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multimode

with **ScanAsyst**

Instruction Manual

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Document Revision History: MultiMode V8 Manual

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Product Names:
NanoScope®
MultiMode™
Dimension®
Dimension® Icon®
BioScope™
BioScope™ Catalyst™
Atomic Force Profiler™ (AFP™)
Dektak®

Software Modes:
TappingMode™
Tapping™
TappingMode+™
LiftMode™
AutoTune™
TurboScan™
Fast HSG™
PhaseImaging™
DekMap 2™
HyperScan™
StepFinder™
SoftScan™
ScanAsyst™
Peak Force Tapping™
PeakForce™ QNM™

Hardware Designs:
TrakScan™
StiffStage™

Hardware Options:
TipX®
Signal Access Module™ and SAM™
Extender™
TipView™
Interleave™
LookAhead™
Quadrex™

Software Options:
NanoScript™
Navigator™
FeatureFind™

Miscellaneous:
NanoProbe®

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Chapter 1   Introduction to the MultiMode SPM

The MultiMode scanning probe microscope (MM-SPM) is designed for imaging small (approximately 15mm diameter) samples using a series of interchangeable scanners. It is able to provide images of features varying in size from the atomic scale to more than 125µm. This manual provides information specific to the MM-SPM. Some basic introductory information is contained in documents: *SPM Training Notebook* (Bruker p/n 004-130-000) and *Basic SPM Training Course* (Bruker p/n 004-131-000). Refer to the *NanoScope Software 8 User Guide* (Bruker p/n 004-1025-000) for additional information. This manual is designed for use with the NanoScope V Controller and NanoScope 8 software. If you use a different controller/software, refer to the appropriate manuals where applicable.

The MM-SPM uses a stationary probe and samples are scanned back and forth beneath the probe. In some Bruker designs—e.g., Dimension Series SPMs— mounted samples are stationary and the probe scans back and forth above them. Typically, MM-SPM samples are attached to round metal disks ("pucks"), which are magnetically attached to the top of the scanner tube. Voltages applied to the scanner electrodes move the sample back and forth and the probe extracts information somewhat similarly to the way a phonograph needle plays a vinyl record.

Figure 1.0a   MultiMode SPM System Components
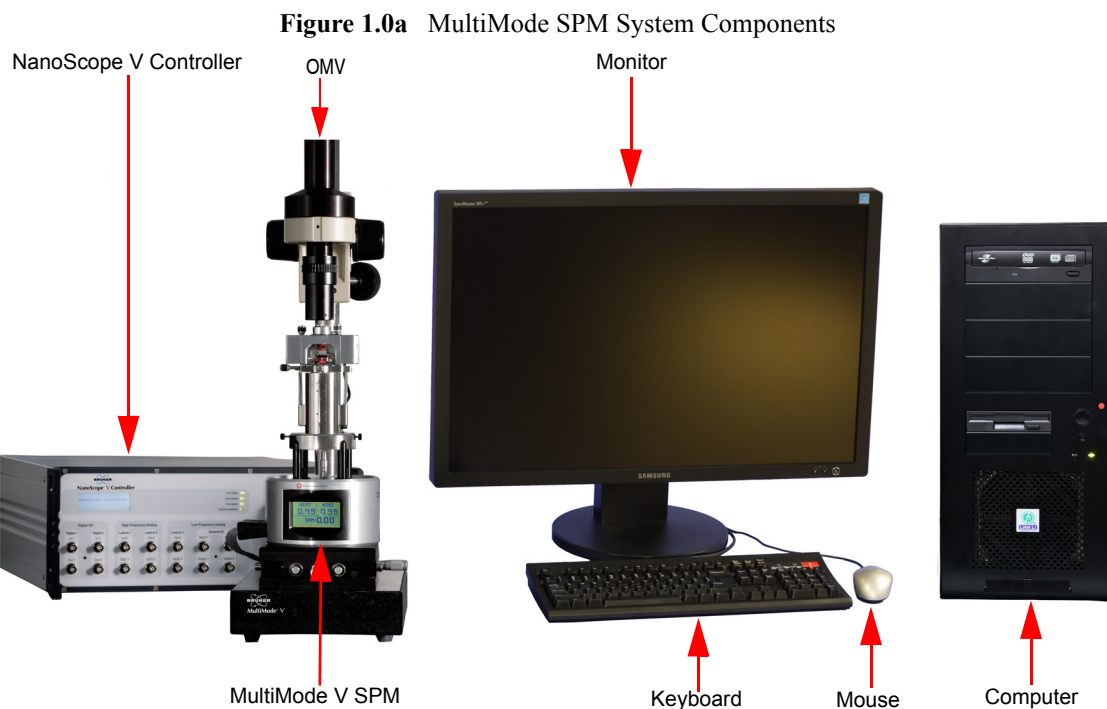
This manual details installation requirements and procedures, maintenance requirements and procedures, and applications used with the MultiMode Scanning Probe Microscope.

This chapter discusses the following:

- **Conventions and Definitions:** Section 1.1

- **Control Station Overview:** Section 1.2

  - **Input and Display Devices:** Section 1.2.1

  - **Computer:** Section 1.2.2

  - **NanoScope V Controller:** Section 1.2.3

- **Facilities Specifications:** Section 1.3

- **Microscope Specifications:** Section 1.4

  - **Image Size and Resolution:** Section 1.4.1

  - **Scanning Techniques with the MultiMode SPM:** Section 1.4.2

- **Terms and Abbreviations:** Section 1.5

- **Maintenance and Troubleshooting:** Section 1.6

1.1 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:
  <u>**Window or Menu Item**</u> / <u>**BUTTON OR PARAMETER NAME**</u> is set to <u>**VALUE**</u>.

- NSV is used to refer to a NanoScope V Controller.

1.2 Control Station Overview

The MultiMode SPM control station consists of three components: input and display devices (keyboard, mouse and monitor), computer and NanoScope V controller.

1.2.1 Input and Display Devices

Input and display devices include a large monitor, keyboard and a mouse, shown in Figure 1.2a. These devices convey information signals to the computer to operate the software and SPM.

CAUTION: The monitor contains a hazardous voltage of 120V.

Figure 1.2a MultiMode V Input and Display Equipment
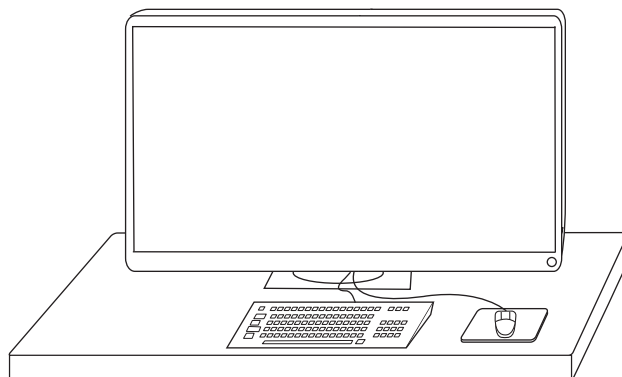
1.2.2 Computer

The computer, shown in Figure 1.2b, is the main control unit of the MultiMode SPM system; it includes a frame grabber, a DVD-RW drive and a custom Digital Signal Processing/Interface (DSP) card. The computer receives data from the input devices, and controls external hardware via the standard ports and the input/output (I/O) bus. The computer isolates the user from direct interaction with the hardware. (See Chapter 5 for a complete description of the computer connections).

Figure 1.2b MultiMode V8 Computer rear view

Power switch

Power cord

Keyboard

Mouse

Monitor

64-pin mini SCSI
(refer to text)
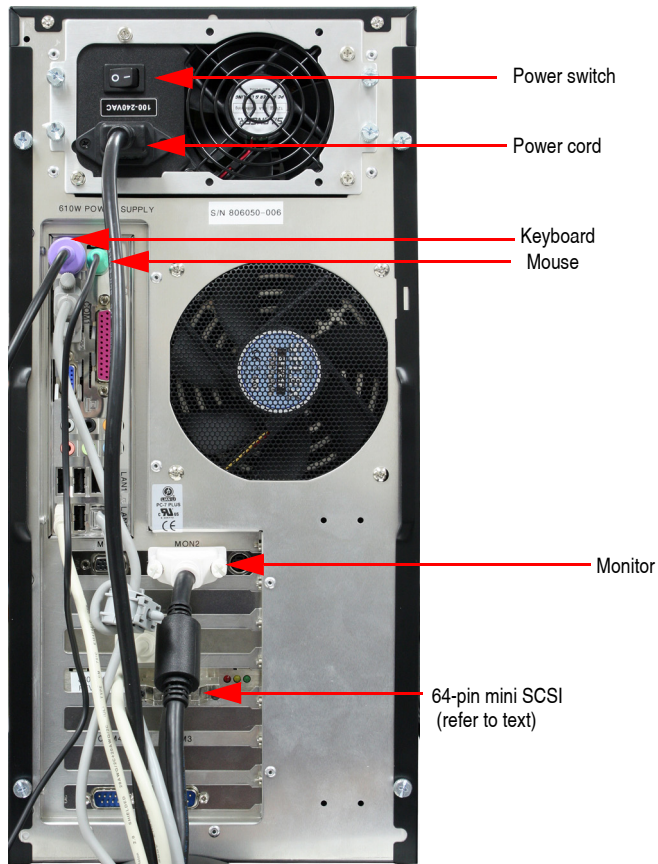
1.2.3 NanoScope V Controller

The NanoScope V Controller, shown in Figure 1.2c, controls the microscope head and scanning. The NanoScope V controller is controlled via a 68-pin micro-SCSI cable connection between the rear of the controller and the computer (see Figure 1.2d).

Figure 1.2c NanoScope V Controller

The controller connects to the side of the MultiMode microscope via a cable from the 37-pin adapter at the front of the controller (see Figure 1.2e).

CAUTION:	Cables should not be removed or installed while power is applied to the system.
	Connecting the 68 pin cable or the 37 pin adapter to the NanoScope V Controller while power is applied may damage the instrument.

Figure 1.2d NanoScope V Controller (back view)

Ventilation Fans, Outflow

Fuses Power Switch

68-pin Micro-SCSI connector to PC

AC Input Requirements

Fuse Ratings

Power Cord
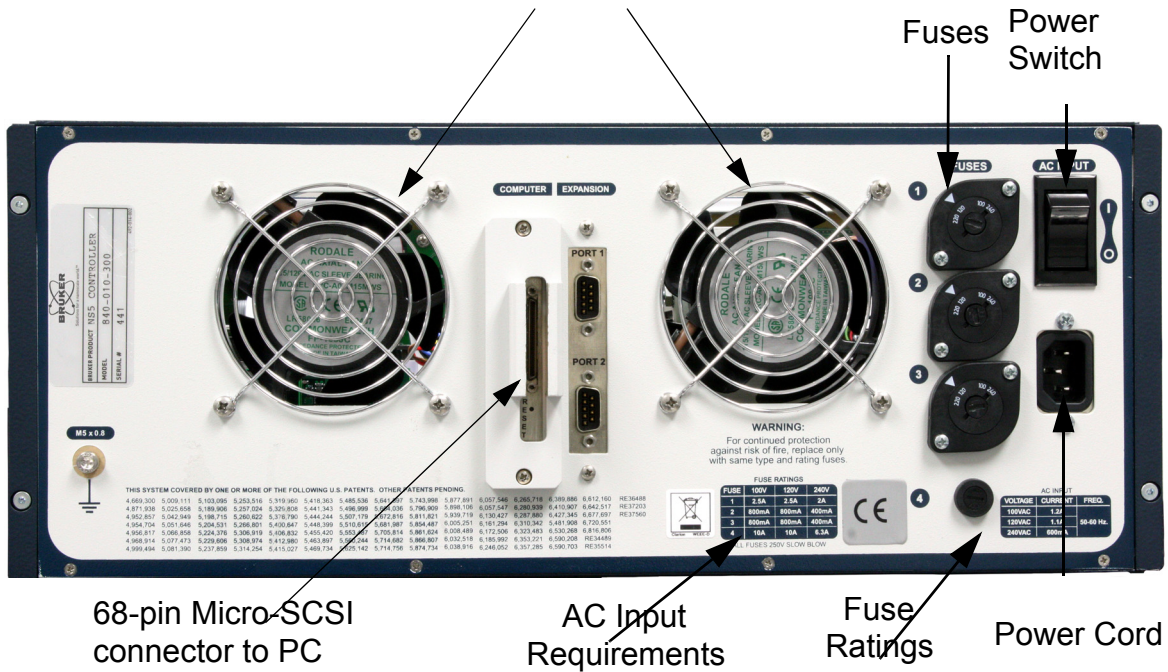
Figure 1.2e Controller (front view)

37 Pin Adapter

> **CAUTION:** The NanoScope Controller will overheat if the controller ventilation holes are blocked or if the controller is exposed to heat from an outside source.

Addition information about the NanoScope V Controller can be found in the *NanoScope V Controller Manual NanoScope Software v 8*, Bruker p/n 004-1030-000.

1.3   Facilities Specifications

Compliance with the requirements and specifications outlined in Chapter 3 is essential before installation and operation of the MultiMode SPM. Chapter 3 details facility site requirements, safety requirements, and configuration options for the MultiMode SPM.

1.4   Microscope Specifications

The MultiMode SPM can be configured with any of several scanners, depending upon the imaging requirements. Generally, the smaller scanners are used for small scan areas or sample feature size. Atomic-scale scans are most often conducted with "A", "E" or "EVLR" scanners. Larger scans are normally performed using "JVLR" scanners. (Ref. Table 4.1a on page 53)

1.4.1   Image Size and Resolution

Images consist of raster-scanned, electronic renderings of sample surfaces at a maximum resolution of 5120 x 5120 points per image. In addition, width-to-height aspect ratio may be specified. The different aspect ratios permit "strip scans" which require less time to capture.

1.4.2   Scanning Techniques with the MultiMode SPM

The MultiMode can operate in several SPM modes. Some of these modes employ Bruker proprietary methods.

- **Contact AFM**—Measures topography by sliding a probe tip across and in contact with the sample. Operates in both air and fluids. See Chapter 9.

- **Tapping Mode AFM**—Measures topography by tapping the surface with an oscillating tip. Tapping mode eliminates shear forces which can damage soft samples and reduce image resolution. Tapping mode is available in air and fluids (patented). This is the technique of choice for most AFM work. See Chapter 10.

- **Phase Imaging**—Provides image contrast caused by differences in surface adhesion and viscoelasticity. See Chapter 10 and Chapter 16.

- **Non-contact AFM**—Measures topography by sensing Van der Waals attractive forces between the surface and the probe tip held above the surface. Provides lower resolution than either contact AFM or tapping mode.

- **LiftMode**—A combined, two-pass technique that separately measures topography (using tapping mode) and another selected property (e.g., magnetic or electric force), using the topographical information to track the probe tip at a constant height above the surface (patented). See Chapter 15.

- **Magnetic Force Microscope (MFM)**—Measures magnetic force gradient distribution above the sample surface. Performed using LiftMode to track topography. See Chapter 16.

- **Lateral Force Microscopy (LFM)**—Measures frictional forces between the probe tip and sample surface. See Chapter 13.

- **Scanning Tunneling Microscopy (STM)**—Measures topography of a conductive sample surface using a tunneling current which varies with the separation between the probe tip and the sample surface. An optional Low-Current STM Converter allows operation in the sub-picoamp tunneling current region which can be useful when scanning poorly conductive samples. Tunneling spectroscopy may also be performed. See Chapter 12.

- **Electrochemical Microscopy (ECSTM and ECAFM)**—Measures the surface structure and properties of conducting materials immersed in electrolyte solutions with or without potential control. See the Electrochemical SPM manuals.

- **Lithography**—Use of a probe tip to mechanically scribe or indent a sample surface. May be used to generate patterns, test for microhardness, etc. Performed using AFM and STM. See the *Command Reference Manual* and *Support Note 225 - Nanoindentation/Nanoscratching*.

- **Torsional Resonance Mode** (TrMode)—Oscillates the cantilever in torsion. See the appropriate *Torsional Resonance Support Note*.

Most of these imaging techniques are discussed in this manual. For additional information, refer to Bruker web site (http://www.bruker.com) to order the necessary support notes or obtain technical support.

1.5   Terms and Abbreviations

This section contains a brief list of terms and abbreviations to assist the reader. Other terms and abbreviations are referenced in the Index at the back of this manual.

AFM—Atomic force microscopy; atomic force microscope.

aliasing—Electronic image error due to differences in resolution between surface features and the pixels used to represent them.

bias—Electrical potential applied to a tip or sample which causes electron flow to ensue from one to the other.

calibration—Measurement of known features to ensure accuracy of SPM images.

cantilever—Flexible portion of probe extending from the substrate and to which the tip is attached.

cantilever tune—Process of finding a cantilever's natural, resonant frequency by exciting the cantilever through a range of frequencies until maximum amplitude (resonance) is obtained.

DSP—Digital signal processor. Computer processor used to control SPM feedback loop.

drive amplitude—Amplitude of the signal used to oscillate a tip in tapping mode.

drive frequency—Frequency of the signal used to oscillate a tip in tapping mode.

engagement—Process of bringing a probe tip and sample together in a controlled manner such that useful information about the surface is obtained without damaging either the tip or the sample.

error—Difference setpoint voltage and detector voltage.

false engagement—Condition due to surface effects or insufficient setpoint (too low during contact AFM; too high during tapping mode) in which the feedback controller attempts imaging a sample that is not engaged with the tip.

feedback—Process of self-correction of the **error** signal.

fluid cell—Accessory used for imaging materials in fluid.

integral gain—Amount of correction applied in response to the average error signal.

LookAhead gain—Amount of correction applied in response to the error signal based upon recorded information from the preceding scan line. This feedback technique is not currently active.

NanoScope—Trademark name applied to Bruker's SPM products.

probe—Integrated mechanical device (including a substrate, cantilever and tip) used for imaging.

probe holder—Removable appliance for mounting SPM probes. On MultiMode SPMs, the probe holder is installed within the head of the microscope. On Dimension Series SPMs, the probe holder attaches to the end of the scanner tube.

proportional gain—Correction applied in response to the error signal in direct proportion to the error.

RMS amplitude—Root mean square (RMS) signal measured at the detector. (tapping mode only.)

SPM—Scanning probe microscopy; scanning probe microscope. A general term encompassing all types of microscopy which utilize a scanned micro-sharpened probe and feedback circuitry to image nanometric phenomena. Includes AFM, ECAFM, ECSTM, EFM, MFM, STM, and many others.

sensitivity—Amount of movement produced by a scanner piezo for a specific amount of voltage.

setpoint—Operator-selected force threshold between tip and sample used as the feedback control loop's target.

spring constant—Amount of force required to bend a cantilever a specific amount (typical units are N/m).

tip holder—See **probe holder**.

1.6   Maintenance and Troubleshooting

Refer to Chapter 19 for detailed information regarding maintenance and troubleshooting issues for the MultiMode SPM.

Chapter 2   Safety

This chapter details the safety requirements involved in use of the MultiMode Scanning Probe Microscope. Specifically, these safety requirements include safety precautions, environmental conditions, and equipment safety applications. Training and compliance with all safety requirements is essential during installation and operation of the MultiMode SPM.

Specifically, this chapter discusses the following areas of information:

- **Aperture Warning Label:** Section 2.5.3

- **Head Serial Number and Contact Information Labels:** Section 2.5.4

2.1   Important Equipment Safety Practices

Here is a summary of precautions to follow during your learning phase. When appropriate safety precautions are observed, the MM-SPM is robust against damage.

2.1.1   Read the manuals!

Even if you have prior experience with the MM-SPM, be sure to read Chapter 1 – Chapter 7 in this manual before doing any imaging work. The remaining chapters describe specific types of imaging. Chapter 9 provides an introduction to contact AFM, which is a good place for beginners to start. Other chapters may be read as required. Consult the *NanoScope Software version 8 User Guide* for a complete explanation of software controls.

2.1.2   Follow good "rules of engagement"

"Engagement" refers to the process of bringing the tip and surface together. This is not a simple process and the software routine for controlling the process is complex. Some probes (especially crystal silicon tapping mode probes) can be broken or damaged if engaged too quickly or with too much force. Ensure that engagement settings never exceed the limits of safety (see Chapter 7) and never attempt to engage manually using coarse adjustment screws.

2.1.3   Never move the head while imaging

The head contains the probe holder, laser and photodiode array. An X-Y translation stage is provided for moving the head and tip several millimeters across the sample for coarse adjustment. Unless using a very flexible cantilever probe such as Silicon Nitride, it is advisable to disengage before using the X-Y stage to reposition the head over long distances. Even for relatively smooth samples and flexible cantilever probes, the head should be moved with great caution while the tip is engaged because tip breakage can occur.

2.1.4   Never leave the controller ON while the computer is turned OFF

When finished with an imaging session, follow the power down sequence: turn off the controller, then turn off the computer. If the controller is left ON for a substantial time without an energized computer, damage to the scanner may result. (This is especially true if the scan has been heavily offset in X and Y.)

2.1.5   Do not unplug cables to/from energized hardware. Turn OFF first

Unplugging or plugging cables on energized hardware is not recommended and may result in damage to the MM-SPM. Always turn OFF hardware before making electrical connections.

2.1.6   Check all connections before hardwiring external equipment

External equipment which is hard-wired into the MM-SPM, such as for EFM and ECSTM imaging, requires special care. To prevent damage to your microscope, always check connections and documented instructions before energizing the system.

2.2   Safety Requirements

Figure 2.2a   Safety Symbols Key

Symbol	Definition
	This symbol identifies conditions or practices that could result in damage to the equipment or other property, and in extreme cases, possible personal injury.
	Ce symbole indique des conditions d'emploi ou des actions pouvant endommager les équipements ou accessoires, et qui, dans les cas extrêmes, peuvent conduire à des dommages corporels.
	Dieses Symbol beschreibt Zustände oder Handlungen die das Gerät oder andere Gegenstände beschädigen können und in Extremfällen zu Verletzungen führen können.
	This symbol identifies conditions or practices that involve potential electric shock hazard.
	Ce symbole indique des conditions d'emploi ou des actions comportant un risque de choc électrique.
	Dieses Symbol beschreibt Zustände oder Handlungen, die einen elekrischen Schock verursachen können.
	This symbol identifies a laser hazard. Exposure could result in eye damage.
	Ce symbole indique un risque lié à un laser. Une exposition à ce laser peut entraîner des blessures aux yeux.
	Dieses Symbol bedeutet "Gefährliche Laserstrahlung". Laserstrahlung kann zu Beschädigung der Augen führen.
	This symbol identifies a thermal hazard. Touching could result in skin burns upon contact.
	Ce symbole indique un risque lié à de hautes têmpratures. Un contact peut entraîner des brûlures de la peau.
	Dieses Symbol bedeutet "Heiße Oberfläche". Berührung kann zu Hautverbrennungen führen.
	This symbol identifies a mechanical crushing hazard. Moving parts can result in serious injury to hands or fingers.
	Ce symbole indique un risque d'écrasement. Déplacer des pièces de l'appareil peut entraîner des blessures sévères des mains ou des doigts.
	Dieses Zeichen bedeutet "Quetschungsgefahr durch mechanisch bewegte Teile". Bewegliche Teile können zu erheblichen Quetschverletzungen von Fingern oder Händen führen.
	This symbol identifies a heavy object. Improper lifting can cause muscle strain or back injury.
	Ce symbole indique un objet lourd. Soulever cet objet de façon incorrecte peut entraîner des froissements musculaires ou des problèmes de dos.
	Dieses Symbol identifiziert ein schweres Objekt. Falsches Anheben kann Muskelzerrungen und Rückenverletzungen verursachen.
	This symbol identifies a corrosive material hazard. Corrosive material can cause serious damage to the equipment.
	Ce symbole indique un risque de la matière corrosive. La matière corrosive peut causer des dommages sérieux au matériel.
	Dieses Symbol bedeutet "Gefahr durch Korrosive Materialien". Korrosive Materialien können erhebliche Schäden am Gerät oder anderen Gegenständen verursachen.

2.3 Safety Precautions

Because the MultiMode SPM features motorized components, it is crucial that operators become familiar with precautions to avoid injury to themselves and/or damage to samples. This section of the manual should be read by ALL persons working with or around the system.

2.3.1 General Operator Safety

⚠️	**WARNING:**	Service and adjustments should be performed only by qualified personnel who are aware of the hazards involved.
	AVERTISSEMENT:	Tout entretien ou réparation doit être effectué par des personnes qualifiées et conscientes des dangers qui peuvent y être associés.
	WARNUNG:	Service- und Einstellarbeiten sollten nur von qualifizierten Personen, die sich der auftretenden Gefahren bewußt sind, durchgeführt werden.

⚠️	**WARNING:**	Follow company and government safety regulations. Keep unauthorized personnel out of the area when working on equipment.
	AVERTISSEMENT:	Il est impératif de suivre les prérogatives imposées tant au niveau gouvernemental qu'au niveau des entreprises. Les personnes non autorisées ne peuvent rester près du système lorsque celui-ci fonctionne.
	WARNUNG:	Befolgen Sie die gesetzlichen Sicherheitsbestimmungen Ihres Landes. Halten Sie nicht authorisierte Personen während des Betriebs vom Gerät fern.

	WARNING:	Voltages supplied to and within certain areas of the system are potentially dangerous and can cause injury to personnel. Power-down all components and unplug from power sources before doing **any** electrical servicing. (Bruker service personnel, *only*).
	AVERTISSEMENT:	Les tensions utilisées dans le système sont potentiellement dangeureuses et peuvent blesser les utilisateurs. Avant toute intervention électrique, ne pas oublier de débrancher le système. (Réservé au personnel de Bruker, seulement.)
	WARNUNG:	Die elektrischen Spannungen, die dem System zugeführt werden, sowie Spannungen im System selbst sind potentiell gefährlich und können zu Verletzungen von Personen führen. Bevor elektrische Servicearbeiten irgendwelcher Art durchgeführt werden ist das System auszuschalten und vom Netz zu trennen. (Nur Bruker Personal.)

	WARNING:	Never alter wiring on the MultiMode SPM.
	AVERTISSEMENT:	Ne jamais toucher les cables et l'installation pneumatique sur le boîtier accoustique du MultiMode.
	WARNUNG:	Ändern Sie niemals etwas am pneumatischen System oder der Verdrahtung der Schallschutzhaube.

	WARNING:	The MultiMode SPM contains a diode laser with an output of less than 1.0mW at 690nm.
	AVERTISSEMENT:	Le microscope "MultiMode SPM" est équipé d'une diode laser dont la puissance de sortie est inférieure à 1.0mW à 690nm.
	WARNUNG:	Das MultiMode SPM ist mit einem Halbleiterlaser ausgerüstet, dessen Ausgangsleistung kleiner ist als 1.0mW bei 690nm.

	WARNING:	Do not use acetone to clean the MultiMode SPM.
	AVERTISSEMENT:	Ne pas utiliser d'acétone pour nettoyer le MultiMode SPM.
	WARNUNG:	Bitte verwenden sie kein Azeton um das MultiMode SPM zu reinigen.

2.3.2 Microscope

To avoid operator injury and equipment damage, observe the following cautions regarding the MultiMode microscope.

⚠️	**CAUTION:**	If you use the equipment in a manner not specified by the manufacturer, you can impair the protection provided by the instrument.

⚡	**WARNING:**	The internal electronics of the microscope, controllers, and peripheral equipment feature high-voltage components. Because there are no user-serviceable parts, do not attempt system repairs. Disconnect faulty components and ship them to Bruker for repair or replacement.
	AVERTISSEMENT:	Les parties électroniques du microscope, du controleur et des équipements périphériques comportent des équipements fonctionnant avec de hauts voltages. Ne pas essayer d'effectuer de réparations, aucune de ces parties n'étant concue pour être réparée par l'utilisateur. Déconnecter les équipements défectueux et les envoyer à Digital Instruments/Bruker pour réparation.
	WARNUNG:	Die Elektronik des Mikroskops selbst, der Steuergeräte und der externen Geräte ist mit Hochspannungselementen ausgestattet. Diese Elemente dürfen nur von geschultem Personal gewartet werden. Versuchen Sie nicht, das System selbst zu reparieren. Trennen Sie fehlerhafte Komponenten vom System, und schicken sie diese zur Reparatur oder zum Umtausch zu Bruker.

WARNING: Do not attempt repairs on electrical components. If it is necessary to enter the electrical chassis for any reason (e.g., to replace a computer card), power-down the entire system and disconnect it from its power source.

AVERTISSEMENT:Ne pas essayer de réparer les parties électroniques. Si il est nécessaire d'accéder au boitier électronique (pour remplacer une carte dans l'ordinateur par exemple), éteindre tout le système et le déconnecter.

WARNUNG: Versuchen Sie nicht, elektrische Komponenten selbst zu reparieren. Falls es aus irgend einem Grund notwendig sein sollte, ein Gehäuse mit elektrischen Bauteilen zu öffnen (z.B., um eine Computer-Karte auszutauschen), schalten Sie das gesamte System ab, und trennen Sie es von der Spannungsquelle.

CAUTION: Avoid spilling fluids onto the microscope stage or into electrical assemblies, particularly the SPM head. If it is necessary to use fluids, apply only small amounts as needed.

ATTENTION: Eviter d'éclabousser la platine du microscope et les assemblages électriques, en particulier la tête du microscope. Si il est nécessaire d'utiliser des liquides, ne les employer qu'en faibles quantités.

VORSICHT: Vermeiden Sie es, Flüssigkeiten auf dem Probentisch oder über elektronische Bauteile, insbesondere den Mikroskopkopf, zu verschütten. Wenn es notwendig ist, Flüssigkeiten zu verwenden, benutzen Sie dem Bedarf entsprechend nur geringe Mengen.

CAUTION: Cables should not be removed or installed while power is applied to the system.

Connecting cables while power is applied may damage the instrument.

2.3.3   Sample Safeguards

⚠	**CAUTION:**	All interlocks are provided to ensure operator and sample safety. **Do not** attempt to bypass interlocks.
	ATTENTION:	Tous les intelocks sont fournis pour assurer toute sécurité à l'utilisateur. **Ne pas** essayer de ne pas les employer.
	VORSICHT:	Alle Sperrvorrichtungen des Systems sind dazu vorgesehen, Personal und Probe zu schützen. Versuchen Sie **nicht**, Sperrvorrichtungen zu umgehen.

2.4   Environmental Conditions

Environmental conditions that may affect the performance of the MultiMode SPM are vibration and noise. See Chapter 3 for detailed condition specifications.

2.5   Hazard Labels

The MultiMode SPM hazard labeling system identifies possible hazard areas. Caution must be taken according to the label warnings when working with associated areas. The following labels appear on the MultiMode SPM:

2.5.1   Laser Explanatory Label

The **Laser Explanatory Label** (see Figure 2.5a and Figure 2.5b) indicates that the area to which the label is affixed is affected by a laser. The Laser Explanatory Label is affixed on the MultiMode SPM base.

2.5.2   Laser Warning Label

The **Laser Warning Label**, on the MultiMode base and shown in Figure 2.5a, indicates that the area to which the label is affixed is affected by a laser.

Figure 2.5a   Laser Explanatory (top) and Laser Warning (bottom) Labels

Figure 2.5b   Laser Explanatory Label
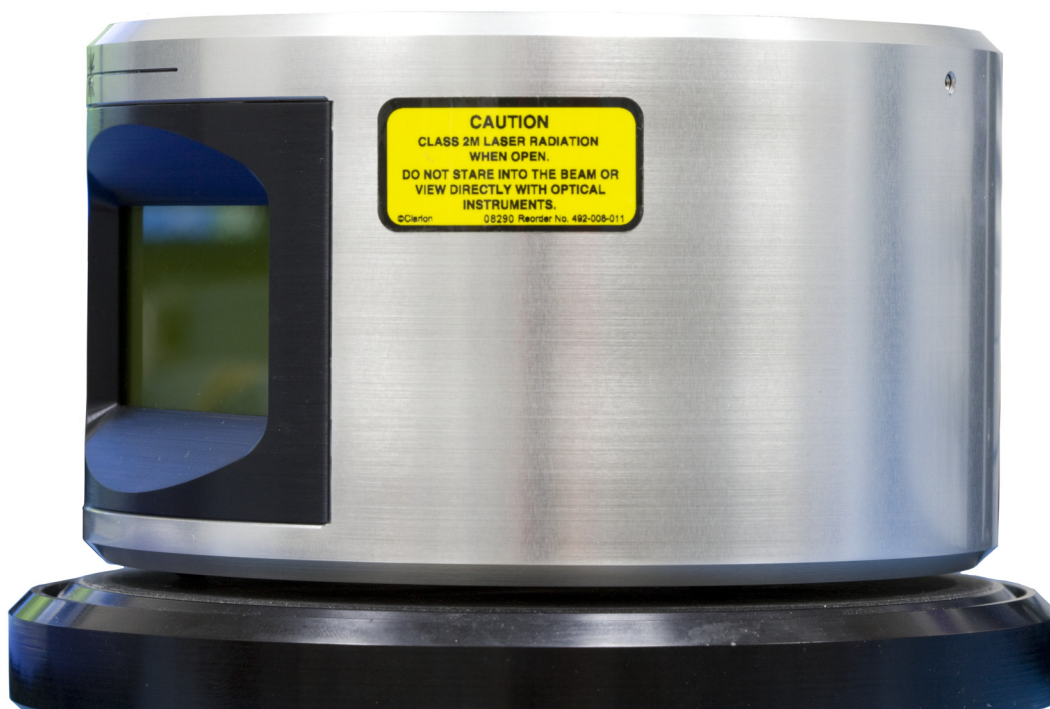
2.5.3   Aperture Warning Label

The **Laser Aperture Warning Label**, shown in Figure 2.5c, is located on the top of the MultiMode head, pointing tword the front opening, where the laser light can exit.

Figure 2.5c   Aperture Warning Label

2.5.4 Head Serial Number and Contact Information Labels

The head serial number, shown in Figure 2.5d, is located on the back of the MultiMode head.

Figure 2.5d Serial number labels

Serial Number

Chapter 3　　　Facilities Requirements

This chapter details facility requirements and configuration options for the MultiMode Scanning Probe Microscope.

This chapter discusses the following:

- **MultiMode SPM Configuration Options:** Section 3.1

    - **VT-103-UNIV Air Table and Acoustic Hood:** Section 3.1.1

    - **VT-102-2 Air Table:** Section 3.1.2

    - **VT-50 Benchtop Vibration Isolation Platform:** Section 3.1.3

    - **TRVI Vibration Isolation Platform:** Section 3.1.4

    - **OMV Optical Viewing System:** Section 3.1.5

- **Computer/Controller Facilities Requirements:** Section 3.2

- **Facilities Requirements Summary:** Section 3.3

- **Environmental Acoustic / Vibration Specifications:** Section 3.4

- **General Facilities Guidelines:** Section 3.5

3.1　MultiMode SPM Configuration Options

The essential components of a MultiMode V8 SPM system are: the SPM, its controller, and a computer with a large (30") monitor. A MultiMode SPM system has no third-party essential components. Optionally, a MultiMode SPM can be housed inside an acoustic hood or placed on a vibration isolation device.

The following are typical configurations for the MultiMode SPM system. This chapter details options for acoustic and mechanical vibration isolation, as well as various options for positioning the control station (computer, control electronics and accessories). Facilities requirements depend on what type of configuration is used.

3.1.1　VT-103-UNIV Air Table and Acoustic Hood

The VT-103-UNIV can be used with the MultiMode. The VT-103-UNIV is an integrated air table and acoustic hood for vibration isolation. The VT-103-UNIV is comprised of an air table on which the MultiMode microscope rests (not shown) and an acoustic hood which can be raised to access the instrument, and lowered to seal the instrument during operation.

> **Note:**　The OMV optical microscope accessory for the MultiMode cannot be placed inside the VT-103 hood. It must be placed on a table adjacent to the NanoScope V Controller.

Figure 3.1a　VT-103-UNIV
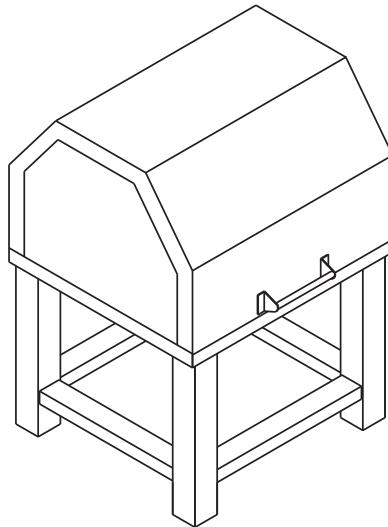
VT-103-UNIV Dimensions

Note: Dimensions and weights are summarized in Section 3.3.

Figure 3.1b VT-103-UNIV (Front View, Hood Closed)
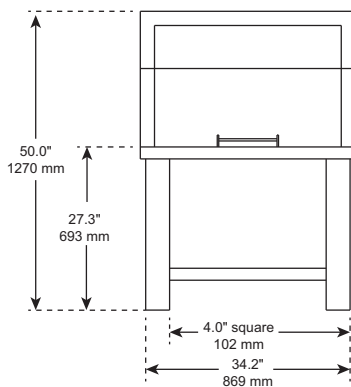
Figure 3.1c VT-103-UNIV (Hood Open)
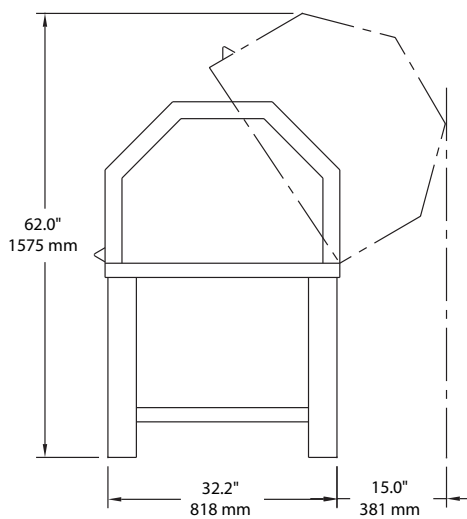
Figure 3.1d VT-103-UNIV (Top View)
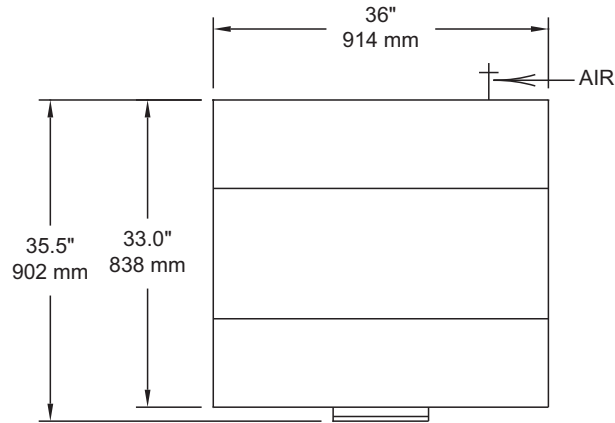
3.1.2 VT-102-2 Air Table

The VT-102-2 air table, shown in Figure 3.1e, can be used with the MultiMode. This configuration consists of two basic elements: the VT-102-2 which is an air table for vibration isolation, and a typical "table top" version of the control station (computer, monitor, control electronics and accessories). The VT-102-2 is a compact air table on which the MultiMode microscope rests and does not include an acoustic hood. The OMV optical microscope accessory can be placed on the VT-102-2 table. Figure 3.1f illustrates the "table top" version of the control station in one of many possible configurations used to position the control station elements. This configuration includes the controller on the far right of the table; the tower computer placed next to it on the table top; and the monitor, keyboard, and mouse located on the far left. Alternatively, the controller and/or computer may be located on the floor or underneath the monitor to minimize footprint.

Figure 3.1e VT-102-2 Air Table
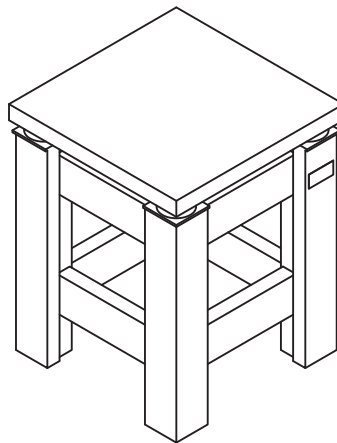
Figure 3.1f VT-102-2 configuration
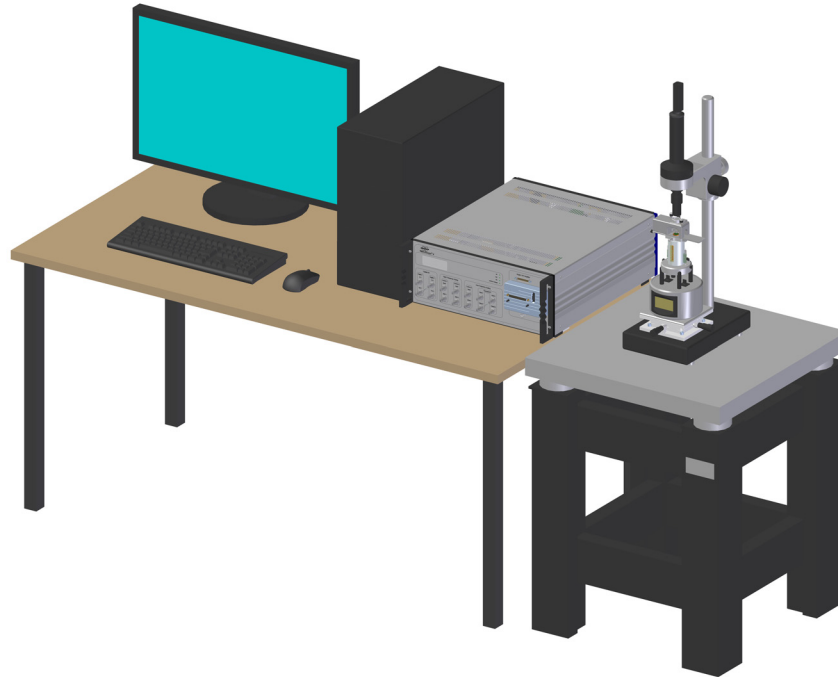
VT-102-2 Dimensions and Utilities

The VT-102-2 vibration isolation table may be used with the MultiMode for selected applications that do not require acoustic isolation for the desired performance level. The table must be moved to its final location before Bruker personnel can install and train on the SPM.

Figure 3.1g VT-102-2 Vibration Isolation Table

Top View   Side and Front View
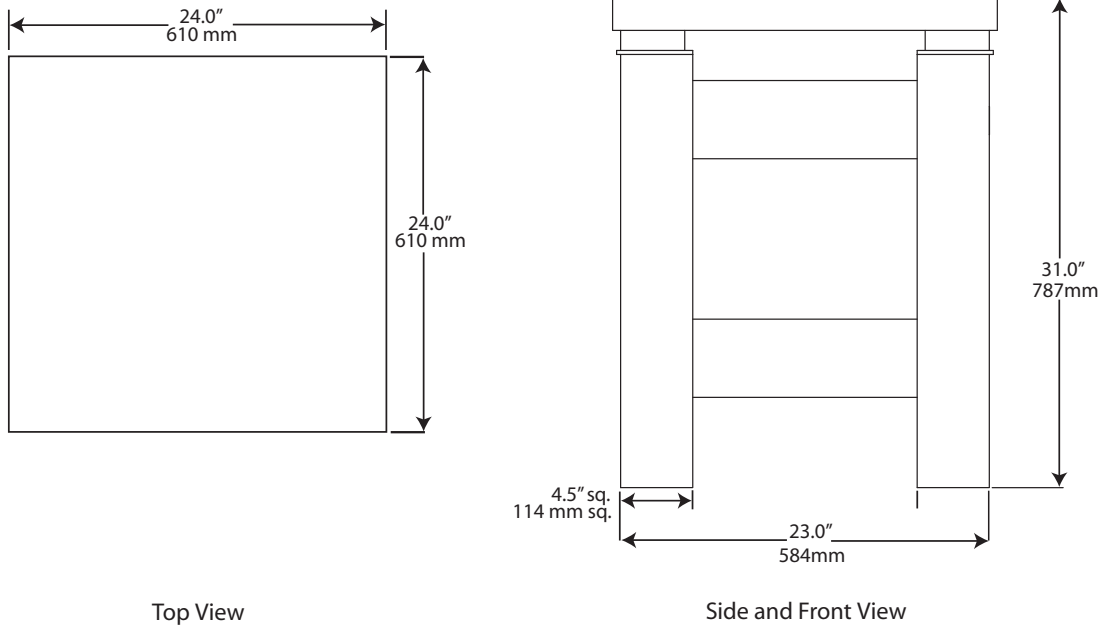
3.1.3 VT-50 Benchtop Vibration Isolation Platform

The VT-50, shown in Figure 3.1h, is a compact vibration isolation platform that offers high performance while not requiring air lines for operation. As a table-top platform, it offers the additional benefit of placing the MultiMode at a convenient height on an existing table. Use of the VT-50 is recommended when a table is available that is highly rigid, adjacent to the workstation, and mechanically separated from the workstation desk. It is not recommended to place the VT-50 on the same desk that is also used for the workstation.

> **Note:** The VT-50 is fully compatible with the OMV optical viewing system given the large dimensions of the isolation platform.

Figure 3.1h   VT-50 Benchtop Vibration Isolation Platform

3.1.4 TRVI Vibration Isolation Platform

The TRVI, shown in Figure 3.1i, is a low cost, highly effective vibration isolation platform with fundamental frequency of oscillation of less than 1Hz in vertical, horizontal and rotational modes. It does not require air lines for operation.

> **Note:** The TRVI is not compatible with the OMV optical viewing system due to inadequate space on the isolation platform.

Figure 3.1i   TRVI Vibration Isolation Platform

3.1.5   OMV Optical Viewing System

The OMV-UNIV optical viewing system, shown in Figure 3.1j, is an aid for aligning MultiMode SPMs. The optical viewing system consists of a camera, which is mounted vertically over the head of the SPM to view both the sample surface and tip.

