image of a PS + LPDE blend.



8.5 ScanAsyst and Peak Force Tapping Mode Parameters

8.5.1 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 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, the probe is lifted by the **Lift Height** distance, the noise measured and then subtracted.

Range and Settings: 0 - 100. If **ScanAsyst AutoConfig Frames** = 0, background subtraction is not done.

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**.

8.5.2 Peak Force Tapping 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 142) 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.

8.5.3 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: 11 V (~0.1375 µm) to 416 V (~5.2 µm).

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

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.

8.5.4 Parameter Visibility

The visibility of various parameters depends on the selected mode. Table 8.5a shows parameter visibility as a function of microscope mode.

Table 8.5a Parameter Visibility

Panel	Parameter	Simple Mode	Expanded Mode	Show All	Other Dependencies
Feedback	**Peak Force Setpoint**	Yes	Yes	Yes	
	Feedback Gain	Yes	Yes	Yes	
	Low Pass Deflection Bandwidth	No	Yes	Yes	
	ScanAsyst Setup	No	No	Yes	
	ScanAsyst Noise Threshold	No	Yes	Yes	
	ScanAsyst Auto Config Frames	Yes	Yes	Yes	
	ScanAsyst Auto Control	Yes	Yes	Yes	
	ScanAsyst Auto Gain	Yes	Yes	Yes	ScanAsyst Auto Control
	ScanAsyst Auto Setpoint	Yes	Yes	Yes	ScanAsyst Auto Control
	ScanAsyst Scan Auto Scan Rate	Yes	Yes	Yes	ScanAsyst Auto Control
	ScanAsyst Auto Z Limit	Yes	Yes	Yes	ScanAsyst Auto Control
Limits	**Z Limit**	Yes	Yes	Yes	
	Deflection Limit	No	Yes	Yes	

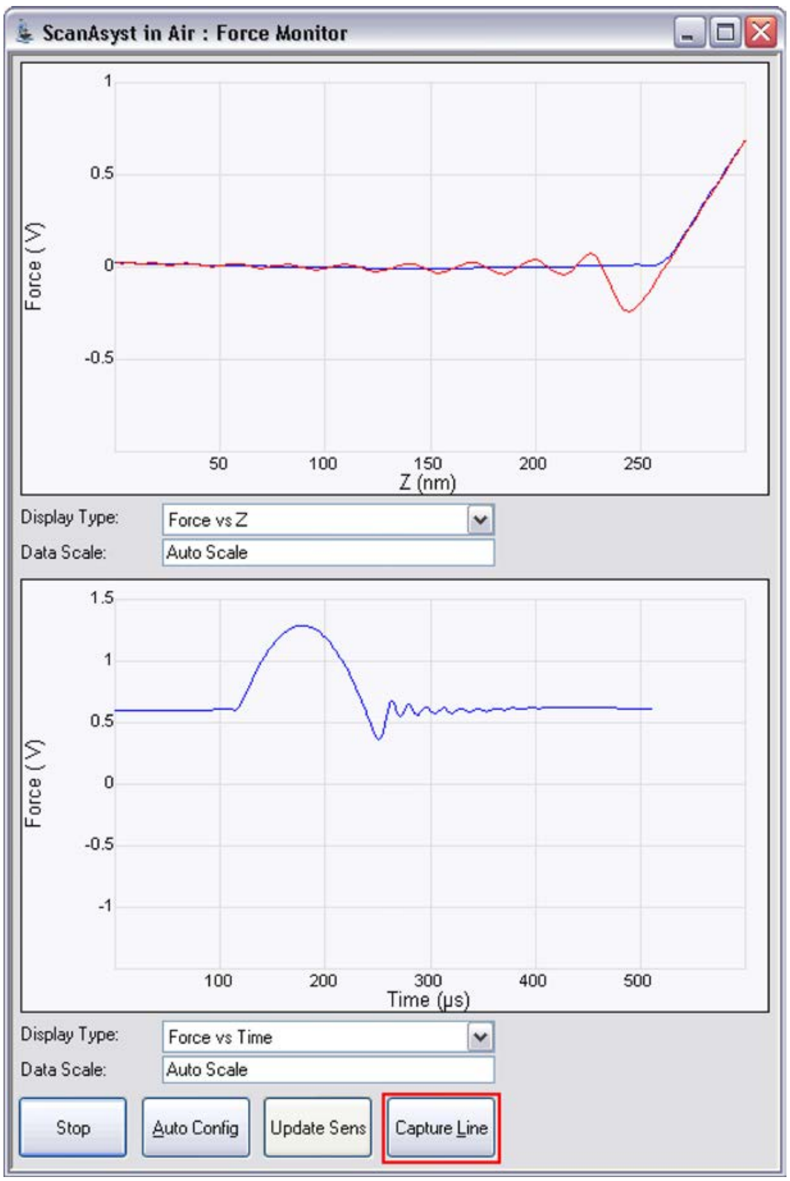
8.6 Capture buttons

The capture buttons 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.