> **Note:**   The OMV-UNIV is compatible with the VT-102-2 and VT-50 isolation platforms. It is incompatible with the VT-103-UNIV and the TRVI isolation platforms due to space restrictions.

Figure 3.1j   OMV Optical Viewing System
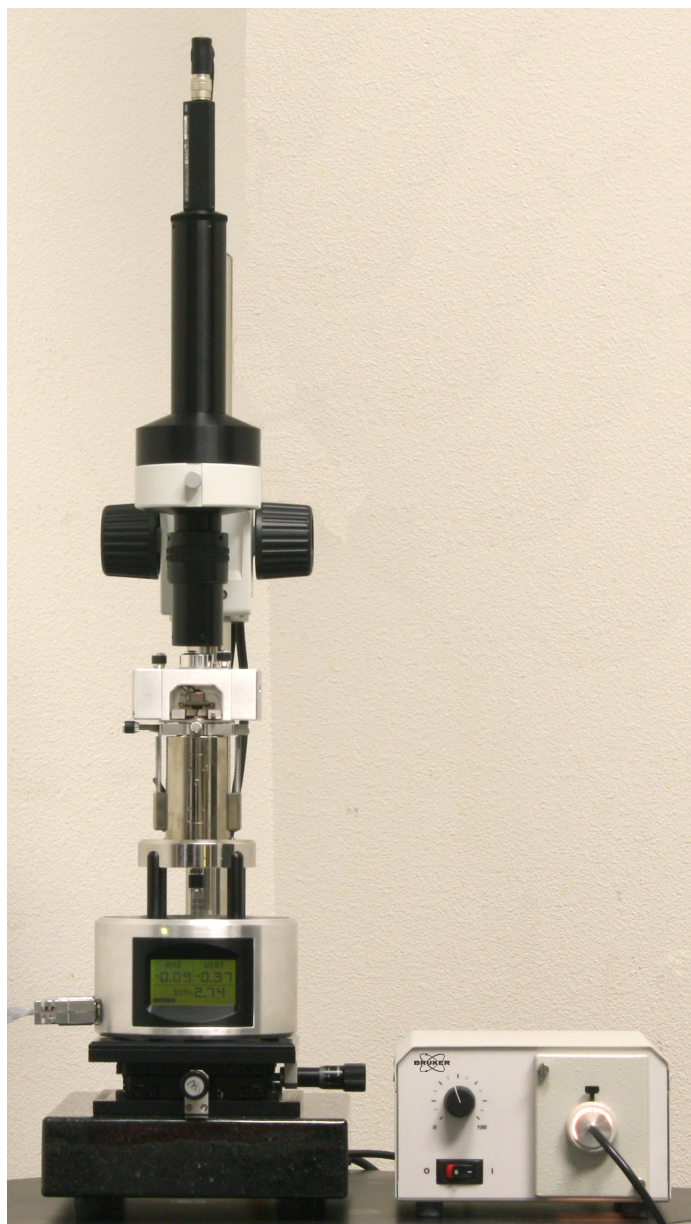
OMV Optical Viewing System Specifications

- Compatible with all TopView AFM, TipView STM and MultiMode configurations

- Allows optical imaging simultaneous with AFM, STM or MultiMode operation

- 10X Nikon objective (working distance = 49.5mm; numerical aperture 0.2; and resolution of 1.6-micron)

- 450X magnification with 14-inch video window

- Erect image (the optical image is not inverted horizontally or vertically)

- 12.7mm travel two-axis stage

- Total vertical range of over 400mm

- Stable, vibration-isolated granite base (additional isolation pad required beneath microscope for atomic resolution)

- Sony high-resolution CCD color camera

3.2   Computer/Controller Facilities Requirements

The customer must supply a table or workstation on which to place the control system.

Figure 3.2a   Table Top Setup
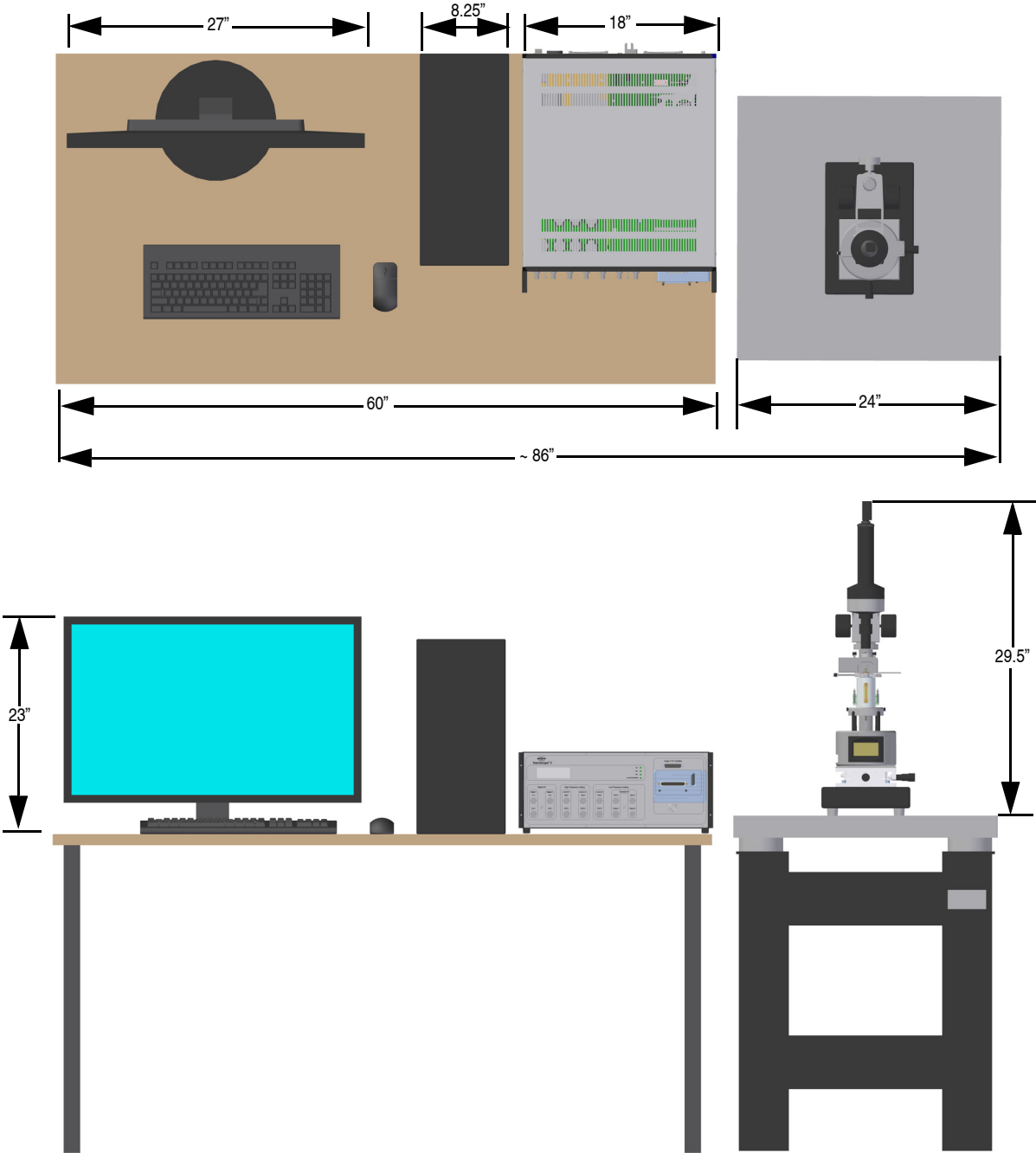
Weight: 120 lbs. nominal total; 100 typically on table top.

Note: Dimensions shown for computer and controller equipment are approximate and subject to change without notice.

3.3 Facilities Requirements Summary

Note: The following summary does not include computer/controller requirements.

Table 3.3a Isolation Table/Hood Facilities Requirements Summary

	MultiMode with VT-103-UNIV Hood / Table	MultiMode with VT-102-2 Table	MultiMode with VT-50
Disconnect or Shutoff	None required	None required	None required
Clean Dry Air for Air Table	Requires user supplied air 60-80 PSI, 1 CFM2	Requires user supplied air 60-80 PSI, 1 CFM2	None required
Approximate Footprints	36" x 33"	24" x 24"	17" x 17"
Min. Door Width	36" minimum	26" minimum	Not applicable
Hood and Table Including SPM	1,000 pounds evenly distributed	650 pounds evenly distributed	85 pounds evenly distributed
Crated Dimensions/Weights of Hoods/ Tables	39" x 39" x 53" 960 pounds	28" x 42" x 37" 650 pounds	23" x 23" x 15" 110 pounds

Table 3.3b MultiMode Facilities Requirements Summary

	MultiMode
Vacuum for Sample Holddown	None required
LAN Connection	None required but network support card included
Electrical	600W; single phase; 100V, 120V or 240V outlet; 50 - 60 Hz dedicated circuit
Exhaust	None required
Environment / Noise	See Page 44
Crated MMAFM System Dimensions/ Weight	54" x 46" x 33" 250 pounds

3.4 Environmental Acoustic / Vibration Specifications

The following conditions must be met in order to guarantee published system specifications:

- Acoustic: Acoustic noise should not exceed 75dBC (Note "C" weighting).

- Vibration: Vibration of the SPM mounting surface should not exceed VC-D in any direction, vertical or horizontal.

Figure 3.4a Vibration Criteria Plot
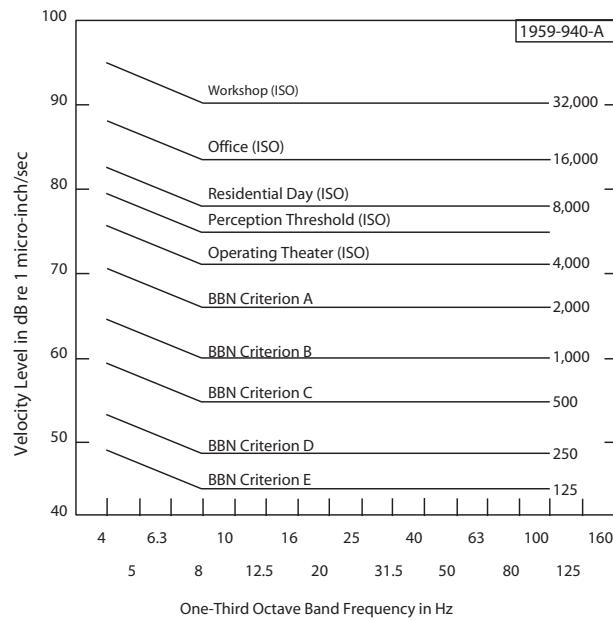
BBN Criterion A - Probe Test Equipment. 100X Microscopes
BBN Criterion B - 500X Microscopes. Aligners, Steppers to 5μm Geometries
BBN Criterion C - 1000X Microscopes. Aligners, Steppers to 1.5μm Geometries
BBN Criterion D - Steppers, E-Beams to 0.3μm Geometries, CD Inspection Equipment.
  Most SEMs to 50,000X
BBN Criterion E - Anticipated Adequate for Future Fabrication and Test Equipment
  for Low Submicron Geometries

3.5   General Facilities Guidelines

The following list contains general facilities recommendations for the MultiMode system:

- Use the ground floor or basement whenever possible.

- Use a sturdy desk or table.

- Do not mount PA/Paging speakers near the AFM. If a speaker is required, use a local volume control instead.

- Keep the telephone ringer on low and install the telephone away from the AFM. Or, turn the telephone ringer off and install a flashing light as a substitute ringer.

- Do not install fluorescent lighting with switching ballasts (also referred to as electronic ballasts). Standard high efficiency ballasts are fine.

- Install baffling in the air ducts to reduce the hissing from the HVAC system or use non-powered HEPA filters. This type of filtering is called Class M7 or 245,000. An in-line fan is installed upstream to provide pressure for the HEPA filter. If installing locally powered HEPA fans, a local on/off switch is needed to turn the fans off while images are captured.

- Add insulation to all walls. If possible, all walls should be hard walls as opposed to temporary walls. Installing insulation in the ceiling helps to dampen the acoustic noise from the roof.

- Temperature is standard laboratory setting +/- 2.5 F°.

- Humidity is standard laboratory setting +/- 10% RH.

Chapter 4   SPM Fundamentals for the MultiMode

The following sections are covered in this chapter:

- **Hardware:** Section 4.1

    - **MultiMode SPM:** Section 4.1.1

    - **SPM Head:** Section 4.1.2

    - **Scanners:** Section 4.1.3

    - **Probe holders:** Section 4.1.4

    - **Probes:** Section 4.1.5

- **Control Mechanisms and Feedback:** Section 4.2

    - **Overview of SPM control mechanisms:** Section 4.2.1

- **Feedback and Gains:** Section 4.3

    - **Proportional and Integral Feedback—An Analogy:** Section 4.3.1

    - **Proportional Feedback and Gain:** Section 4.3.2

    - **Integral Feedback and Gain:** Section 4.3.3

    - **LookAhead Feedback and Gain:** Section 4.3.4

    - **Completing the Analogy—Feedback Gains in SPM:** Section 4.3.5

    - **Setpoint:** Section 4.3.6

    - **More about Feedback and Images:** Section 4.3.7

    - **What Data Type of Image?:** Section 4.3.8

- **Control Parameters and Feedback:** Section 4.4

  - **Reexamining the Control Loop:** Section 4.4.1

  - **General Description of Main Menu Items:** Section 4.4.2

  - **User Example:** Section 4.4.3

  - **Review of General Operating Concepts:** Section 4.4.4

- **Review of Tapping Mode AFM:** Section 4.5

  - **General Operating Concepts:** Section 4.5.1

  - **Optimizing the Tapping Mode AFM Signal after Engagement:** Section 4.5.2

4.1 Hardware

This section describes the MultiMode SPM and system hardware. The MultiMode SPM consists of six components: SPM, controller, computer, keyboard, mouse and monitor.

Figure 4.1a MultiMode SPM System Hardware
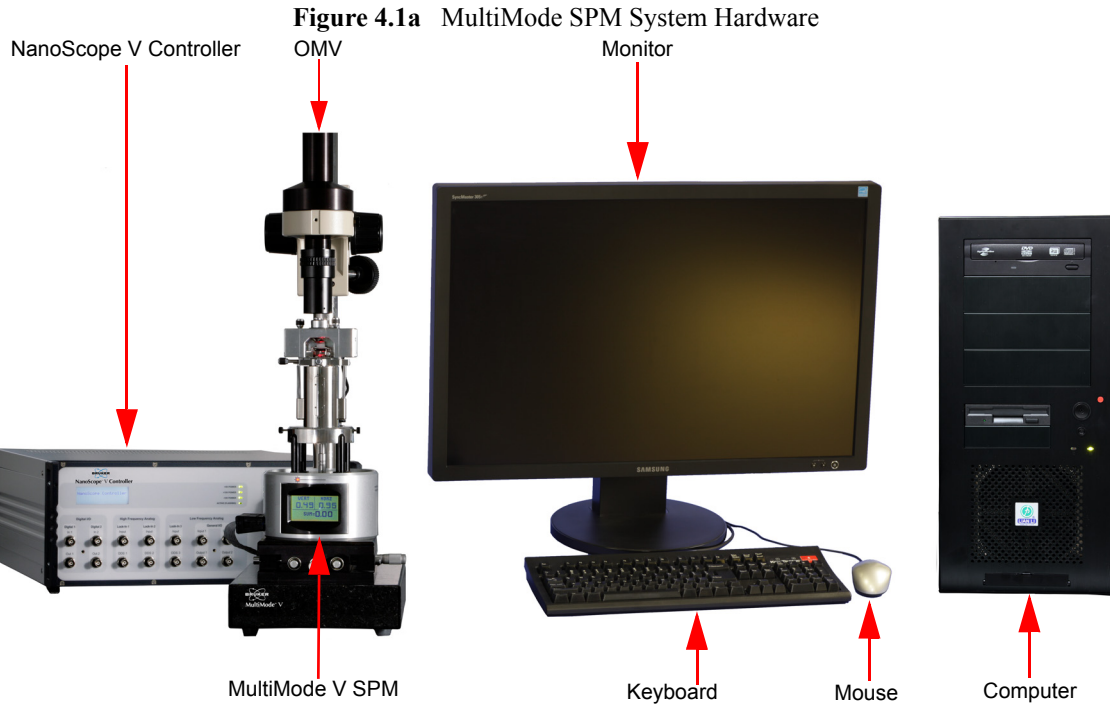
4.1.1 MultiMode SPM

The central component of the system is the SPM, shown in Figure 4.1b.

Figure 4.1b   MultiMode SPM

Photodiode vertical
adjustment knob

Laser adjustment knobs

SPM probe

Sample

Head

Probe holder

Stage controls

Retaining springs

Scanner
(Shown: "EV", model AS-12)

Motor drive
coupling

Scanner support ring

Mode selector
switch

Motor control
switch

RMS/VERT
(tapping/contact)

VERT/HORZ
(tapping/contact)

Stabilizing screw

Signal sum display

Base
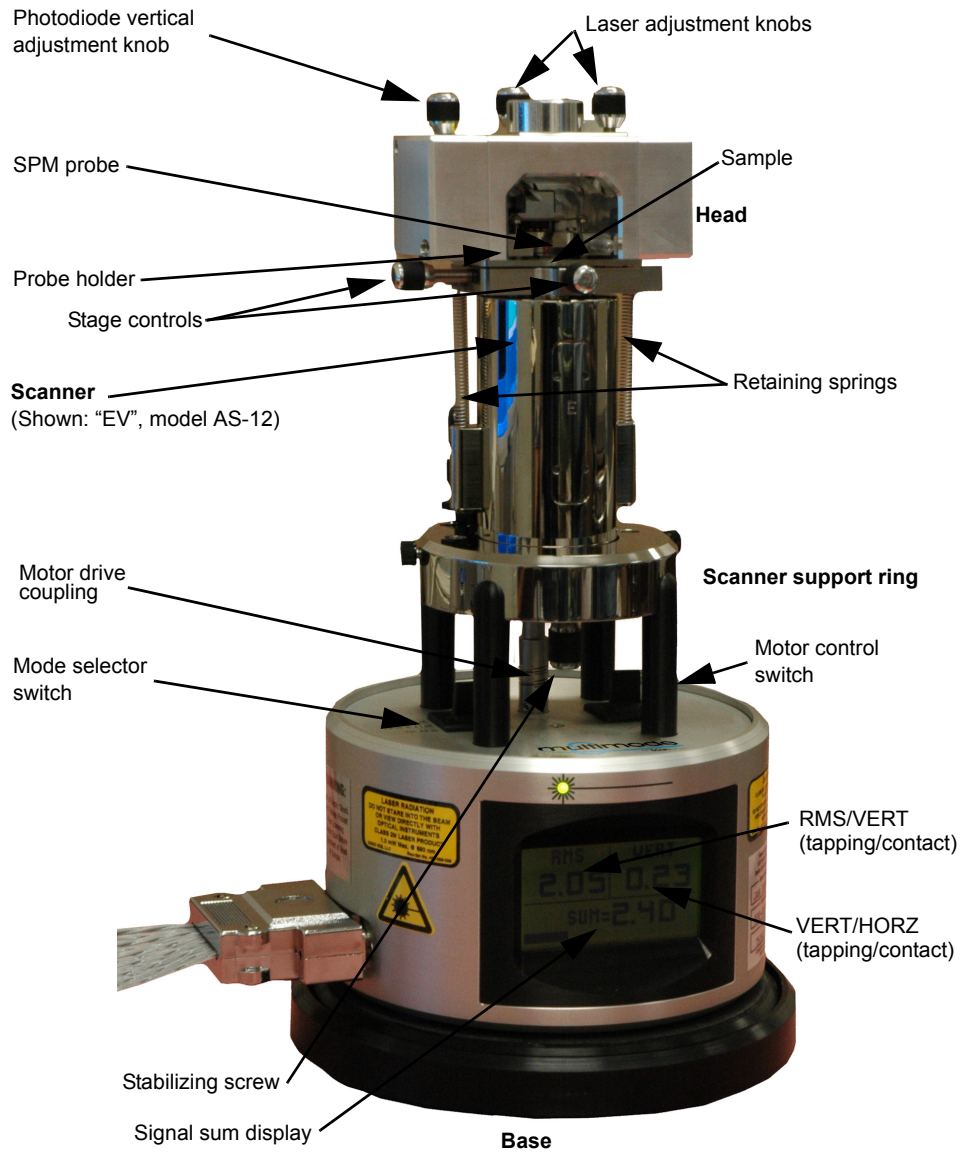
4.1.2 SPM Head

Figure 4.1c illustrates an MM-SPM head with various adjustment knobs. The head and attached X-Y stage are kinematically mated to the scanner via three contact points. A pair of retaining springs secures the head, allowing it to be raised and lowered using adjustment screws threaded through the scanner body. On older models, two screws are manually adjusted by the operator; the rear-most screw is motorized. Newer, "vertical" scanners use a single-screw motorized adjustment.

Figure 4.1c   MultiMode SPM Head and Major Components
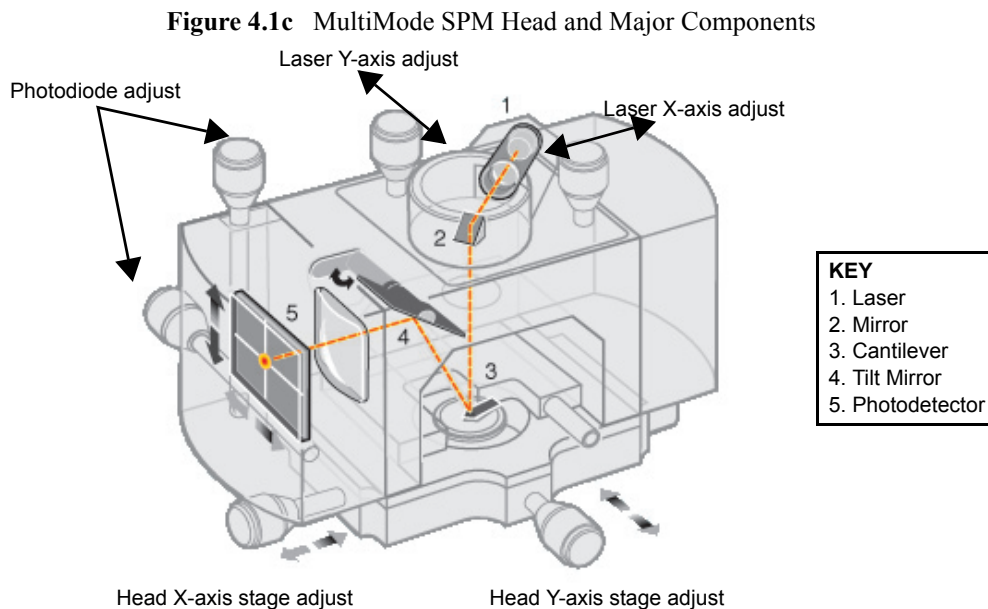
Photodiode array—Figure 4.1d shows the arrangement of the photodiode elements in the MultiMode head. The four elements of the quad photodiode (position sensitive detector) provide different information depending on the operating mode. In all modes, the four elements combine to form the SUM signal. The differential signal between the top two elements (A) and the two bottom elements (B) provides a measure of the vertical deflection of the cantilever. This differential signal is used in Contact and TappingMode AFM. Similarly, the differential signal between the sum of two left photodiodes (C) and the sum of the two right photodiodes (D) provides a measure of the torsional deflection of the cantilever and is used in Lateral Force Microscopy (**Image data type: Friction**), Torsional Resonance mode and HarmoniX mode.

Figure 4.1d Quad Photodetector Arrangement
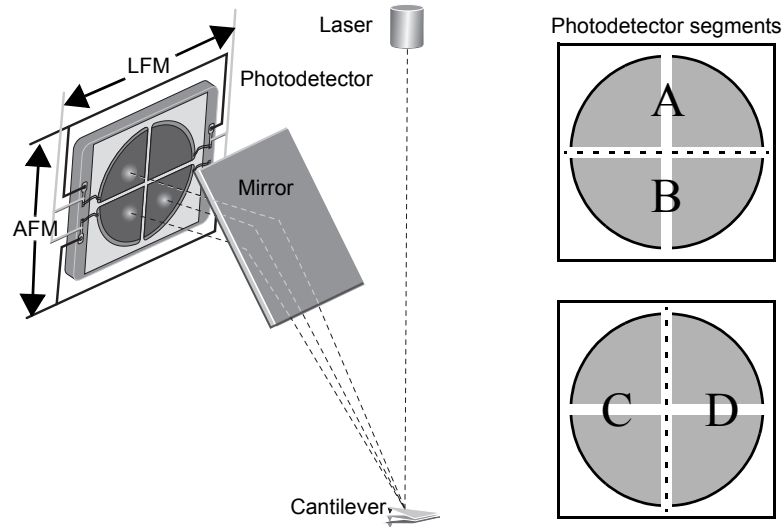
4.1.3 Scanners

Figure 4.1e below shows some of the interchangeable scanners which are used for different sample sizes or resolution (see chart). Longer scanners, e.g., type "J," yield larger field scan sizes; shorter scanners, e.g., type "A," are limited to smaller images but provide better resolution down to the atomic-scale. Smaller scanners tend to be more noise-free at acoustic frequencies because of their compact size and rigidity. Larger scanner offer wider scans, while requiring extra noise dampening precautions at smaller scan sizes of high resolution.

Figure 4.1e Various Scanners Available with the MultiMode SPM

AS-12VLR
(vertical "E")
or
AS-130VLR

AS-12
("E") or
AS-0.5

Note: All scanners are interchangeable.

Every scanner exhibits unique piezo properties which are characterized in a parameter file. When a scanner is changed, the parameter file for the new scanner must also be changed to ensure maximum accuracy at any scan size. Loading a new parameter file requires only a few seconds. Table 4.1a describes the range capabilities of MultiMode SPM scanners.

Table 4.1a MultiMode SPM Scanner Specifications

Model	Scan Size	Vertical Range
AS-0.5 ("A")	0.4µm x 0.4µm	0.4µm
AS-12 ("E")	10µm x 10µm	2.5µm
AS-12VLR ("E" vertical)	10µm x 10µm	2.5µm
AS-130VLR ("J" vertical)	125µm x 125µm	5.0µm

Figure 4.1f depicts the electrode configuration used on a typical scanner piezo tube. Electrodes are oriented as shown when the MultiMode is viewed from the front. AC signals applied to conductive areas of the tube create piezo movement along the three major axes. With the **Scan Angle** parameter in the control panel set to **0.00**, the fast-scan direction is in the direction of the X-axis.

Figure 4.1f Typical Scanner Piezo Tube and X-Y-Z Electrical Configurations
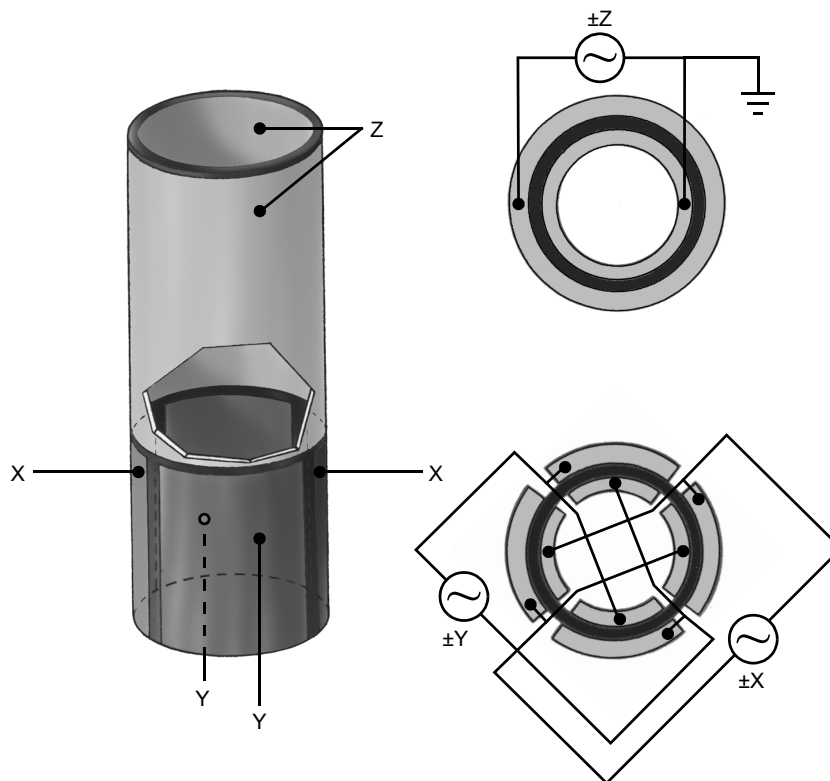
AC voltages applied to the scanner crystal X-Y axes produce a raster-type scan motion represented in Figure 4.1g. The horizontal axis presented on the display monitor is referred to as the "fast axis" (in this example, the X-axis, although either axis may be designated as the "fast axis.") and scans at a **Scan Rate** entered by the user. The orthogonal axis is known as the "slow axis" (in this example, the Y-axis).

Figure 4.1g   Voltages Applied to the X- and Y-axes Produce a Raster Scan Pattern
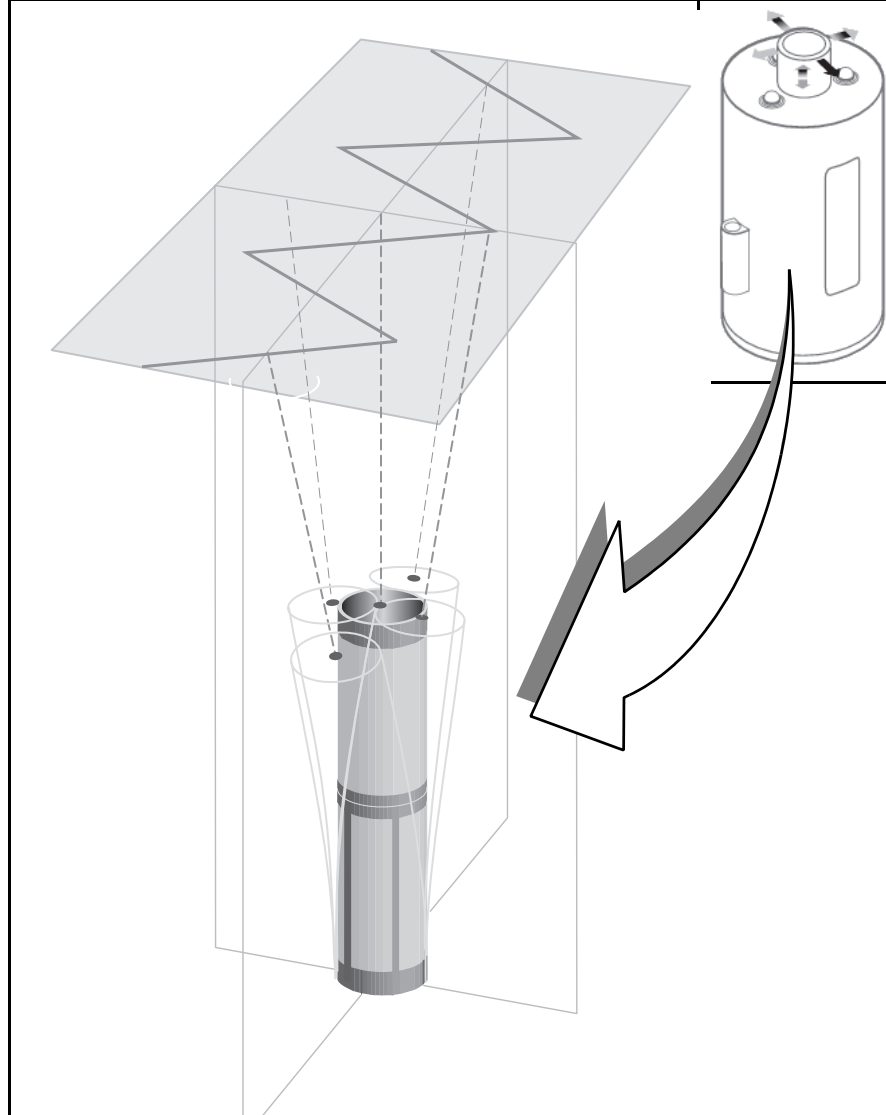
4.1.4 Probe holders

Probe holders might also be referred to as "tip" or "cantilever" holder. "Probe holder" is the preferred nomenclature.

The sample and mode of SPM to be performed dictate the choice of probe and probe holder. For example, if contact AFM is to be used for imaging, a silicon nitride probe mounted in a standard probe holder is the usual choice. If TappingMode is to be used for imaging a biological specimen in fluid, a special fluid cell is employed. STM utilizes a special probe holder, having a tiny tube holder adapted for holding wire tips. Examples of probe holders are shown in Figure 4.1h.

Figure 4.1h Various probe holders for the MultiMode SPM
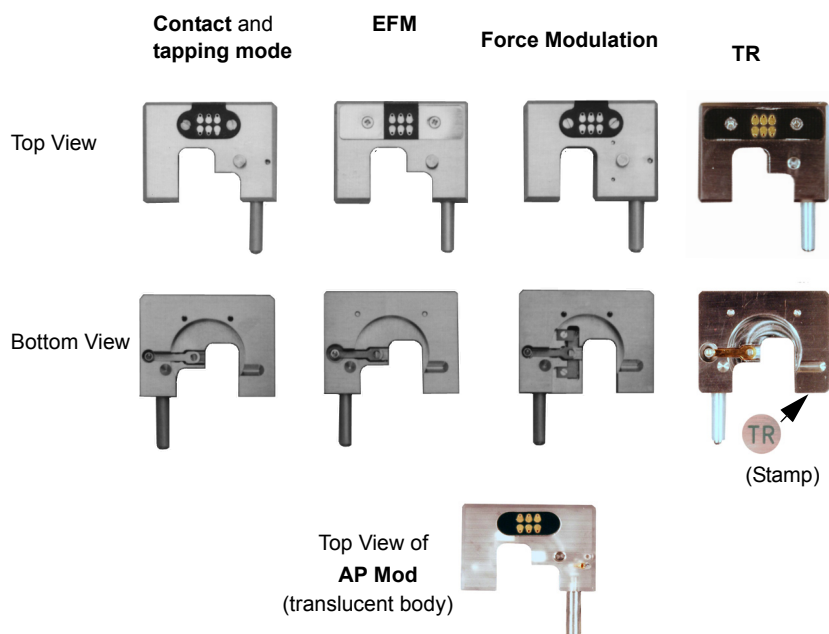
	CAUTION:	Some early-model AFM probe holders may electrically short out power supplies when used with MM-SPMs. If you suspect your tipholder is from an older AFM, check with Bruker or your international representative *before* using.
	ATTENTION:	Certains modèles de support de bras de leviers pour AFM de contact peuvent endommager les alimentations électriques quand ils sont utilisés avec un MultiMode (MM-SPMs). En cas de doute, contacter Bruker ou son représentant légal pour verification.
	VORSICHT:	Einige ältere Ausführungen des AFM-Spitzenhalters können einen Kurzschluß verursachen falls sie mit einem MultiMode-AFM verwendet werden. Bitte wenden Sie sich im Zweifelsfall an Bruker bzw. an die für Sie zuständige Bruker-Vertretung.

4.1.5 Probes

Probes come in a variety of sizes, shapes and materials and are chosen according to the type of imaging being performed.

Cantilevered Probes

Most SPM work is done using cantilevered probes. A sharp tip is located near the end of a flexible cantilever which is held by a rigid substrate. In contact AFM, the cantilever acts as a nanometric spring and surface forces can be measured as the tip follows the sample surface and causes the cantilever to flex. In tapping mode, the probe is oscillated (up and down) near the resonant frequency of the cantilever and oscillation amplitude and/or phase of the cantilever motion are monitored.

Figure 4.1i   Two Types of Cantilevered Probes
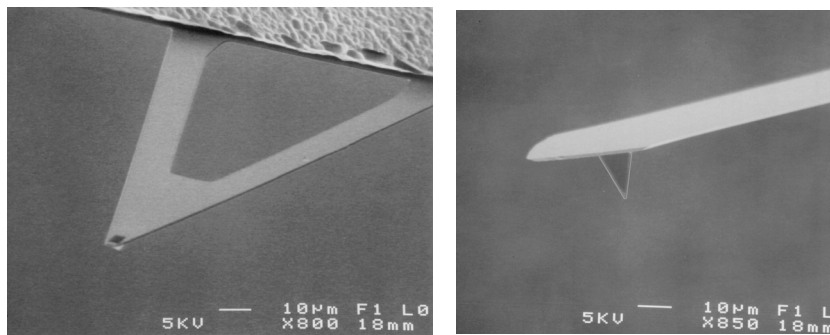
Silicon Nitride   Etched Crystal Silicon

Cantilevered Probes—silicon nitride

Most contact AFM is conducted with silicon nitride tip probes. These probes are very flexible (low spring constant) which makes them easier to use and more "forgiving" than stiffer etched crystal silicon cantilevers. Silicon nitride probes are offered in a variety of sizes and coatings to accommodate the sample being imaged. A concern with silicon nitride tips is that they are easily captured by the sample's surface tension (capillary) properties; that is, trapped within a microscopic layer of condensed atmospheric water vapor on the sample surface. This surface tension effect exerts considerable force at the probe's tip. Although this may not be a problem on harder samples, it may deform softer samples. Adjustment of the **Setpoint** parameter can offset much of this force; however, it may still prove troublesome on delicate samples. Silicon nitride tips are usually not used in tapping mode except in fluids.

Cantilevered Probes—etched crystal silicon

Tapping mode, a Bruker proprietary form of imaging, can minimize contact forces. In tapping mode, an etched crystal silicon tip cantilever probe is oscillated near its resonant frequency. Because the cantilever is relatively stiff and oscillates at a high frequency, it possesses sufficient energy to break free of surface tension forces. An etched silicon probe is more brittle and less forgiving than silicon nitride. The operator must exercise care while setting up the tip and sample.

Cantilevered Probes—MFM

A variation of the tapping mode tip is the MFM probe. This is an etched crystal silicon tapping mode probe with a magnetic coating on the tip. As the magnetized tip oscillates within magnetic fields on or above the sample surface, the cantilever's phase and frequency are altered by the magnetic field. The phase and/or frequency changes provide a measure of magnetic field strength and are used to produce images of magnetic fields or domains.

Cantilevered Probes—EFM

Similar to MFM (see above), EFM is also performed using special tips. Tips are electrically charged and are used to obtain surface potential maps of the specimen. EFM techniques can produce images of the sample's electrical domains.

Specialized Probes

New AFM analyses and methods mat require new probes. Many specialized probes exist and others are being developed. Some specialized probes/analyses include:

- Scanning capacitance microscopy (SCM)—Tip acts as an RF antenna to image microcapacitance phenomena. MFM tips may be used for SCM.

- Chemically doped tips—Tip is doped with a chemical species of interest to the investigator. The chemically doped tip measures chemical bonding forces on sample surface, images receptor sites on biological membranes, etc.

- Focused ion beam (FIB)—tapping mode tips up to 6 μm in length are produced from crystalline silicon using a focused ion beam. Improved aspect ratio provides improved angular resolution on steep side walls.

4.2   Control Mechanisms and Feedback

"Feedback" refers to using the actual result of an action to determine what adjustment or new action is required to produce the desired result. We use feedback in everyday life. If the volume of the radio is too high, this "result" is used as feedback for us to reduce the volume. The new volume may be to loud or too soft and this result is used as "feedback" for the next adjustment. Feedback may come from several sources such as adjusting your driving speed based on feedback from your speedometer, speed limit signs, traffic conditions, etc. Countless examples of feedback applications exist. To produce useful results, the SPM must control the tip-sample interaction with great precision. The control is accomplished by use of electronic feedback. The "result" is the probe spacing from the sample or contact force and the "action" is the adjustment required to make a correction to the desired value. Although signal processing and the specifics of feedback vary with the imaging mode, the feedback loop performs essentially the same function for all modes.

4.2.1   Overview of SPM control mechanisms

The MMSPM uses an "optical lever" to determine probe to sample spacing. A laser beam is reflected off the back of the cantilever probe and positioned onto a photodetector. The relatively long distance between the cantilever and the photodetector acts like a "lever". Very small changes of the cantilever are "leveraged" to produce relatively large changes at the photodetector. While scanning, the cantilever will respond to changes in the sample (by flexing more or less during contact imaging or oscillating with different amplitude or phase during tapping mode imaging). During setup for imaging, the target location or oscillation characteristics of the beam on the photodetector are set by the user established "setpoint". As tracing is performed, the location or characteristics of the beam may move off the setpoint due to the variations in the sample. The variation from the setpoint is an "error signal". The error signal is used in the feedback system to make changes which will reduce the error.

Figure 4.2a   Optical Lever for Monitoring Tip Movement
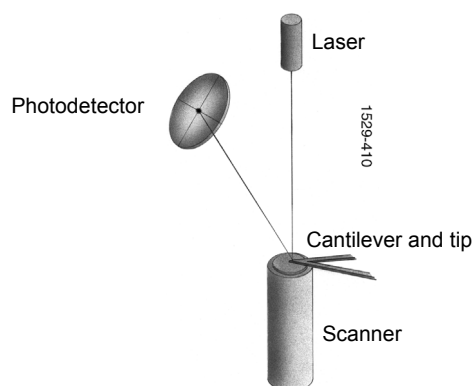
During tracing, error signals are continuously being generated and used by the feedback control system to make corrections. The control system utilizes the error signals in two control methods -- Proportional and Integral. The user controls how much of each method is used by inputting "gain" values.

4.3   Feedback and Gains

The feedback system used to control tip-sample interactions and render images must be optimized for each new sample. This is accomplished by adjusting gains in the SPM's feedback circuit. This section discusses gains and how they are used within the SPM system.

4.3.1   Proportional and Integral Feedback—An Analogy

To provide a simplified explanation of proportional and integral feedback and how they are used to control the SPM, we use the analogy of a hot air balloon carrying three balloonists. Each rider controls a separate gas valve. Each valve is opened partially and connected directly to the burner so opening or closing any valve will cause the balloon to rise or fall respectively. The balloonists decide to open or close valves based on their rules or observations. Some valves may be adjusted more open while others are closed and the balloon will rise or fall depending on the net effect of all three valves. Mounted beneath the balloon's gondola is a camera which photographs the ground below. The balloon's objective is to obtain detailed photographs of the surface. To obtain the highest resolution images, the balloon must track the surface as closely as possible without crashing into it. The problem for the balloonists is to control the balloon's position relative to the ground as accurately as possible.
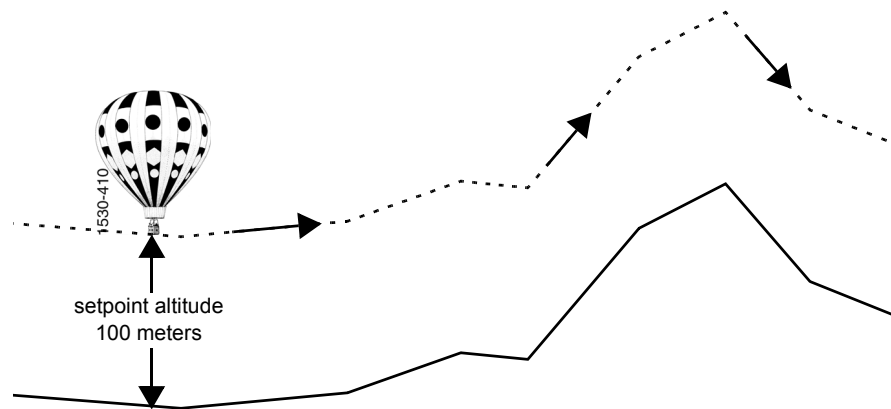
Because the balloon will drift slightly up and down due to the effects of wind and temperature, the balloonists must establish some minimum altitude as a safety zone. Let us call this the "setpoint" altitude, and let us assume that it is set at an altitude of 100m.

> **Note:**   For the sake of simplicity, "setpoint" in this analogy applies to the balloon's altitude. In SPM, "setpoint" corresponds to the probe's height above the sample (which may cause the probe tip to be in contact or out of contact with the sample).

When the terrain is flat, the balloonists need only ensure a constant supply of gas is supplied to the balloon's burners to keep the balloon aloft. As the terrain becomes hilly, the task becomes more complex. If the terrain rises, the balloonists must respond by opening gas valves to the burner to lift the balloon. As the balloon clears the hill and terrain drops away, the balloonists must close the

valves to reduce height and continue tracking the terrain. The way of the balloonists' respond to terrain can be modeled in terms of three types of feedback: *proportional*, *integral* and *LookAhead*.