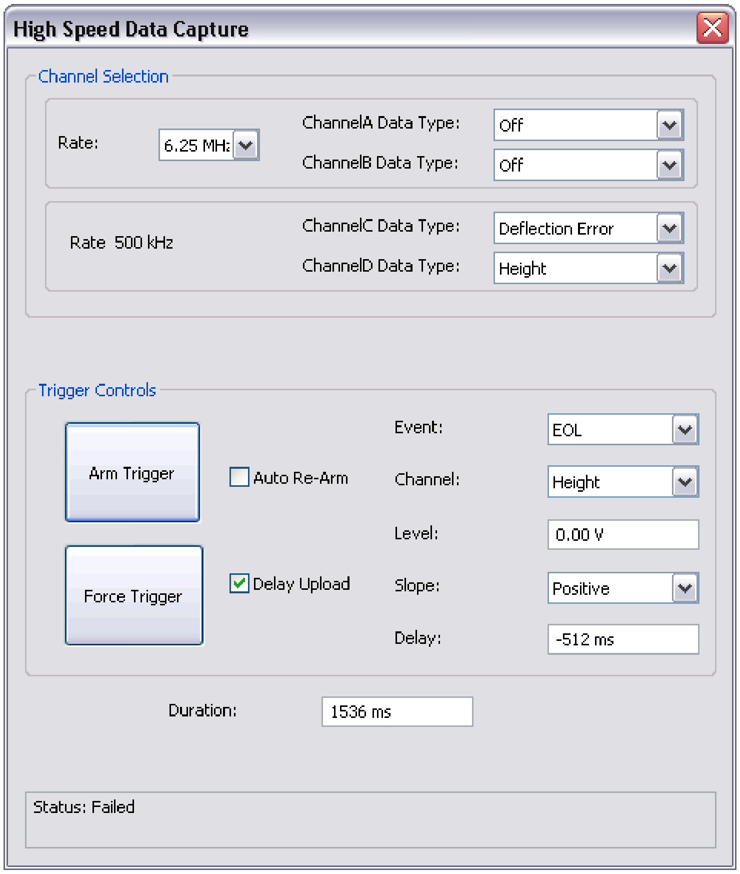
2. When you are in a region of interest, click the **CAPTURE LINE** button, shown in Figure 8.6a, to capture a scan line.

Figure 8.6a **CAPTURE LINE** button



3. The **High Speed Data Capture** window, shown in Figure 8.6b, 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 8.6b High speed data capture is complete. However, the data is not immediately transferred to the PC.