4.3.2 Proportional Feedback and Gain

Proportional means that a correction is done proportionally in response to the error signal. In the case of our first balloonist, Peter, this means producing hot air in proportion to the balloon's altitude above the terrain: where the terrain rises sharply and the error signal becomes large, Peter uses large amounts of gas to lift the balloon; where the terrain is relatively flat and the error signal remains small, Peter supplies a small, steady amount of gas to maintain the setpoint altitude above the surface. The degree to which Peter reacts to an error signal is called "gain".
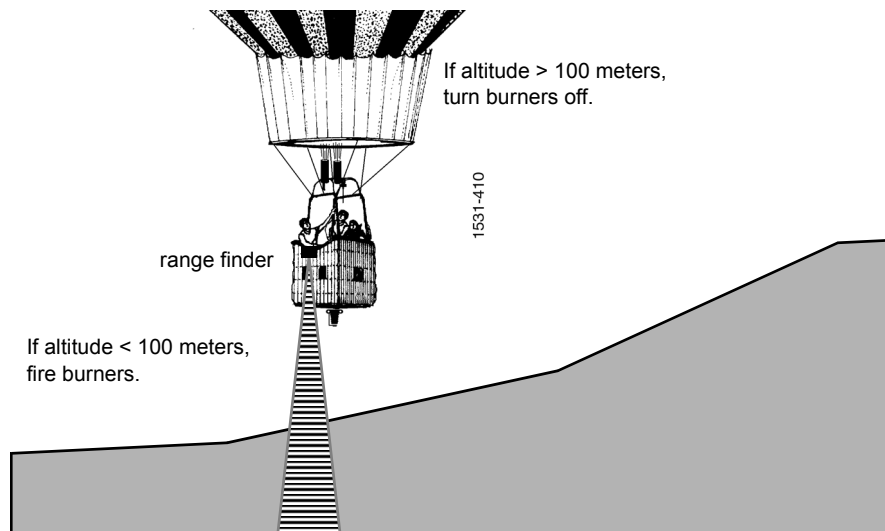
A simple feedback loop is illustrated in this analogy: suppose Peter uses a range finder every 30 seconds to determine the distance between the balloon and ground. If the balloon is below its setpoint clearance, he adjusts his valve to send more gas to the burner. If the balloon is above its setpoint altitude, he reduces gas to the burners to lower the balloon. Peter is given instruction on how much to adjust the valve as the error signal changes. The instruction is called "gain" and is really nothing more than a number. Since Peter deals with proportional feedback, his instruction is "proportional gain." The higher the proportional gain, the more Peter reacts to changes in altitude. For example, at a proportional gain of **1**, if the balloon is 20, 30, 40 meters too low, he increases gas flow by 10, 15 and 20 liters per second respectively. If proportional gain is **2**, the flow rates would be doubled to 20, 30 and 40 liters per second respectively. Although this sort of feedback and gain works well for simple situations, it does not function as well for more complex ones. Using proportional feedback alone, some residual error can remain. The balloon might approach but not quite reach the setpoint of 100 meters or it might end up changing altitude between a little above and a little below the setpoint. Other feedback methods can be used eliminate the residual error.

4.3.3 Integral Feedback and Gain

Integral feedback is based on the cumulative error signal. Peter (proportional feedback) is making burner adjustments based on the error signal. If the balloon is traveling over a random topography, the balloon clearance will be randomly too much or too little so the error signal will be randomly positive or negative. Peter will be adjusting his valve to try to bring the error signal to zero. We know that Peter, doing his very best, will usually be a little off the target setpoint with a tendency to be off in one direction or another (i.e., mostly too high or mostly too low). Irene, the second balloonist will watch for the tendency and use it to derive *integral* feedback to be used to correct for Peter's tendency (the proportional feedback residual error).

Let us assume that Irene checks the range finder every 30 seconds and looks at the difference between the reading and the setpoint clearance of 100 meters. Since Peter is doing a decent job over the random terrain, the error signals is usually fairly small and ranges between +5 and -5 meters. (clearances of 105 and 95 meters). When Irene looks at the average of the last 10 readings, she gets an integral feedback error signal of -1 (on average, the balloon has had a clearance of 99 meters). Irene reacts by opening her valve to send more gas to burner to correct the integral feedback error. After the next 30 seconds, Irene looks at the average of the 10 most recent error signals and adjusts her burner to correct for the new integral feedback error. The effect of integral feedback is to reduce total error by addressing errors over a longer period of time. Integral feedback tends to smooth out the short-term, fluctuating effects and residual error of proportional gain while reducing the setpoint error. Like Peter, Irene has instruction on how to react to integral error feedback and like Peter, the instruction is "gain". Integral gain operates based on integral feedback identically to how proportional gain operates based on proportional feedback. If integral gain is set too high, there is a tendency to overshoot the setpoint. Integral gain is highly sensitive and must be used with care.

4.3.4 LookAhead Feedback and Gain

[Some past systems have included *LookAhead* feedback but there has been some question over the usefulness of this type control. Present systems do not include LookAhead feedback but this description is included for completeness and the eventuality that the control will be reinstated.] The third balloonist, Larry, employs LookAhead feedback. For our analogy, Larry uses a map to anticipate the rise and fall of the terrain. When his map indicates a mountain is approaching, he opens his valve to the burners to lift the balloon. When a valley is indicated on the map, he closes his valve to lower the balloon. The effect of LookAhead gain is to make corrections based on prior knowledge in anticipation of the need for correction. In SPM implementation, the "map" which is used for LookAhead feedback is the preceding trace. Essentially, LookAhead feedback makes corrections in the anticipation that the current trace will be similar to the previous trace. The LookAhead feedback is most useful when there is small variation between adjacent traces. As with previous descriptions of gain, LookAhead gain is the "instruction" on how to react to LookAhead error.

4.3.5 Completing the Analogy—Feedback Gains in SPM

Feedback gains used to control an SPM's probe tip are like to those in the hot air balloon analogy. In the case of a probe tip, the objective is similar to the balloon objective: the operator assigns a setpoint value corresponding to a certain amount of tip-sample force, then adjusts gains for the feedback to track the surface as closely as possible while maintaining the setpoint. Instead of gas-fired burners, the Z-axis piezo crystal uses voltage to raise and lower the probe. The **Scan Rate** parameter must be also be considered. Just as a balloonist would find it difficult to track rough terrain in a fast-moving balloon, the microscopist could have difficulty imaging when Scan rate is set too high. If the balloon clearance setpoint is too low there is a risk of a crash. If the AFM setpoint is improperly set a poor image or damage to the probe or sample might occur.

4.3.6 Setpoint

In our ballooning example, "setpoint" referred to the desired balloon to ground clearance to be maintained. In scanning probe microscopy, "setpoint" refers to tip-sample force. There are two ways of defining setpoint, depending upon whether contact AFM or tapping mode imaging is being performed. In contact AFM, setpoint is determined by the amount of cantilever flexion—as the setpoint increases, the cantilever flexes more and tip-sample forces increase. In tapping mode, setpoint is determined by the RMS amplitude of the oscillating tip—as setpoint decreases, RMS amplitude decreases, but tip-sample forces increase.

> **Note:** An oscillating tip in tapping mode attains its fullest amplitude when it is in free air and not interacting with a sample. As the oscillating tip is brought near the sample, the RMS amplitude decreases due to interference with the sample during intermittent contact. The RMS oscillation is reduced more and more as the probe is moved closer and closer to the sample and the interference increases. A tapping mode **Setpoint** of **0.00** means moving the probe so close to the sample that the RMS amplitude is zero -- that is, the tip is not oscillating at all but remains in full contact with the sample. In tapping mode, reducing setpoint (i.e., the RMS amplitude of the oscillation) increases tip-sample forces … this is opposite to the effect of making setpoint changes in contact AFM.

4.3.7 More about Feedback and Images

Bruker's signal feedback architecture is described in numerous domestic and international patents. The basic feedback processes may be described as follows:

LookAhead

[As stated earlier, recent software implementations do not include LookAhead feedback but this explanation is retained for information only.] As described in the earlier analogy, having a record of previous flights over terrain enabled three balloonists to anticipate the rises and falls of the terrain below them. Similarly, the feedback controller relies upon data from the previous (immediately adjacent) scan line as a predictor of features likely to be encountered in the current scan. The prediction is most reliable on samples which contain regular, periodic features (e.g., gratings) since

scan lines change relatively little from scan-to-scan. Consider, for example, scan lines tracing the surface of a penny.
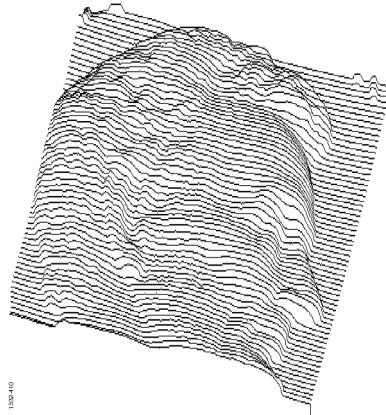
Although this scan is much larger than normally found in SPM, it illustrates how an adjacent, lagging scan line can be used to determine local scan lines on regular surfaces. In most places (e.g., the forehead), each scan line changes little from the line next to it. In some local areas (such as under the nose) there are small, sudden changes; however, these are relatively isolated. In contrast, a similar trace of an irregular, random surface would reveal that each scan line bears little resemblance to its adjacent line.

The purpose of **LookAhead gain** is to take full advantage of regular features by using a previously scanned line to predict the next one. In NanoScope software, the **LookAhead gain** value may be adjusted between a range of **0** (off) to **1.0** (maximum). As values are adjusted upward from **0**, the **LookAhead gain** is weighted to apply more data from the adjacent (lagging) line. **LookAhead gain** is useful for surfaces with regular or repeated features.

When **LookAhead gain** is switched on (> **0**), it is the first gain calculated in the feedback process and is used to weight the integral gain as follows:

$$G_{\text{int}}^{LA} = z_x + (z_{(x),(y-1)} - z_{(x+1),(y-1)})G_{\text{LA}}$$

That is, the **LookAhead**-weighted integral gain, G_{int}^{LA}, is calculated by subtracting the adjacent pixel's Z-axis value from the one immediately next to it, then multiplying this difference by the **LookAhead gain** value and summing the product with the current data value. A diagram of the affected pixels appears as shown here:
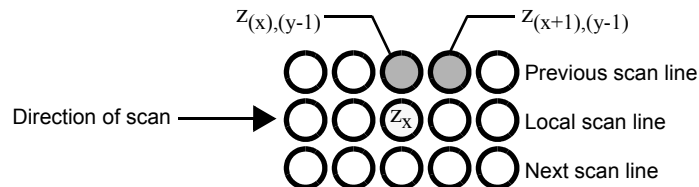
> **Note:** Although the **LookAhead gain** value can be set by the operator to values between **0** and **1.0**, these are *not* the values plugged into the equation. This is a digital signal feedback process and the actual value multiplied varies between 0 and 2^{24}. A similar relation is used for both **Integral** and **Proportional gain**.

At start-up, there is no information yet recorded from an adjacent scan line; therefore, the **LookAhead gain** is effectively **0** until three lines have been scanned. This allows the system to stabilize and acquire data.

Integral

The second step in the feedback process uses integral gain to correct for error by averaging (integrating) the total error. In the ballooning example, Irene kept a running average of the balloon's altitude error, then responded by firing the burners or turning them off to bring the balloon closer to the setpoint altitude. Similarly, the SPM's feedback process maintains a running average of the error and responds to it. As we have seen, enabling the **LookAhead gain** by setting it > **0** conditions the **Integral gain** entered by the operator. If **LookAhead gain** is turned off (= **0**), integral gain enters the feedback process unchanged. The integral gain is then used to calculate a running average of error as follows:

$$z_{\text{acc}}^{\text{new}} = \text{error} \cdot G_{\text{int}}^{LA} + z_{\text{acc}}^{\text{old}}$$

where $z_{\text{acc}}^{\text{new}}$ is the new average error calculated by adding the old average error $z_{\text{acc}}^{\text{old}}$ to the product of the integral gain times the error. The running average represented by $z_{\text{acc}}^{\text{new}}$ maintains itself continually until one or more of the major scanning parameters is changed by the operator. Whenever major scan parameters are changed (e.g., **Setpoint**), the error accumulator is reset and begins a new running average. With the average error calculated, the feedback system makes a final error correction based upon proportional gain.

Proportional

The final step in the feedback process uses proportional gain to complete error correction. Recall that proportional gain responds to error *in proportion* to how much it differs from the setpoint. Proportional gain is used to calculate the correction voltage sent to the Z-axis piezo according to the relation

$$\text{z voltage} = z_{\text{acc}}^{\text{new}} + \text{error} \cdot G_{\text{prop}}$$

As suggested in the equation, the bulk of error correction has been completed prior to implementing proportional gain. **Proportional gain** produces a smaller effect than either **Integral** or **LookAhead gain**. Nevertheless, the system can be driven into oscillation whenever any gain is set too large, including **Proportional gain**.

REMINDER: Gain values entered on the **Feedback** panel do not directly translate to any real quantity, but are merely self-referencing; e.g., a **Proportional gain** value of **1.0** is <u>not</u> the same as **Integral** or **LookAhead gain** values of **1.0**.

4.3.8 What Data Type of Image?

Bruker SPM technology includes many types of microscopy, including: ECSTM, contact AFM, ECAFM, tapping mode in air, tapping mode in fluids, amplitude and phase magnetic force microscopy (MFM), surface potential and field gradient electric force microscopy (EFM), lateral force microscopy (LFM), force modulation imaging, scanning capacitance microscopy (SCM), thermal imaging, HarmoniX^{TM} and force volume imaging as well as variations and combinations of the above. New types of SPM are developed and added continually.

The NanoScope system allows up to eight simultaneous image channels, plus auxiliary channels. Each of the image **Channel** control panels (**Channel 1**, **2**,. . . **8**) contains a **Data Type** parameter specifying the type of image to be shown on that channel. The **Data Type**, in turn, is determined by the currently selected microscope (**Tools**> **Select Microscope**) and **Microscope Mode** shown on the **Other** panel (**EXPANDED** mode). For example, although **Height** data can be displayed for most types of imagery, only tapping mode displays **Amplitude** data. Similarly, only contact AFM displays **Friction** data. Whenever the **AFM mode** and **Data type** parameters are changed, some new portion of the feedback signal is utilized and/or processed differently. Some users employ the NanoScope's auxiliary channels to generate new types of images from the feedback system.

To better understand what is being viewed when selecting different **Data Type**s, consider the diagram below:
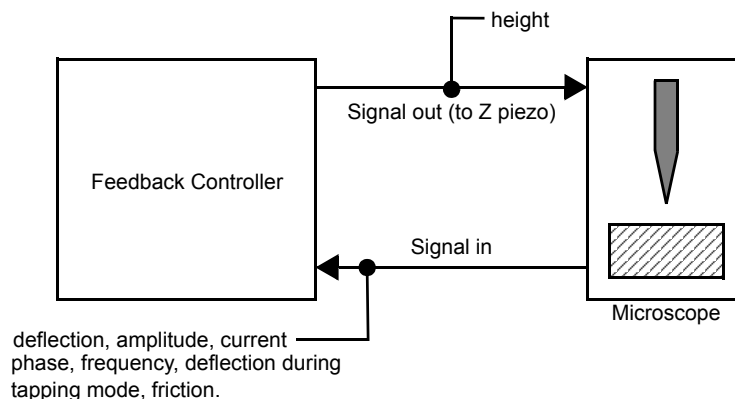
The diagram illustrates that the primary input from the SPM (line 0) is deflection, amplitude or current depending upon whether the data type is Contact, Tapping or STM. In Tapping Mode, the same line conveys **Amplitude** data. In addition, the auxiliary channels exist to input other feedback parameters.

4.4 Control Parameters and Feedback

The feedback methods described in Section 4.3 provide a general basis for imaging sample surfaces; however, large differences in samples require additional controls to obtain optimal images. The NanoScope imaging system utilizes over 1000 parameters. Most of these parameters are never seen by users. This section provides a brief description of some of the more commonly used parameters.

4.4.1 Reexamining the Control Loop

The NanoScope control system performs two main functions: 1) it generates drive voltages to control the X-Y scans of the piezoelectric transducer; 2) it maintains an incoming analog signal from the microscope detection circuitry at a constant value. The analog signal is controlled by a feedback system. The computer is programmed to read voltage from a comparator circuit through an analog-to-digital (A/D) converter and to keep the two inputs of the comparator circuit equal (0V). An output voltage generated by the computer moves the piezoelectric transducer in the Z direction to correct for differences read into the A/D converter. The voltages applied to the X, Y and Z piezo elements are interpreted as imaging data.

4.4.2 General Description of Main Menu Items

This section is a general overview of various Real-time menu items. Refer to the *Command Reference Manual* or the *NanoScope Software 8 User Guide* for more information about these settings.

Scan Size—Size of the scan along one side of a square. If the scan is non-square (as determined by the **Aspect Ratio** parameter), the value entered is the longer of the two sides.

Aspect Ratio—Determines whether the scan is to be square (**Aspect Ratio 1:1**), or non-square (**Aspect ratio 2:1**, **4:1**, **8:1**, **16:1**, **32:1**, **64:1**, **128:1** or **256:1**). The number of lines limits the **Aspect ratio** parameter. You can scan a minimum of two lines.

X Offset; Y Offset—These controls allow selection of the center of the area to be scanned.

Scan Angle—Combines X-axis and Y-axis drive voltages, causing the piezo to scan the sample at variable X-Y angles.

Scan Rate—Sets the number of lines scanned per second in the fast scan (X-axis on display monitor) direction.

Samples/Lines—Sets the number of pixels displayed per line.

Line—Sets the number of lines scanned per frame.

Slow Scan Axis—Starts and stops the slow scan (Y-axis on display monitor). This control is used to allow the user to check for lateral mechanical drift in the microscope or assist in tuning the feedback gains. Always set to **Enable** unless checking for drift or tuning gains.

Z limit—Limits the amount of drive voltage available to the Z piezo circuit. The Z control system uses a 16-bit D/A converter which drives an amplifier capable of outputting voltages from +220V to -220V. This means that the resolution of the control over the Z direction is approximately 6.7mV per bit (440V divided by 65536). This setting defaults to 440V automatically. Reducing the Z limit is useful, when using a "E" or "J" scanner, if scanning samples with relatively small Z features (less than 10 nm peak-to-valley). For example, setting the Z limit to 55V means that 55 Volts is divided by the same 16-bit digital control. This gives eight times finer control over the Z direction of the scanner.

Integral Gain and **Proportional Gain**—Control the response time of the feedback loop. The feedback loop tries to keep the output of the SPM equal to the setpoint reference chosen by moving the piezo in Z to keep the SPM's output at the setpoint reference. Piezoelectric transducers have a characteristic response time to the feedback voltage applied. The gains are values that magnify the difference read at the A/D convertor. The magnified difference results in a larger than required compensation to be made. The large compensation causes the piezo scanner to move faster in Z which is needed to compensate for the mechanical hysteresis of the piezo element. The effect is smoothed because the piezo is adjusted at up to four times the rate of the display.

4.4.3 User Example

Try this experiment with an easy sample to see how changing parameters influence an image. A good choice of sample is a diffraction grating or the calibration reference supplied with the system.

- Display both the input and the output of the feedback loop. This means setting the display to show both **Height** data and the appropriate microscope signal (STM = **Current**, Contact AFM = **Deflection**; Tapping Mode AFM = **Amplitude**).

- Engage the microscope and reduce the gains until they are close to zero. The input display data will become larger in Z, and the height data will blur or become smeared. Raise the gains using the left and right arrow keys until the input voltage is minimized.

- Try increasing and decreasing the **Scan Rate** parameter. This will increase or decrease the traveling velocity of the tip. Note that it will be necessary to increase the gain settings at faster scan speeds and decrease the gains at slower scan speeds.

4.4.4 Review of General Operating Concepts

The AFM system is comprised of the scanner and the AFM detection system. The scanner houses the piezoelectric transducer. The piezo element physically moves the sample in the X, Y and Z direction. The detection system consists of a laser which generates a spot of light that is reflected off of a microfabricated cantilever onto a mirror and finally into a photodetector (see Figure 4.4a). The position of the spot is determined by circuitry which generates a voltage from the difference between the photodiode segments (A - B). The circuit outputs a voltage ranging from +10V to -10V depending on the position of the spot on the two photodiodes.

The AFM system maintains the tip at the end of the cantilever in contact with the sample surface. The sample is scanned under the tip in X and Y. Features on the sample surface deflect the cantilever, which in turn change the position of the laser spot on the photodiodes. This position change is read by the feedback loop. The feedback loop moves the sample in Z to restore the spot to its original position.(see Figure 4.4a).

1. A flat portion of the sample surface is scanned beneath the tip left-to-right, maintaining the laser beam at the center of the photodiode array.

2. As the tip encounters a raised feature, the cantilever is pushed up and the laser beam is deflected onto the "A" portion of the array. With the "A" photodiode receiving an increased portion of the laser light, its voltage increases while portion "B's" decreases (A > B).

3. The Vertical Deflection (A-B) voltage differential is sensed by the feedback electronics, causing a dropped voltage to the Z piezo crystal—the piezo retracts. As the Z piezo retracts, the cantilever recenters the laser beam onto the photodiode array (A = B).

4. As the tip encounters a decline in the sample topology, the tip drops. This directs more of the beam onto the "B" portion of the photodiode array. With the "B" photodiode receiving an increased portion of the laser light, its voltage increases while portion "A's" decreases (A < B).

5. Again, the Vertical Deflection (A-B) voltage differential is sensed by the feedback electronics, increasing voltage to the Z piezo crystal—the piezo extends. As the Z piezo extends, the tip is pushed down until the laser beam recenters on the photodiode array (A = B).

Figure 4.4a Contact AFM Concepts (Steps 1-5 exaggerated.)
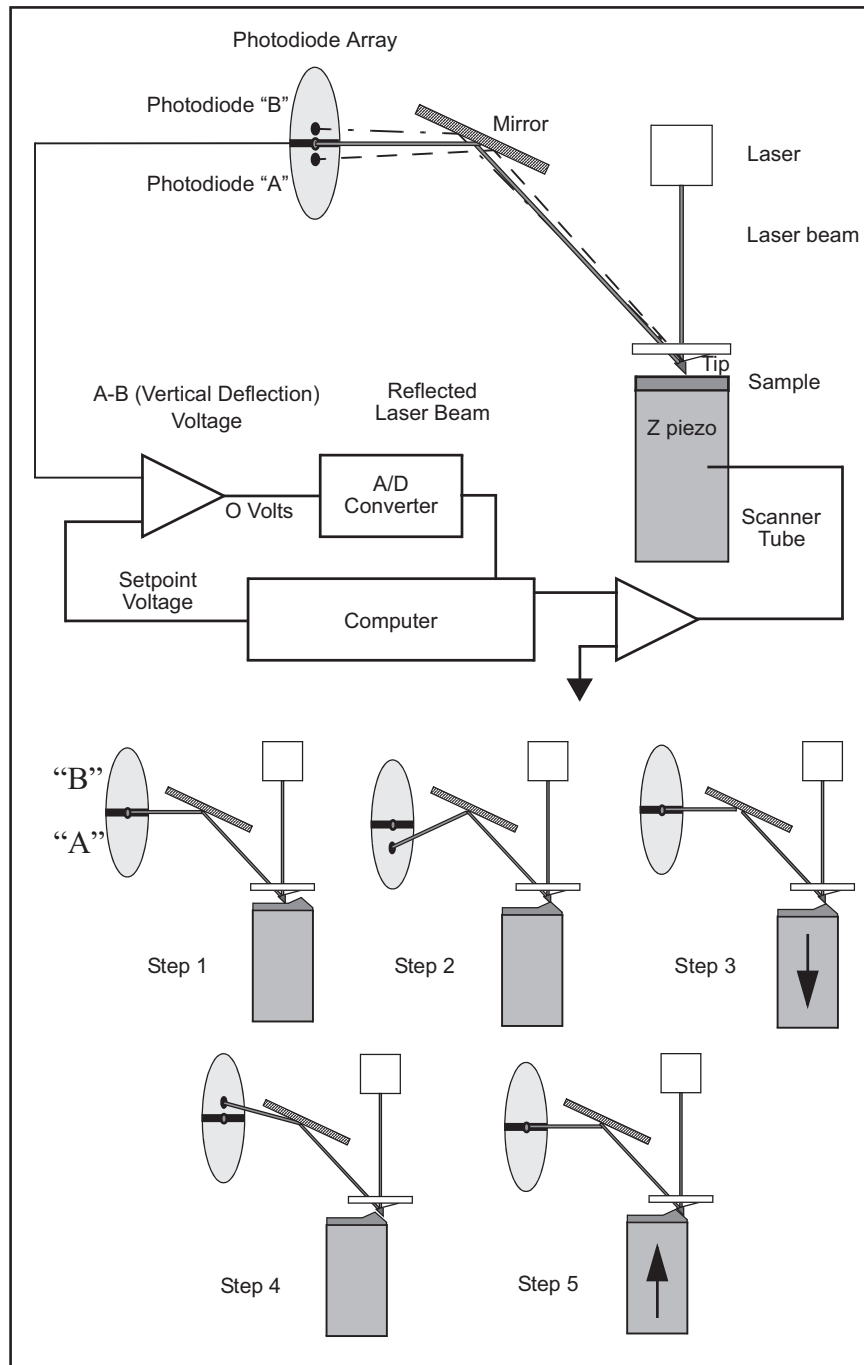
The AFM always first engages in the repulsive region of its operating range. In other words, the cantilever exerts a positive pressure on the sample surface. The AFM block diagram shows the relationship between the cantilever movement and the laser spot on the photodiode array. The diagram shows that the spot moves onto "A" when the cantilever is pushed up. The initial setup is to have the **Vertical Deflection** (A-B) voltage about 2-3V *more negative* than the **Setpoint** voltage.

Bruker recommends starting with the **Setpoint** voltage set to 0V and the **Vertical Deflection** (A-B) set to -2V. The reason for this is that 0V is the middle of the control range. The indication of a good engagement is a distinct jump of about 1V from the **Vertical Deflection** (A-B) voltage to the **Setpoint** voltage.

The displayed height image is an average of the corrections made to Z in a given display period (number of samples menu item). The two gains are set to values to effectively "tune" the feedback response to the particular sample topology. This will set response time of the system so that there is no difference between the SPM's signal and the setpoint reference during scanning.

Proportional Gain—The computer multiplies the value read from the comparison circuit each time the A/D converter is read by this number. It is the high frequency feedback control.

Integral Gain—The average of A/D readings is multiplied by this number. This is the low frequency feedback control.

One of the easiest ways to set the gains properly is to view the input of the feedback loop in the scope window. This means displaying the STM current, the AFM deflection, or the TMAFM amplitude signal. Then raise the gain values until the input of the feedback is minimized. Note that this will result in an image that shows only large transitions in Z; this is normal. There will always be a time lag between the input and the output (height data) of the feedback loop. Usually, the **Integral Gain** is the most sensitive control. Raise the gains together until the input signal (current, deflection or amplitude) is minimum. Don't set them so high as to cause oscillations in the image. Oscillations are an indication of too much feedback correction voltage sent to Z. This is generally known as feedback oscillation. The **Proportional Gain** can usually be set about 20 percent higher then the **Integral Gain**, but it is not required.

4.5   Review of Tapping Mode AFM

4.5.1 General Operating Concepts

One advantage of Tapping Mode AFM is an reduction of frictional forces which exert torque on the cantilever. Unlike traditional contact AFM which maintains a constant cantilever deflection, the Tapping Mode feedback loop keeps the cantilever oscillating at a constant amplitude. The tip on the cantilever is oscillated at a frequency near the cantilever resonance. A laser beam is reflected off a microfabricated cantilever, onto a mirror, then onto a photodiode array. As the cantilever vibrates, the laser spot oscillates across the photodiode array and produces an AC voltage. The signal from the photodiodes is rectified, then lowpass filtered into a DC voltage (**RMS Ampl.**). The RMS amplitude is proportional to the amount of cantilever motion.

The feedback system compares the RMS amplitude to the setpoint voltage. The two voltages are kept equal by controlling the amplitude of cantilever movement. The sample surface is in close proximity to the cantilever such that the tip touches the surface only at the lowest point of its oscillation. The RMS voltage is reduced to the setpoint voltage by the feedback loop moving the sample nearer to the tip. The sample reduces the cantilever movement until the desired RMS voltage is reached. The oscillation amplitude of the cantilever is held constant by the piezo moving the sample in Z as it is simultaneously translated in X and Y.

Engagement in Tapping Mode AFM requires that the setpoint voltage be smaller than the RMS voltage when the probe to sample distance is large (far from engaged). The sample is raised until the RMS reaches the setpoint (see Figure 4.5a and Section 4.5.2 for explanation).

Figure 4.5a TappingMode AFM Concepts
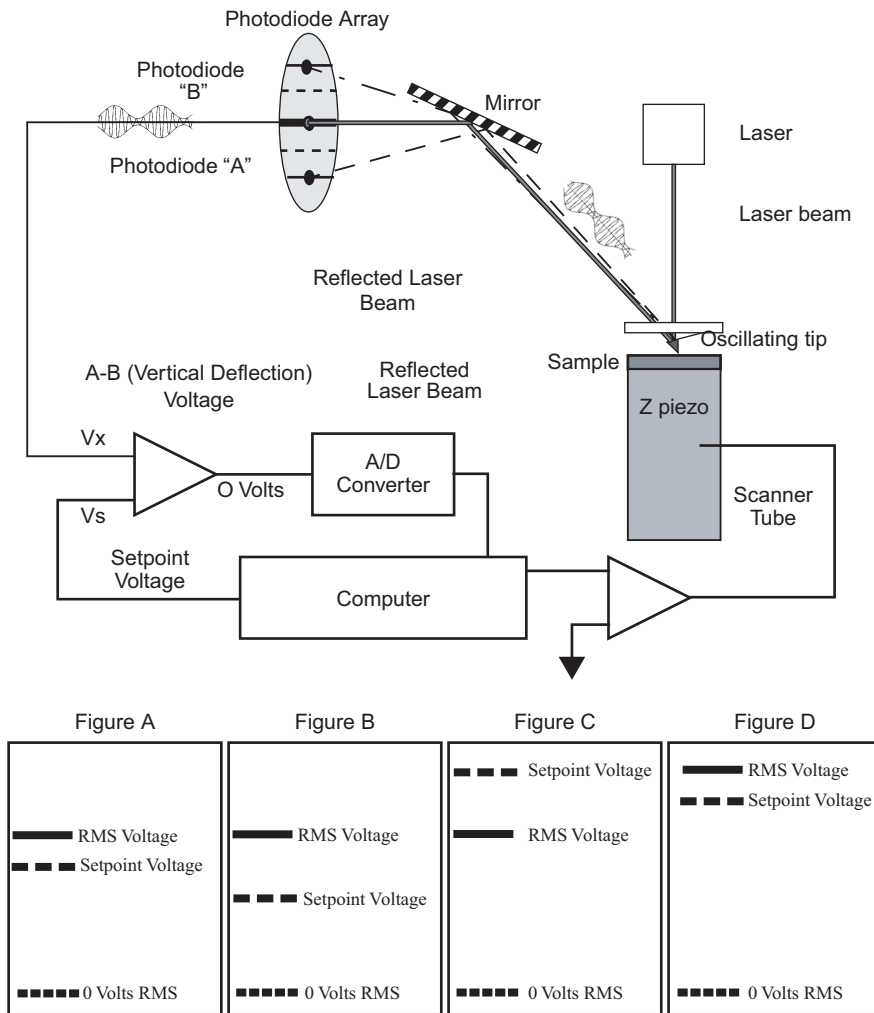
Figure 4.5a shows the relationship between the RMS and the setpoint voltage during the engage cycle. The initial setpoint voltage is determined by the computer rather than the user. The computer sets the setpoint equal to a percent (specified as a multiplier under **Engage Setpoint**) of the RMS amplitude. The sample is raised until the RMS matches the setpoint. The computer then tests for true engagement as follows: 1) the motor halts the sample's rise; 2) the setpoint is decreased slightly; 3) the feedback control monitors movement of the Z piezo. Depending upon the tip's relationship to the sample, one of the two following conditions will result:

- A small piezo movement indicates the cantilever is truly engaged with the sample surface.

- A large Z piezo movement indicates that the cantilever is being damped by air trapped between the cantilever and sample surface (not in contact with the actual, solid surface)—this is a false engagement condition. The setpoint is readjusted and the engage cycle repeated until the computer reads a small change in Z when the setpoint voltage is lowered further. One symptom of a false engagement is the "tip travel μm"

display (at the bottom of the Real Time Status window) stops momentarily, then starts again.

4.5.2 Optimizing the Tapping Mode AFM Signal after Engagement

The figures on the bottom of Figure 4.5a show the relationship between the RMS and the setpoint voltages.

There are some basic rules to remember:

1. The setpoint voltage is always lower than the RMS voltage.

2. The difference between the RMS voltage when the tip is off the surface and the setpoint voltage dictates the amount of damping or "tapping force." The larger the difference, the greater the tapping force.

3. The RMS voltage relates to the amount of kinetic energy in the cantilever (figs. A and D). This is important to note because some samples are stickier than others. If the RMS amplitude is too small, the tip may stick and be held to the sample surface.

The initial setup for tapping mode AFM is to:

4. Tune the cantilever at its resonance.

    a. Select **Cantilever Tune** from the **Microscope** > **Cantilever Tune** pop-down menu, or click on the **Cantilever Tune** icon in the **Workflow Toolbar**. For **Auto Tune Controls**, make sure the **Start Frequency** is at the lower end of the range for the particular cantilever and the **End Frequency** is at the higher end of the range. **Target Amplitude** should be **500 mV**.

    b. Set the **Peak Offset** to **3-10 percent**. The **Peak Offset** is the percentage of the cantilever's free-air resonant frequency to be automatically offset. Peak offset is used to compensate for changes in resonance before engagement due to the tip's interaction with the surface after engagement. *Range and Settings*: 0-50 percent. Positive values offset the **Drive frequency** to lower values (the normal direction of shift during engagement).

    c. Click **Auto Tune**. A "**Tuning**..." sign should appear and then disappear once Auto Tune is done. When done, **EXIT** the **Cantilever Tune** menu.

5. Engage the microscope.

Chapter 5 Setup & Installation

The following sections describe the setup and installation of your MultiMode microscope:

- **Installing the MultiMode SPM:** Section 5.1

- **Component List:** Section 5.2

  - **Unpack The System:** Section 5.2.1

  - **Vibration Isolation:** Section 5.2.2

  - **System Power Up:** Section 5.2.3

5.1   Installing the MultiMode SPM

Set up the computer, main controller and monitor as described in this chapter. Refer to Chapter 3 for facilities requirements and environmental specifications.

5.2   Component List

If this is a first-time installation, inventory all received parts and components against the shipping checklist received with the system.

5.2.1 Unpack The System

The NanoScope system is normally shipped in several boxes.

1. Clear a table for the microscope. The table should be at least 76 x 152cm (30" x 60"). The table should be level, of sturdy construction, and located where it will not be vibrated or jarred by foot traffic. It should be located away from sources of heat and cold; avoid windows, air conditioning and heating ducts, blowers, etc.

2. First remove the computer and controller. Heavy hardware should be removed carefully from each box and may require more than one person to handle the hardware safely.

Figure 5.2a Hardware Setup
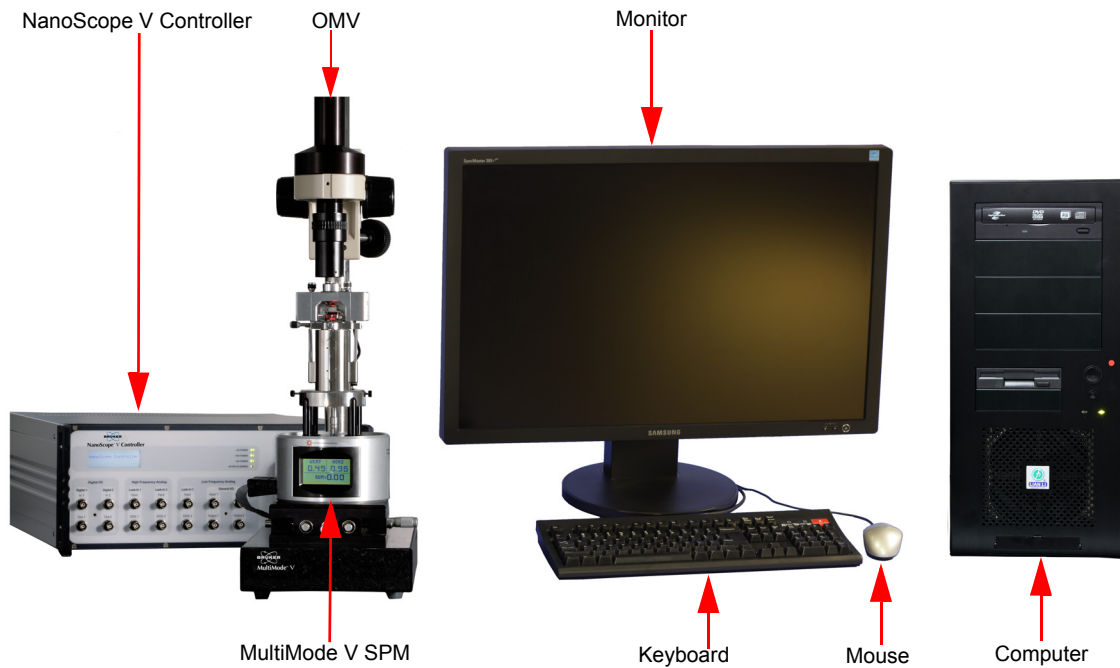
NanoScope V Controller   OMV   Monitor

MultiMode V SPM   Keyboard   Mouse   Computer

⚠	**CAUTION:**	The NanoScope Controller will overheat if the controller ventilation holes are blocked or if the controller is exposed to heat from an outside source.

3. Remove the remaining components and locate them using the layout above as a guide. Vibrational noise can affect scans. All units are supplied with a vibration isolation pad to set the SPM on. The pad is placed under the SPM to reduce vibrational noise. Some users find it advantageous to set the SPM with vibration pad on the floor or on a heavy concrete cinder block. If a tripod vibration isolator or similar mount is used, locate it next to the table.

Cabling

1. Before connecting cables, review the following precautions:

	CAUTION:	DO NOT power anything until all cables are connected and double-checked. Power cords should be connected to their source of power last.
	CAUTION:	Verify the power disconnection device is easily located.

	CAUTION:	DO NOT route cables through pinch-points or over sharp edges. The table should be pulled away from walls so that cables will not be pinched. The cable between the controller and SPM carries up to 440V and must be protected from chaffing.

2. Connect computer cables (monitor, keyboard, mouse and controller) as shown below (see Figure 5.2b).

Figure 5.2b MultiMode V8 Computer rear view
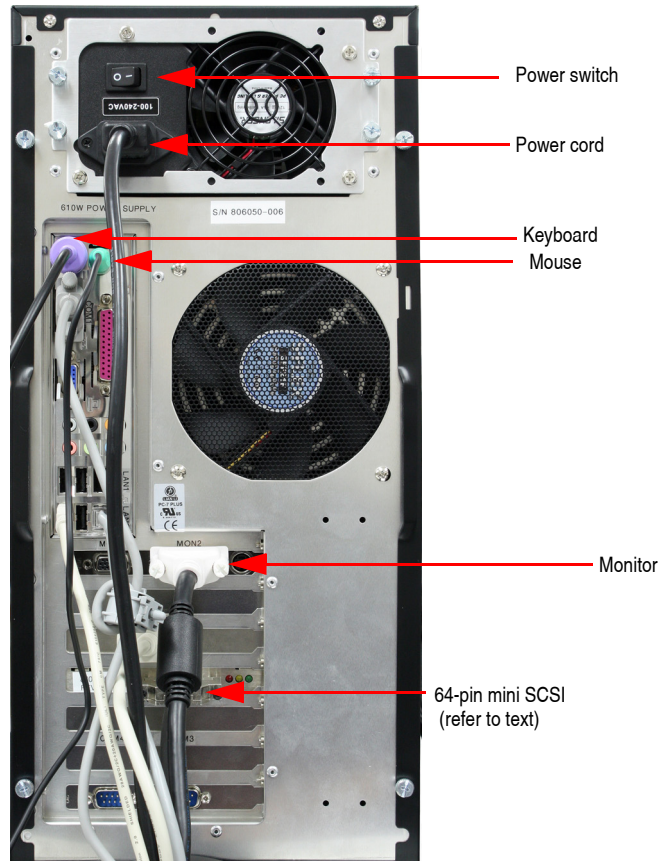
> **Note:** Printers are also user-supplied. Serial ports COM1 and COM2 may be configured by the user for peripheral equipment.

3. Connect cables to the back of the Controller: the power cable should be connected to the controller's receptacle on one side, the controller-to-computer cable on the other. Connect the computer-to-controller cable to the back of the computer using the connector labeled "To NanoScope Controller."

4. The SPM is connected to the front of the controller with the 37-pin ribbon connector through a required cabling adapter. Verify that the ribbon cable is securely connected; otherwise, the microscope may not engage or may exhibit other problems.

5. For vertical scanners, a stabilizing screw will be used to secure the scanner. The stabilizing screw is located near the front of the base. It will be easier to mount the scanner if you first identify the screw and the mounting hole.

Figure 5.2c Pre-position stabilizing screwv

Stabilizing
screw

6. Install the scanner on the support ring with the scanner cap upright.

Figure 5.2d Align scanner drive screw with coupling on base
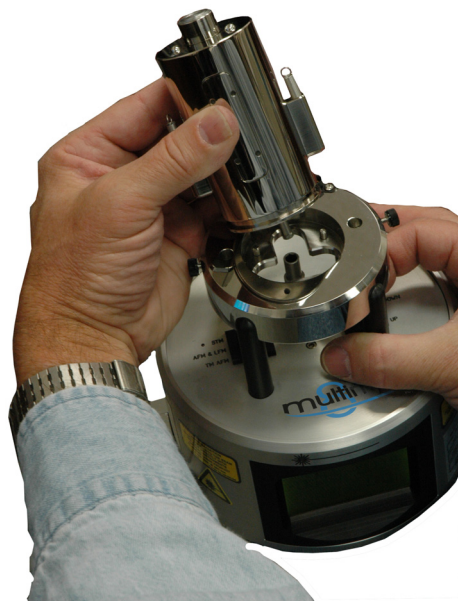
7. If the motorized scanner screw will not seat securely into the coupling on the base, manually rotate the screw slightly until it aligns and seats with the coupling. Alternatively, if the MultiMode has power, press lightly down on the scanner while holding the motor switch on the base in the "up" position until the scanner seats properly. If installing a non-vertical engagement scanner, attach retaining springs to the support ring by inserting into holes and securing with thumbscrews. Secure the scanner to the base by inserting the stabilizing screw (if a vertical engage scanner). Plug the scanner into the receptacle on the support ring as shown below.

Figure 5.2e   Installing the Scanner
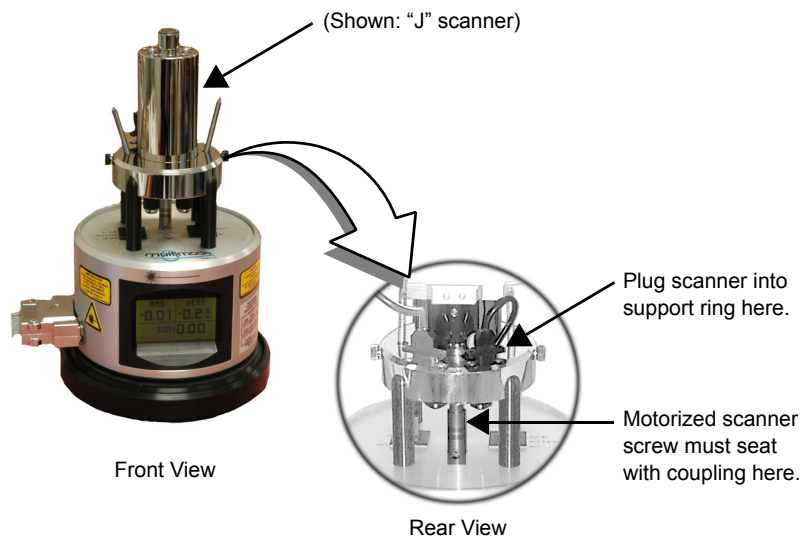
8. Carefully set the MultiMode's head onto the SPM scanner. The head should mate kinematically with the scanner's contact balls. Secure the head using both retaining springs by stretching the springs up and hooking them over the posts located on both sides of the head.

Figure 5.2f   Attach springs to head
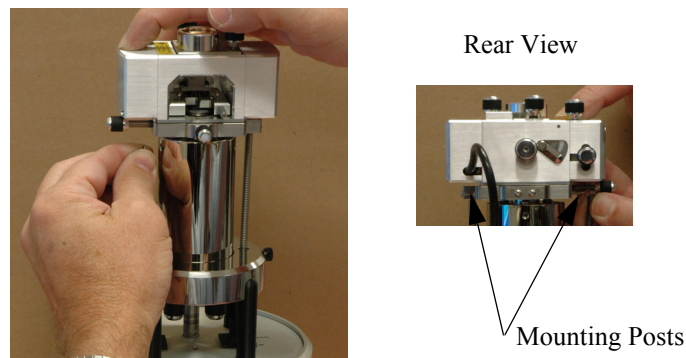
9. Plug the head into the receptacle on the support ring as shown in Figure 5.2g, below.

Figure 5.2g   Installing cable plugs

Cables plugged in
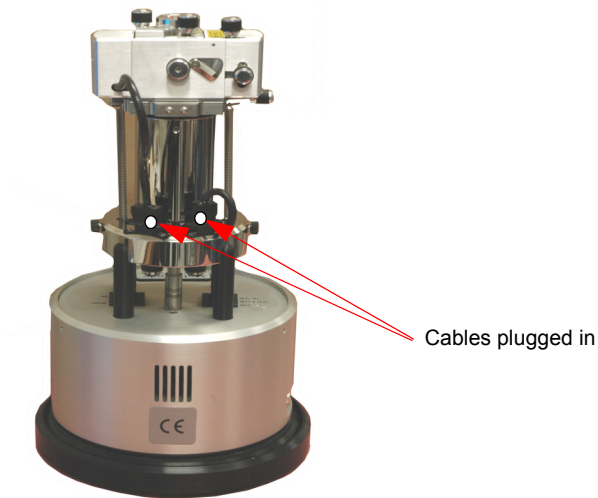
10. Recheck all cable connections. Review the steps above one last time to ensure that all connections are properly made.

5.2.2 Vibration Isolation

The microscope must be isolated from sources of vibration in the acoustic and sub-acoustic frequencies. This requirement can be relaxed somewhat for large-scale images, but atomic-scale imaging is sensitive to ordinary room vibrations.

Adequate vibration isolation can usually be obtained with the vibration isolation pad supplied with the system. In many cases, the pad provides enough vibration isolation to run the microscope on the table, but the pads are especially effective when placed on a massive object isolated from the fan noise/vibration of the computer and controller.

Another effective vibration isolation system consists of a large mass, 20 or more pounds, suspended from elastic bungee cords. The mass should stretch the cords at least one foot, but not so much that the cords reach their elastic limit. The SPM should be placed on the large mass having a natural frequency of about 1 Hz or less both vertically and horizontally. Test this by gently pushing on the mass and measure the rate at which it swings or bounces. A ready-to-use tripod isolation system is available from Bruker (model TRVI). Experience suggests that air tables often have poor horizontal isolation. If an air table is used, it may require additional horizontal isolation for atomic-scale images.