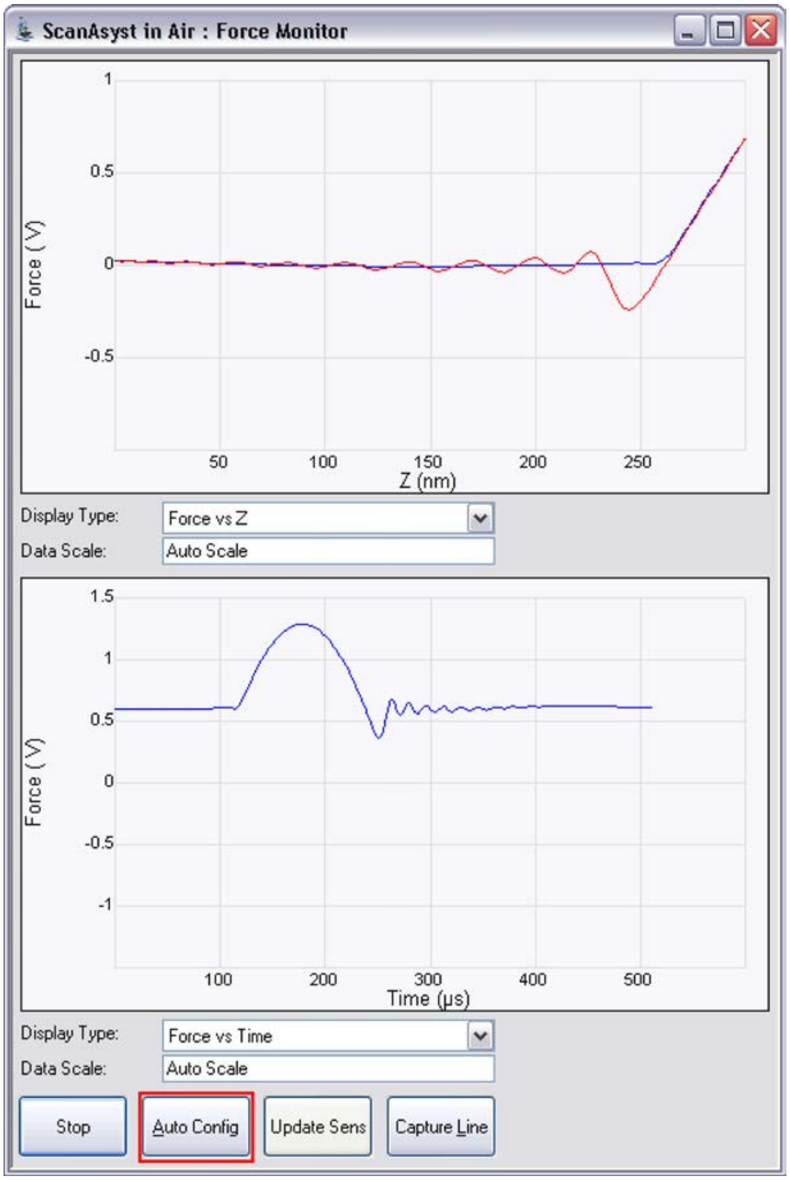
4. Click the **UPLOAD DATA** button to transfer the captured data to the computer. While the data transfer process takes place, the scan data will look corrupted because the DSP time is shared between PeakForce QNM properties computation and data transfer.

8.7 Optimizing a ScanAsyst image

If your force curves show background noise or otherwise need improvement, click **AUTO CONFIG** 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.

Figure 8.7a The **AUTOCONFIG** button



8.8 Advanced Atomic Force Operation

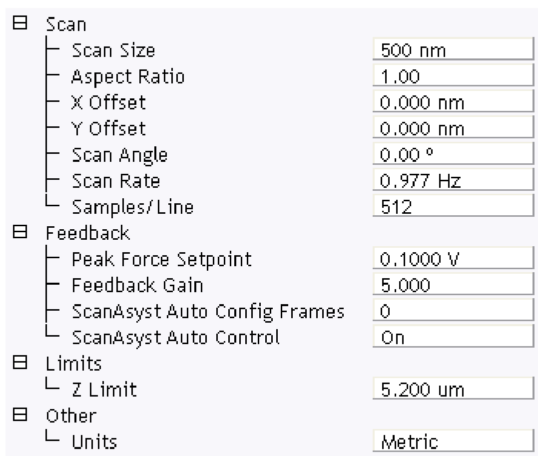
8.8.1 Displaying Parameters

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 8.8a, displays the minimum number of parameters needed to make an image.

Figure 8.8a The **SIMPLE MODE** view of the **Scan Parameter List** for **ScanAsyst in Air**



Expanded Mode

1. The **EXPANDED MODE** view, shown in Figure 8.8b, increases the number of displayed parameters enabling expert users to fine tune an image.

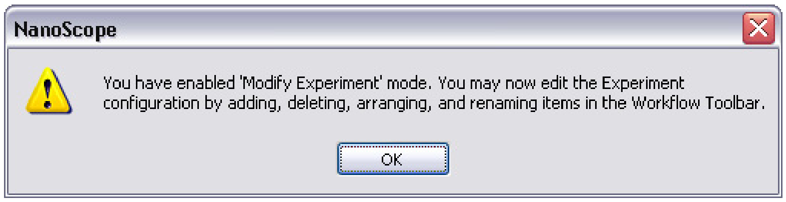
Figure 8.8b The **EXPANDED MODE** view of the **Scan Parameter List** for **ScanAsyst in Air**