As a final note, the best way to reduce interference from vibrations is to eliminate as many sources of vibration as possible. To reduce vibrations transmitted along cables, eliminate tension in the cables and keep them away from fans and other noise sources. Also, keep the microscope away from sources of acoustic noise. Loud noises (including conversation) can disrupt atomic images, so it is best to isolate the SPM as much as possible. Use the noise reduction hood supplied with the system to alleviate acoustic noise.

5.2.3 System Power Up

Prepare the System for Power-up

1. Verify that all cables are connected properly, especially the NanoScope V Controller to the computer.

2. Verify the following electrical requirements:

   - 600W; single phase

   - 100V, 120V or 240V outlet

   - dedicated circuit, if available

   - 50/60Hz

3. Verify that the power cord is plugged into a grounded power receptacle.

⚠️	**CAUTION:**	Cables should not be removed or installed while power is applied to the system. This restriction does not apply to the small black cable that connects the microscope head to the stage control electronics.

Power-up Sequence

1. Turn on the computer.

2. Turn on the monitor.

3. Turn on the controller.

Chapter 6   Cantilever Preparation

The MultiMode Scanning Probe Microscope is supplied with etched silicon cantilever substrates for TappingMode AFM and silicon nitride cantilevers for Contact AFM modes. The cantilever probes should be inspected under the microscope when used for the first time to gain a better understanding of how the probes and substrates are connected and separated. The procedure for removing individual substrates from the wafer varies depending on the wafer. It is easier to accomplish this task with the aid of a stereo microscope with 50 – 70x magnification.

This chapter addresses the following:

- **Silicon Cantilever Substrates:** Section 6.1

    - **Wafer Tool Kit:** Section 6.1.1

    - **Cantilever Preparation:** Section 6.1.2

    - **Tip Shape of Etched Silicon Probes:** Section 6.1.3

- **Silicon Nitride Cantilever Substrates:** Section 6.2

    - **Tip Shape of Silicon Nitride Probes:** Section 6.2.1

6.1   Silicon Cantilever Substrates

⚠	**CAUTION:** The cantilevers are stored tip-side-up, and the silicon is very brittle. The cantilever will break off of the substrate if contacted.

6.1.1   Wafer Tool Kit

A wafer tool kit for working with silicon cantilever substrates is included with the MultiMode SPM system.

6.1.2   Cantilever Preparation

To prepare the cantilever for imaging:.

1. View the wafer with an optical microscope to determine the orientation of the cantilever substrates and to inspect the cantilevers themselves.

    Note: A 10-70X stereo microscope is useful for this task. The cantilevers are tip-side-up when viewed in the wafer holder.

2. Disconnect the substrate from the bulk of the wafer by pressing down gently on the non-cantilever end of the substrate or using sharp-pointed tweezers to carefully break the two substrate supporting arms connecting the substrate to the silicon wafer frame (see Figure 6.1a).

    Note: The supporting arms connecting the substrate to the bulk of the wafer shatter when pressure is applied. It may be convenient to break several substrates from the wafer at one time. Extras may be safely stored in a specially prepared petri dish.

3. At the bottom of the petri dish, place X4-grade, GEL-PAK adhesive strips.

4. Place the substrates, tips facing up, on the adhesive to permit easy removal of the substrates when needed.

    Note: Cover the petri dish when not in use.

Figure 6.1a Silicon Cantilever Substrates in Wafer

Cantilever   Press Here
To Break
Out Substrate   Substrate
Supporting
Arm   Cantilever
Substrate
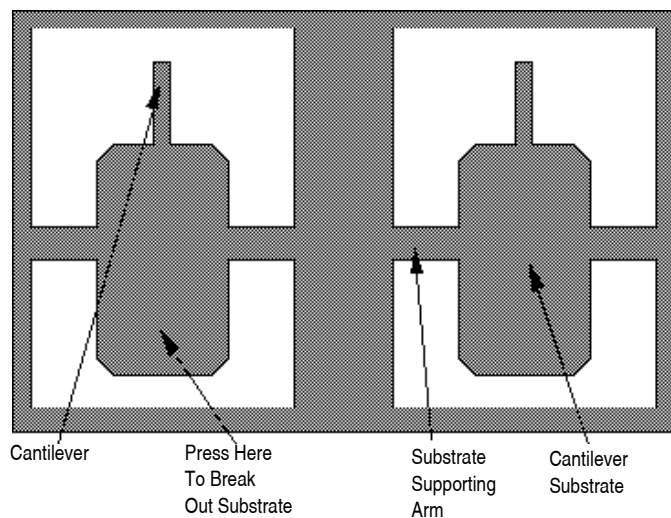
5. Use the curved, sharp-pointed tweezers to remove the cantilever substrate from the wafer container, grasping the sides of the substrate away from the lever and probe tip.

> **Note:** It may be helpful to tip the substrate to one side to help grasp it in the tweezers.

⚠	**CAUTION:** Silicon is extremely brittle. Be very careful to avoid any contact with the probe lever because it will immediately snap off.

6.1.3 Tip Shape of Etched Silicon Probes

Etched silicon probes provide the most consistent tip sharpness of the probes presently available. There are subtleties in general shape that produce different effects from the etched silicon tips when imaging samples with steep walls over steps of 100 nm to several microns in height (see Figure 6.1b).

Figure 6.1b Theoretical Tip Shape of Silicon Probes
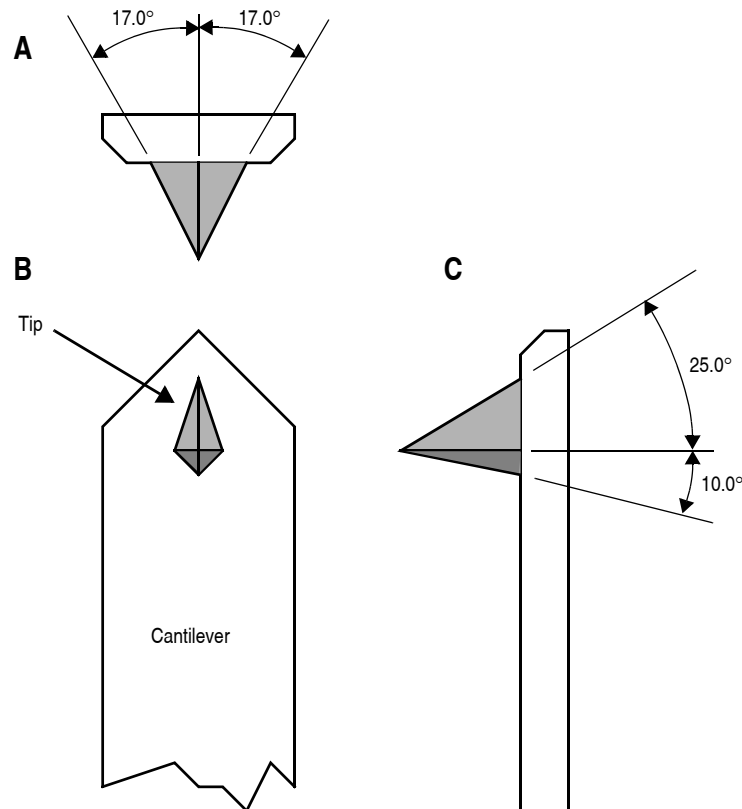
The present process creates a tip which is symmetric from side-to-side with a 17 ± 2 ° half cone angle (see A of Figure 6.1b) and asymmetric from front-to-back, along the length of the lever (see C of Figure 6.1b).

In addition, the substrate mounting angle also affects the interaction of the tip shape with the surface. Along the front edge of the tip, the half angle is nominally 25°, while at the back edge of the tip, the half angle is approximately 10°. Neither of these angles account for the tilt of the substrate. With the mounting angle of the substrate factored in, the front edge of the tip is 35° and the back edge of the tip is zero degrees. From the tip side, the cross-section of the tip near the lever is approximated by an inverted 'kite' shape. All of these subtleties arise from the etching process used to make the tip, which employs caustic solutions to perform wet anisotropic etching of the silicon.

Figure 6.1c Silicon Probe Tip Profile Artifact (Front-to-Back)

Scan line produced using a theoretical
probe tip shape on a 1 - 2 μm deep
vertical wall trench

Scan direction = 0 deg.

10°

55° 80°

Scan Line Profile

1 μm - 2 μm Deep Trench

Note: Any wall angle on the left wall that is > 55 deg.
will be shown as 55 deg. in the image.
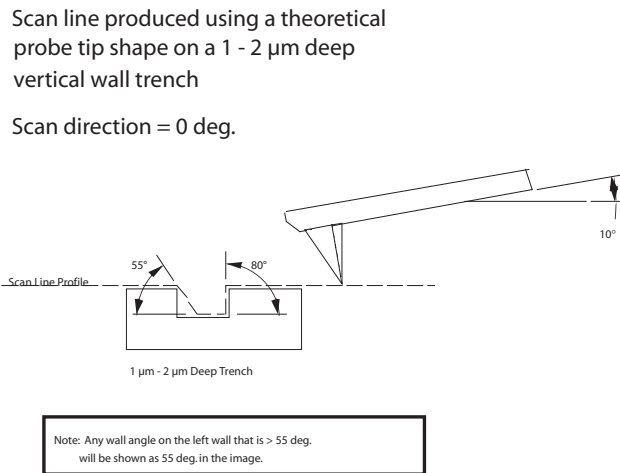
To measure sidewall angles, the best orientation of the sample uses the back edge of the tip (that which faces back towards the cantilever substrate) to measure step angles (see Figure 6.1c). Using the back edge, step angles approaching 90 degrees can be measured routinely, depending on the step height.

> **CAUTION:** Ensure that the area of measurement offers sufficient clearance so that other faces and edges of the tip and lever do not interfere with the measurement.

This method does not work well in small openings of less than 5 microns where, depending on the depth of the step, other tip edges may contact other faces of the small opening. Wall angle measurements are best measured in open areas for these reasons.

Figure 6.1d   Silicon Probe Tip Step Profile Artifact (Side-to-Side)

Scan line produced using theoretical
probe tip shape on a 1 - 2 μm deep
vertical wall trench

Scan direction = 90 deg.

Scan Line Profile

73°   73°

1 - 2 μm Deep Trench
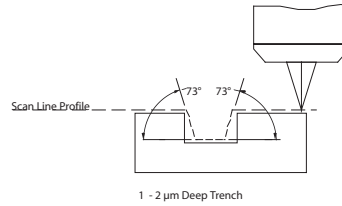
Note: Any wall angle that is > 73 deg.
          will be shown as 73 deg. in the image.

Measurements of line pitch are often best measured using the side-to-side faces of the tip, which exhibits symmetry. Because of the approximate 17° half angle of the tip, the line or space measurement is best done at the top of the line for simplification of the measurement artifacts (see Figure 6.1e).

Figure 6.1e   Common Silicon Probe Profile (Resultant Scan Artifact)

Subsequent scan line produced by using
the realistic probe tip shape

10°

55°   70 - 80°

Scan Line Profile

1 μm - 2 μm Deep Trench
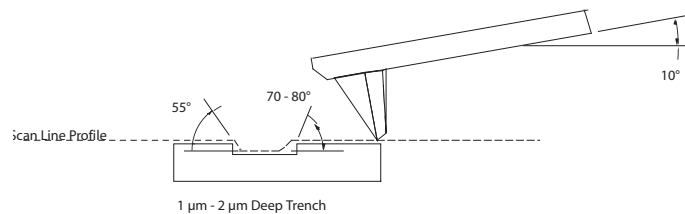
Note: Any wall angle on the left wall that is > 55 deg.
          will be shown as 55 deg. in the image.
          Any wall angle on the right wall that is >70-80 deg.
          will be shown as 70 -> 80 deg. in the image.

Figure 6.1e depicts the resultant effect of the angled back ridge on the step angle measurement for a deeper trench depth. This is tip and topography dependent.

In addition to microscopic scale shape characteristics (see Figure 6.1b - Figure 6.1e), another factor which can affect the wall angle over shorter (nominal 100 nm) step height measurements is the shaped cusp at the end of the tip. The shaped cusp at the end of the tip is formed to increase the sharpness of the tip point to a length of 100 nm from the end of the tip. It is formed in such a manner that the radius of curvature of a silicon tip can be in the range of 5 – 10nm (on a very good tip).

6.2   Silicon Nitride Cantilever Substrates

The following procedure details how to remove Silicon Nitride cantilever substrates.

1. Verify that the wafer is oriented with the tips facing upward (gold coated surface down).

2. Inspect the wafer with an optical microscope.

> **Note:**   A 10 -70X stereo microscope is useful for becoming familiar with the styles and orientation of the cantilevers on the probe substrate. Included with the AFM microscope is a tool kit containing: wafer tweezers, substrate tweezers, regular tweezers, and a pin-vise.

3. Remove the Pyrex strips by resting the silicon ring on a glass slide or ruler.

4. Apply downward pressure with the tweezers until the strip breaks free from the silicon ring (see Figure 6.2a).

Figure 6.2a   Silicon Nitride Cantilevers in a Wafer
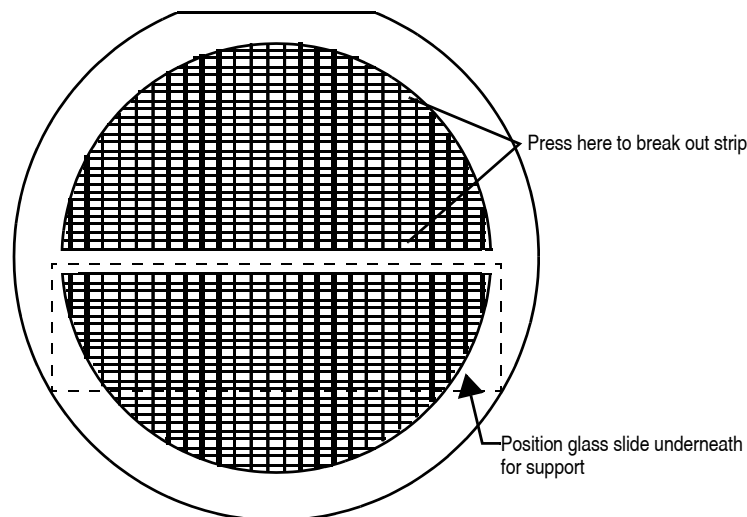
⚠	**CAUTION:**	Be careful to avoid pushing strips together as the cantilevers are between the strips. All cantilevers on one side of both strips could break off if the strips are inadvertently pushed together.

5. Place the strip down on a white piece of paper to inspect it under the microscope.

⚠	**CAUTION:**	Verify that the cantilevers are on the top side of the strip. The cantilevers are on the same side as the reinforcing ring, while the saw-cuts are on the opposite side from the cantilevers. Between each one on the down side of the strip there should be a saw-cut almost through the Pyrex.

6. Handling the strip by its ends, place it on a glass slide (taped down to the edge of a table) with the substrate or spacer piece to be removed hanging over the edge (see Figure 6.2b). The saw cut should be approximately on the edge of the slide.

7. While holding down the next substrate on the strip with the wooden end of a cotton swab, grip the overhanging piece with a pair of wide tweezers and rotate downward until the cantilever substrate breaks off.

8. Repeat this process until as many cantilever substrates as required are removed.

Figure 6.2b Substrate Break-off
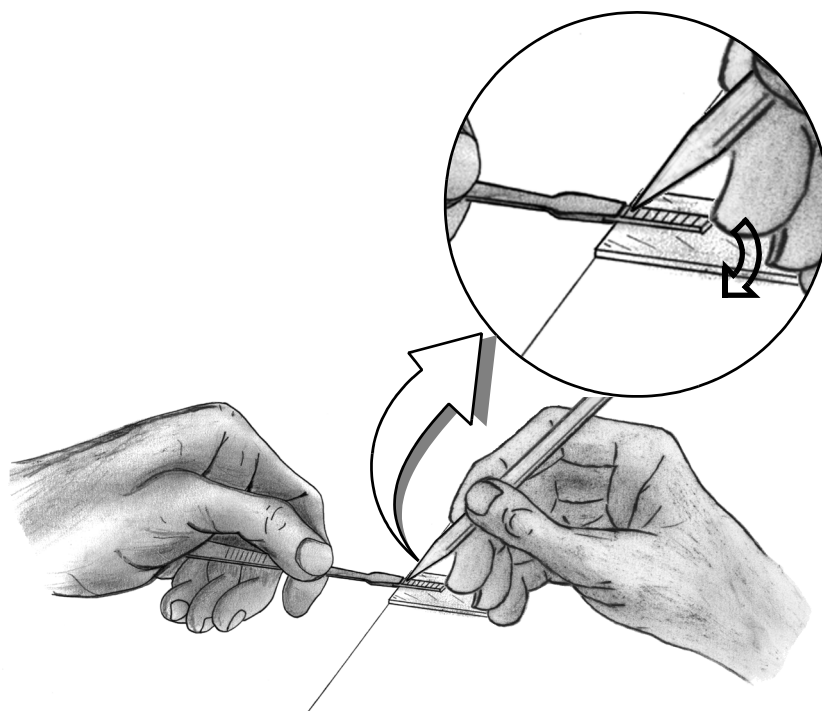
> **Note:** Extra substrates are easily stored in a covered petri dish. The shipped substrates are secured with X0-grade, GEL-PAK™ adhesive strips. The strips are used to permit easy removal of the substrates. If GEL-PAK adhesive strips cannot be found, a simple substitute is the adhesive area from a Post-it note.

Each substrate has two cantilevers on each end of the substrate. Both 100 and 200µm length cantilevers with two different leg widths are provided. When ready to use a cantilever substrate, it may be desirable to remove the unused cantilevers from that substrate, but it is not necessary. For most applications use the 200µm cantilever with the wider legs. For atomic scale images, the 100µm triangular cantilever with the wider legs yields good results.

6.2.1 Tip Shape of Silicon Nitride Probes

Silicon nitride probes provide low cost and durable probes suitable for contact mode imaging. There are some subtleties in general shape that should be understood to gain the best advantage from the silicon nitride tips when imaging samples with steps of 0.1 to several microns in height. The probe tip is approximated by a pyramid formed by intersecting <111> planes in silicon. The approximate shape of the tip is shown in Figure 6.2c along with dimensions and approximate values for spring constants and resonant frequencies.

Figure 6.2c   Silicon Nitride Cantilevers

Lever Type	A	B	C	D	Spring constant (N/m)	Measured Fr (kHz)
100 μm Wide	115	122	21	60	0.58**	40
200 μm Wide	193	205	36	113	0.12**	12.3-22.1 †
100 μm Narrow	115	122	15	69	0.38**	-
200 μm Narrow	193	205	20	150	0.06**	-
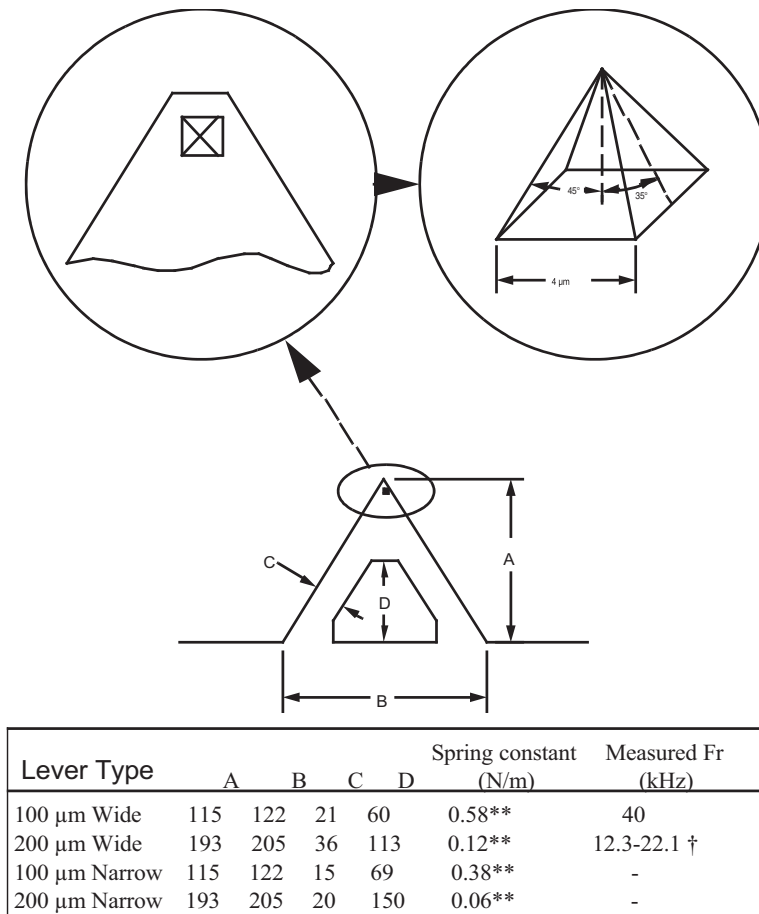
Because the Silicon Nitride probe tips have lower aspect ratios than single-crystal etched silicon probes, the steepest measurable step wall angle is appreciably lower. The highest measurable angle using silicon nitride probes is approximately 65° (see Figure 6.2d) using the inner face of the tip (towards the cantilever holder). The steepest measurable angle from side to side (parallel to the edge of the probe's substrate) is approximately 55°. Both of these figures assume that the measurement does not have interference from other edges.

Figure 6.2d   Silicon Nitride Cantilevers (profile)
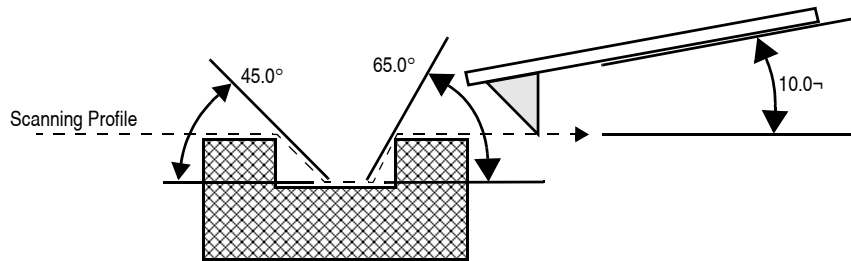
There are two types of silicon nitride cantilever probes available: standard and oxide-sharpened tip processes. The standard devices have the nitride deposited directly into the etched silicon mold pit formed by the intersecting <111> planes, and have points that are slightly rounded with respect to the tips produced using the oxidation sharpening process.

The oxide-sharpened silicon nitride probes have a thermally grown silicon dioxide film deposited in the mold pit used to shape the nitride tip, prior to silicon nitride film deposition. The oxide has two effects: it shapes the inner contours of the pyramidal pit so that a slight cusp forms at the point of the pyramid, and the oxide protects the tip from excessive exposure to a long duration wet silicon etch used to free the cantilevers from the silicon substrate. The result is a noticeably sharper point at the end of the pyramid. Regrettably, along with the increased sharpness of the tip comes a slight increase in double tip effect experienced with the oxide sharpened process.

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:

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
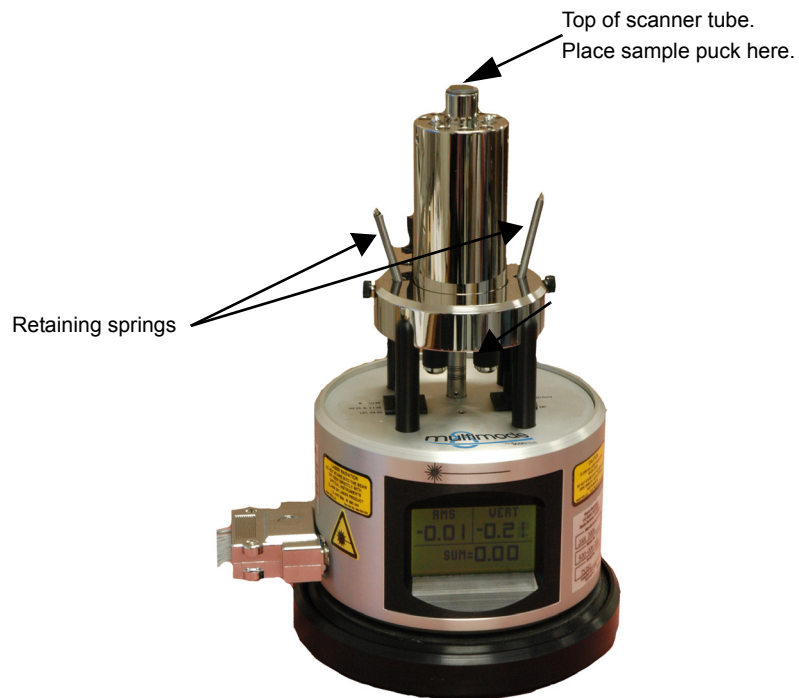
Reinstall the Head

- 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

Reattach retaining springs (2)
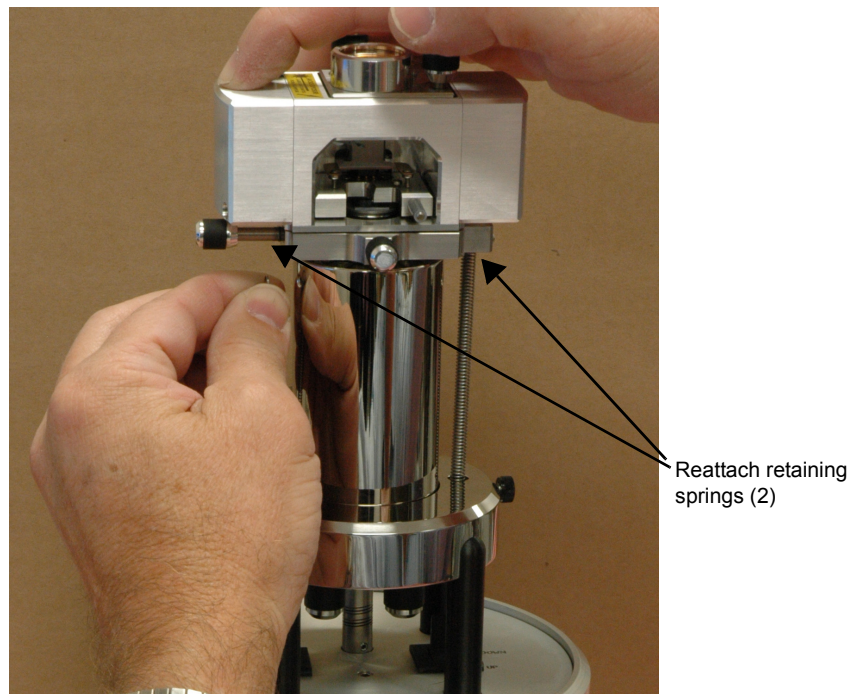
Check Head for Free Vertical Movement

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
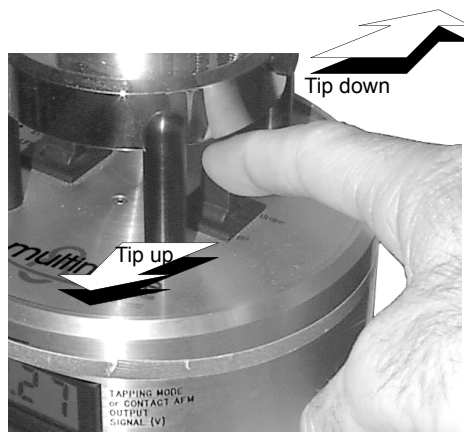
7.1.3 Load Probe in Probe holder

- **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

Handle probes with tweezers.

Apply gentle downward
pressure to lift spring clip.

Spring clip

(Underside of AFM probe holder)

Locate probe flush against
inside edges of groove.
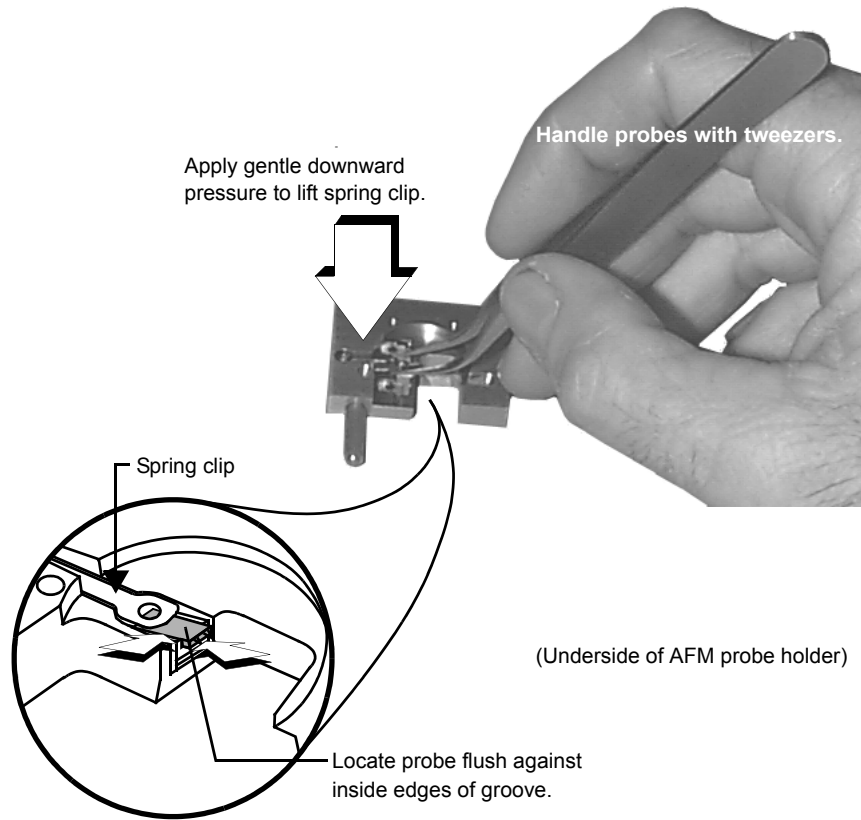
Figure 7.1f   Underside Detail of Fluid Cell

Probe

Wire clip

Lift wire clip by pressing
plunger on opposite side
of probe holder. Insert probe
with tweezers, then release
clip.
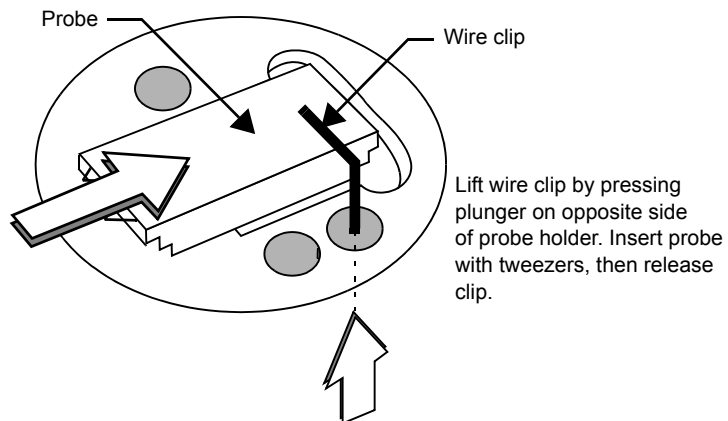
Load Probe in Probe holder

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

Rotate clamping screw CW to secure Probe Holder

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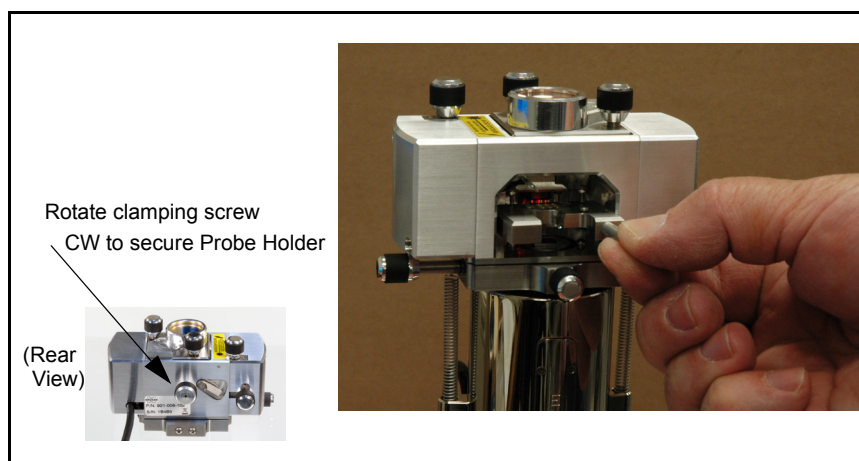
After the probe holder is loaded with a probe, the probe holder will be placed inside the SPM head and clamped into position using the clamping screw at rear of head.

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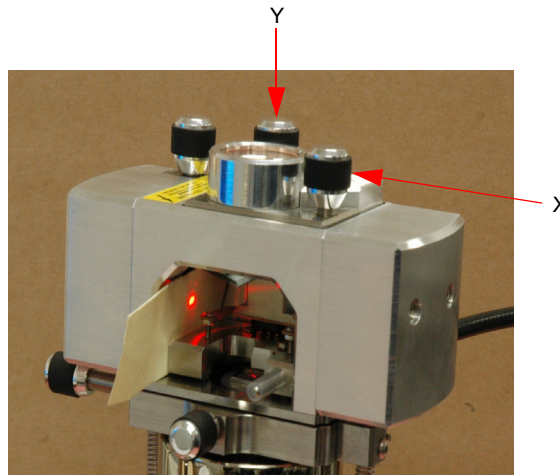
7. Reposition the laser with the screws on top of the optical head, if necessary.

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
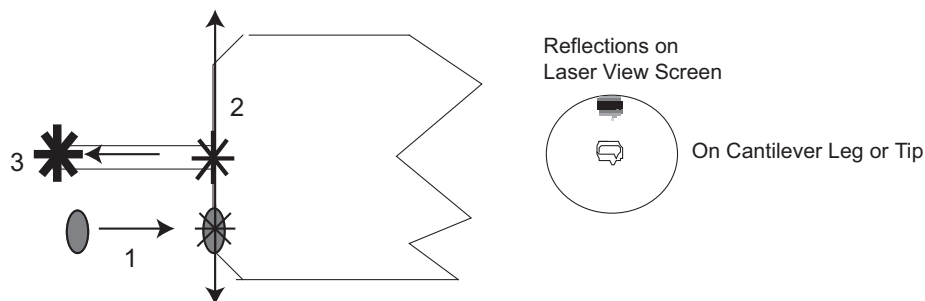
4. Turn the back-left laser control knob clockwise or counter-clockwise to move the laser in the Y direction (parallel to the substrate edge and perpendicular to the cantilever) until the beam crosses the cantilever and a shadow appears over the laser spot on the surface below. Turning the back-left 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
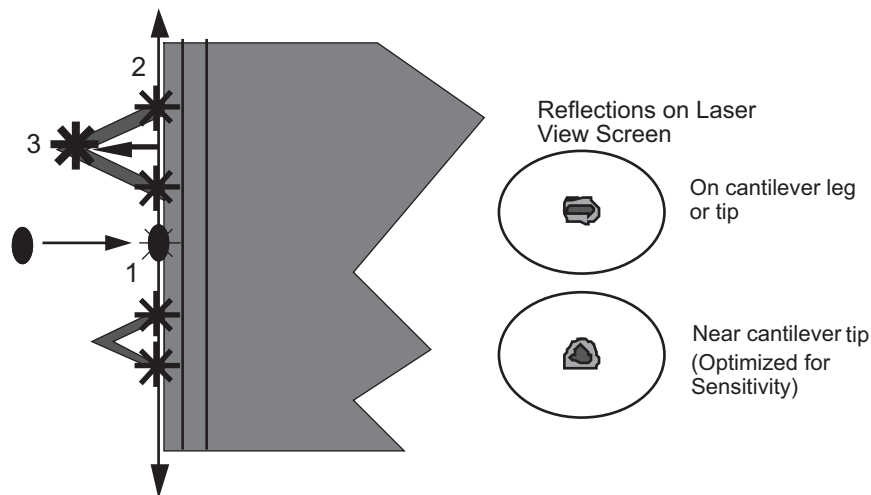
4. With the laser positioned at the edge of the substrate, use the back-left laser control knob to move in the Y direction (parallel to the edge of the substrate) until the laser is positioned at the base of a desired cantilever. Ensure that the cantilever selected is appropriate for the analysis which 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

(Front-Top View)

(Rear View)
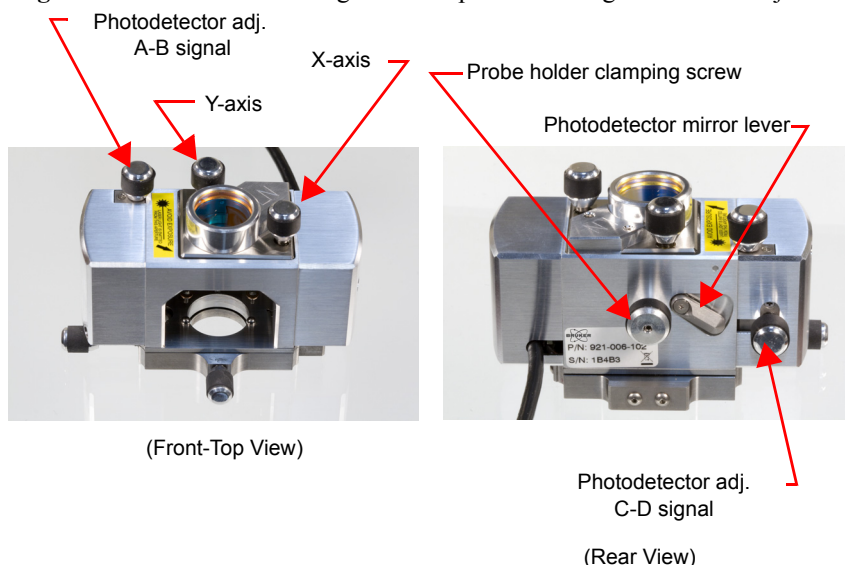
This adjustment is much less sensitive than the laser position adjustments. The maximized sum signal should be approximately **4.0 - 9.0V** for silicon nitride cantilevers. The value of this signal varies with many factors. It is important to note that it is possible to see a large response on the bar 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

Menu Bar
Toolbar
Workflow Toolbar
Scan Parameters List
Video Window
Status Bar
Real-time Status Window
Image Thumbnails
Image Windows
Scope Windows
Client Window
Browse Window
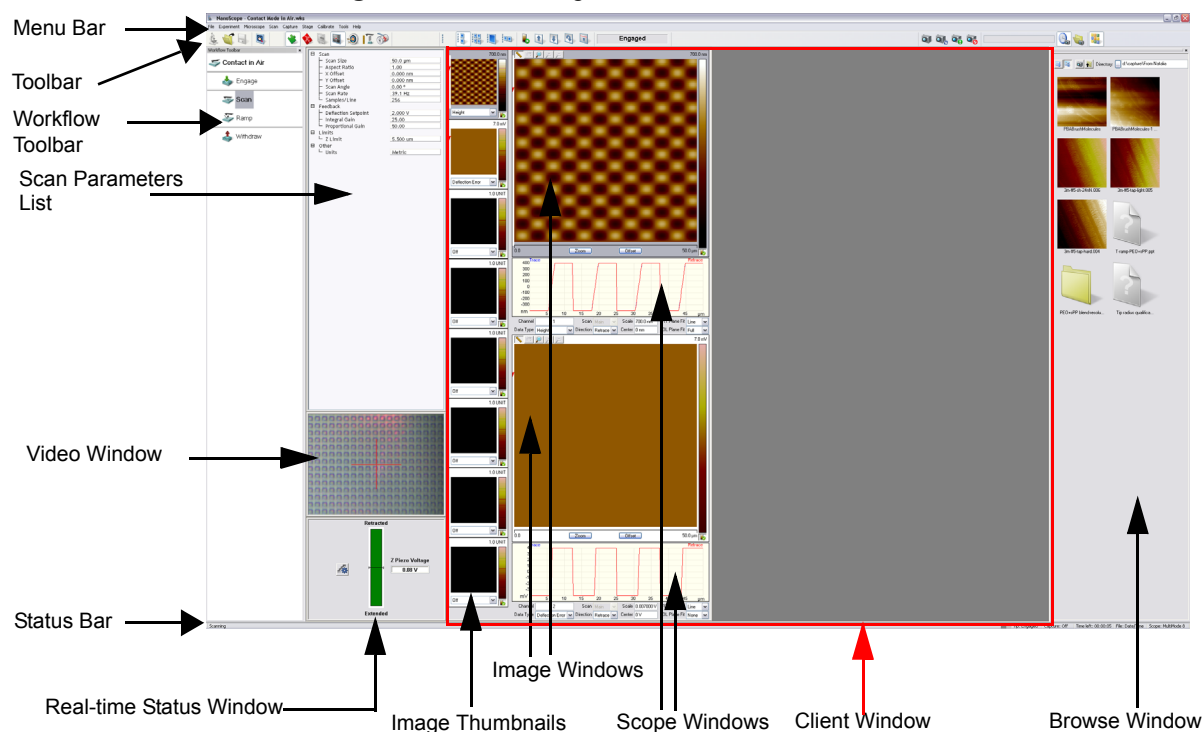
3. Set the microscope configuration by selecting **Tools > SELECT MICROSCOPE...** from the menu bar. See Figure 7.3b.

Figure 7.3b SELECT MICROSCOPE from the Tools menu
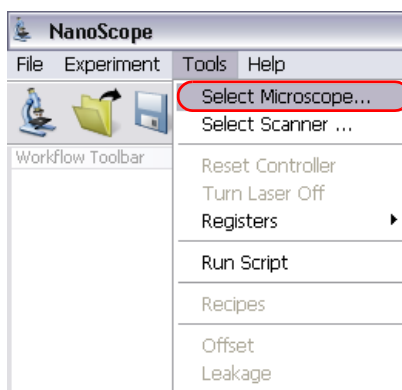
4. This opens the **Microscope Select** window, shown in Figure 7.3c.

Figure 7.3c   Microscope Select Dialog Box
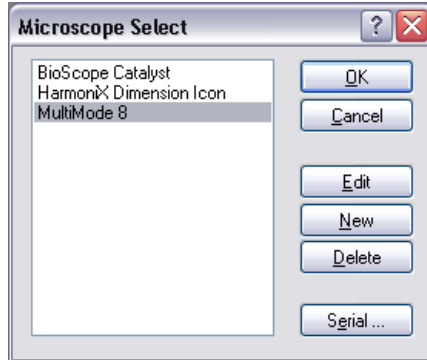
5. Click **NEW** if this is a new microscope, **EDIT** if you wish to modify a configured microscope, **DELETE** to remove a microscope configuration or **CANCEL** to exit.

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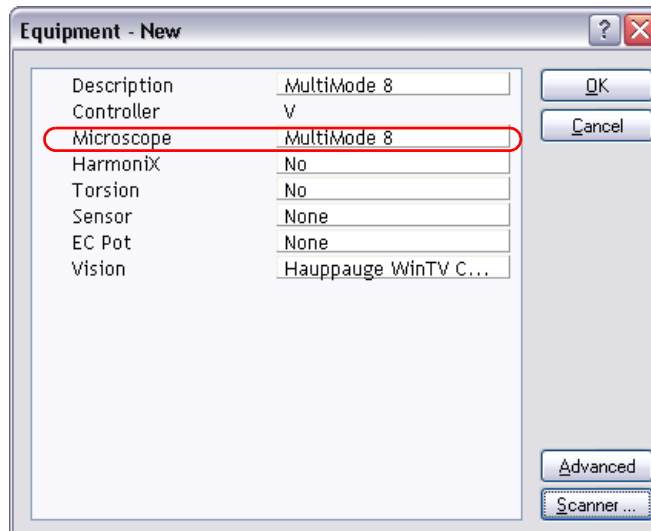
7. Select **MULTIMODE V** from the drop down **Microscope** menu. This step is not necessary if the MultiMode has not been reconfigured since the last use.

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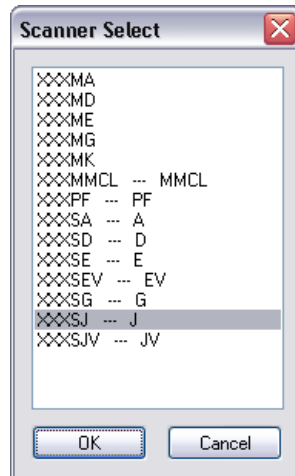
10. Select the SCANNER and click OK. This returns you to the **Microscope Select** window, shown in Figure 7.3c.

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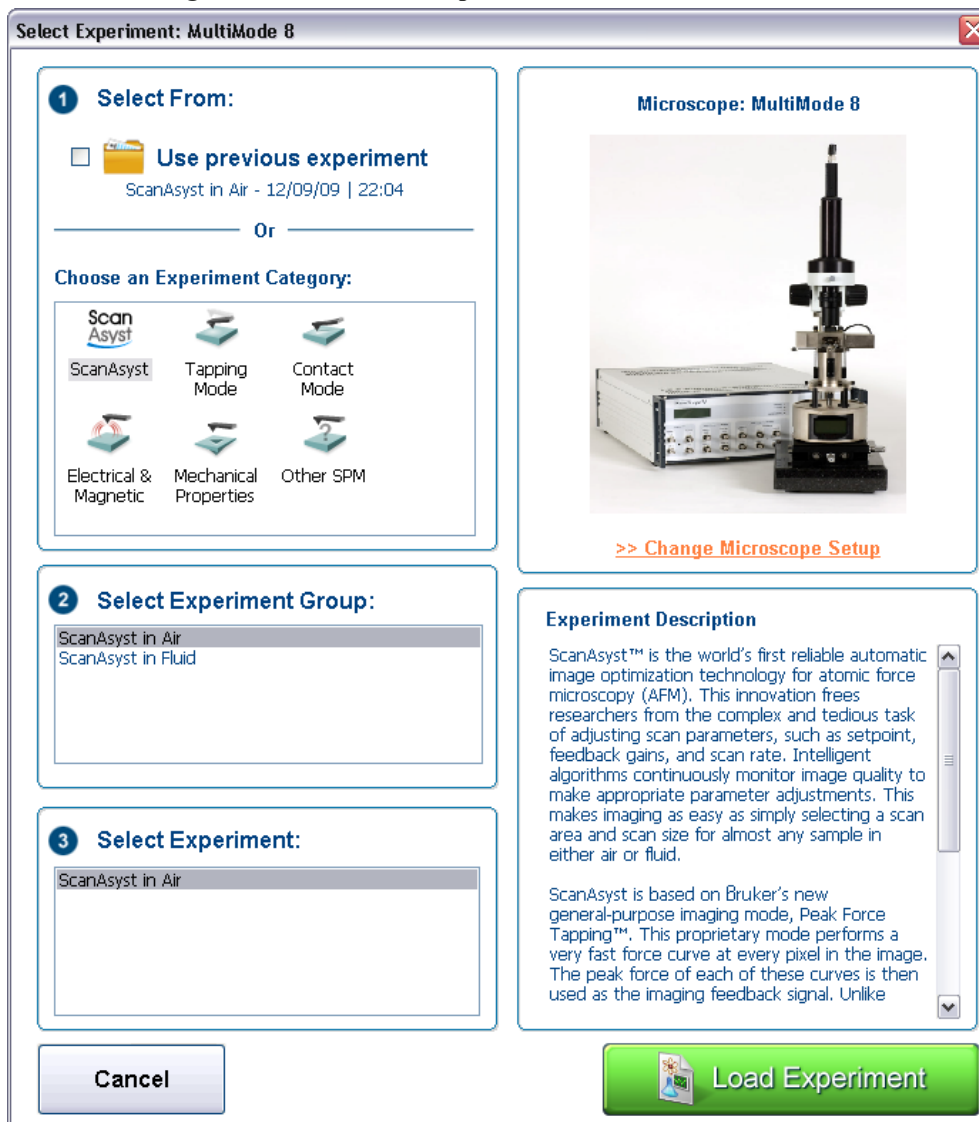
2. Select the **Experiment Category** and **Experiment**.

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
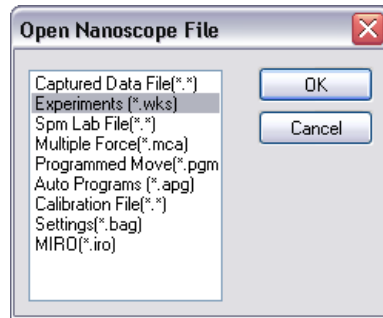
7.4   MultiMode SPM Voltage Meters

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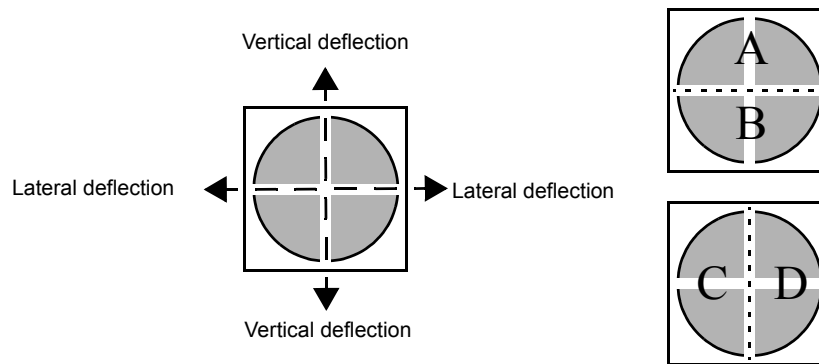
- The meters will display differently depending on which operating mode (see Figure 7.4b) has been selected using the switch on the base. In addition, an LED indicator light will change color to red in contact mode and green in tapping mode.

Figure 7.4b   Mode selector switch

Red = Contact
Green = Tapping

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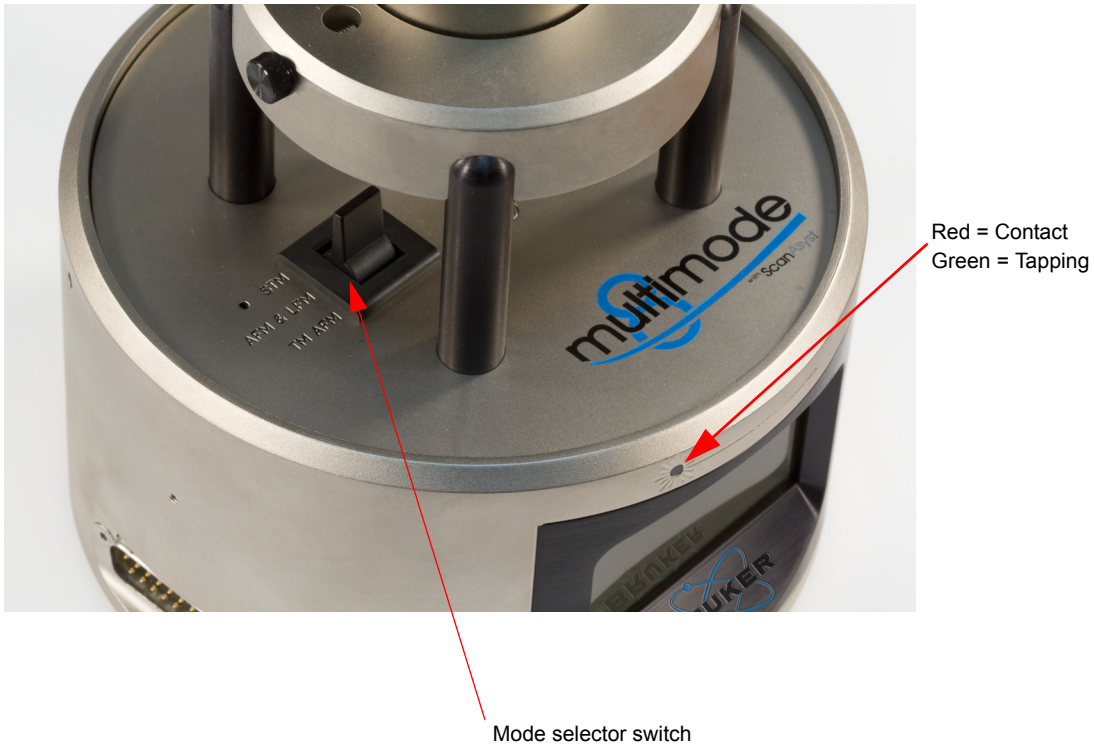
- In AFM & LFM (Contact) mode, the display will show values for VERT, HORZ and SUM. See Figure 7.4c.

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.
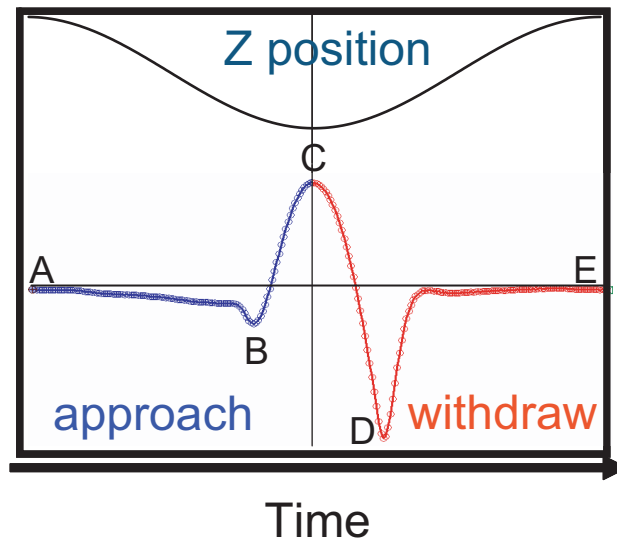
8.2.2 Force curves

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

Z position
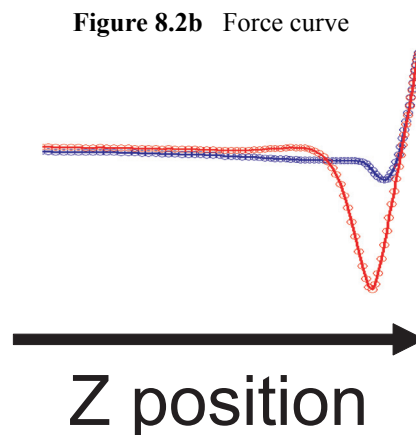
8.3 Probe Selection

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

Red = Contact
Green = Tapping

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

Photodetector Vertical Adjustment Knob
(A - B)

Tilt Mirror Lever

Photodetector Horizontal Adjustment Knob
(C-D)
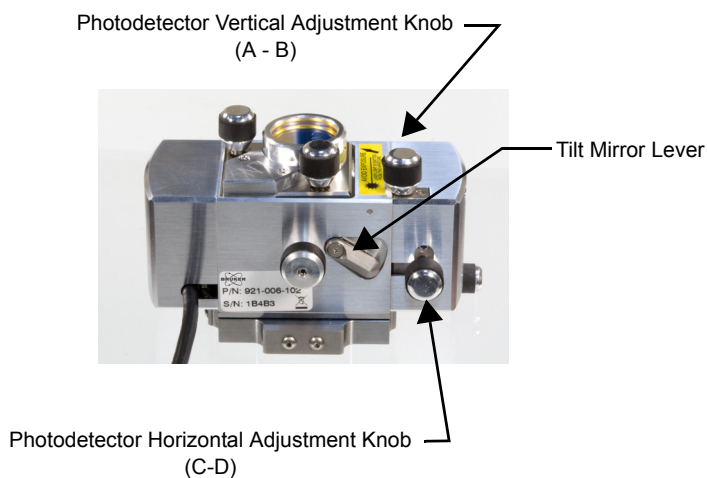
Refer to Figure 8.4c. For laser aligning screws atop the SPM head, the right-front screw moves the laser spot left-to-right (horizontally or along the X-axis). Turning this screw clockwise moves the laser spot to the right. The left-rear laser aligning screw moves the laser spot top-to-bottom (vertically 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
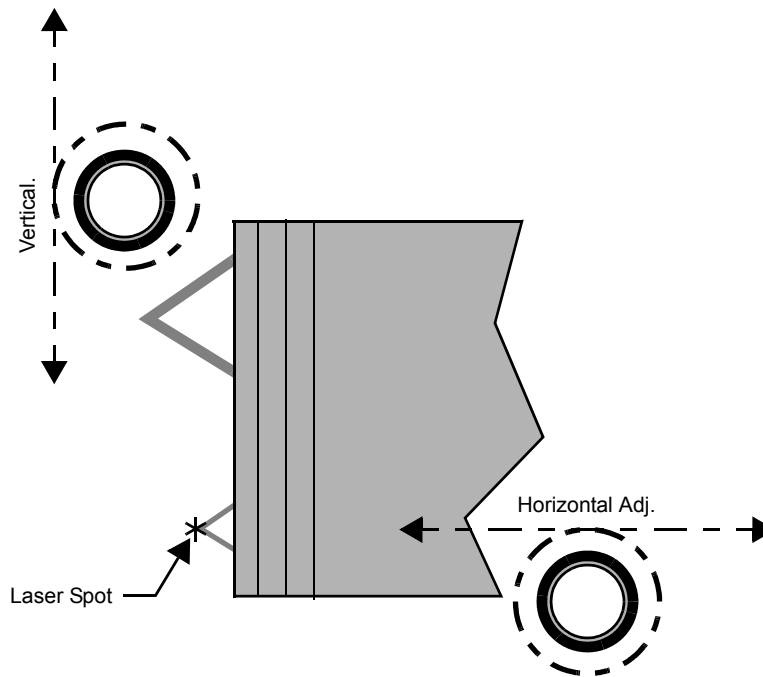
8.4.3 Signal Settings

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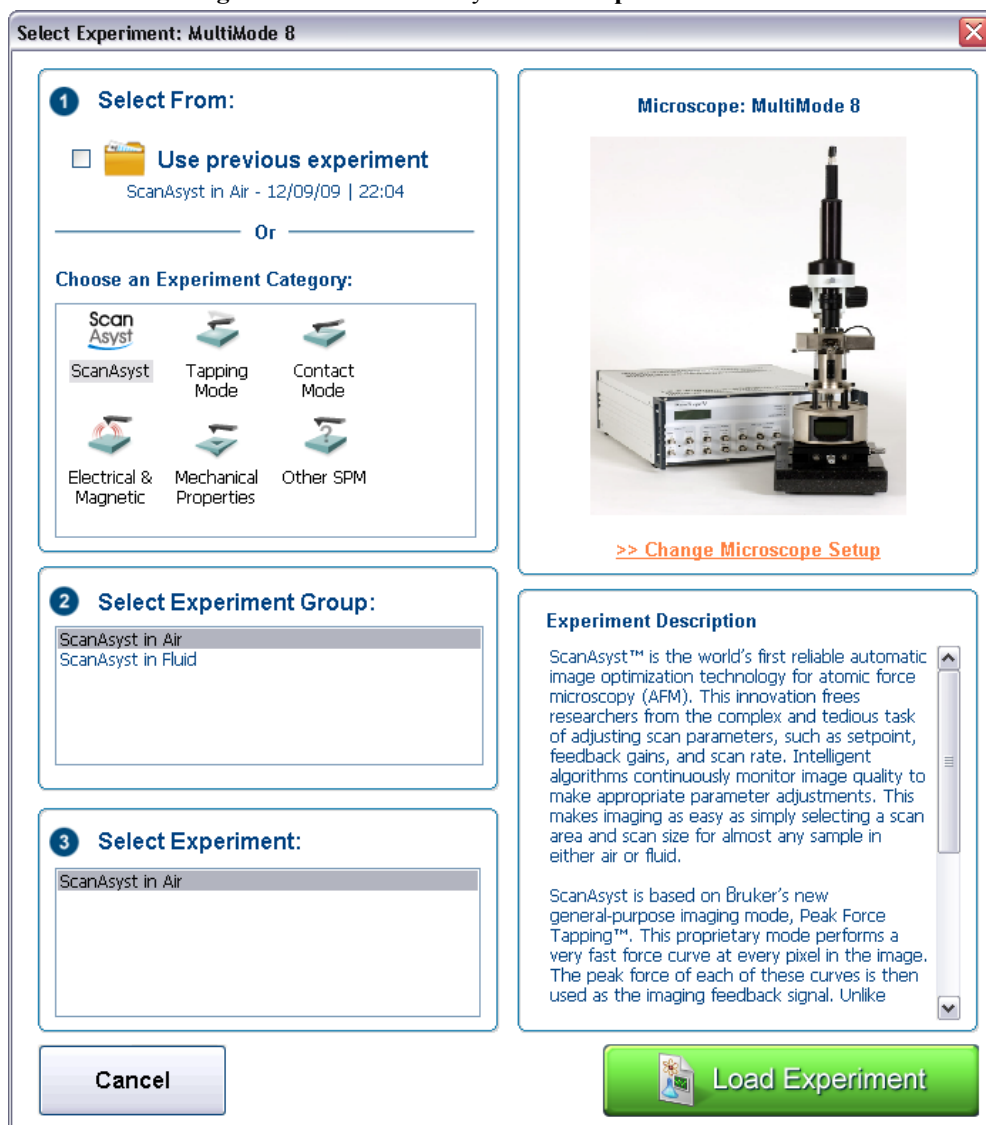
2. Select **SCANASYST** in the **Choose an Experiment Catagory** panel.

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
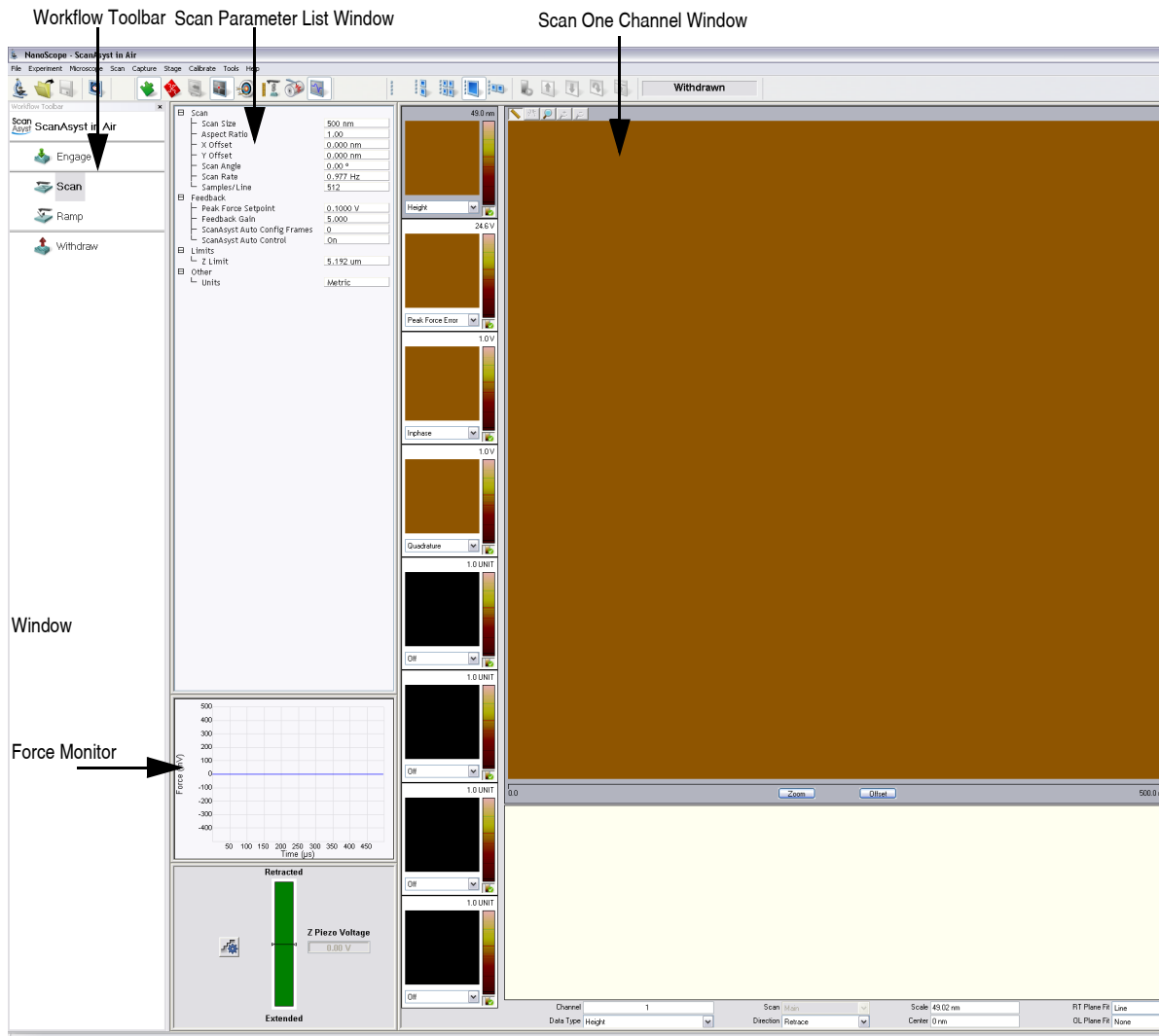
8.4.7 Position Tip with OMV

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

Channel	1	Scan	Main	Scale	50.00 nm	RT Plane Fit	Line
Data Type	Height Sensor	Direction	Retrace	Center	0 nm	OL Plane Fit	None
Channel	2	Scan	Main	Scale	24.58 V	RT Plane Fit	Offset
Data Type	Peak Force Error	Direction	Retrace	Center	0 V	OL Plane Fit	None
Channel	3	Scan	Main	Scale	1.000 V	RT Plane Fit	Line
Data Type	Inphase	Direction	Retrace	Center	0 V	OL Plane Fit	None
Channel	4	Scan	Main	Scale	1.000 V	RT Plane Fit	Line
Data Type	Quadrature	Direction	Retrace	Center	0 V	OL Plane Fit	None

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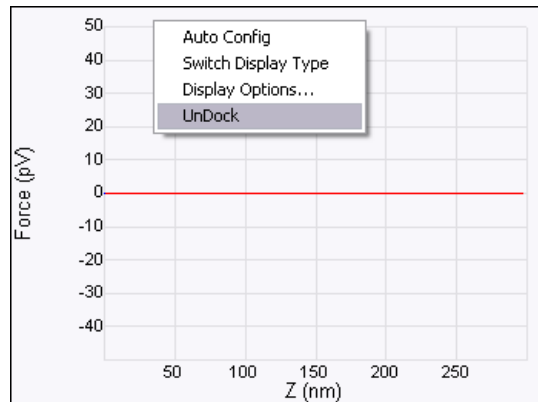
2. Select one plot to be **FORCE VS. TIME** and the other to be **FORCE VS. Z**.

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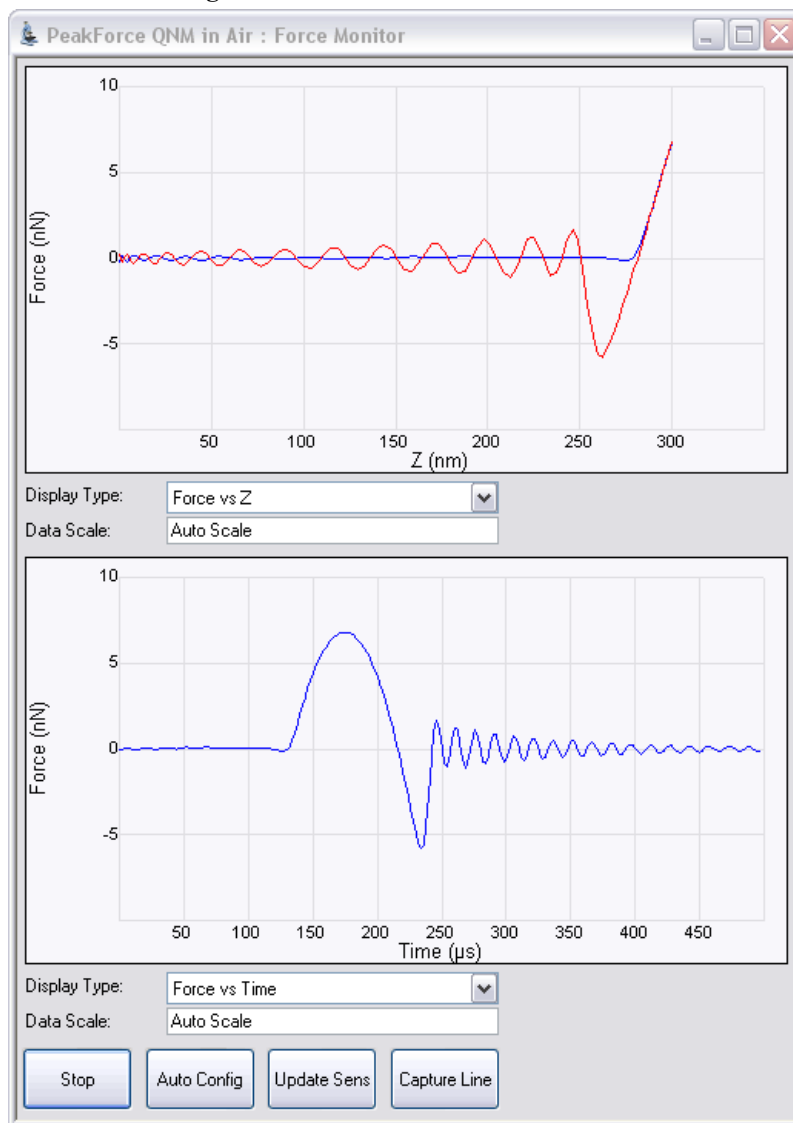
4. The **HEIGHT** channel in the **Scan** window, shown in Figure 8.4j, will display a topographical image of your sample.

Figure 8.4j   Height Image of a PS + LPDE blend.
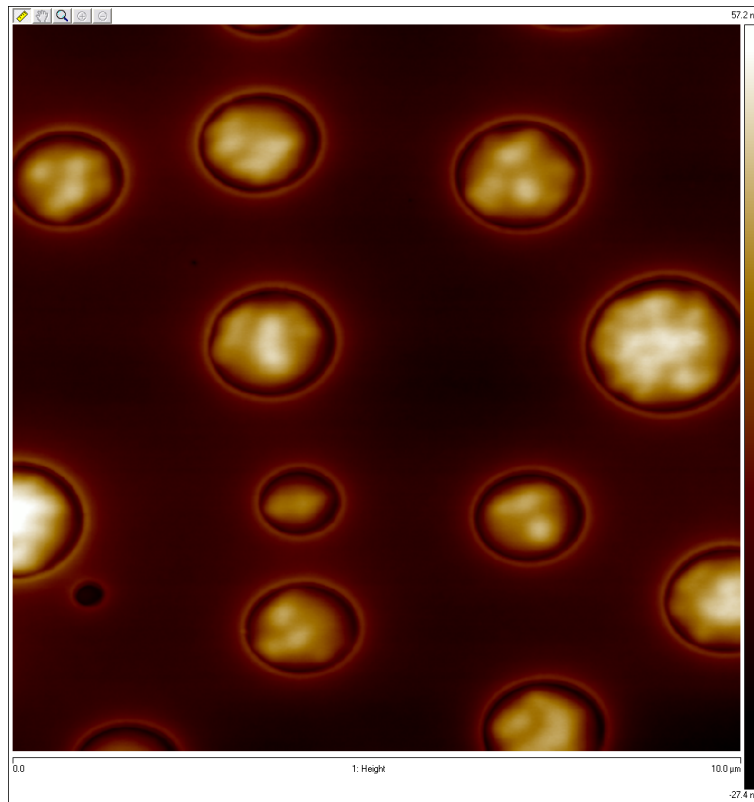
8.5   ScanAsyst and Peak Force Tapping Mode Parameters

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
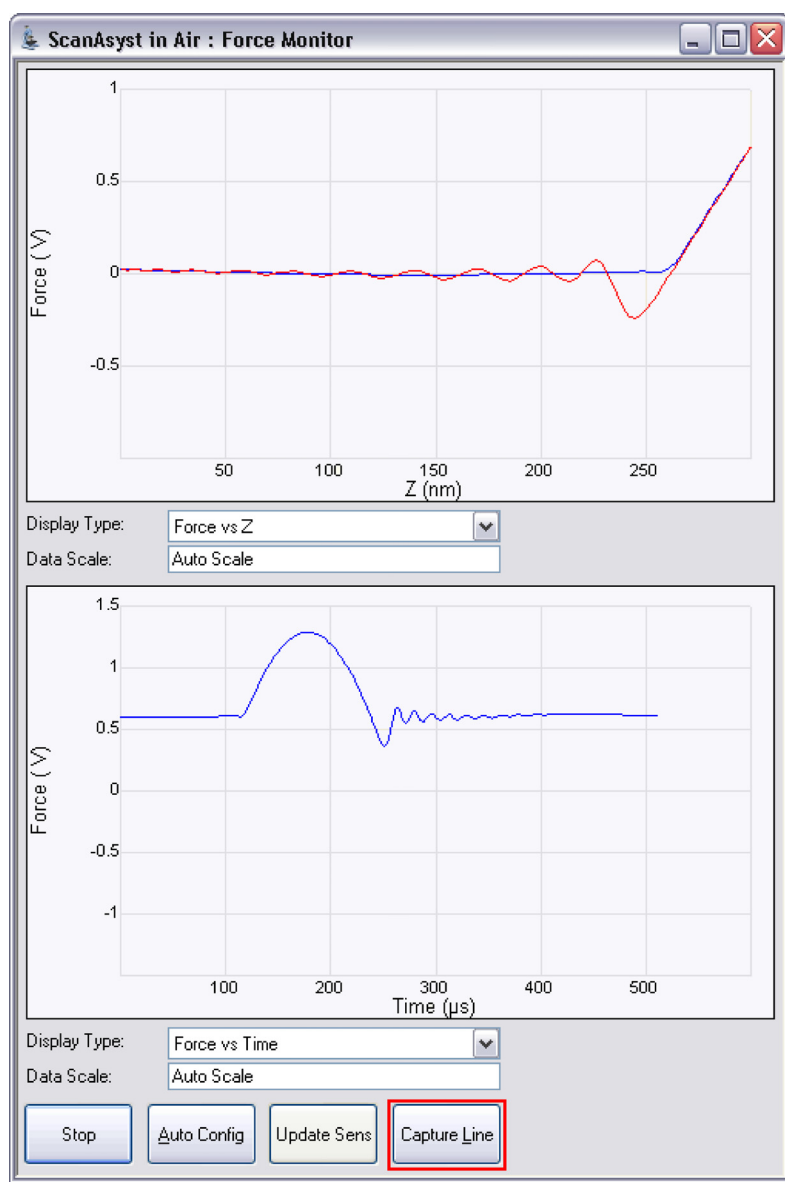
3. The **High Speed Data Capture** window, shown in Figure 8.6b, will open and the **Status** will change when the data has been captured. **UPLOAD DATA** to the PC when the capture is complete. When **CAPTURE LINE** is used this way, the off-line NanoScope Analysis software will correctly associate 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.
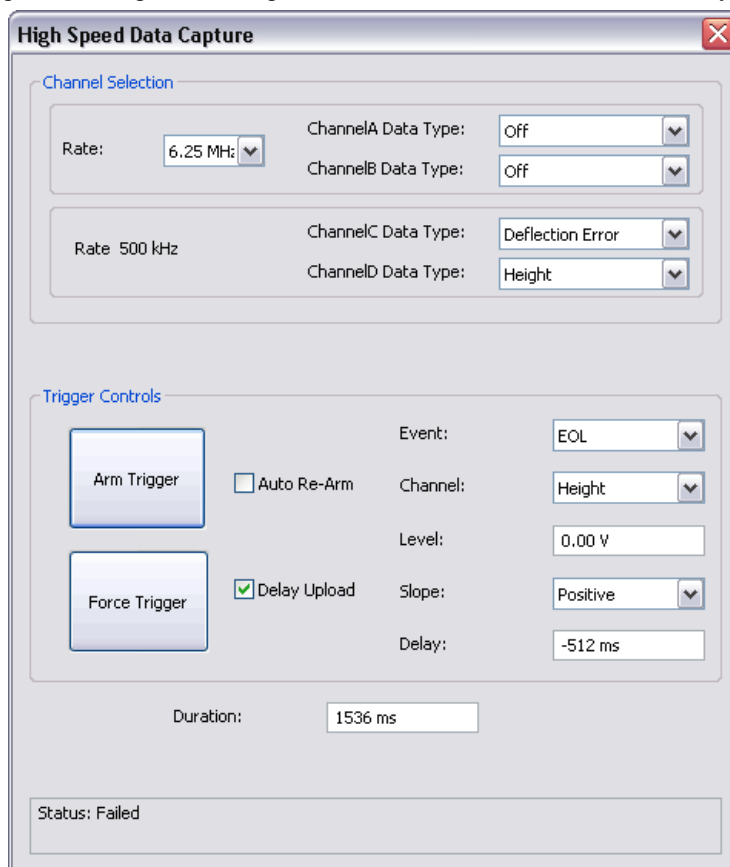
4. Click the **UPLOAD DATA** button to transfer the captured data to the computer. While the data transfer process takes place, the scan data will look corrupted because the DSP time is shared between PeakForce QNM properties computation and data transfer.

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
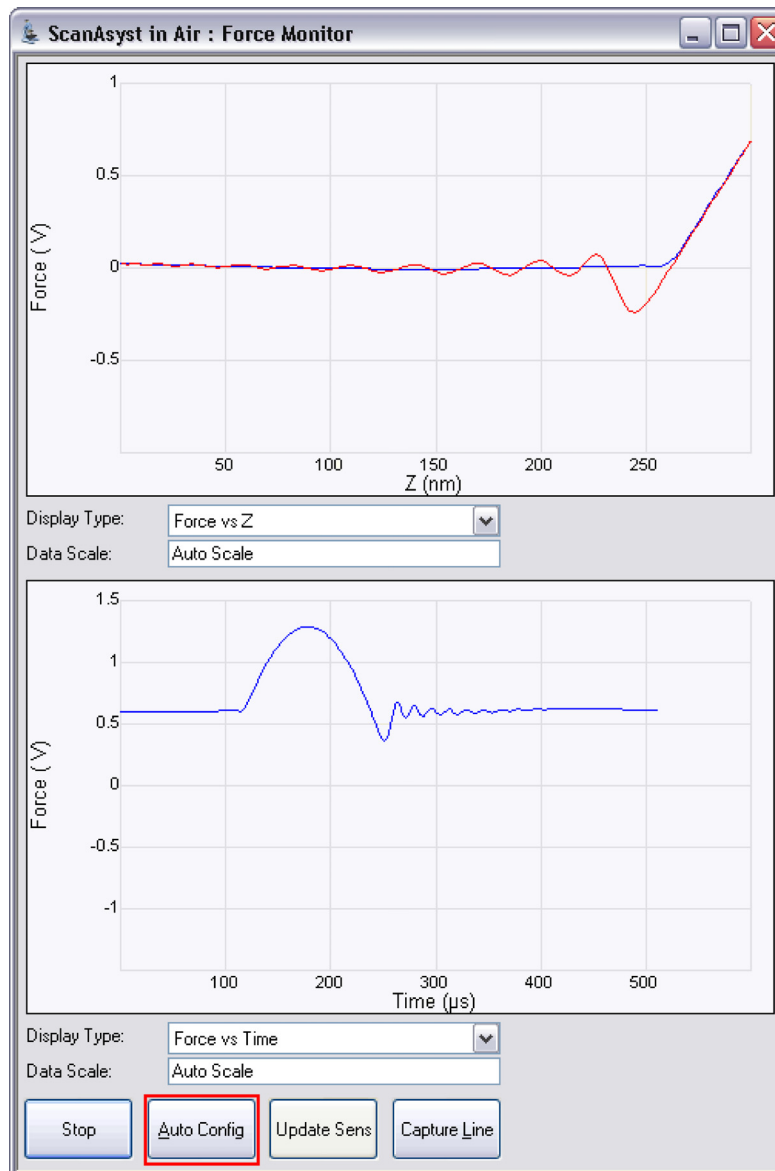
8.8 Advanced Atomic Force Operation

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**

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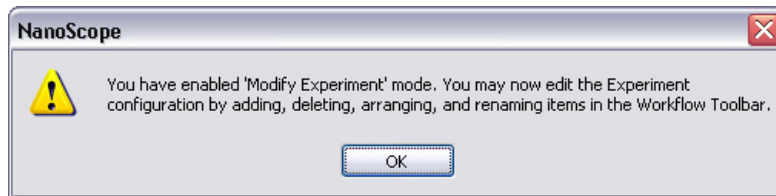
2. Click **OK** to open the **Configure Experiment** window, shown in Figure 8.8d.

Figure 8.8d The **Configure Experiment** Window
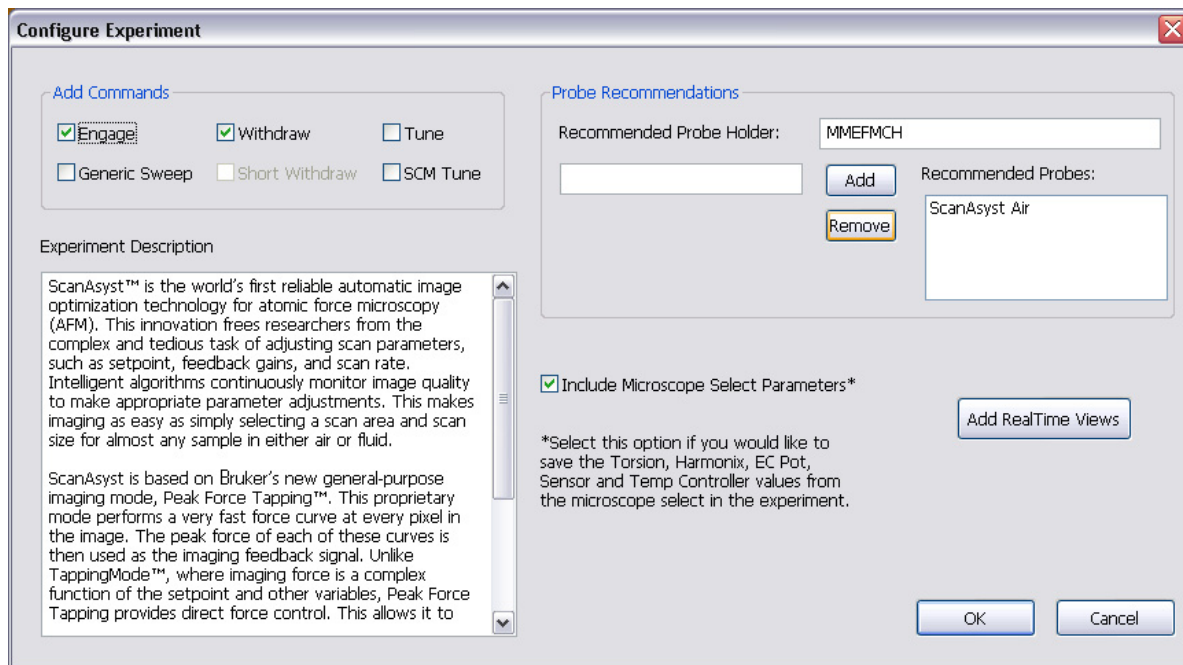
3. Click **CANCEL** which will close the **Configure Experiment** window.

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
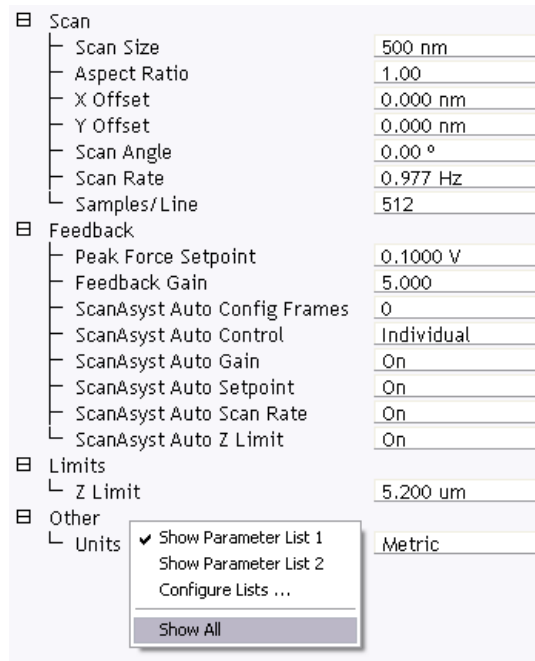
This makes all **Scan Parameters** visible along with two check boxes, the left, green, check box for the SIMPLE MODE and the right, red, check box for the EXPANDED MODE. See Figure 8.8f.

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.

Chapter 9   Contact AFM Mode

This chapter covers procedures for operating the MultiMode SPM in Contact AFM Mode. It is assumed the operator has previously prepared a Contact Mode probe and aligned the MultiMode head per instructions provided in Chapter 7 of this manual. Specific information regarding tip preparation is provided in Chapter 7; Appendix A of the *Command Reference Manual* contains further information regarding tips.

The following sections are included in this chapter:

- **Preparation Prior to Imaging:** Section 9.1

  - **Adjust the Detector Offsets:** Section 9.1.1

  - **Signal Settings:** Section 9.1.2

  - **Adjust tip height above sample surface:** Section 9.1.3

  - **Position Tip with OMV:** Section 9.1.4

  - **Select Mode of Operation:** Section 9.1.5

  - **Set Initial Scan Parameters:** Section 9.1.6

  - **Engage:** Section 9.1.7

  - **Adjust Setpoint with Force Calibration:** Section 9.1.8

- **Advanced Atomic Force Operation:** Section 9.2

  - **Displaying Parameters:** Section 9.2.1

  - **Optimization of Scanning Parameters:** Section 9.2.2

- **Force Calibration Mode:** Section 9.3

- **Beyond the Basics of AFM Operation:** Section 9.4

  - **Cantilever Selection:** Section 9.4.1

- **Optimization of Scanning Parameters:** Section 9.5

- **Data type:** Section 9.5.1

- **Gain settings:** Section 9.5.2

- **Scan size and Scan rate:** Section 9.5.3

- **Deflection Setpoint:** Section 9.5.4

- **Lowpass filter (LP deflection and LP friction):** Section 9.5.5

9.1 Preparation Prior to Imaging

9.1.1 Adjust the Detector Offsets

Verify that the MultiMode head has been fitted with a Contact Mode 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 9.1a). The detector adjustment screws are at the left side of the head.

Figure 9.1a   Photodetector Mirror Adjustments—Rear View
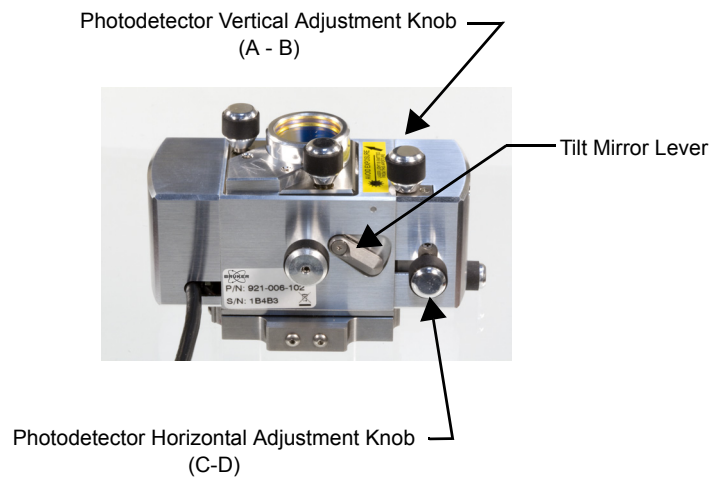
Refer to Figure 9.1b. For laser aligning screws atop the SPM head, the right-front screw moves the laser spot left-to-right (horizontally or along the X-axis). Turning this screw clockwise moves the laser spot to the right. The left-rear laser aligning screw moves the laser spot top-to-bottom (vertically 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 9.1b Laser Adjustment Knobs—Top View
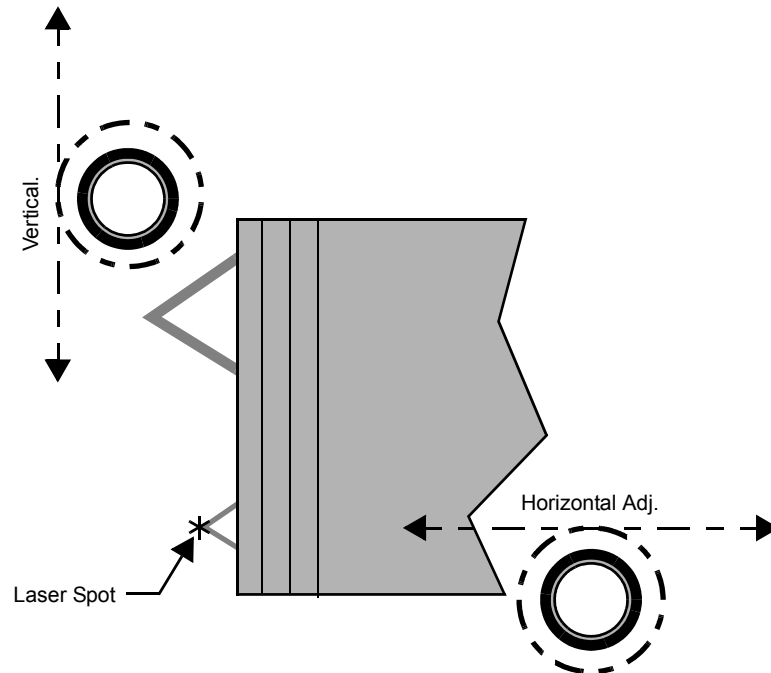
9.1.2 Signal Settings

In Contact AFM Mode, the vertical deflection (**Vert Defl.**) signal is used to provide the dynamic feedback signal for surface height tracking. The horizontal deflection (**Horiz Defl.**) is used for lateral force measurements in Contact Mode LFM, torsional resonance mode and HarmoniX. When disengaged in Contact AFM Mode and preparing for engagement, set the detector **Vert Defl.** adjustment to between **-3.0** and **-2.0V** for (SiNi cantilevers) or **-0.5** and **-1.0** (Si cantilevers). This is recommended for a 0.0V **Setpoint.** If performing LFM, set **Horiz Defl.** to **-1.0** to **+1.0V.**

The difference between the vertical deflection before engage and the setpoint is related to the force. A larger, more positive setpoint voltage results in a larger contact force.

> **Note:** Large offsets are not recommended between engage and disengage (2V) with etched silicon cantilevers in Contact Mode (450µm long only) because breakage is likely. With etched silicon contact probes, it may be necessary to reduce the offset between engage and disengage to a value of 1.0—1.5V. The large difference is recommended for first-time use of silicon nitride cantilevers.

9.1.3 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.

9.1.4 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**.

9.1.5 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 9.1c.