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
Tip Velocity	0.977 µm/s
Samples/Line	512
Lines	512
Slow Scan Axis	Enabled
Feedback	
Peak Force Setpoint	0.1000 V
Feedback Gain	5.000
LP Deflection BW	40.00 kHz
ScanAsyst Noise Threshold	1.00 nm
ScanAsyst Auto Config Frames	0
ScanAsyst Auto Control	Individual
ScanAsyst Auto Gain	On
ScanAsyst Auto Setpoint	On
ScanAsyst Auto Scan Rate	On
ScanAsyst Auto Z Limit	On
Peak Force Tapping Control	
Peak Force Amplitude	150 nm
Lift Height	300 nm
Limits	
Z Limit	5.200 um
Deflection Limit	24.58 V
Other	
LP Deflection	Enabled
Tip Bias Control	Ground
Sample Bias Control	Ground
Units	Metric
Bidirectional Scan	Disabled
Tip Serial Number	
Output 1 Data Type	Off
Output 2 Data Type	Off

Show All

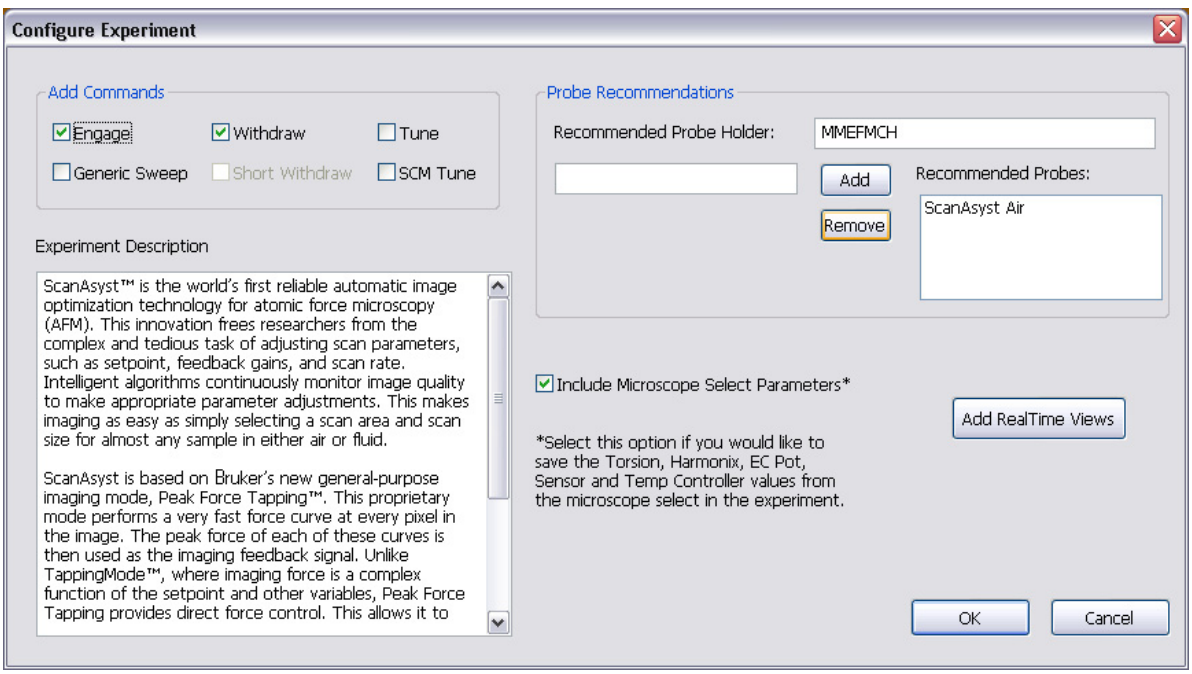
1. From the Menu bar, click **EXPERIMENT** > **CONFIGURE EXPERIMENT**. This opens an information window, shown in Figure 8.8c.

Figure 8.8c The Configure Experiment information window



2. Click **OK** to open the **Configure Experiment** window, shown in Figure 8.8d.

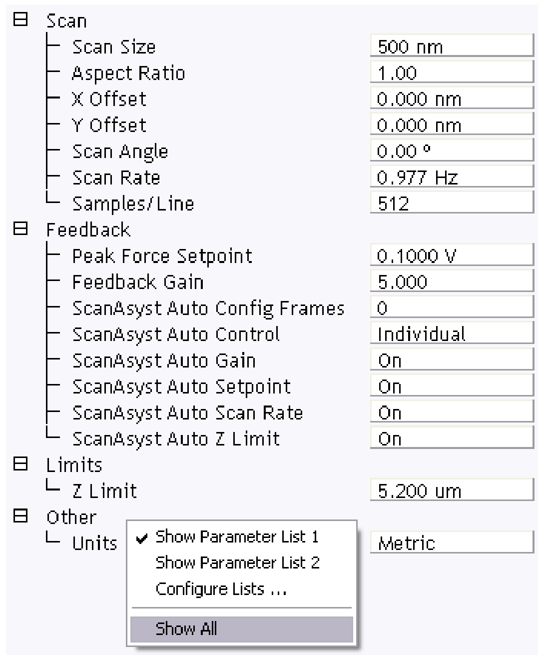
Figure 8.8d The **Configure Experiment** Window