Figure 9.1c The Contact Mode **Select Experiment** window
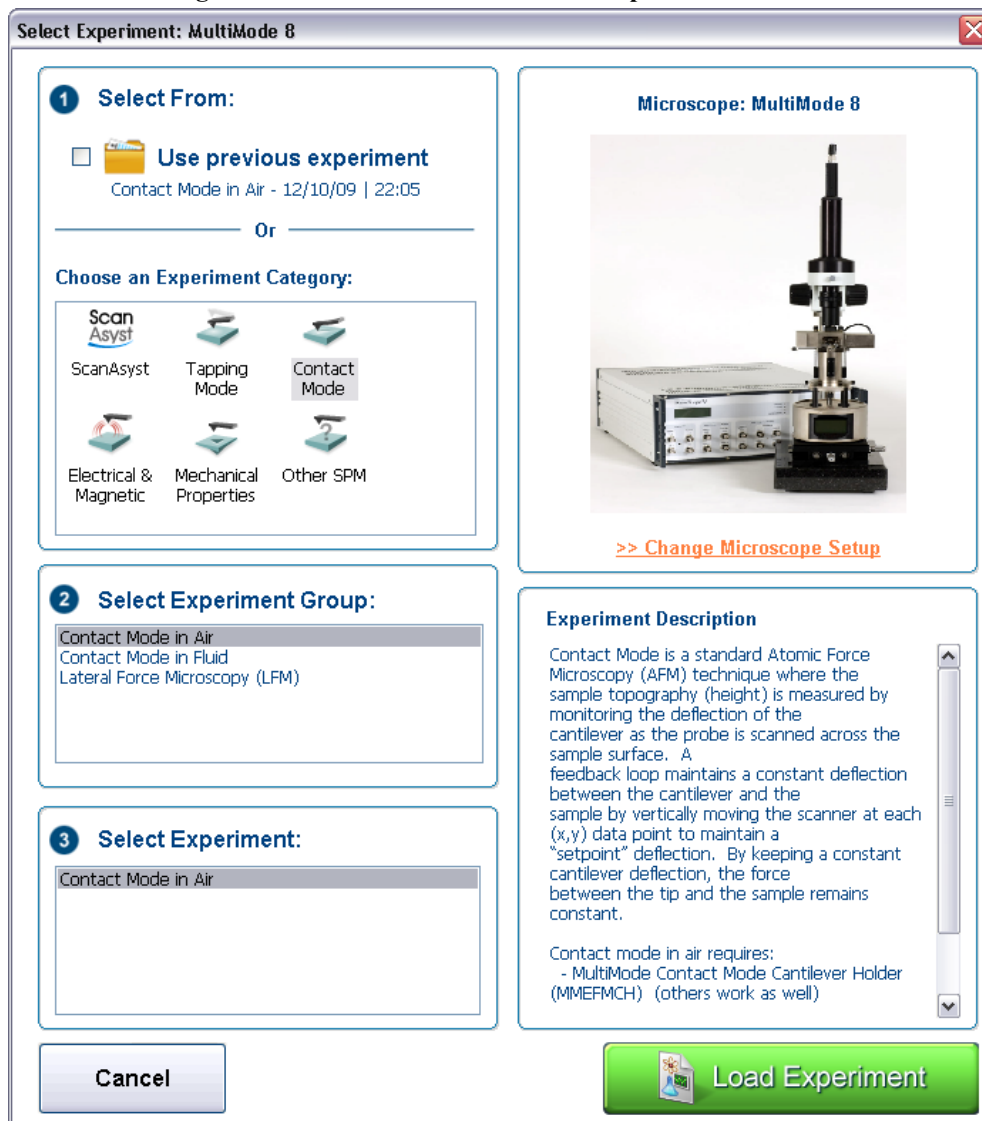
2. Select CONTACT MODE in the **Choose an Experiment Catagory** panel.

3. Select CONTACT MODE IN AIR in the **Select Experiment Group** panel.

4. Select CONTACT MODE IN AIR in the **Select Experiment** panel and click LOAD EXPERIMENT.

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** windows and the **Scan Parameters List** window, shown in Figure 9.1d.

Figure 9.1d Contact Mode in Air (Simple Mode) configuration
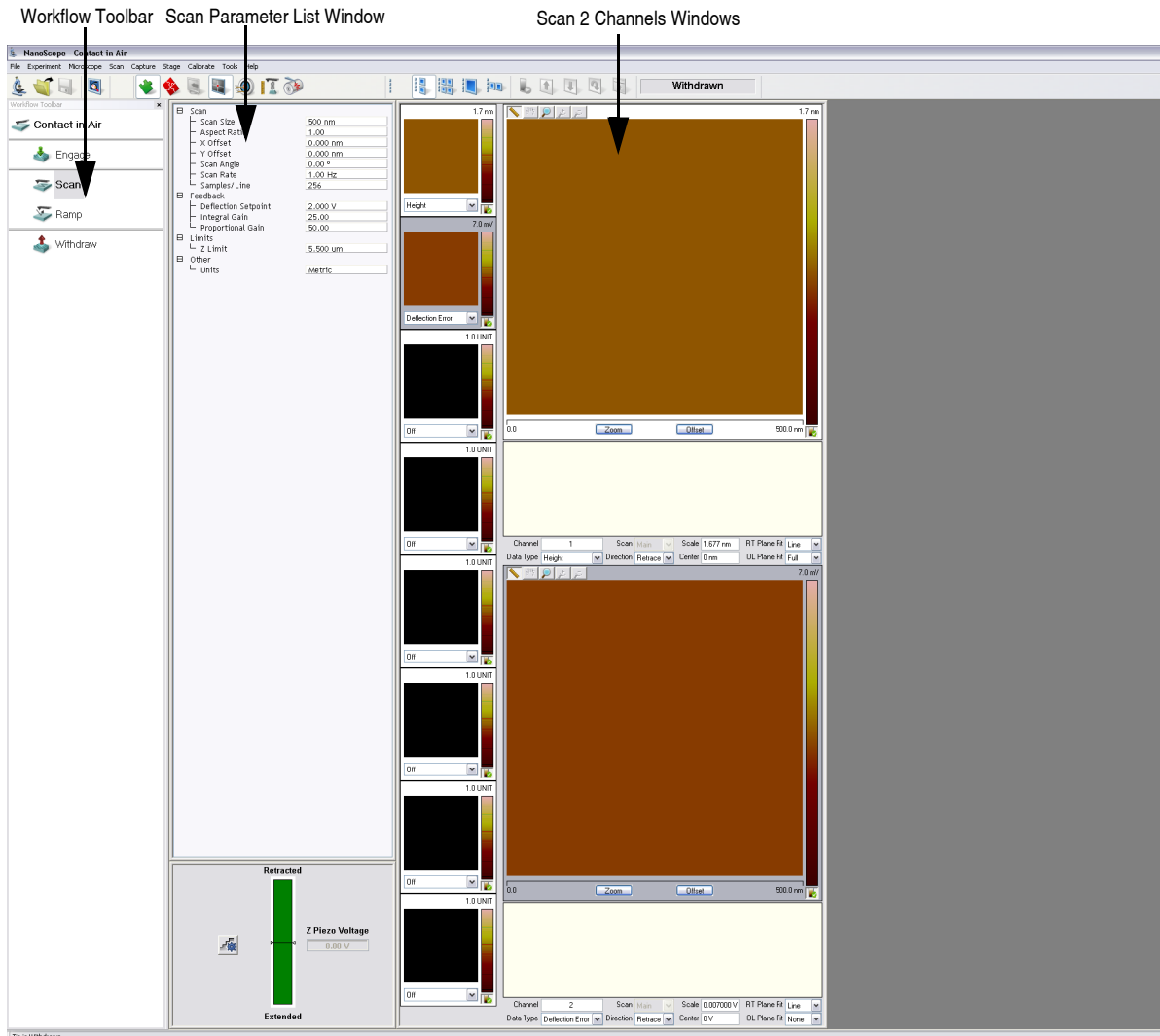
9.1.6   Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 9.1e).

1. Set the **Scan Rate** to **1 Hz**.

2. Set the **Scan Size** to **100nm**.

3. Set the **Scan Angle** to **0** degrees.

4. Ensure that the **Slow Scan Axis** is **ENABLED**.

Feedback Panel

1. Set the **Integral Gain** to **2** and the **Proportional Gain** to **5** (see Figure 9.1e).

2. Set the **Deflection Setpoint** to **0.0V**.

Other Panel

1. Set the **Units** to **METRIC**.

Figure 9.1e   Suggested Contact Settings

Limits

1. Set **Deflection Limit** and **Z Limit** to maximum values. Maximum values can be determined by entering very large numbers which exceed the program's acceptable limits and which the program will correct to an acceptable number. If the program accepts the value you enter, it not large enough.

Figure 9.1f Other Controls Panel Settings for Initial Setup (Contact Mode).
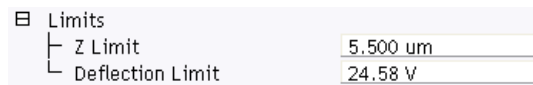
Channels 1 and 2

1. Set the **Channel 1 Data Type** to **HEIGHT** (see Figure 9.1g).

2. Set **Data Scale** to a reasonable value for the sample.

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

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

4. Set the **Channel 2** through **Channel 8 Data Type** to **OFF**.

Figure 9.1g Suggested Channel Settings
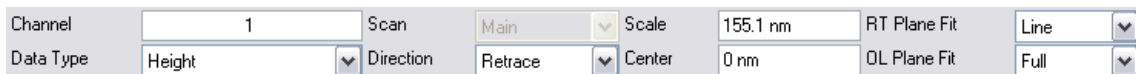
9.1.7 Engage

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

A pre-engage check, shown in Figure 9.1h, followed by Z-stage motor sound should be observed. If for any reason the engage aborts because the SPM head is still too far away from the surface, click on the **ABORT** button and readjust the screws to start the tip closer to the sample surface.

Figure 9.1h Pre-Engage Check
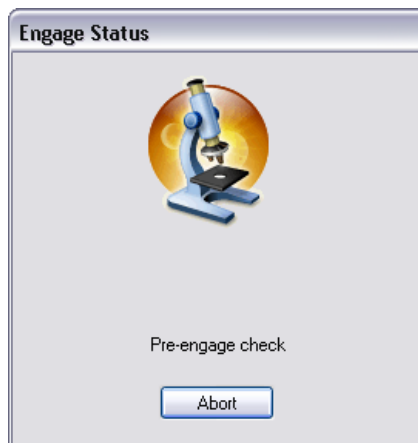
Assuming the tip is properly positioned before engaging again, an image should begin to appear on the image monitor.

After good engagement is obtained, gradually reduce the setpoint offset (disengage-minus-setpoint) to give a smaller contact force. A large offset between the setpoint and disengaged signal is recommended during a first try, because sometimes there are difficulties in engagement that can be reduced with a larger offset. On subsequent engagements, try smaller signal offsets as confidence in use of the instrument is gained.

9.1.8 Adjust Setpoint with Force Calibration

The most accurate way to minimize the **Setpoint** value is by Force Calibration using the Ramp, mode. This consists of obtaining a force plot, then adjusting the **Setpoint** value until the tip almost pulls free of the surface. In this way, it is possible to minimize tip-sample forces and reduce wear on both the tip and surface. Refer to Chapter 14 for details.

9.2   Advanced Atomic Force Operation

9.2.1   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 9.2a, displays the minimum number of parameters needed to make an image.

Figure 9.2a   The **SIMPLE MODE** view of the **Scan Parameter List** in **Contact Mode**

Expanded Mode

1. The **EXPANDED MODE** view, shown in Figure 9.2b, increases the number of displayed parameters enabling expert users to fine tune an image.

Figure 9.2b The **EXPANDED MODE** view of the **Scan Parameter List** in **Contact Mode**

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	1.00 Hz
Tip Velocity	1.00 µm/s
Samples/Line	256
Lines	256
Slow Scan Axis	Enabled
Feedback	
SPM Feedback	Deflection
Deflection Setpoint	0.4999 V
Integral Gain	2.000
Proportional Gain	5.000
Analog3	0 V
Analog4	0 V
LP Deflection BW	2.500 kHz
LP Friction BW	2.000 kHz
Interleave	
Limits	
Z Limit	5.500 um
Deflection Limit	24.58 V
Other	
LP Deflection	Enabled
LP Friction	Enabled
Tip Bias Control	Ground
Sample Bias Control	Ground
Units	Metric
Minimum Engage Gain	2.00
Tip Serial Number	
Parameter Update FB	Off
Output 1 Data Type	Off
Output 2 Data Type	Off

9.2.2 Optimization of Scanning Parameters

Careful selection of scan parameters is important in the successful application of Contact Mode AFM. In most cases, optimal parameter selection depends on the sample. It is beneficial to experiment with a range of values within each parameter, however, please review discussions of the scan parameters in the Realtime control panels before making bold changes. The following section analyzes the effects of the most important parameters.

Data Type

Data Type is the first parameter set because the settings of other parameters depend on it. The **Data Type** parameter in the **Channel** control panels selects the type of data collected by the system. **Height** data corresponds to the change in piezo height needed to keep the cantilever deflection constant. **Deflection Error** data comes from the differential signal off of the top and bottom photodiode segments relative to the **Deflection Setpoint**.

The scan parameters required to collect accurate height data are different from the optimal parameters for deflection data. To collect height data, the feedback gains must be high so that the tip tracks the sample surface with minimal cantilever deflection. The position of the piezo during the scan reflects the height of the sample. Topographical **Deflection Error** data is only reasonable on very smooth, flat samples. Topographical **Deflection Error** data is used infrequently and only for high resolution work.

Collect **Deflection Error** data with low feedback gains to ensure the piezo remains at a constant position relative to the sample. In this case, the tip and cantilever are deflected by the features on the sample surface. The output fluctuations in the cantilever deflection voltage from the top and bottom photodiode segments are recorded as a measure of the variation in the sample surface. **Deflection Error** data is not automatically calibrated in units of distance. You must measure the sensitivity using the procedures discussed in Chapter 14.

Deflection Error data collected with high feedback gains essentially equals the derivative of the height. This is commonly referred to as the error-signal. The error-signal provides a sensitive edge-detection technique and can be very helpful in visualizing fine details in topography that are difficult to see in regular height data. Using two channels, you must capture both height and deflection data simultaneously. **Deflection Error** (error-signal) data alone does not yield quantitative height information.

Gain Settings

The **Integral** and **Proportional** gains in the **Feedback** panel determine the feedback on the piezo height. The feedback loop keeps the deflection signal constant by adjusting the height of the piezo tube. If the gains are high, as they should be for **Height** data, the piezo height changes to keep the cantilever deflection nearly constant. If the gains are low, as they should be for topographical **Deflection Error** data, the cantilever deflects from its nominal position as it encounters features in the sample.

In general, set the gain settings as follows:

1. Set the **Integral** and **Proportional gain**s to **2-5** to start scanning.

2. To optimize the gains for **Height** data, increase the **Integral Gain** until the piezo begins to oscillate, then eliminate the oscillations by reducing the gain with 2-3 clicks of the left arrow key.

3. Repeat the process for the **Proportional Gain**.

> **Note:** Piezo oscillations typically cause high frequency wavy lines in the Realtime image.

4. For **Deflection Error** data, engage the microscope with the gains high, then lower them as much as possible without losing contact with the sample once the system begins scanning.

Scan Size and Scan Rate

In general, decrease the **Scan Rate** as the **Scan Size** increases. Use scan rates of **1.5 - 2.5 Hz** for large scans on samples with tall features. High scan rates help reduce drift, but use high scan rates only on flat samples with small scan sizes. When first using the system, vary the scan rate enough to observe a change in the image quality.

Deflection Setpoint

The **Deflection Setpoint** parameter defines the desired voltage (and, therefore, the desired deflection or force of the cantilever) for the feedback loop. The setpoint voltage constantly compares to the present vertical deflection signal of the photodiode to calculate the desired change in the piezo position. When the gain values are high, and when the **Data Type** is set to **Height**, the Z piezo position changes to keep the photodiode output signal close to the **Deflection Setpoint**; therefore, the cantilever deflection remains nearly constant.
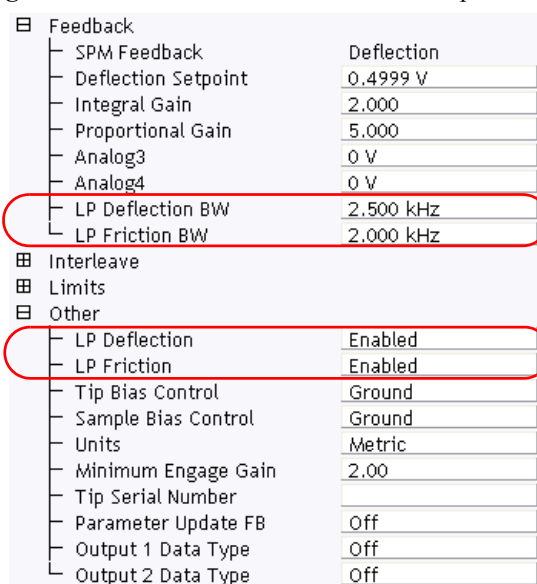
Adjust the **Deflection Setpoint** to increase or decrease the cantilever deflection and, therefore, the contact force of the tip on the sample. The **Ramp Mode** menu allows you to adjust the setpoint while viewing a graph of the tip position versus the deflection voltage. Using this procedure, described in detail in Chapter 14, minimizes the contact force of the tip on the sample. This is especially important on soft materials such as biological samples.

Lowpass Filter (LP Deflection and LP Friction)

LP Deflection (**Other > LP Deflection**, see Figure 9.2c) invokes a user programmable low pass filter to remove high frequency noise from the real-time data. The filter operates on the collected data regardless of scan direction. The cutoff frequency, shown in Figure 9.2c, can be set from 2-20kHz. The filter affects feedback in Contact mode. For faster scanning, set the **LP Deflection BW** to higher values.

LP Friction (**Other > LP Friction**, see Figure 9.2c) invokes a user programmable low pass filter to remove high frequency noise from the friction data channel. The LP Friction filter does not affect feedback or scanning speed. The cutoff frequency, shown in Figure 9.2c, can be set from 2-20kHz.

Figure 9.2c LP Deflection and LP Friction parameters

9.3 Force Calibration Mode

The Ramp Mode allows you to check the interaction between the cantilever and the sample surface. For detailed information regarding force ramps, see Chapter 14.

9.4 Beyond the Basics of AFM Operation

The above section provided a quick introduction to setup of the MultiMode microscope in the Contact AFM Mode. Although a great deal can be accomplished from this starting point, there is far more to the operation of the AFM. More tip-sample interaction information is in your *NanoScope Software User Guide* regarding the precise measurement of tip-sample forces.

Cantilever selection is an important area and it will become more important as tip and cantilever technology continues to develop. A clear understanding of the parameters in the control panels allows the user to tune the microscope to a wide variety of samples. Force mode imaging provides a great deal of information if the user fully understands its nuances. For more information regarding force mode imaging, see Chapter 14 in this manual.

9.4.1 Cantilever Selection

Two basic cantilever configurations are available for the Contact AFM Mode. Traditional triangular silicon nitride cantilevers have been used successfully for years. They are robust and relatively inexpensive. Etched silicon cantilevers with integral tips provide another scanning option. They have a higher aspect ratio and a smaller radius of curvature than the silicon nitride cantilevers.

Silicon nitride cantilevers for this system are available in two process variations: standard and sharpened. Usually, the standard silicon nitride cantilevers and resonant mode etched silicon probes are shipped with the MultiMode. Sharpened silicon nitride cantilevers have an almost identical appearance, but have slightly sharper tips. Note that this system does not require the "stand-alone" type silicon nitride probes which are used in some older, interferometric microscope heads; however, they may still be used.

Each silicon nitride cantilever substrate includes four cantilever probes with different sizes and spring-constants. Two of the cantilevers on each substrate measure 115µm from the substrate to the apex of the triangular cantilever (these are referred to as 100µm cantilevers) while the other two cantilevers measure 193µm from the substrate to the apex of the triangular cantilever (these are referred to as 200µm cantilevers). Both cantilever lengths are available with wide legs and narrow legs; however, thickness of both cantilevers is the same. The calculated spring constants for common cantilever configurations are listed below. These values are approximate; some variability will occur. The tabulated values should be used to approximate the contact force unless more accurate values are measured by the user.

Table 9.4a Cantilever Spring Constants

Cantilever Type	k (N/m) (narrow legs)	k (N/m) (wide legs)
115µm (triangular)	0.32	0.58
196µm (triangular)	0.06	0.12

The 100µm wide-legged cantilever can be used on most samples. If the image degrades rapidly because the probe is damaging the sample surface, it may be beneficial to switch to a cantilever with a lower spring-constant. Cantilevers with smaller spring-constants should be used on softer samples which will be destroyed by imaging with high-contact forces.

9.5   Optimization of Scanning Parameters

Careful selection of the scan parameters is important to the successful application of Contact AFM Mode. In most cases, the optimal parameter selection depends on the sample. All parameters in the **Realtime** control panel are discussed in your software manual. The user is encouraged to review these descriptions carefully. This section analyzes the effects of the most important parameters.

9.5.1   Data type

Data Type is the first parameter to set because the settings of other parameters depend on it. The **Data Type** parameter in the **Channel** control panels selects the type of data that is collected by the system. **Height** data corresponds to the change in piezo height needed to keep the cantilever deflection constant. **Deflection** data comes from the differential signal off of the top and bottom photodiode segments. The **Sensitivity** parameter in the **Force Mode Calibration** panel must be determined, as described in the discussion of **Force Calibration** mode, before deflection data is accurate.

The scan parameters required to collect good height data are different from the optimal parameters for deflection data. To collect height data, the feedback gains must be high so that the tip tracks the sample surface with minimal cantilever deflection. The position of the piezo during the scan reflects the height of the sample. Deflection data should be collected with low feedback gains so the piezo remains at a constant position relative to the sample. In this case, the tip and cantilever will be deflected by the features on the sample surface. The output fluctuations in the cantilever deflection voltage from the top and bottom photodiode segments are recorded as a measure of the variation in the sample surface. To collect accurate topographical data, the **Data Type** parameter should be set to **Height** in most instances.

Deflection data collected with high feedback gains is essentially the derivative of the height. This is commonly referred to as the error-signal mode. The error-signal mode provides a sensitive edge-detection technique. Using dual screen mode, it is possible to capture both **Height** and **Deflection** data simultaneously.

9.5.2   Gain settings

The **Integral** and **Proportional** gains in the **Feedback Controls** panel control the feedback on the piezo height. The feedback loop tries to keep the deflection signal constant by adjusting the height of the piezo tube. If the gains are high, as they should be for **Height** data, the piezo height will change to keep the cantilever deflection nearly constant. If the gains are low, as they should be for topographical **Deflection** data, the cantilever will deflect from its nominal position as features in the sample are encountered. In general, the **Integral** and **Proportional Gain** can be set to **2–3** to start scanning. To optimize the gains for height data, increase the **Integral Gain** until the piezo begins to oscillate, then eliminate the oscillations by reducing the gain with two or three clicks of the left-arrow key. Repeat the process for the **Proportional Gain**. Piezo oscillations typically cause high frequency wavy lines in the Realtime image. For deflection data, engage the microscope with the gains high, then lower them as much as possible once the system is scanning. Use scope mode while adjusting gains until trace and retrace nearly match without wavelike oscillations induced by gains set too high.

9.5.3 Scan size and Scan rate

In general, the **Scan Rate** must be decreased as the **Scan Size** is increased. Scan rates of 1.5–2.5Hz should be used for large scans on samples with tall features. High scan rates help reduce drift, but they can be used only on very flat samples with small scan sizes.

Notice the tip velocity parameter value. It is approximately (scan size) x 2 x (scan rate). Once the gains are optimized, record the tip velocity. You can then change scan size then adjust the tip velocity to return it to the previous value. This will automatically adjust the scan rate and should produce good tracking of surface features which are similar at different scan sizes.

9.5.4 Deflection Setpoint

The **Deflection Setpoint** parameter defines the desired voltage (and, therefore, the desired deflection of the cantilever) for the feedback loop. The setpoint voltage is constantly compared to the present photodiode cantilever deflection voltage to calculate the desired change in the piezo position. When the gain values are high, as they should be when the **Data type** is set to **Height**, the Z piezo position changes to keep the photodiode output signal close to the **Setpoint**; therefore, the cantilever deflection remains nearly constant. When the gain values are low (as they should be when the **Data type** is set to **Deflection**) the piezo height response is slow and the photodiode signal varies around the **Setpoint** value.

In Contact Mode, increased **Deflection Setpoint** yields higher tip-sample forces. The **Deflection Setpoint** can be adjusted to increase or decrease the cantilever deflection and, therefore, the contact force of the tip on the sample. This is especially important on soft materials such as biological samples.

9.5.5 Lowpass filter (LP deflection and LP friction)

LP Deflection (**Feedback > LP Deflection**) invokes a user programmable analog low pass filter to remove high frequency noise from the realtime data. The filter operates on the collected data regardless of the scan direction. The cutoff frequency can be set 2-20kHz. The filter affects feedback in contact mode. For faster scanning, set the LP filter to higher values.

LP Friction (**Feedback > LP Friction**) invokes a low pass filter to remove high frequency noise from the friction data channel. The LP Friction filter does not affect feedback nor the scanning speed. The cutoff frequency can be set 2-20kHz.

Chapter 10 Tapping Mode AFM

This chapter covers procedures for operating the MultiMode SPM using tapping mode in air. It is assumed that the operator has previously prepared a tapping mode probe tip and aligned the SPM head per instructions provided in Chapter 7 of this manual.

The following sections are included in this chapter:

- **Principles of Tapping Mode:** Section 10.1

- **Basic TappingMode AFM Operation:** Section 10.2

  - **Select the Microscope:** Section 10.2.1

  - **Select Mode of Operation:** Section 10.2.2

  - **Check Parameters:** Section 10.2.3

  - **Adjust Laser and Photodetector:** Section 10.2.4

  - **Additional preparations:** Section 10.2.5

  - **Tune the Cantilever:** Section 10.2.6

  - **Automatic Tuning:** Section 10.2.7

  - **Set Initial Scan Parameters:** Section 10.2.8

  - **Engage:** Section 10.2.9

  - **Optimize Scan Parameters:** Section 10.2.10

  - **Withdraw the Tip:** Section 10.2.11

- **Advanced TappingMode AFM Operation:** Section 10.3

  - **Resonating Techniques:** Section 10.3.1

  - **Cantilever Oscillation:** Section 10.3.2

  - **Decreasing the Cantilever Drive Frequency:** Section 10.3.3

10.1 Principles of Tapping Mode

Figure 10.1a depicts a cantilever oscillating in free air at its resonant frequency. A piezo stack excites the cantilever substrate vertically, causing the cantilever to move up and down. As the cantilever moves vertically, the reflected laser beam, or "return signal," deflects in a regular pattern over a photodiode array, generating a sinusoidal, electronic signal. This signal is converted to a root mean square (RMS) amplitude value, which is displayed in volts on the upper left (Tapping Mode Output Signal) meter located on the front of the MultiMode base.

Figure 10.1a   Tapping Cantilever in Free Air
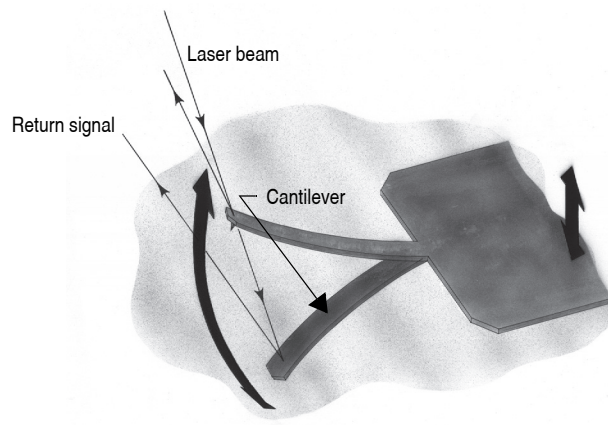
Figure 10.1b represents the same cantilever at the sample surface. Although the piezo stack continues to excite the cantilever substrate with the same energy, the tip is deflected in its encounter with the surface. The reflected laser beam reveals information about the vertical height of the sample surface and characteristics of the sample material itself. These material characteristics include elasticity, magnetism, and presence of electrical forces.

Figure 10.1b   Tapping Cantilever on Sample Surface
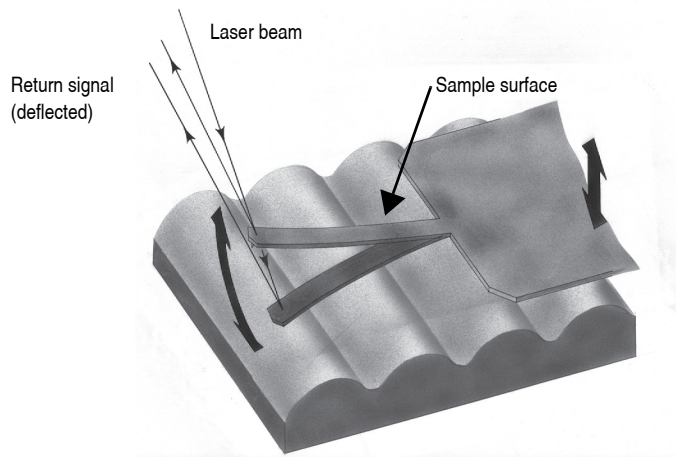
> **Note:**   Deflection of the cantilever and return signal are exaggerated in the figure for illustrative purposes.

10.2   Basic TappingMode AFM Operation

The following is a general outline of basic operational procedures involved in TappingMode AFM. For more detailed instructions, refer to Chapter 7 of this manual.

10.2.1 Select the Microscope

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

10.2.2 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 10.2a.

Figure 10.2a The **Select Experiment** window
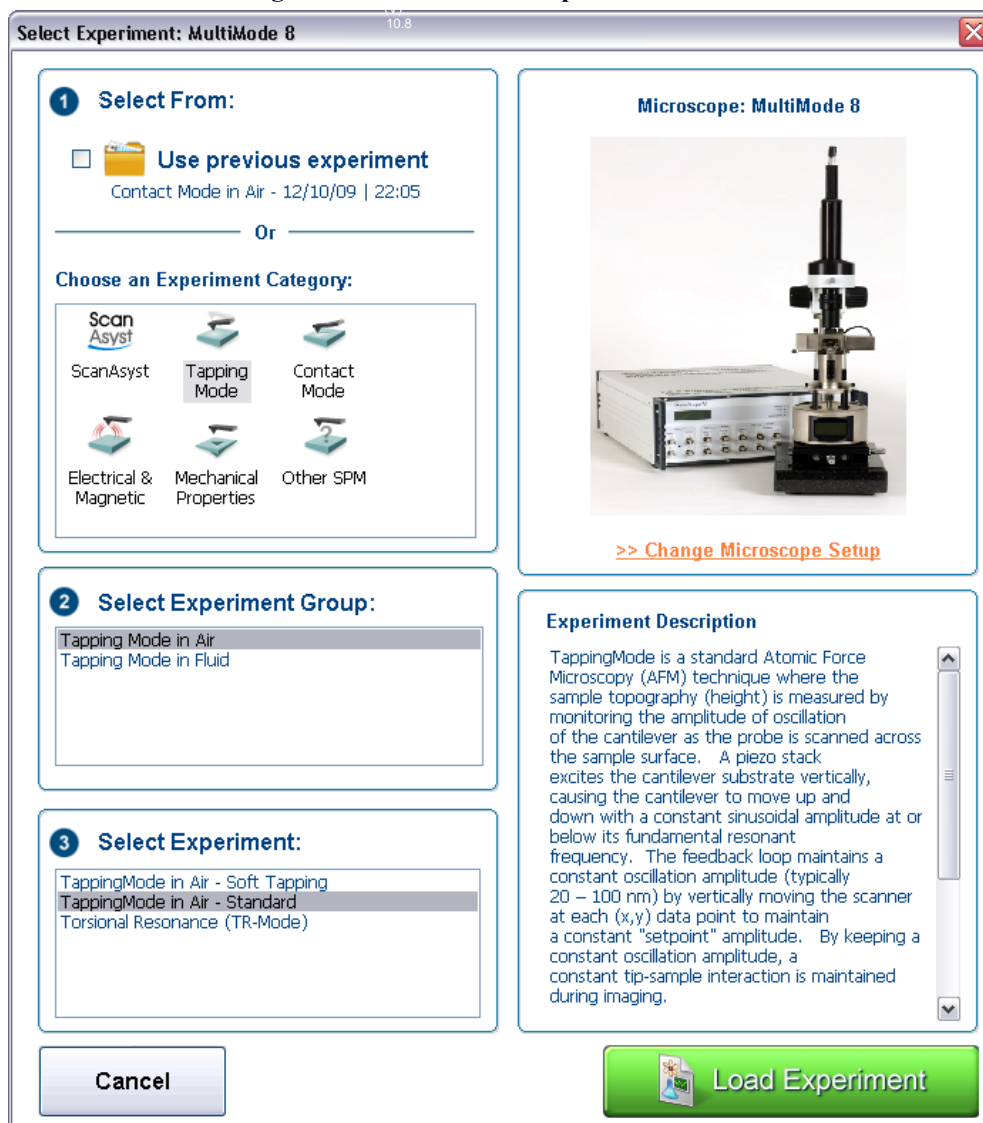
2. Select TAPPINGMODE AFM in the **Choose an Experiment Catagory** panel.

3. Select **TAPPINGMODE IN AIR** in the **Select Experiment Group** panel.

4. Select either **TAPPINGMODE IN AIR - SOFT TAPPING** or **TAPPINGMODE IN AIR - STANDARD** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** windows and the **Scan Parameters List** window, shown in Figure 10.2b.

Figure 10.2b Tapping Mode (Simple Mode) configuration
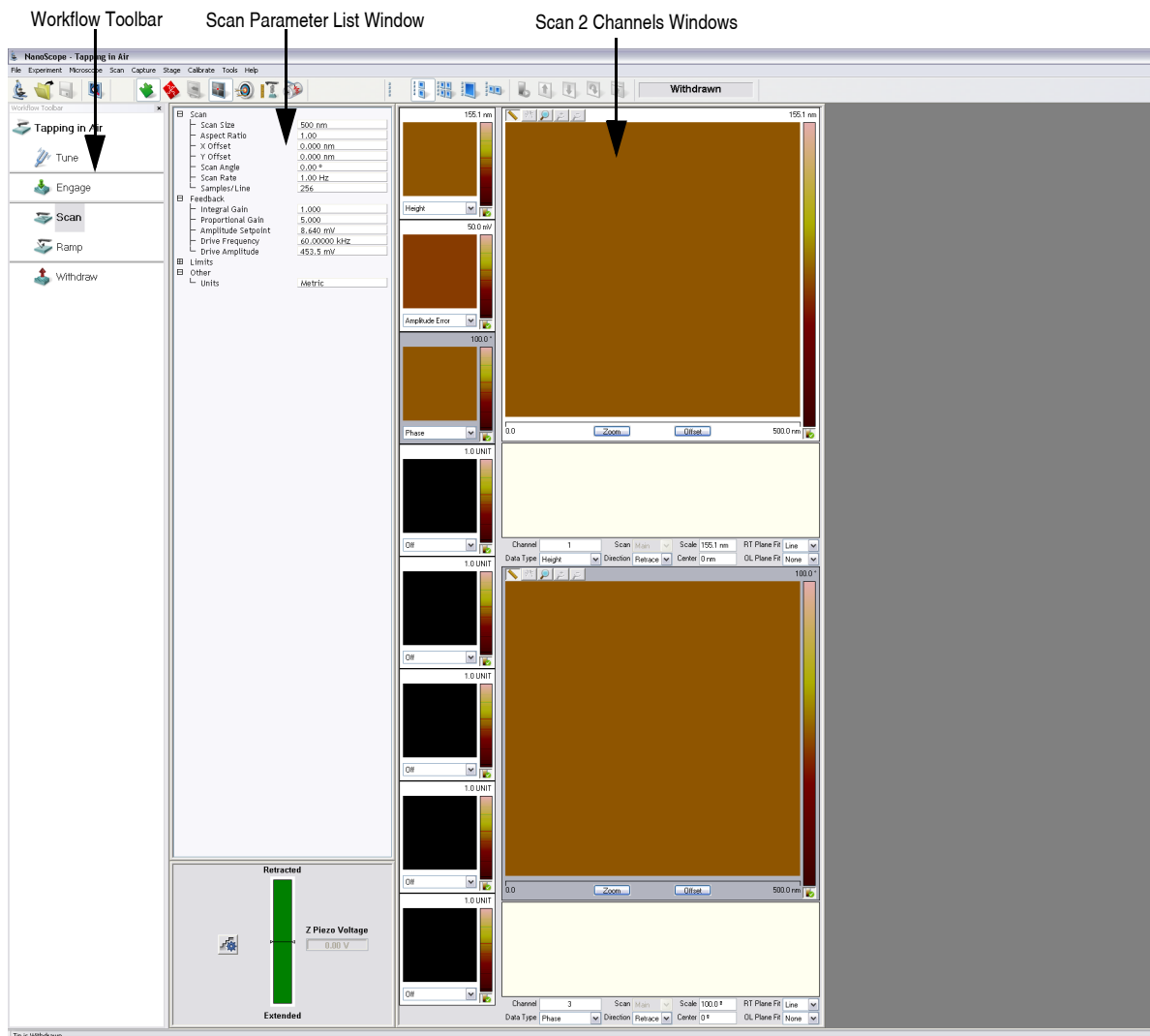
10.2.3 Check Parameters

Check that the **Realtime** control panel parameters are set within reasonable limits for tapping mode operation. If you are uncertain what parameter settings to start with, try the values below:

Panel	Parameter	Setting
Scan	**Scan Size**	1µm
	X Offset	0.00nm
	Y Offset	0.00nm
	Scan Angle	0.00°
	Scan Rate	1.00Hz
	Sample/Line	256
	Slow Scan Axis	Enabled
Feedback	**Integral Gain**	4.00 (see "d")
	Proportional Gain	8.00 (see "d")
	Amplitude Setpoint	a
	Drive Frequency	b
	Drive Amplitude	c
Other	**Microscope Mode**	Tapping
	Engage Setpoint	1.00
Interleave	**Interleave Mode**	Disabled
Limits	Five settings	Set to max (see "e")

a. **Amplitude Setpoint** is initially set by the software during engagement. It may be adjusted up or down afterward.

b. **Drive Frequency** is determined during Cantilever Tune (Section 10.2.6).

c. **Drive Amplitude** is determined during Cantilever Tune (Section 10.2.6).

d. Gains vary with the size of the scanner. The values given are typical for small scanners and should be reduced for larger scanners. Use the Scope window while scanning to optimize gains for good surface tracking. Gains for tapping mode are typically about 10% of gains for contact imaging.

e. Input very large values so maximum acceptable value will be determined by program when the input value exceeds the max acceptable. The **Amplitude Range** setting is the peak to peak photodetector output resulting from cantilever oscillation.

10.2.4 Adjust Laser and Photodetector

Verify that the probe holder has been fitted with a tapping mode, single crystal silicon probe and aligned per instructions provided in Chapter 7 of this manual. Photodetector voltage values are displayed on meters mounted on the front of the MultiMode base. The laser photodetector is adjusted using the photodetector adjustment knobs on the left-top and left-rear of the head (see Figure 10.2c below). Recall that RMS amplitude is an AC signal which does not have any real meaning until cantilever tuning is completed (see Section 10.2.5). The laser spot should be approximately centered on the photodetector prior to entering the cantilever **Tune** routine.

Figure 10.2c Photodetector adjustment knobs and SPM voltage meters.

Photodetector adjustment knobs.

Photodetector output signal (RMS AC volts). This value is directly influenced by the Drive amplitude parameter when tip is at resonance.

Sum signal. This value changes according to how much of the beam falls on the photodetector.

RMS	VERT
0.26	*0.00*

SUM
4.0

Deflection voltage. This changes according to how beam is centered vertically. At 0.00 Volts beam is centered.

Figure 10.2d   Voltage meters on the MultiMode base reveal a great deal about the amplitude and alignment of the tapping mode laser signal on a tuned tip operating at its resonant frequency.

Small laser signal amplitude yields low output voltage.

RMS
0.06

VERT
9.91

SUM
1.2

Sum voltage low due to misalignment of laser on cantilever or laser on photodetector.

Vertical voltage difference high because laser signal falls on bottom half of photodetector only.

Larger laser signal amplitude yields higher output voltage.

RMS
0.28

VERT
0.00

SUM
3.8

Sum voltage higher due to stronger laser signal. All of beam falls on photodetector.

Well centered laser beam yields a vertical voltage difference of zero.

Note: The actual value of **SUM** will also depend upon the type of probe in use. Uncoated silicon probes are less reflective and will produce a smaller **SUM**.

10.2.5 Additional preparations

In TappingMode, the RMS amplitude representing AC voltage signal is used to provide the dynamic feedback signal for surface height tracking. The vertical deflection signal (displayed on the "Vertical or Horizontal Difference" meter) should be close to zero (± 1.0V) prior to running cantilever **Tune** and/or attempting engagement. If you are uncertain how to set parameters for the **Channel 1** panel, try the following settings:

Panel	Parameter	Setting
Channel 1	**Data Type**	Height
	Scale	(see note)
	Direction	Retrace
	Scan	Main
	Realtime Plane Fit	Line
	Offline Plane Fit	None

> **Note:**   Setting depends upon sample height. A data **Scale** value of 4.50µm is the maximum for most "J" scanners. Always start with a low data **Scale** value if feature height is unknown, then increase until the image is optimized. On the 200nm step height calibration sample, a reasonable data **Scale** setting would be 300nm initially. The data **Scale** setting does not offset the scan but how the data is displayed. It is used similarly to a contrast control.

10.2.6 Tune the Cantilever

This section describes steps required to find the resonance peak of the cantilever and adjust the oscillation voltage so the cantilever will vibrate at an appropriate amplitude. A range of vibration frequencies will be applied to the cantilever to determine the frequency which produces the largest response (the resonant frequency). In most instances, the resonant peak will have a sharp Gaussian distribution but at times the peak can be somewhat asymmetric. The system will tolerate some deviation in the shape of the peak.

1. Select **Microscope** > **Cantilever Tune** or click the **TUNE** icon on the Workflow Toolbar.

2. The initial **Cantilever Tune** panel, shown in Figure 10.2e, appears with the **Frequency Sweep** (a plot of cantilever response as a function of applied oscillation frequency).

3. Choose either the automatic or manual tuning method (see **Automatic Tuning:** Section 10.2.7 and **Manual Cantilever Tuning:** Section 10.3.4).

10.2.7 Automatic Tuning

For most purposes, the **Auto Tune** function will suffice.

- Ensure that **Mode** is set to **MAIN** (not **INTERLEAVE**).

- Verify the starting parameters. The nominal resonant frequency of a probe can be found on the probe box label. Ensure the start and end frequencies correlate with this value.

- Click **Auto Tune**. The computer and controller begin automatic tuning. See Figure 10.2e.

Figure 10.2e   Auto Tune Control Panel
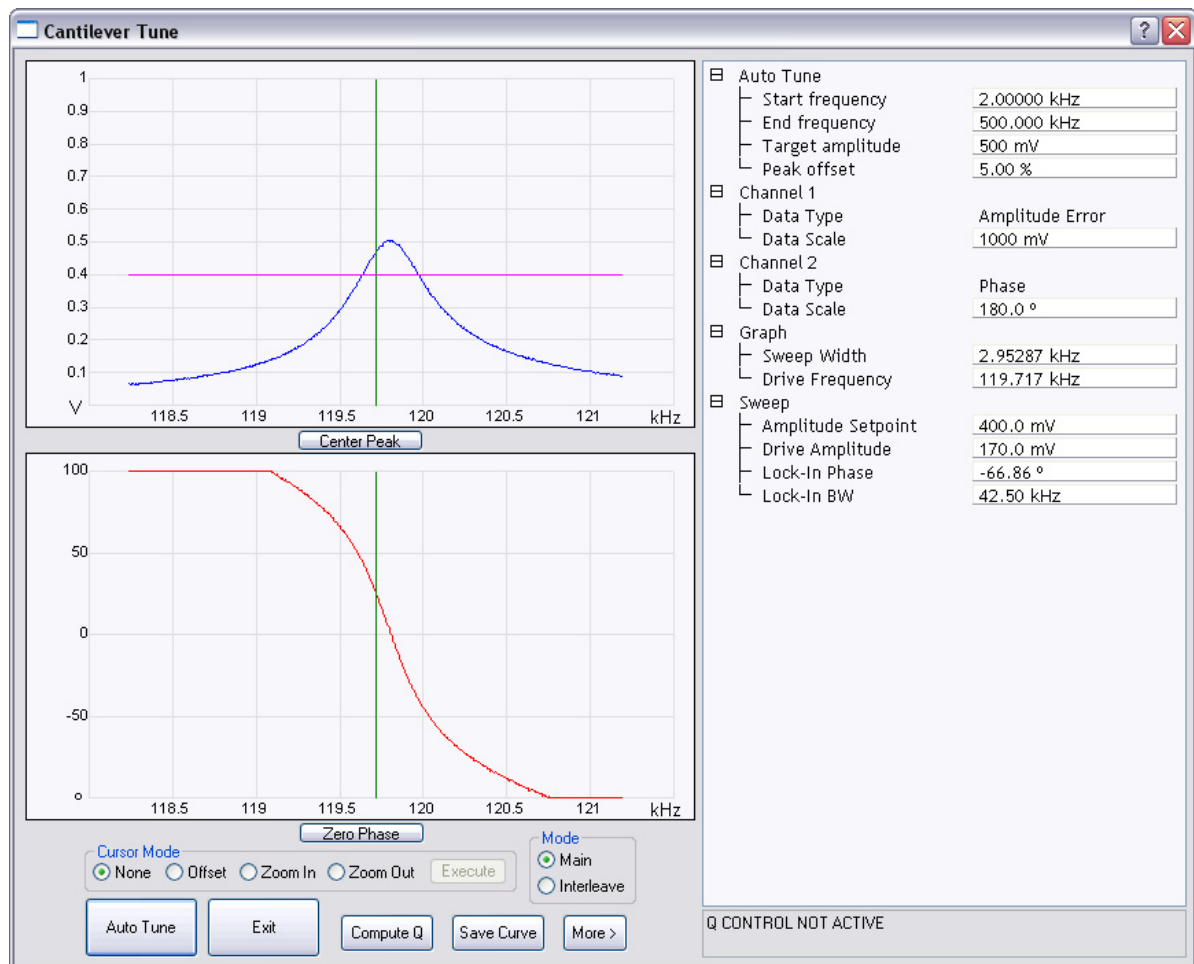
10.2.8 Set Initial Scan Parameters

Scan Panel

In the **Scan Controls** panel, set the following initial scan parameters (see Figure 10.2f).

1. Set the **Scan Rate** to **1 Hz**.

2. Set the **Scan Size** to **500 nm**.

3. Set the **Scan Angle** to **0**.

Figure 10.2f   Suggested Tapping Mode in Air Scan Settings

Feedback Panel

1. Set the **Integral gain** to **0.5** and the **Proportional gain** to **5** (see Figure 10.2f).

   Note: The **Drive Frequency** and **Drive Amplitude** values were determined during the Cantilever Tune procedure. It is not necessary to enter a value for the **Amplitude Setpoint**; this will be determined automatically during the engage process.