3. Click **CANCEL** which will close the **Configure Experiment** window.

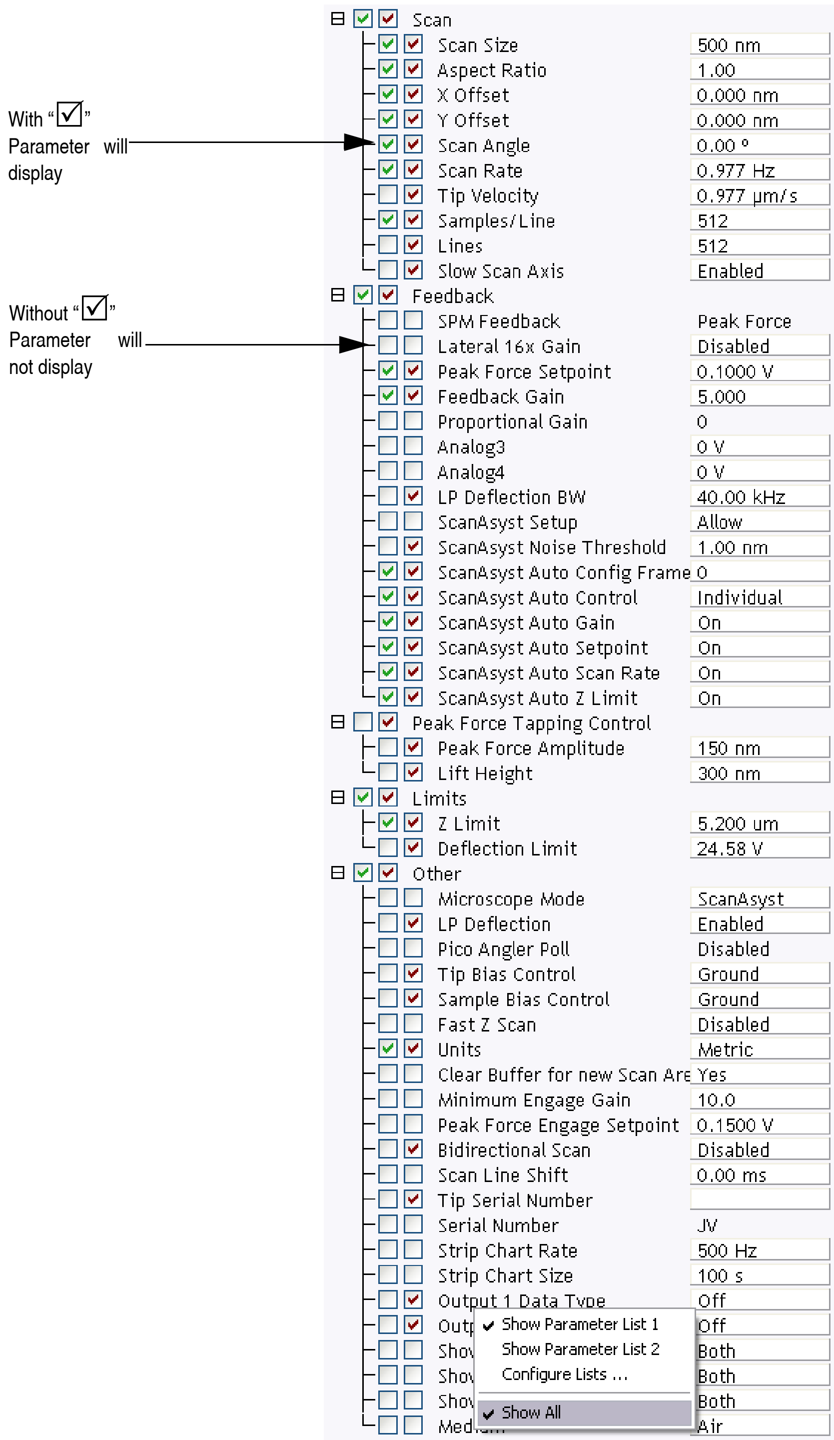
4. Right-click in the **Scan Parameter List** panel and select **SHOW ALL**, shown in Figure 8.8e.

Figure 8.8e Select **SHOW ALL** items



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 8.8f.

Figure 8.8f Enable Parameters



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.