Other Panel

1. Set the **Units** to **METRIC**.

Channels

1. Set the **Channel 1 Data Type** to HEIGHT (see Figure 10.2g).

2. Set the **Channel 2 Data Type** to AMPLITUDE ERROR.

3. Set the **Channel 3 Data Type** to PHASE.

4. Set **Data Scale** to a reasonable value for the sample.

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

5. Set **Line direction** to either **Trace** or **Retrace**.

6. Set the **Channel 4** through **Channel 8 Data Type** to OFF.

Figure 10.2g Suggested Channel Settings

Channel	1	Scan	Main	Scale	155.1 nm	RT Plane Fit	Line
Data Type	Height	Direction	Retrace	Center	0 nm	OL Plane Fit	None
Channel	2	Scan	Main	Scale	50.00 mV	RT Plane Fit	Line
Data Type	Amplitude Error	Direction	Retrace	Center	0 mV	OL Plane Fit	None
Channel	3	Scan	Main	Scale	100.0 º	RT Plane Fit	Line
Data Type	Phase	Direction	Retrace	Center	0 º	OL Plane Fit	None

10.2.9 Engage

1. Move the probe to the area of interest using the X-Y translation knobs on the MultiMode head.

2. Use the meters to verify that the vertical deflection is between -1 and +1, the RMS amplitude (top left meter) is 1-2V, and the sum voltage is greater than 1V. Do not readjust the RMS (see note).

> **Note:** Auto-tune will set the RMS to be approximately 90-95% of the target amplitude. For example: tuning with a target amplitude of 500 mV causes the RMS on the display meter set to approximately 480 mv.

3. Select **Microscope** > **Engage** or click the ENGAGE icon on the toolbar. A pre-engage check begins, followed by Z-stepper motor motion. If for any reason the engage aborts because the tip is still too far away from the surface, use the coarse adjustment screws and/or the Tip Down motor control switch to bring the tip and surface closer together. If all goes well after re-engaging, a well-formed image will begin to appear.

If it becomes necessary to image another part of the sample, execute a **Withdraw** command before moving the X-Y translation stage; otherwise, the tip may be damaged. After the tip is engaged, the

control panel values can be readjusted to provide the desired scan parameters. Refer to Section 10.2.10 below for scan parameter optimization.

> **Note:** After the tip engages, adjust the control panel values to provide the desired scan parameters. Refer to Section 10.3.5 for scan parameter optimization.

4. After engaging, it may be necessary to recheck the Cantilever Tune parameters. Select **MICROSCOPE > CANTILEVER TUNE** or click the **TUNE** icon in the **Workflow Toolbar**. In the **Tip offset** box, shown in Figure 10.2h, enter **200nm** and click **OK**. Adjust the **Drive Frequency** if necessary.

Figure 10.2h Start Sweep On Surface
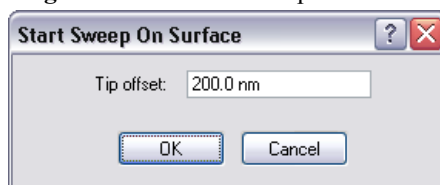
10.2.10 Optimize Scan Parameters

The procedure to optimize the scan parameters is similar to that of Contact Mode.

1. In the **Scan** panel, set **Slow Scan Axis** to **DISABLED**. This function is available in the Expanded Mode.

2. Observe the agreement between the Trace and Retrace lines. Adjust the **Scan Rate, Gains,** and **Amplitude Setpoint** to bring these two lines in to coincidence. A more detailed discussion of these parameters can be found in Section 10.3.

10.2.11 Withdraw the Tip

1. Select **RealTime** > **Withdraw** or click the **WITHDRAW** icon in the **Workflow Toolbar**. The SPM will stop scanning and then ascend approximately 10μm. If more clearance between tip and sample is desired, toggle the **Up / Down** switch on the top-right side of the MultiMode base.

2. Replace or move the sample.

> **CAUTION:** Never withdraw samples without verifying that the tip has adequate clearance during the entire sample removal sequence.

10.3   Advanced TappingMode AFM Operation

This section discusses the more subtle aspects involved in operating the MultiMode in TappingMode.

10.3.1 Resonating Techniques

Without a thorough understanding of principles associated with cantilever resonating techniques, you may generate distorted data. Understanding the **Cantilever Tune** process and the effects of real-time scan parameters is critical for effective operation of the microscope. It is also important to understand similarities and differences between **Ramp Mode** in **Contact AFM** and **Ramp Mode** in **TappingMode** (see Chapter 14).

10.3.2 Cantilever Oscillation

The response of the cantilever to inputs plays an important role in the operation of the MultiMode SPM while in TappingMode. There is an important trade-off between the response time of the cantilever and the force applied to the sample. The cantilever does not respond instantly to perturbations in oscillation amplitude. The cantilever drive system pumps energy gradually into the cantilever oscillation. Figure 10.3a illustrates a typical response curve of the cantilever amplitude as a function of time. To demonstrate the conflicting requirements, the performance of the system is analyzed at two operating points.

Figure 10.3a   Cantilever Response Curve
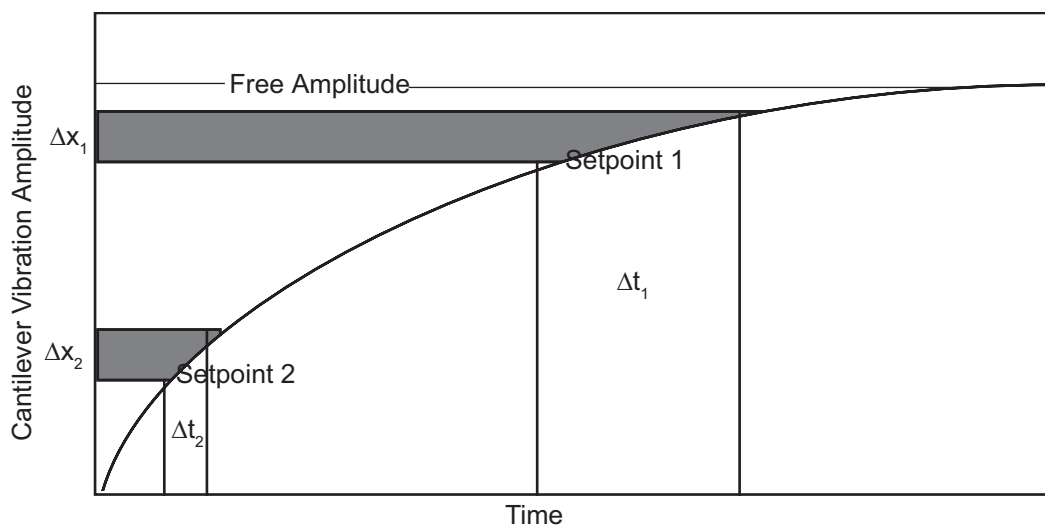
At Setpoint 1 the operating point is only slightly lower than the free oscillation amplitude. This has the advantage of dissipating very little energy to the sample surface. The disadvantage is that the system takes longer to recover from a given perturbation in the amplitude. Consider the situation where the tip travels off a step with a height of Δx. At Setpoint 1 it takes longer for the amplitude of

the cantilever oscillation to increase; therefore, the feedback system is slow in responding to the error created by going off of the step. At operating Setpoint 2 the cantilever amplitude builds up more rapidly. The feedback system senses the error caused by going off of the step and responds more rapidly. However, more energy transfers to the sample surface while scanning at this operating point.

The nature of the sample influences the decision between response time and contact force. For example, harder samples can withstand higher contact forces, so the response time improves by lowering the **Amplitude Setpoint**. Soft samples that are relatively flat should run with higher **Amplitude Setpoint** values to reduce the energy imparted to the sample. In general, the solution to the problem is to decrease the scan rate and increase the feedback gains. In some situations, the feedback gains cannot increase without causing piezo oscillations; in such cases there is no choice but to reduce the scan rate.

Inadequate response time typically occurs when the tip encounters a low point in the sample. The amplitude of the cantilever oscillation decreases very quickly when taller portions of the sample are encountered. As a result, the system response is markedly different depending on whether the tip is climbing or descending a feature in the sample. For this reason, viewing the **Scope** trace (below the scan) is very useful when setting scan parameters. As the tip descends, features are evaluated by comparing the **Trace** and **Retrace**. Figure 10.3b illustrates the effects of poorly selected scan parameters on a calibration standard that includes a series of sharp-walled pits. Regardless of the scan direction, the tip does not track the wall of the pit when the tip encounters a pit. However, it does track the surface closely when moving out of the pit.

Figure 10.3b   Scope Trace with High Scan Rate
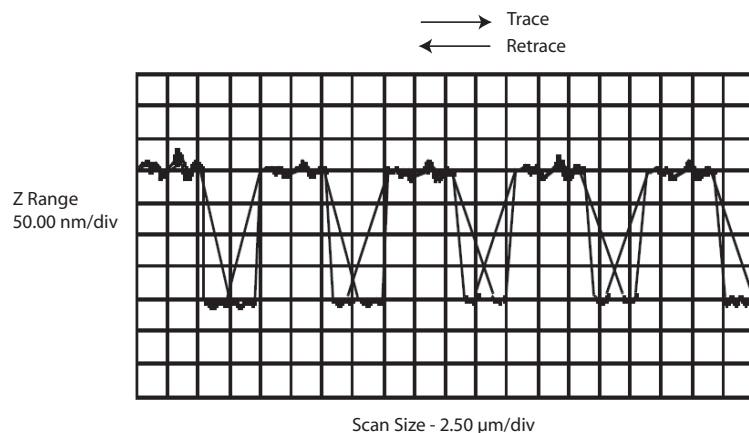
Figure 10.3c depicts the same sample with a slight increase in the **Integral Gain** and a twofold decrease in the **Scan Rate**. The tip now tracks the surface when it descends into the pit as well as when it exits. The **Trace** and **Retrace** lines now coincide closely.

Figure 10.3c Scope Trace with Correct Scan Rate

Z Range
50.00 nm/div

Scan Size - 2.50 µm/div
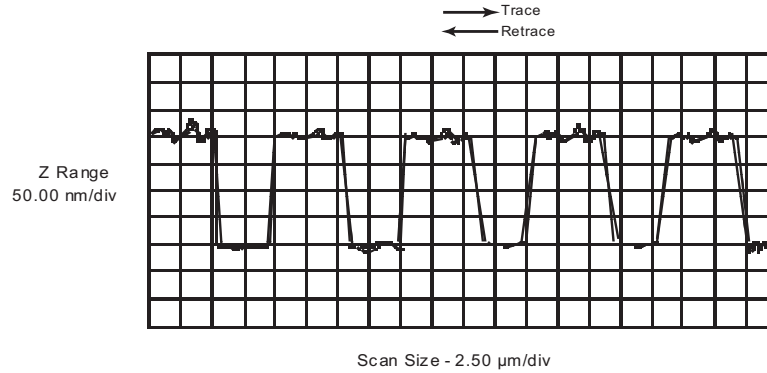
10.3.3 Decreasing the Cantilever Drive Frequency

The **Drive Frequency** selected to oscillate the cantilever plays an important role in the performance of the microscope while in TappingMode. As a first step it is important to determine the resonant frequency of the cantilever, but the **Drive Frequency** can be further tuned to improve scanning performance.

The microscope produces better data in TappingMode when the **Drive Frequency** is set lower than the resonant peak of the cantilever. The **Drive Frequency** is set such that it coincides with a 1-10 percent decrease in the oscillation amplitude by setting **Peak Offset** in the **Auto Tune** controls to the desired percent. Figure 10.3d shows a suggested operating region. This is a suggestion based on our observations; users are encouraged to experiment with the microscope and decide what produces the best results.

Figure 10.3d   Suggested Range of Drive Frequencies

Drive Frequency should be within this range.

Cantilever
Response
12.54 nm/div

Setpoint

Center Frequency - 338.8723 kHz
5.0 kHz/div
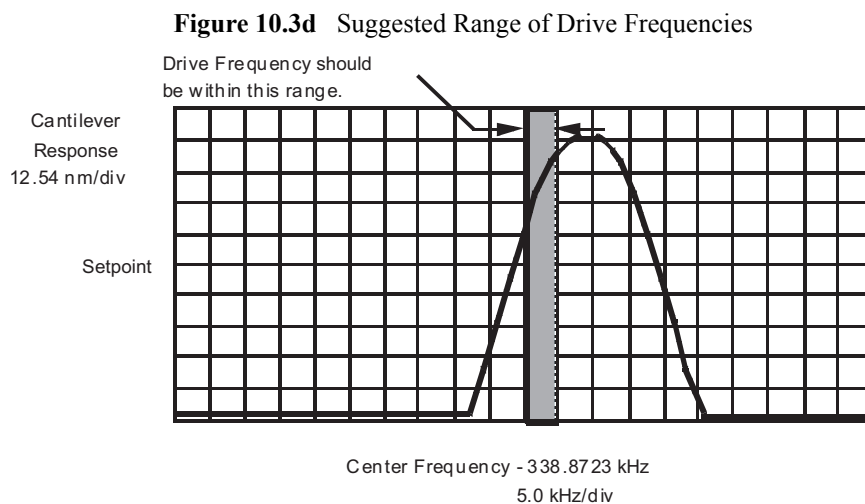
10.3.4 Manual Cantilever Tuning

With Force Modulation or Fluid Tapping applications, it may be useful to tune the cantilever manually. MFM and EFM imaging often offset the drive frequency above or below the resonant frequency.

> **Note:** More than one type of cantilever exists. Cantilevers can have different dimensions and different resonant frequencies. The box containing the probe will have a label indicating the nominal resonant frequency of the probes. Certain parameter values, particularly the center frequency and the sweep width used in the following example, apply to a particular cantilever type. In the following example, nominal parameter values will vary depending on the actual cantilever used.

1. In the **Graph** panel of the **Cantilever Tune** window, set the **Drive Frequency** parameter to a value near the center of the resonant frequencies range specified for the wafer. For example, if the frequency range is specified as 240—420 KHz, select a drive frequency of **330 KHz**.

2. Start with a **Drive Amplitude** of about 50 mV. It is possible to detach the cantilever from the substrate by applying too large of a drive amplitude; therefore, it is important to exercise some caution when adjusting the **Drive Amplitude** parameter. Note that the **Drive Amplitude** is set automatically during the engage process.

3. Set the **Sweep Width** to the same value as the **Center Frequency**.

4. The **Sweep Width** must be large enough to cover the frequency range specified for the wafer.

5. Zero the **Amplitude Setpoint**. Note that the **Amplitude Setpoint** will be reset automatically during the engage process.

6. Set the **Amplitude Range** to **4000 mV**.

7. Center the peak on the frequency sweep plot shown using the **ZOOM IN** and **OFFSET** commands after identifying the maximum amplitude peak with the lowest frequency in the frequency response plot.

   > **Note:** The **OFFSET** command sets the center frequency equal to the cursor position to shift the plot. The **ZOOM IN** command decreases the sweep width and shifts the center frequency value to stretch the plot.

8. Increase the **Amplitude Setpoint** until the peak appears.

9. Continue to **ZOOM IN** and center the peak until the peak coincides with the vertical center line within **10 Hz**. The value displayed for center frequency is now used as the resonant frequency of the cantilever.

   > **Note:** The system works well in TappingMode if the center frequency is at, or below, the peak in the resonance plot. The center frequency can decrease to the point

where the oscillation amplitude reaches 90 percent of the maximum value. Operate at a frequency lower than the resonant frequency to avoid shifting the resonant frequency upon approach of the tip to the surface (see Figure 10.3f).

Figure 10.3e Cantilever Tune Control Panels for Main Controls
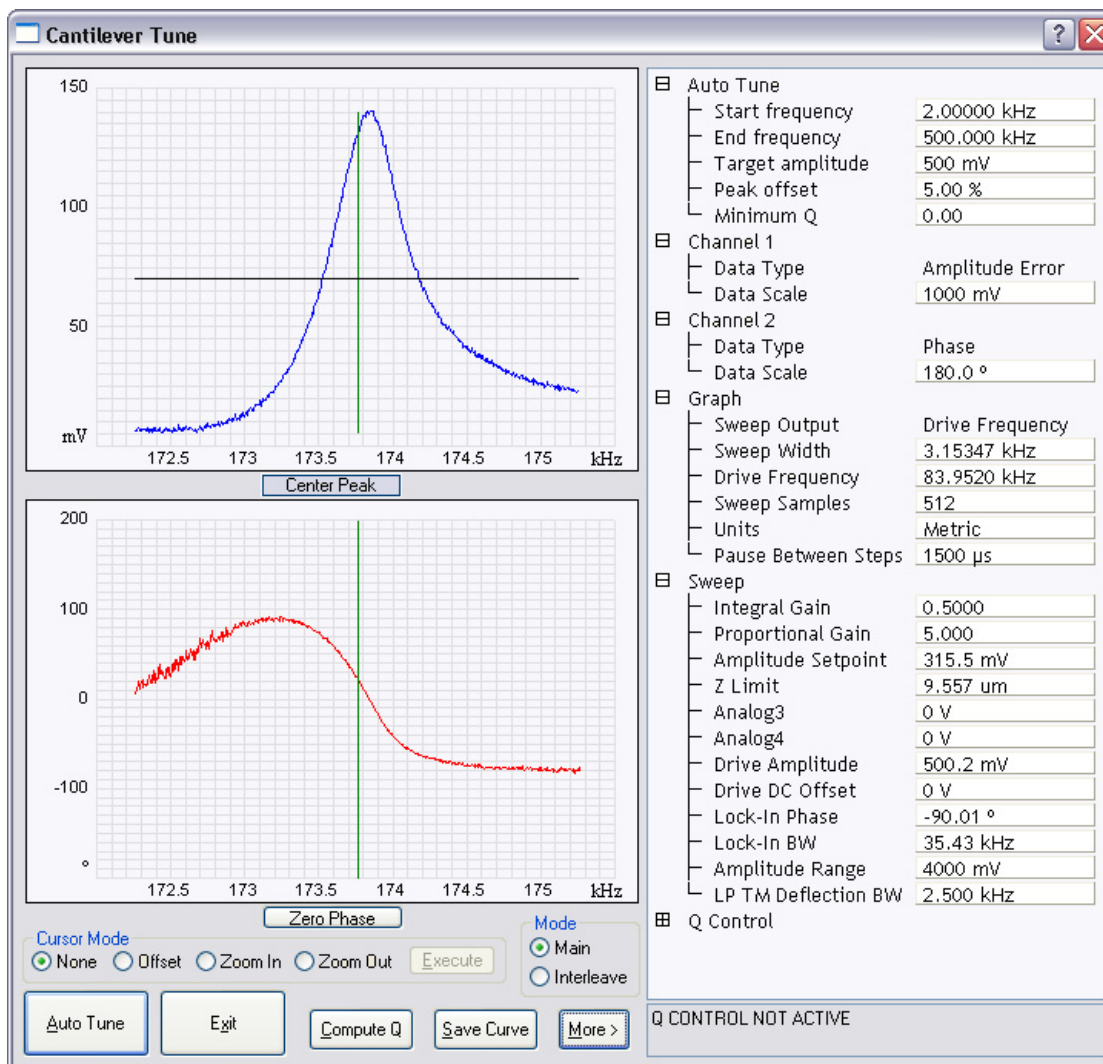
10. Specify the **RMS amplitude** after tuning the cantilever to its resonant frequency. The desired operating amplitude depends on the sample and other scanning conditions.

Figure 10.3f Cantilever Tune Frequency Sweep

Frequency Sweep

Cantilever Response

Setpoint

Center Frequency - 337.8723 KHz
0.05 KHz/div
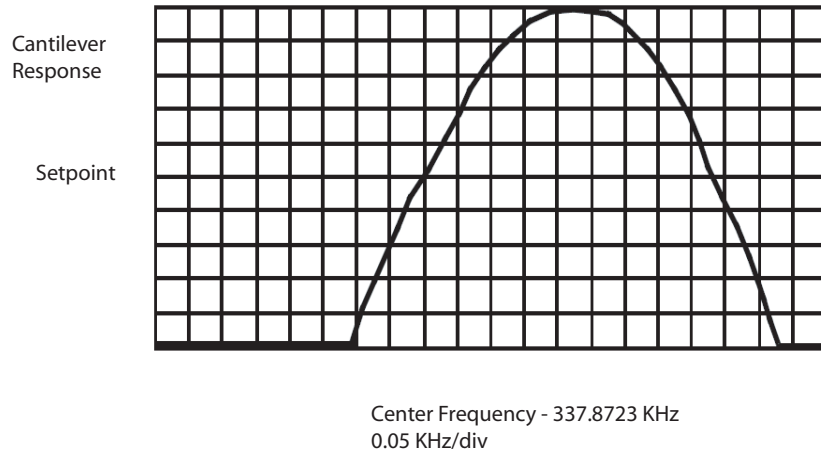
11. Click **AUTO TUNE**. The parameters set in the **Cantilever Tune** control panel appear in the **Realtime** control panel.

12. Click **ABORT TUNING** to exit the **Cantilever Tune** command and leave the parameters unchanged.

10.3.5 Optimization of Scanning Parameters

The user is encouraged to review Section 9.2.2 which discusses parameter optimization for Contact AFM.

10.3.6 Data Type

Data Type is the first parameter to set because the values of other parameters depend on it. The **Data Type** parameter in the **Channel 1**, **Channel 2** and **Channel 3** panels selects the type of data collected by the system. **Height** data corresponds to the change in piezo height needed to keep the amplitude of the cantilever constant. **Amplitude** data measures the change in amplitude relative to the amplitude setpoint.

The scan parameters required to collect good **Height** data are different than the optimal parameters for **Amplitude** data. To collect **Height** data while tracking the sample surface with minimal change in the tip's oscillation amplitude, the feedback gains must be high.

> **CAUTION:**   Do not conduct TappingMode microscopy with low feedback gain values, as this will cause damage to both the tip and sample. The maximum amplitude of the cantilever oscillation is not sufficient to track tall features.

Amplitude data collected with high feedback gains is the derivative of the height. **Amplitude** mode provides a sensitive edge detection technique. It is possible to capture both **Height** and **Amplitude** data simultaneously.

10.3.7 Gain Settings

The **Integral** and **Proportional Gains** on the **Feedback** panel must be high enough to force the feedback system to track the sample surface. When scanning in TappingMode, set the **Integral** and **Proportional Gains** to lower values than those values used in Contact mode. The **Proportional Gain** can usually be set 30%-100% higher than the **Integral Gain**. To optimize the gains, increase the **Integral Gain** until the piezo begins to oscillate (feedback oscillation usually occurs with **Integral gains** of **10-20)**, then eliminate the oscillations by reducing the gain with two or three clicks of the left arrow key. Repeat the process for the **Proportional Gain**.

10.3.8 Scan Size, Scan Rate, and Setpoint

The **Scan Size**, **Scan Rate**, and **Setpoint** values affect data output differently. As in Contact mode, decrease the **Scan Rate** as the **Scan Size** is increased. Use **Scan Rates** of **0.5-1.0 Hz** for large scans on samples with tall features. High scan rates help reduce drift, but only use them on flat samples with small scan sizes.

The **Amplitude Setpoint** parameter defines the desired voltage for the feedback loop. The **Amplitude Setpoint** voltage is constantly compared to the present **RMS amplitude** voltage to calculate the desired change in the piezo position. When the gain values are high, as they should be when the **Data Type** is set to **Height**, the Z piezo position changes to keep the amplitude voltage close to the **Amplitude Setpoint**; therefore, the oscillation amplitude remains nearly constant.

As discussed above, changing the **Amplitude Setpoint** alters the response of the cantilever oscillation and changes the amount of force applied to the sample surface.

The force applied by the tip on the sample increases as the **Amplitude Setpoint** is decreased. To minimize the force on the sample:

- Increase the **Amplitude Setpoint** using the right arrow key on the computer keyboard until the tip is no longer interacting with the surface. This can be observed by monitoring the trace and retrace lines in the oscilloscope display and by monitoring the **Z Center Position**. When the tip is no longer interacting with the surface, the Z Center Position will move to the retracted limit.

- Slowly decrease the amplitude setpoint using the left arrow key on the keyboard until features appear in the trace and retrace lines in the oscilloscope display.

10.3.9 Surface Tune

It is often desirable to check the cantilever tune after engaging.

A warning, shown in Figure 10.2h, appears indicating that cantilever tuning while engaged can result in damage to the tip or sample. This warning prompts you to enter a **Tip offset**. This is the distance above the sample that the tip will be raised before displaying the tuning spectrum. Use a minimum **Tip offset** of **100-200nm**.

Observe the plot of amplitude vs. driving frequency to see if the cantilever tuning curve shifted during the engagement. The drive frequency can be adjusted to compensate for such a shift by using the **Offset** command located in the **Cantilever Tune** plot. The phrase response curve can be adjusted as well.

10.4   Troubleshooting

10.4.1   Frequency Response Plot

If a peak in the frequency response plot does not appear, perform the following steps:

1. Increase **Sweep Width** to the maximum value.

2. Increase the **Drive Amplitude** in intervals of 200-300mV until you have reached 2-3V.

3. If the peak still has not appeared, then increase the **Sweep Width** by first increasing the **Center Frequency**, then maximizing the **Sweep Width**. If there is still no peak on the response plot, check the laser alignment.

10.4.2   Engaging the Sample

If the engage aborts because the tip is still too far away from the surface, use the Tip Down motor control to bring the tip and surface closer together. After successful re-engaging, a well formed image appears.

10.4.3   Cantilever Will Not Tune

If the Cantilever Tune fails, check the following:

- Ensure the laser is properly aligned at the free end of the cantilever (the end furthest from the substrate).

- Ensure there is good physical coupling between the probe and the probe holder. Make sure that the groove the probe is held in is free of debris, and that the probe substrate is in firm contact with the back wall of the groove and is pushed firmly against one side of the groove.

Chapter 11 Fluid Operation

This chapter addresses scanning probe microscope (SPM) imaging of samples in fluid using a MultiMode. Refer to the following for your specific area of interest:

- **Introduction:**

- **General Fluid Operation:**

  - **Clean Fluid Cell and O-ring:**

  - **Select Mode of Operation:**

  - **Select the Probe:**

  - **Remove Organic Contamination from the Tip:**

  - **Load the Fluid Cell with a Probe:**

  - **Sample Mounting:**

  - **Align the Laser:**

  - **Adjust the Detector Offsets and Setpoint (Contact Mode):**

  - **Engage the Surface:**

  - **Adjust Scan Parameters (Contact Mode):**

  - **Clean and Dry Parts When Done:**

- **Tapping Mode in Fluids:**

  - **Procedure for Tapping Mode Imaging in Fluid:**

  - **Optimizing Image Quality:**

- **Troubleshooting Tips:**

  - **Cantilever Tune Plot Looks Poor: Loose Probe:**

  - **Laser Sum Signal Absent or Weak: Air Bubbles:**

11.1 Introduction

Imaging of samples in fluid is a growing application of AFM technology. This may be prompted by a desire to minimize surface forces on delicate samples, the need to observe biological specimens in their natural, fluid environments, and/or the necessity to make real time observations of samples undergoing electrochemical reactions (ECAFM). In order to conduct ECAFM observations with electrical potentials, it is necessary to connect an external potentiostat unit. Contact Bruker for more information.

Imaging samples under fluid eliminates attractive forces due to surface tension. This enables the sample surface to be imaged with a minimum of cantilever tip force—a decided advantage when imaging biological specimens and delicate materials.

Essentially, the procedure for observing samples under fluid is the same as that for Contact Mode or TappingMode AFM in air; however, special hardware is utilized to contain the fluid. In addition, minor adjustments must be made to correct for refractive effects as the laser beam transits air-fluid boundaries.

This chapter describes Contact Mode and TappingMode AFM operation of the MultiMode SPM in fluid, including loading the probe into the probe holder, mounting the probe holder into the head and aligning the laser on the cantilever portion of the probe and then engaging the probe tip with the sample.

This chapter assumes familiarity with Contact Mode and TappingMode AFM operation of the MultiMode in air. If you are not familiar with air operation of the MultiMode, refer to the procedures outlined in Chapter 9 and Chapter 10 before attempting to operate the MultiMode AFM with a fluid cell.

11.2 General Fluid Operation

The fluid cell consists of a small glass assembly with a wire clip for holding an AFM probe (see Figure 11.2a). The glass surfaces provide a flat interface so that the AFM laser beam may pass into the fluid without being distorted by an unstable fluid surface. The probe is mounted in a rectangular groove on the bottom of the fluid cell, and held in place by a gold-coated wire clip. A circular groove surrounds the probe and is used to seat the (optional) O-ring. Additionally, there are at least two fluid ports located on the front side of the fluid cell. These ports allow for the introduction and removal of fluid.

Figure 11.2a   EC TappingMode Fluid Cell

There are two fluid cells that are commonly used for fluid imaging:

- Model MTFML is a fluid cell that can be used for imaging samples in fluid using either Tapping Mode or Contact Mode.

- Model MMTMEC is a fluid cell that can be used for imaging samples in fluid using either Tapping Mode or Contact Mode and has additional port for the insertion of a reference electrode and a counter electrode.

  Note: It is necessary to connect an external potentiostat to perform electrochemical measurements. Contact Bruker for more information.

The fluid cell comes with the following accessories:

- 10 glass cover slips (for use as sample substrates)

- silicone tubing

- 5cc syringe

- 4 male Luer fittings

- 2 female Luer fittings

- 2 tube clamps

- 6 silicone O-rings

- 6 fluorosilicon O-rings

- Silicone grease

- Lens paper

The male Luer fittings can be inserted into the fluid ports on the front of the fluid cell, and the opposite side of the fitting is connected to the silicone tubing. The female Luer fittings connect the silicone tubing to the syringe.

Use of the O-rings is optional. Instructions for operating the fluid cell with and without an O-ring are detailed in this chapter. The previous O-ring design was based on a circular cross-section. The new O-ring design has an "S" shaped cross-section with thin, flexible walls. This new design makes insertion of the O-ring easier, reducing the chances of leaking and minimizing imaging artifacts caused by lateral friction.

11.2.1 Clean Fluid Cell and O-ring

To reduce contamination problems and to obtain high-quality images, clean the fluid cell, and O-ring if applicable, as follows:

1. While soaking the fluid cell and O-ring in warm, soapy water, place a few drops of liquid dish soap on them. Use nonabrasive soap to prevent scratching the glass surfaces.

2. Gently rub the fluid cell and O-ring with a cotton swab or finger. Avoid scratching the glass surface with abrasive material.

3. Using distilled water, rinse the fluid cell and O-ring of all soap.

4. Using 0.2µm-filtered, compressed air or dry nitrogen, blow dry the fluid cell until all moisture evaporates.

11.2.2 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 11.2b.

Figure 11.2b   The Contact Mode in Fluid **Select Experiment** window
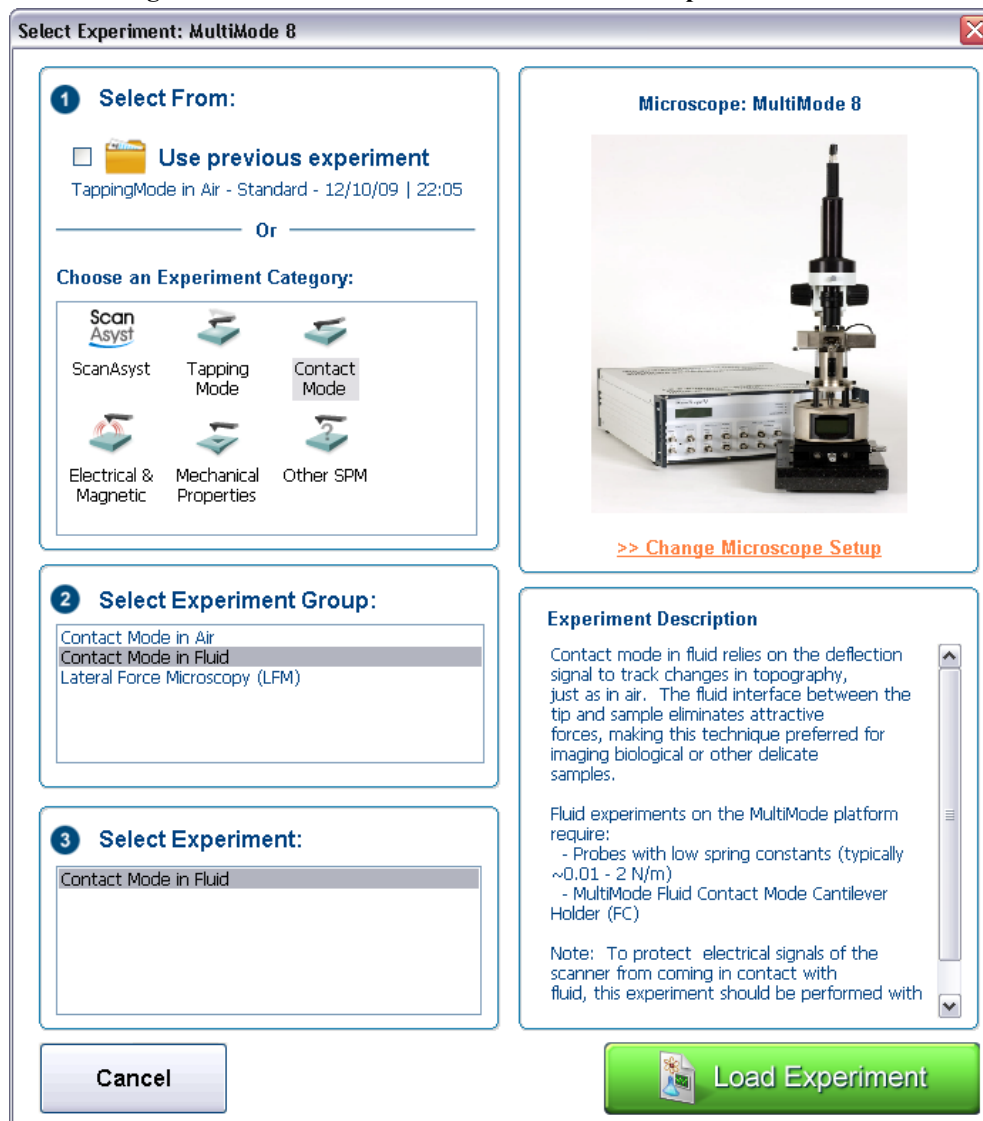
2. Chose a mode that supports imaging in fluid: SCANASYST, TAPPING MODE OR CONTACT MODE in the **Choose an Experiment Catagory** panel.

3. Select SCANASYST IN FLUID, TAPPINGMODE IN FLUID or CONTACT MODE IN FLUID in the **Select Experiment Group** panel.

4. Select SCANASYST IN FLUID, TAPPINGMODE IN FLUID or CONTACT MODE IN FLUID in the **Select Experiment** panel and click LOAD EXPERIMENT.

5. This opens the **Workflow Toolbar**, the **Scan 1** or **2 Channels** (depending on the mode) windows and the **Scan Parameters List** window, shown in Figure 11.2c.

Figure 11.2c   Contact Mode in Fluid (Simple) configuration
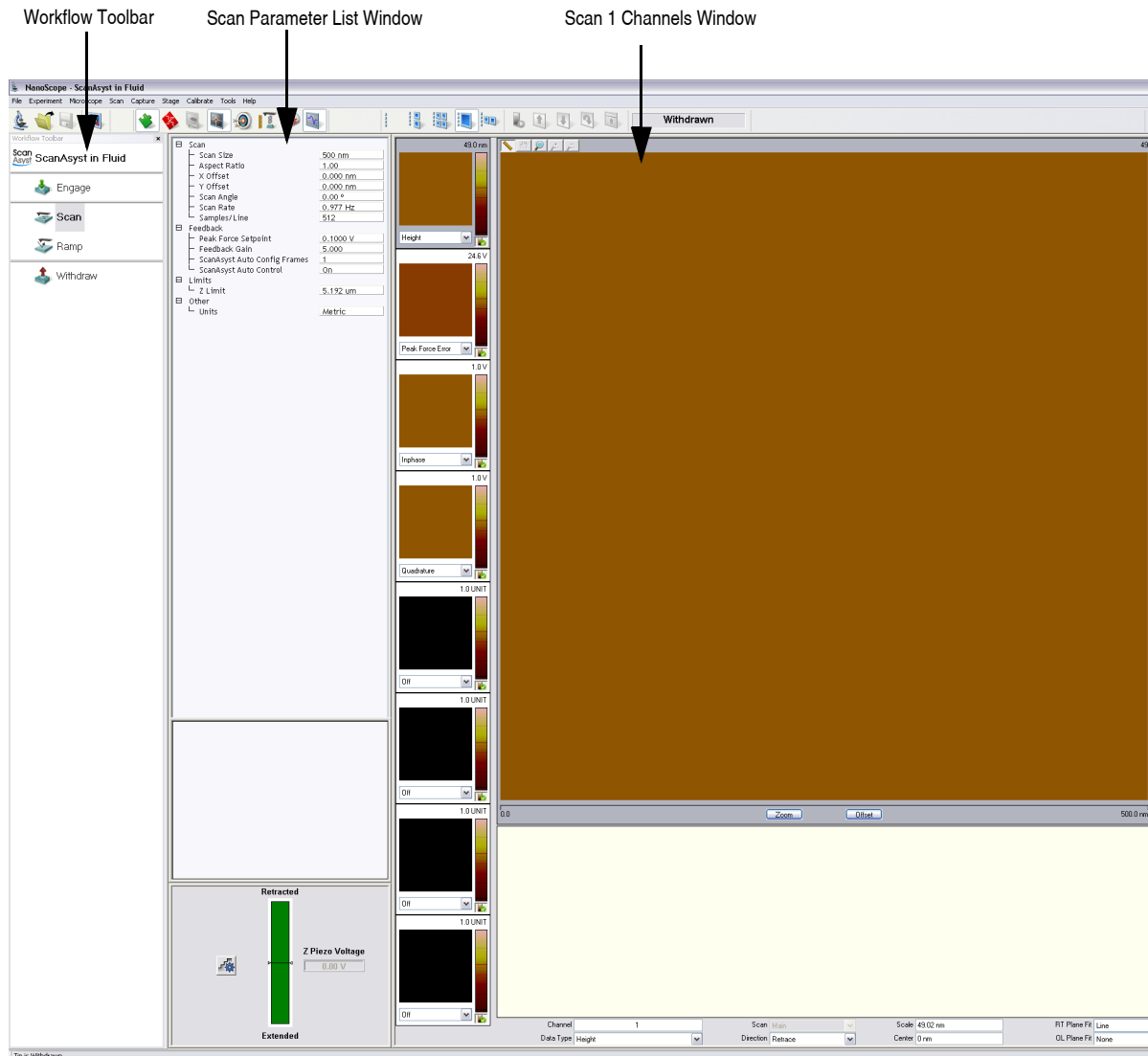
11.2.3 Select the Probe

AFM probes featuring low stiffness cantilevers produce the best results for biological applications. We recommend sharpened tips to start: 100μm ("short"), V-shaped cantilevers with oxide-sharpened silicon nitride tips; for example, models NP-S (standard) or NP-STT (oriented twin tip). Stiffer etched silicon probes (model FESP, single beam, 225μm long) used at very small oscillation amplitudes also obtain good results. Experiment to find which probes work best for your sample.

> **Note:** For additional information on selecting a probe, please refer to the applications note "*Choosing AFM Probes for Biological Applications.*"

11.2.4 Remove Organic Contamination from the Tip

Contaminants on the tip may limit AFM resolution. You may use ultraviolet (UV) light to remove contaminants, as follows:

1. Place the fluid cell with installed tip face-up on a clean surface.

2. Position a UV lamp very close (3-5mm) to the fluid cell and irradiate the probe for 15-30 minutes at full intensity.

> **Note:** Washing probes in 1-5% SDS (Sodium Dodecyl Sulfate) is also effective. Glass areas may be cleaned using isopropyl alcohol or acetone.

11.2.5 Load the Fluid Cell with a Probe

The probe is held in a small pocket on the bottom side of the fluid cell by a gold-plated, stainless steel wire clip. A tiny coil spring mounted on the top of the fluid cell holds the wire clip against the probe.

Load a probe into the fluid cell by performing the following procedure:

1. Hold the fluid cell with one hand and use the other hand to gently raise the wire clip by pressing the spring from beneath.

> **Note:** Do not press harder than enough to completely compress the spring!

2. Use the clear ball to the side of the spring to rotate the wire clip away from the pocket.

3. Use tweezers to slide a probe into the pocket. Lower the clip to hold the probe (see Figure 11.2d).

4. Verify that the probe is squarely set against one side of the pocket and flush against the back. Verify the probe is held firmly by the wire.

⚠️	**CAUTION:**	Avoid scratching the fluid cell glass surface with the tweezers or the probe, especially in the area under the probe.

Figure 11.2d Load Probe into Fluid Cell
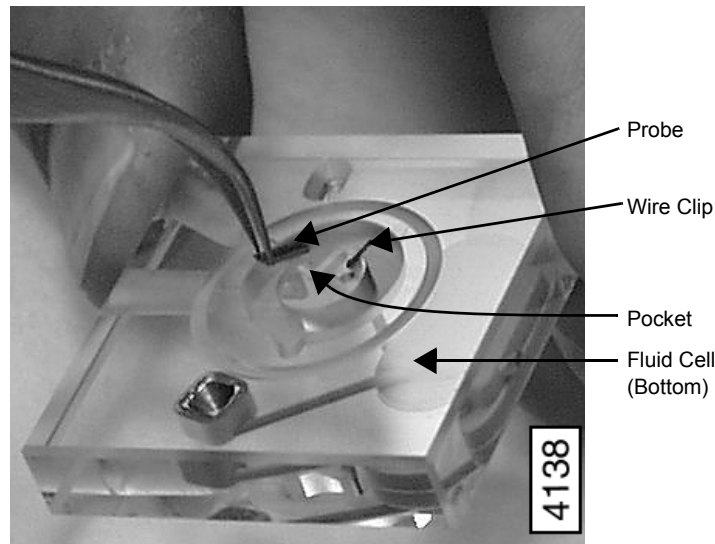
11.2.6 Sample Mounting

Secure a sample support (e.g., mica or a glass cover slip) to a magnetic stainless steel sample puck. Supports may be secured to the puck with epoxy. Select epoxy as follows:

- For non-critical applications, use Devcon 2-Ton Epoxy or 5-Minute Epoxy.

- For applications where contamination control is more critical, use a more inert, solvent-free epoxy such as Master Bond EP21LV or EP21AR or a hot melt adhesive.

  Note: Follow the manufacturer's directions for mixing and curing to obtain the best resistance to leaching and chemical attack.

> **CAUTION:**   Do not use cyanoacrylate glue (e.g., SuperGlue™) for mounting samples in fluid.

Method 1 (with an O-ring)

The O-ring protects the AFM scanner tube from spilled liquids. To mount the sample with an O-ring:

1. Install the protective O-ring into the fluid cell. Insert the O-ring into the recessed groove in the underside of the fluid cell. The O-ring slides up into the recessed groove.

2. Install the sample or sample support onto the scanner and insure the scanner is lowered enough to avoid damaging the probe when the head is mounted.

3. Mount the head carefully to avoid damaging the sample and secure the head in place with the two mounting springs.

4. Install the fluid cell in the AFM head and tighten the clamp to hold the fluid cell in place. Ensure that the O-ring is positioned properly between the sample and fluid cell. Position the fluid cell in the head to ensure that the O-ring creates a seal on the sample around the periphery of the fluid cell and does not overlap any edges of the sample. If necessary, use the XY translation knobs on the head to center the O-ring.

Figure 11.2e Fill the Fluid Cell with Liquid
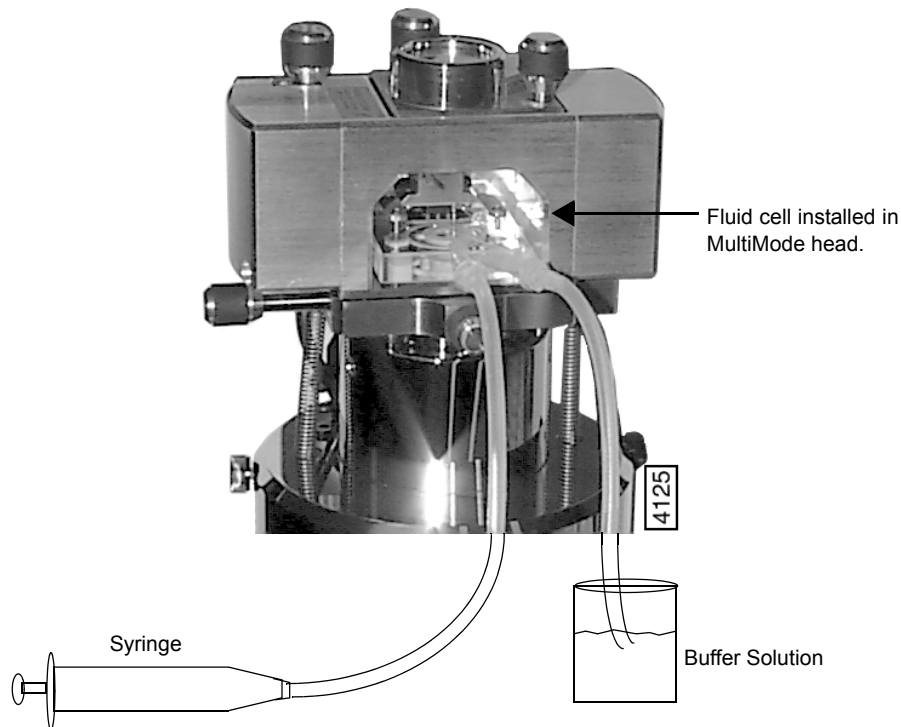
5. Verify the head is leveled side-to-side and that the head is tilted slightly forward, so the tip is level when it contacts the surface.

   Note: If you are using a vertical-engage scanner, leveling problems are eliminated.

6. Using the **Tip Down** switch on the MultiMode base (and the coarse adjustment screws if not using a vertical engage scanner), lower the tip toward the sample until the tip is within ~30µm of the surface. This will bring the fluid cell close enough to the surface to ensure that the O-ring will create a seal against the sample.

7. Fill the fluid cell with liquid.

Note: To minimize the risk of fluid leakage, draw fluid to the fluid cell using the following technique:

• Attach a piece of silicone tubing to one of the fluid ports using a male Luer fitting. Place the free end of the tubing into a beaker containing the buffer.

• Attach a piece of silicone tubing to the other fluid port using a male Luer fitting.

• Fill the fluid cell with fluid by withdrawing the plunger on the syringe. This will draw fluid from the beaker containing the buffer through the fluid cell.

• Check for leaks and wick away any spilled liquid with filter paper until AFM components are dry.

8. Bubbles inside the fluid cell near or on the probe can interfere with the laser beam. Remove bubbles by performing the following procedure:

   a. Observe the fluid cell and probe through the viewing port using the OMV.

   b. Rapidly pull liquid through the cell with a syringe. If sufficient force is applied, the bubbles will be carried out of the fluid cell.

Method 2: Without an O-ring

⚠️	**CAUTION:**	Without the O-ring, this method poses a potential spill hazard to microscope electronics and must always be undertaken with extreme caution. Limit the volume of liquid in the fluid cell to 30-50µl when possible.

In many cases, it is possible to image a sample under a drop of fluid without use of a closed fluid cell. This technique is recommended for aqueous buffers when evaporation is not an issue (for example, in short experiments involving no use of volatile solvents).

Limit risk of leakage as follows:

• Use a sample support larger than the puck.

• Use the recommended adhesive Loctite 770 to affix a Teflon cover over the steel sample puck. The Teflon should extend slightly over the edge of the sample puck. Use epoxy to attach the sample support to the Teflon.

The hydrophobic Teflon helps confine the solution without installing an O-ring (see Figure 11.2f).

Figure 11.2f Stainless Steel Sample Puck with Teflon Cover
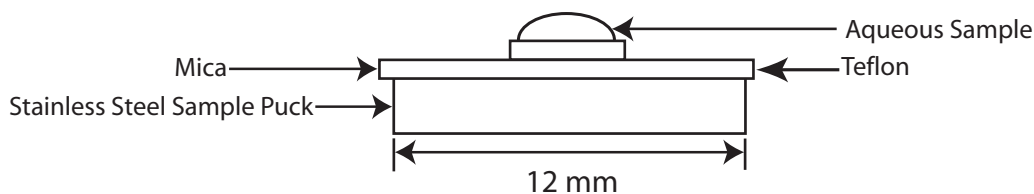
You may load the sample on the support now, or you may inject it when the fluid cell is installed inside the AFM head.

Two variations of this method are possible: starting with a dry sample and starting with a sample in solution.

1. Complete the following if starting with a dry sample:

    a. Attach the sample support (e.g., mica) to a puck.

    b. Deposit your sample on the support.

    Note: It is easier to first align the AFM head and mounted fluid cell (probe installed) with the dry sample puck before installing the liquid-coated sample. Allow 0.5mm clearance between the tip and dry substrate surface.

    c. Occasionally air bubbles form in the fluid cell and block laser light. Reduce the chance of forming bubbles as follows:

    •   Before installing the fluid cell into the head, insert a syringe filled with liquid solution into a fluid port, or connect the syringe to the inlet tubing on the fluid cell.

    •   Push enough fluid through the fluid chamber to flood the fluid cell port with buffer solution, allowing liquid to drip out the bottom of the cell.

    •   Leave the buffer-filled syringe inserted. A small amount of buffer solution should be held to the bottom of the cell by surface tension (see Figure 11.2g).

Figure 11.2g Flush the Fluid Cell Before Installation to Reduce Bubble Formation
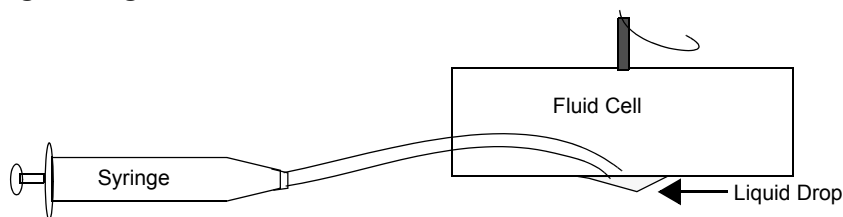
Note: The liquid is held by surface tension between the sample surface and the fluid cell (see Figure 11.2h). Because the aqueous sample changes the light deflection, the laser photodetector must be re-adjusted.

Figure 11.2h Imaging a Sample Covered by a Drop of Fluid
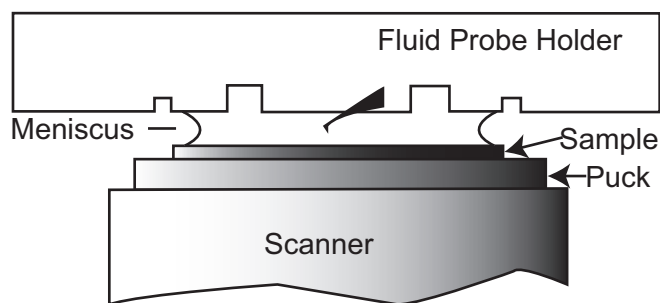
2. Complete the following if starting with a sample in solution:

    a. Place 30-40μl of your sample on the support mounted on the puck (the liquid should form a small dome over the support). During this placement, the sample should adhere to the support (e.g., mica).

    b. Install the sample support on the AFM scanner.

    c. Carefully install the fluid cell inside the head.

       • Inspect from the front to make sure the fluid is well confined to the mica area only.

       • Aim the laser on the cantilever and adjust the photodiode detector position (see Section 11.2.8).

    Note: Always verify that the microscope is dry and that all MultiMode surfaces are free of spilled fluid. Wick away moisture and droplets with filter paper.

    Note: Over time, evaporation of the fluid may necessitate replenishing the fluid cell using a standard micropipette or syringe.

11.2.7 Align the Laser

Use either of the techniques for aligning the laser on the probe (see Chapter 7, **Laser Alignment: Section 7.2.**) The following considerations apply:

- Refraction causes the laser beam path to bend slightly when entering and exiting the fluid surrounding an immersed probe as compared to the path in air. However, the basic process is essentially the same.

- It may be necessary to adjust the tilt of the mirror to position the laser on the photodetector. The laser position on the tip will not change with the introduction of fluid but the mirror will require adjustment.

- Although the top surface of the fluid cell has an anti-reflection coating, a faint reflection may still be visible. This reflection from the glass surface does not affect operation of the MultiMode, but it can be a source of confusion when aligning the laser. Ignore this faint reflection and focus on the much brighter reflection from the cantilever.

- The **Sum** signal on the display monitor typically displays less than **1V** when the laser is not aligned on the probe. The **Sum** signal should rise well above **1V** when the laser is reflecting off the probe.

11.2.8 Adjust the Detector Offsets and Setpoint (Contact Mode)

- Typically, samples are softer in liquid than in air. Before engaging, verify that there is not so large a difference between the setpoint and the vertical deflection signal as to damage the sample.

- Verify reasonable values for scan parameters (e.g., **Scan Rate**, **Scan Size**, **Integral Gain** and **Proportional Gain**). Gains are typically lower in fluid than in air.

- Once engaged, reduce the setpoint to minimize the tracking force.

11.2.9 Engage the Surface

1. Using the **Motor down** switch (and coarse adjustment screws if not using a vertical engage scanner), lower the tip until it is just above the level of the sample surface.

   • The **Motor down** switch is located on the MultiMode base.

2. Select **MICROSCOPE > ENGAGE** or click the **ENGAGE** icon on the toolbar. A pre-engage check begins, followed by Z-stage motor motion.

   • The motor begins to move the AFM head and probe down to the sample.

   • When the tip reaches the surface, the system should automatically stop, beep, and begin to image the sample.

   • In Contact Mode, if the system engages immediately or before the tip reaches the surface, try increasing the **Setpoint** approximately **2.0V**, then repeat this step.

11.2.10   Adjust Scan Parameters (Contact Mode)

1. Once engaged, adjust the scan parameters to obtain the best image.

   Note: This procedure is similar to operation in air, except that samples are often softer in fluid.   Adjusting the applied force can be critical.

2. To avoid sample damage, reduce the **Deflection Setpoint** as low as possible:

   a. Stop when the tip pulls off the surface and the **Z Center Position** in the **RealTime Status** window jumps to **Limit** (**-220V**).

3. Increase the setpoint until the tip begins to touch the surface again and an image appears.

   • As an alternative, adjust the setpoint using Force Calibration. Refer to Chapter 14 for details.

   • Because the tip typically adheres to the sample surface much less in fluid than in air, it is possible to image at much smaller contact forces in liquid.

   • The optimal integral and proportional gains and scan rate may be different from air operation, because the dynamics of the cantilever change in fluid.

4. Set the two gains as high as possible (starting with the integral gain) without causing oscillation distortion to appear in your image.

5. Observe the agreement between the trace and retrace lines and adjust the **Setpoint**, **Gains** and **Scan Rate** to bring the trace and retrace lines into coincidence. This procedure will be similar to operation in air, with the following exceptions:

- Samples are generally softer in fluids. Adjusting the applied forces becomes more critical.

- Scan rates tend to be slower in fluid.

6. Choose a scan rate that is sufficiently slow to image without degrading your data.

11.2.11   Clean and Dry Parts When Done

1. When sample imaging is complete, drain the fluid cell and carefully remove it from the head. Avoid spilling fluid.

2. Rinse and dry the fluid cell, and O-ring if applicable, to prevent the buildup of salts or other contaminants on these parts.

	CAUTION:	When cleaning the fluid cell, use care to avoid scratching the glass surfaces in the center of the fluid cell where the laser beam passes.

11.3 Tapping Mode in Fluids

Operation of tapping mode in fluid provides the same advantages of tapping mode in air, with the additional ability to image samples under native liquid conditions. In fluid tapping mode, the probe is oscillated so that it only intermittently contacts the sample surface. This can reduce or eliminate lateral forces that can damage soft or fragile samples in Contact Mode. The following sections provide general instructions for tapping mode imaging in fluid.

> **Note:** Before attempting tapping mode in fluids, it is recommended that the user becomes familiar with standard tapping mode operation in air (see Chapter 10) and Contact Mode in fluid.

11.3.1 Procedure for Tapping Mode Imaging in Fluid

1. Follow the directions described in the Contact Mode section (see **Section 11.2.1** on page 194) to clean the fluid cell and O-ring. Clean and mount a probe and then the sample.

2. Align the laser on the free end of the cantilever portion of the probe.

3. Adjust the mirror to maximize **SUM.**

4. Center the photodiode to give a deflection signal near **0V**.

5. Set up the system for tapping mode operation:

6. Set the mode selection switch on the left side of the MultiMode base to **TM AFM**.

7. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 11.3a.

**Figure 11.3a**   The TAPPINGMODE IN FLUID **Select Experiment** window
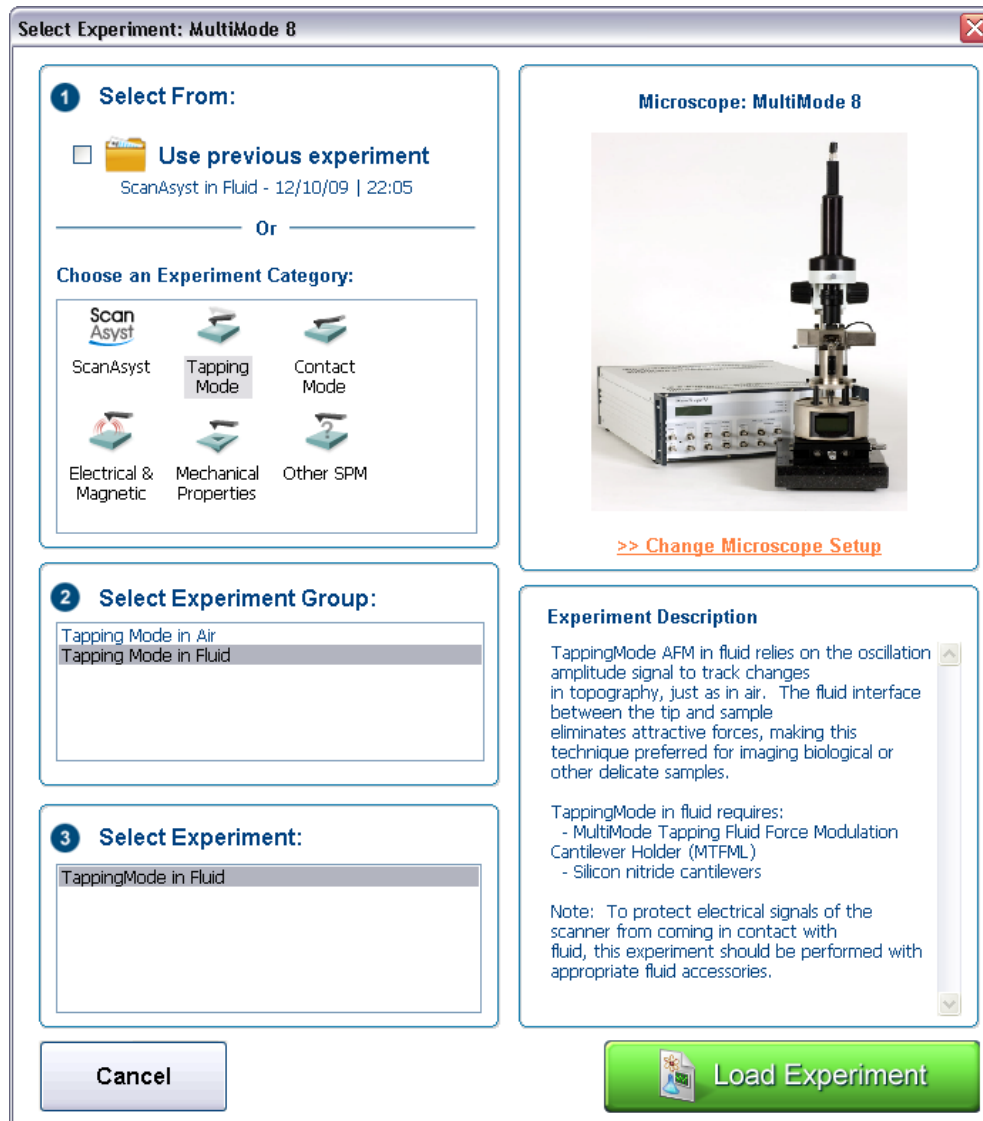
8. Select TAPPINGMODE AFM in the **Choose an Experiment Catagory** panel.

9. Select TAPPINGMODE IN FLUID in the **Select Experiment Group** panel.

10. Select TAPPINGMODE IN FLUID in the **Select Experiment** panel and click LOAD EXPERIMENT.

11. Manual Cantilever Tune:

- This is similar to the Cantilever Tune process used for standard tapping mode in air. Unlike operation in air, the cantilever resonance will be largely damped by liquid and the Auto Tune function cannot be used.

- When viewing a wide bandwidth cantilever tune sweep, observe that there is no single, well-defined resonance peak, but, rather a number of broader maxima. Manually select a peak.

- Select **MICROSCOPE > CANTILEVER TUNE** or click the **TUNE** icon on the NanoScope toolbar to select a drive frequency. The optimal drive frequency can depend upon sample, fluid and fluid volume inside the fluid cell. Experiment to find the best drive frequency for specific imaging conditions. Two frequency ranges that are commonly used are 16-19kHz and 8-12kHz; higher frequencies have also been used. Start with a **Sweep width** of 20kHz. Users of TappingMode in air will notice that there is not a single well-defined resonance, but instead a large number of broad peaks. The peaks are resonances of the fluid cell and fluid, and do not usually depend so much on the cantilever dimensions.

  Note: For the short, narrow Silicon Nitride probe recommended for soft samples (e.g., model DNP), the resonant frequency in fluid is a broad peak centered around 10kHz (see Figure 11.3b). Best results are achieved by tuning the cantilever to a peak between 7-12kHz. Higher and lower frequencies have also been used depending on the type of probe employed. Start with a **Sweep Width** of **20kHz** and a **Drive Frequency** of **10kHz** in the **Sweep** panel.

Figure 11.3b A 100μm, Narrow-legged, Si_3N_4 Cantilever Fluid Tune Curve
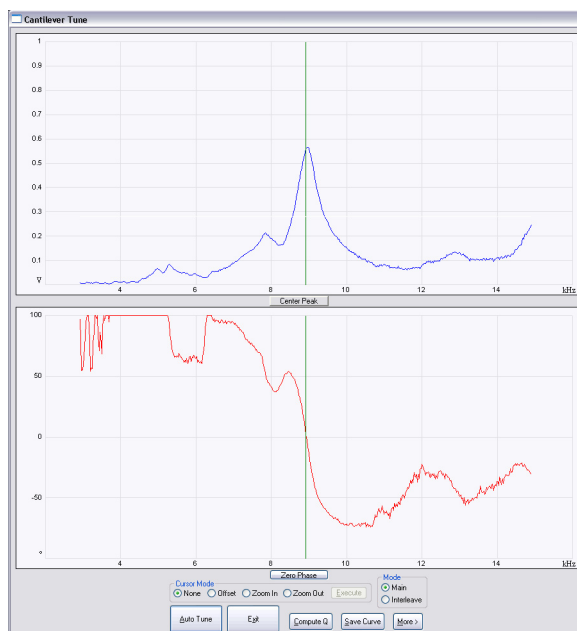
- Manually adjust the **Zoom In** and **Offset** functions in the **Cantilever Tune** window.

Note: If the expected peak does not appear in the spectrum, choose another peak, engage on the surface and disengage immediately. With the tip closer to the surface, the peak at 8 to 9 kHz appears.

Adjust the **Drive Amplitude** until a desired probe RMS amplitude is obtained.

- An RMS amplitude of **150 mV** is appropriate for soft samples and typically results from a **Drive Amplitude** of **250-500mV**. A **Drive Amplitude** >**0.3V** generally works poorly with soft samples.

- For rougher samples, target **300-600 mV** RMS amplitude. Experiment to find what level of probe oscillation gives best results for specific applications.

a. Set the **Scan Rate** to **1Hz**.

b. Set the **Integral Gain** to **0.5**.

c. Set the **Proportional Gain** to **0.7**.

d. Set the data **Scale** to **50nm**.

e. Select the Channel 1 **Data Type** as **HEIGHT**.

f. Select the Channel 2 **Data Type** as **AMPLITUDE ERROR**.

g. Optional: Select the Channel 3 **Data Type** as **PHASE**.

- The RMS amplitude must be adjusted when the AFM tip is near the sample surface (<50μm).

- Verify that the tip is very close to the sample surface when tuning the probe in fluid.

- Adjust and optimize these settings for each imaging condition and sample.

12. Center the laser spot on the photodiode detector.

- Adjust the photodiode until deflection is roughly zero.

- The deflection signal can drift when the probe is first in fluid, so it is best to adjust just prior to engaging.

13. Click the **ENGAGE** icon on the toolbar.to bring the tip into tapping range.

- The NanoScope software automatically selects a setpoint, then stops the engagement when the surface is detected.

14. Adjust the setpoint when engaged.

- The best topographic images are usually obtained at setpoints 10-20 percent less than the RMS amplitude before engaging.

- The setpoint may be optimized by observing and optimizing the image quality.

- It may be necessary to check the cantilever tune after engaging. Click the **TUNE** icon on the NanoScope toolbar. When prompted for a **Tip Offset**, enter **100-200nm**. Observe the cantilever tune spectrum and the offset the **Drive Frequency** if required.

11.3.2 Optimizing Image Quality

Adjust the setpoint by monitoring image quality, as follows:

1. Select an appropriate Scan size. Wait until the current scan line is over a tall feature, then **DISABLE** the **Slow Scan Axis** (Expanded Mode). Check the agreement between the Trace and Retrace displays.

2. Increase the setpoint in small increments until the probe pulls off the surface and the **Z Center** position voltage goes to retracted (i.e., **-220V**).

3. Reduce the setpoint in small increments until an image appears.

4. Continue reducing the setpoint until the image is optimized.

   - The best images are obtained at setpoints just below where an image appears.

   - The NanoScope Controller attempts to keep the cantilever oscillation amplitude constant during the scan.

5. Optimize the **Integral gain** and **Proportional gain** so the **Height** trace shows the sharpest contrast and there are minimal variations in the **Amplitude** trace (the error signal).

6. Optimize the **Scan rate** to obtain the sharpest image.

11.4 Troubleshooting Tips

11.4.1 Cantilever Tune Plot Looks Poor: Loose Probe

The **Cantilever Tune** plot can be used as a diagnostic tool. Become familiar with its characteristics when good images are obtained. If the plot looks substantially different from previous successful experiments, there may be a problem with the fluid cell. For example, the probe may be loose in its holder.

Check the clip which holds the probe in place, and verify the probe is not loose. Contact Bruker for assistance if needed.

11.4.2 Laser Sum Signal Absent or Weak: Air Bubbles

Verify that all bubbles are removed from the probe. Bubbles may attach themselves to the probe, causing the laser beam to be diffracted. While bubbles can sometimes be removed by forcing fluid through the fluid cell, it is often necessary to dry the fluid cell with absorbent paper (e.g., Kimwipe).

Degassing the fluid prior to use in the AFM reduces bubble problems.

11.4.3 Poor Image Quality

Contaminated Tip

Some types of samples (e.g., certain proteins) may adhere to the cantilever or tip. This reduces resolution, resulting in blurred images.

If tip contamination is a problem, it is necessary to protect the tip against contamination. There are two ways this may be accomplished:

1. If the sample is adhered to a surface through adsorption (e.g., diffusion of protein onto mica):

    a. Diffuse the sample substance into the substrate, then rinse with buffer.

    b. Lower the tip into a fluid containing little or no stray substances which may adhere to the tip.

2. If the sample is short-lived and must be imaged quickly, it may be possible to mask the tip against contamination by bringing the tip into gentle contact with an uncontaminated substrate surface:

    a. Put the MultiMode into Contact Mode and engage the substrate surface using a **0.1nm Scan Size**.

    b. While the tip is kept in gentle contact with the substrate surface, add the sample substance to be imaged and allow it to diffuse/settle onto the substrate.

    c. After a diffusion/settling period has lapsed, quickly lift the tip from the substrate surface.

    d. Switch to **Tapping Mode** and image the sample before it becomes contaminated.

Dull Tip

Change to a new probe. AFM probe tips can become dull during use and some unused tips may be defective. Check the probe type being used. Oxide sharpened silicon nitride probes are usually much sharper than standard silicon nitride probes.

Multiple Tip

Change or clean the AFM tip. Probes can have multiple protrusions at the apex of the tip, which result in image artifacts. Features on the surface appear two or more times in an image, usually separated by several nanometers.

11.4.4 Lost Particulate Samples: Attracted to Cantilever

Some particulate samples such as proteins may prove difficult to find directly beneath a cantilever if the cantilever has remained stationary during a diffusion or settling period. This may be due to the fact that some types of particulates are more attracted to the cantilever than to the substrate intended to support them. The result is a "shadow" on the substrate directly beneath the cantilever where fewer sample individuals can be located; they are stuck to the cantilever instead. If you suspect this problem, shift the imaging site to a new location away from where the probe had remained motionless.

11.4.5 Drift in AFM Image Because O-ring Slid Across Sample Surface

Set up the fluid cell so there is minimal lateral movement of the optical head with respect to the sample once the O-ring is installed. Keep the head level while positioning the tip close to the surface to minimize tip lateral motion during engagement. Other countermeasures:

- Unlike three-point supports, the vertical engage scanner allows the tip to approach samples without lateral offset, eliminating stress on the O-ring during engagement. For more detail, contact Bruker.

- Lightly coating the area of the O-ring which contacts the sample surface with silicone grease from the fluid cell kit allows the O-ring to slide across the surface, minimizing lateral stress. This also forms a fluid-tight seal between the O-ring and sample. However, some solvents (i.e., nonpolar organic solvents) may dissolve some of the lubricant into the fluid.

- If not using a vertical engage scanner, when positioning the fluid cell over the sample surface, adjust the positioning knob at the base of the optical head to move it slightly forward. This will counter some of the lateral stress on the O-ring resulting from the optical head moving back during engagement.

11.5 General Notes on Sample Binding

Samples for AFM imaging should be immobilized on a rigid substrate. Macroscopic samples (biomaterials, crystals, polymer membranes, etc.) can be attached directly to a stainless steel sample disk with an adhesive. Dissolved or suspended samples like cells, proteins, DNA, etc. are usually bound to a flat substrate like mica or glass, for example. Many different sample preparations have been developed and SPM applications articles are an excellent source of

information on sample binding. For a list of articles describing biological applications of AFM, including sample preparation techniques, contact Bruker.

Binding Specimens to Mica

The following procedures will describe how to bind two different samples to a mica substrate. Mica is commonly used because atomically flat substrates can be simply and inexpensively prepared. In aqueous solutions, the mica cleavage surface becomes negatively charged. Specimen binding is usually accomplished using electrostatic attraction between charges on the specimen and those on the mica surface. Proteins, for example, can usually be made to stick to mica by operating at a pH where they exhibit positively charged domains. DNA, on the other hand, is negatively charged and can be bound either by altering the mica surface charge from negative to positive (using a silanization process) or by dissolving the DNA in a divalent metal counter ion (e.g. Mg^{++}, Ni^{++}). Both of these techniques are discussed separately below.

Many other techniques are being developed for chemically modifying mica, glass and other substrates to bind a variety of biological samples. Contact Bruker for a bibliography of references on imaging of biological specimens.

11.6 Lysozyme on Mica—A Model Procedure for Protein Binding

11.6.1 Protein Binding Theory

All proteins contain free amino groups that become positively charged at sufficiently low pH. If sufficient free amino groups are located on the outside surface of the protein, then the protein will bind to a negatively charged mica surface. Proteins will become positively charged at pH below their isoelectric point and are then able to bind to mica. The protein lysozyme, for example, becomes sufficiently positively charged to bind to mica at pH 6. This is shown schematically below in Figure 11.6a.

Figure 11.6a   Proteins will typically bind to negatively charged mica when the pH is reduced below the protein's isoelectric point, pI
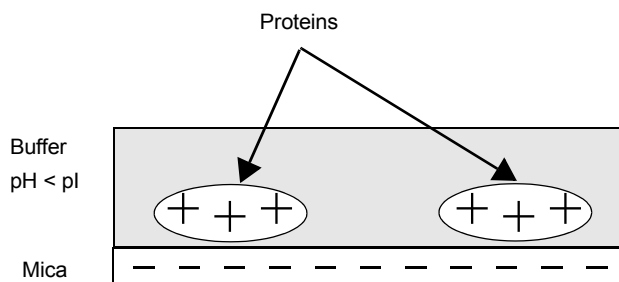
11.6.2 Protein Binding Procedure

The following section gives a detailed procedure for preparing and imaging the protein lysozyme by tapping mode in fluid. The procedure was kindly provided by Monika Fritz at the University of California, Santa Barbara and is described in the following paper:

- Radmacher, M., M. Fritz, H.G. Hansma, P.K. Hansma (1994). "Direct Observation of Enzyme Activity with Atomic Force Microscopy." *Science* **265**, 1577.

1. Obtain the required materials:

   - Deionized water

   - Mica substrates

   - Lysozyme protein L-6876 from Sigma Chemical

   - Phosphate buffer solution, 10 mM KH_2PO_4, 150 mM KCl, pH 6 (buffer may be adjusted for other proteins)

   - Tapping Mode Fluid Cell, Model MTFML

   - Cantilevers (Oxide-Sharpened Silicon Nitride tips, Model NP-S, work well)

   - Source of filtered (0.2 μm), compressed air or dry nitrogen

   - UV lamp, high-intensity; Oriel Mod. 6035 pencil-style spectral calibration lamp or equivalent (optional for cantilever cleaning).

   - Syringes: (1) 1 cc; (2) 5 cc; Micropipettes

   - Fluid cell liquid lines (silicone tubing and fittings)

   - Fluid cell o-ring (optional, see **Method 1 (with an O-ring):**).

   - Tubing clamps (for liquid lines).

   - Filter paper

2. Dissolve the lysozyme in phosphate buffer (PBS) solution to a concentration of 1μg per ml (this concentration provides a convenient coverage for AFM imaging and may be used for a variety of similar size samples). This mixture should be drawn into a clean, 1 cc syringe and capped. Prepare another 5 cc syringe of straight buffer solution.

3. Prepare the fluid cell for tapping mode in fluid operation. Clean the fluid cell and load a cantilever. For best results, clean the cantilever with UV light.

4. Cleave a fresh mica surface by first pressing some adhesive tape against the top mica surfaces, then peeling off the tape.

5. Deposit 50µl of protein solution on the freshly cleaved mica.

6. Allow 20-30 minutes for the protein solution to bind to the mica substrate. Binding time may vary with different samples. For longer binding times, put the mica in a covered dish with a wet piece of filter paper to keep the liquid from evaporating.

7. Rinse the sample with a large quantity of buffer to remove unbound protein. Leave a drop of buffer on the mica.

8. Mount the sample on the scanner end cap. Seal the fluid cell and fill with buffer.

9. After the fluid cell has been flushed with buffer solution, reclamp the drain line. This is important for low-noise, low drift imaging. The sample is now ready for tapping mode imaging. A good tapping mode image of lysozyme protein on mica is shown in Figure 11.6b

Figure 11.6b Tapping mode image of lysozyme in buffer solution using above sample preparation (Scan size = 500nm).
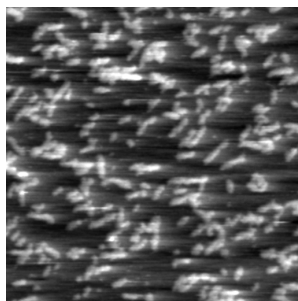
Note: It is also possible to prepare samples inside the fluid cell by flowing the protein solution through the fluid cell. In this case, it may be helpful to engage the tip in contact mode with a zero **Scan size** to protect proteins from binding to the tip.

11.7 Binding DNA to Mica

11.7.1 DNA Binding Theory

DNA and mica are both negatively charged, and so it is necessary to modify the mica surface or the DNA counter ion to allow binding. The counterion method is done by adsorbing the DNA onto the mica in the presence of a divalent (+2 charged) ion, like Ni^{+2}. The divalent ion will serve as a counterion on the negatively charged DNA backbone and will also provide additional charge to bind the mica. This is shown schematically in Figure 11.7a.

Figure 11.7a Negatively charged DNA may be bound to negatively charged mica in the presence of divalent counterions, such as Ni^{+2}.
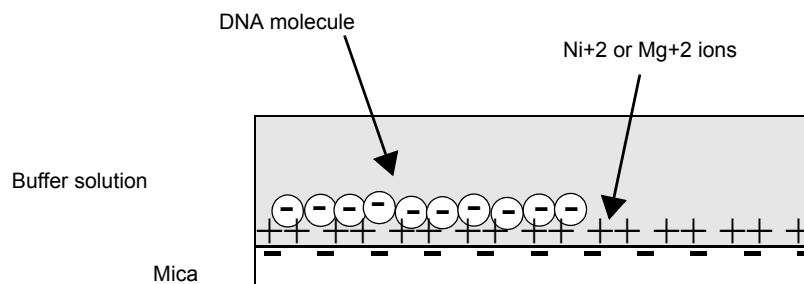
11.7.2 DNA Binding Procedure

The following procedure is adapted from these sources:

- Dunlap, D.D., A. Maggi, M.R. Soria & L. Monaco (1997) "Nanoscopic Structure of DNA Condensed for Gene Delivery." *Nucl. Acids Res.* **25**, 3095.

- Kasas, S., N.H. Thomson, B.L. Smith, H.G. Hasma, X. Zhu, M. Guthold, C. Bustamante, E.T. Kool, M. Kashlev & P.K. Hasma (1997) "*Escherichia coli* RNA polymerase activity observed using atomic force microscopy." *Biochemistry* **36**, 461.

- Lyubchenko, Y.L. & L.S. Shlyakhtenko (1997) "Direct Visualization of Supercoiled DNA *in situ* with Atomic Force Microscopy." *Proc. Natl. Acad. Sci. USA* **94**, 496.

Many other references regarding DNA imaging are listed in the Biological Applications Bibliography; call Bruker for a copy.

1. Obtain the required materials:

    - Mica substrates

    - DNA: BlueScript II SK9(+) double stranded plasmid DNA, 2961 base pairs, 1mg/ml in 10mM Tris, 1mM ethylenediaminetetraacetic acid (EDTA) from Stratagene, La Jolla, CA.

    - Buffer solution: 10 mM HEPES and 5 mM $NiCl_2$ pH 7.6 (for loose binding and air imaging), or $NiCl_2$ (for tight binding and fluid imaging)

2. Dilute DNA in buffer solution to a final concentration of 2.5 ng/µl.

3. Glue a piece of mica to a metal support as described in **Section 11.2.6** on page 198. Cleave the mica substrate with a piece of adhesive tape. Place 30µl of the DNA solution in the center of the mica disk. The DNA will bind to the mica within 1 minute.

4. Load the prepared sample onto the AFM scanner and assemble the fluid cell, as previously described in **Section 11.2.6** on page 198. It may be helpful to wait for the temperature of the buffer to stabilize (20 minutes or more) before imaging.

The sample is now ready for tapping mode imaging.

Chapter 12 Scanning Tunneling Microscopy (STM)

STM relies on "tunneling current" between the probe and the sample to sense the topography of the sample. The STM probe, a sharp metal tip (in the best case, atomically sharp), is positioned a few atomic diameters above a conducting sample which is electrically biased with respect to the tip. At a distance under 1 nanometer ($0.001\,\mu m$), a tunneling current will flow from sample to tip. In operation, the bias voltages typically range from 10 to 1000mV while the tunneling currents vary from 0.2 to 10nA. The tunneling current changes exponentially with the tip-sample separation, typically decreasing by a factor of two as the separation is increased 0.2nm. The exponential relationship between the tip separation and the tunneling current makes the tunneling current an excellent parameter for sensing the tip-to-sample separation. In essence, a reproduction of the sample surface is produced by scanning the tip over the sample surface and sensing the tunneling current.

The following sections are included in this chapter:

- **STM Introduction:** Section 12.1

  - **Overview of STM:** Section 12.1.1

  - **STM Hardware:** Section 12.1.2

  - **Sample Surface:** Section 12.1.3

  - **Vibration isolation:** Section 12.1.4

- **Basic STM Operation:** Section 12.2

  - **Select Mode of Operation:** Section 12.2.1

  - **Prepare the sample:** Section 12.2.2

  - **Prepare the microscope:** Section 12.2.3

  - **Set Initial Scan Parameters:** Section 12.2.4

  - **Engage:** Section 12.2.5

  - **Adjust Scan Parameters:** Section 12.2.6

12.1 STM Introduction

12.1.1 Overview of STM

The STM option is generally employed under the following conditions:

- When atomic/molecular resolution is desired.

- For samples having deeply relieved features, where AFM probes may not be able to penetrate, and/or where feature verticality is very close to 90 degrees.

- On polished samples, where it is necessary to image different layers having similar topography but different electrical conductivities.

- Under conditions where contact with the sample surface is prohibited.

STM relies on a precise scanning technique to produce very high-resolution, three-dimensional images of sample surfaces. The STM scans the sample surface beneath the tip in a raster pattern while sensing and outputting the tunneling current to the NanoScope control station. The digital signal processor (DSP) in the workstation controls the Z position of the piezo based on the tunneling current error signal. The STM operates in both "constant height" and "constant current" data modes, depending on gain settings on the **Feedback** panel. The DSP always adjusts the height of the tip based on the tunneling current error signal, but if the feedback gains are low, the piezo remains at a nearly constant height while tunneling current data is collected. With the gains high, the piezo height changes to keep the tunneling current nearly constant, and changes in piezo height are used to construct the image. The exponential relationship between tip-sample separation and tunneling current allows the tip height to be controlled very precisely. For example, if the tunneling current stays within 20% of the setpoint value (the current to be maintained by the feedback system), the variation in the tip-sample separation is less than 0.02nm.

12.1.2 STM Hardware

Some individual STM components are described below:

STM Converter head and tipholder—To perform STM, you need to add a MultiMode STM convertor head, shown in Figure 12.1a. The STM converter head (so-called because it "converts" the MultiMode to a scanning tunneling microscope) consists of a rigid ring bisected by a solid support for the tipholder. Because of the converter head's compact construction, it holds the STM tip rigid, minimizing vibrational noise. Mounted within the converter head is a circuit which contains the preamplifier for the tunneling current and provides interconnections to the MultiMode SPM electronics.

Figure 12.1a   STM Converter Head
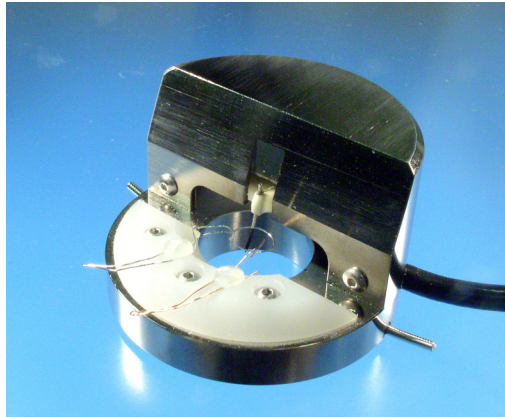
Figure 12.1b   SECPM Head

SECPM Gain Switch
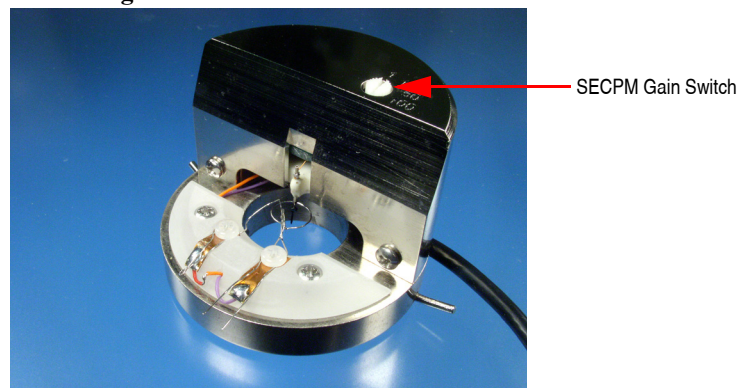
There are three types of STM converter heads available: a standard version which measures current in the nanoampere range (Model #MMSTMC/MMSTMEC, shown in Figure 12.1a), a special, low-current version which measures current in the picoampere range (Model #MMSTMLC/MMSTMLCE), and an electrochemistry version (Model #MMSECPM, shown in Figure 12.1b). The low-current converter head includes an in-line electronics package.

> **Note:**   The STM microscope heads were not designed to operate in UHV.

STM Probes—Probes for the NanoScope STM must be 0.01" in diameter to fit into the tipholder. The two most commonly used tips are made from either a platinum iridium (PtIr, see below) alloy or tungsten. In general, most of the discussion in this manual involving tips and noise reduction applies to both types of tips, but there are some applications that are tip-specific.

PtIr Probes—PtIr probes are mechanically formed and can be purchased directly from Bruker. PtIr probes seem to give better atomic resolution than tungsten, most likely due to the lower reactivity of platinum. The PtIr probes are not as uniformly shaped as the tungsten tips. The quality of the mechanically-formed PtIr probes will vary.

Tungsten Probes—Tungsten probes are electrochemically etched from tungsten wire (see **Etching Tungsten Tips:** Section 12.7). Tungsten probes are more uniformly shaped. They may perform better on samples with steeply sloped features.

12.1.3 Sample Surface

Samples to be imaged with a scanning tunneling microscope must conduct electricity. In many cases nonconductive samples can be coated with a thin layer of a conductive material to facilitate imaging. The sample surface must be conductive enough to allow a few nanoamps of current to flow from the bias voltage source to the area to be scanned. STMs have been used to scan gold, silver, platinum, nickel, copper under oil, chrome plating, doped silicon under oil, conducting polymers, amorphous carbon, blue diamond, diamond-like carbon films, carbon fibers, graphite, iron-oxide compounds, semi-metals, doped semiconductors (molybdenum disulfide), cobalt-chromium compounds, stainless steel and liquid crystals. Oxide layers more than a few atoms thick on the sample tend to affect the scanning and wear down the tip as it is dragged through the oxide. The feedback loop will extend the tip until a tunneling current flows, even if it must push the tip through an oxide layer.

For samples that tend to oxidize, tunneling under oil or scanning in a glove box filled with inert gas can improve the imaging. Silicon oil or paraffin oil (mineral oil) also works well with some samples. One minor problem involved with the use of oil is the increased difficulty in the coarse positioning of the tip. The reflection of the tip comes off the liquid instead of the surface of the sample. It is difficult to tell when the tip is close to the sample surface. The best approach is to lower the tip until it just touches the surface of the oil, then select **Engage**.

12.1.4 Vibration isolation

The microscope should be isolated from sources of vibration in the acoustic and subacoustic frequencies. This requirement can be relaxed somewhat for large-scale images, but atomic-scale work is very sensitive to ordinary room vibrations.

As a final note, the best way to reduce coupling from vibrations is to eliminate as many sources of vibration as possible. Remember that vibrations can be transmitted to the microscope over the cable. Avoid tension in the cable and keep it away from fans and other noise sources. Also, keep the microscope away from sources of acoustic noise. Loud conversation can disrupt atomic scale images. Air currents can also disturb atomic images, so it may be necessary to run the microscope

with a cover on (noting that a cover will trap heat and may cause substantial thermal drift during imaging).

12.2 Basic STM Operation

This section explains how to use the MultiMode to image a conductive sample.

12.2.1 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 12.2a.

Figure 12.2a The **Select Experiment** window
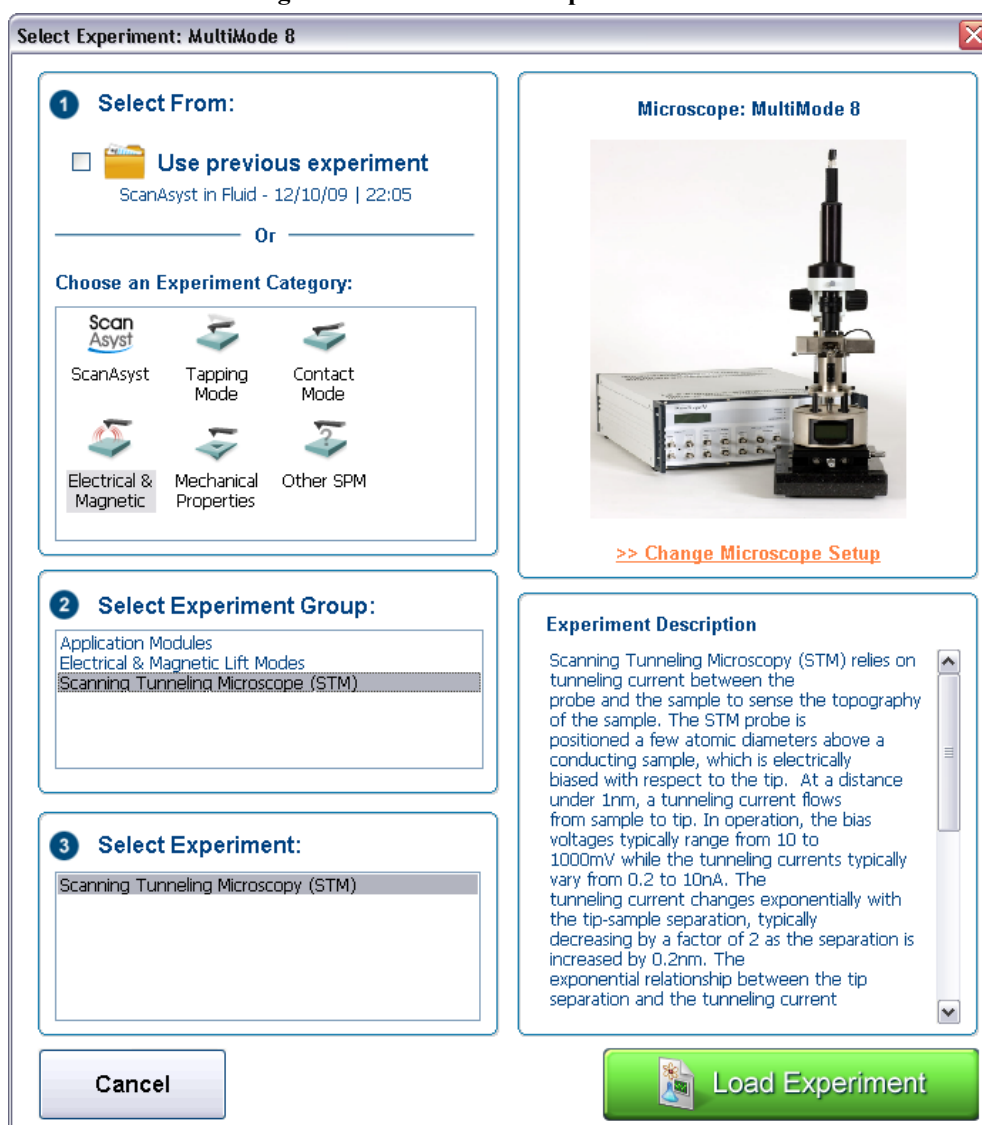
2. Select ELECTRICAL & MAGNETIC in the **Choose an Experiment Catagory** panel.

3. Select **S**CANNING **T**UNNELING **M**ICROSCOPY **(STM)** in the **Select Experiment Group** panel.

4. Select **S**CANNING **T**UNNELING **M**ICROSCOPY **(STM)** in the **Select Experiment** panel and click **L**OAD **E**XPERIMENT.

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** windows and the **Scan Parameters List** window, shown in Figure 12.2b.

Figure 12.2b STM in Air (Simple Mode) configuration
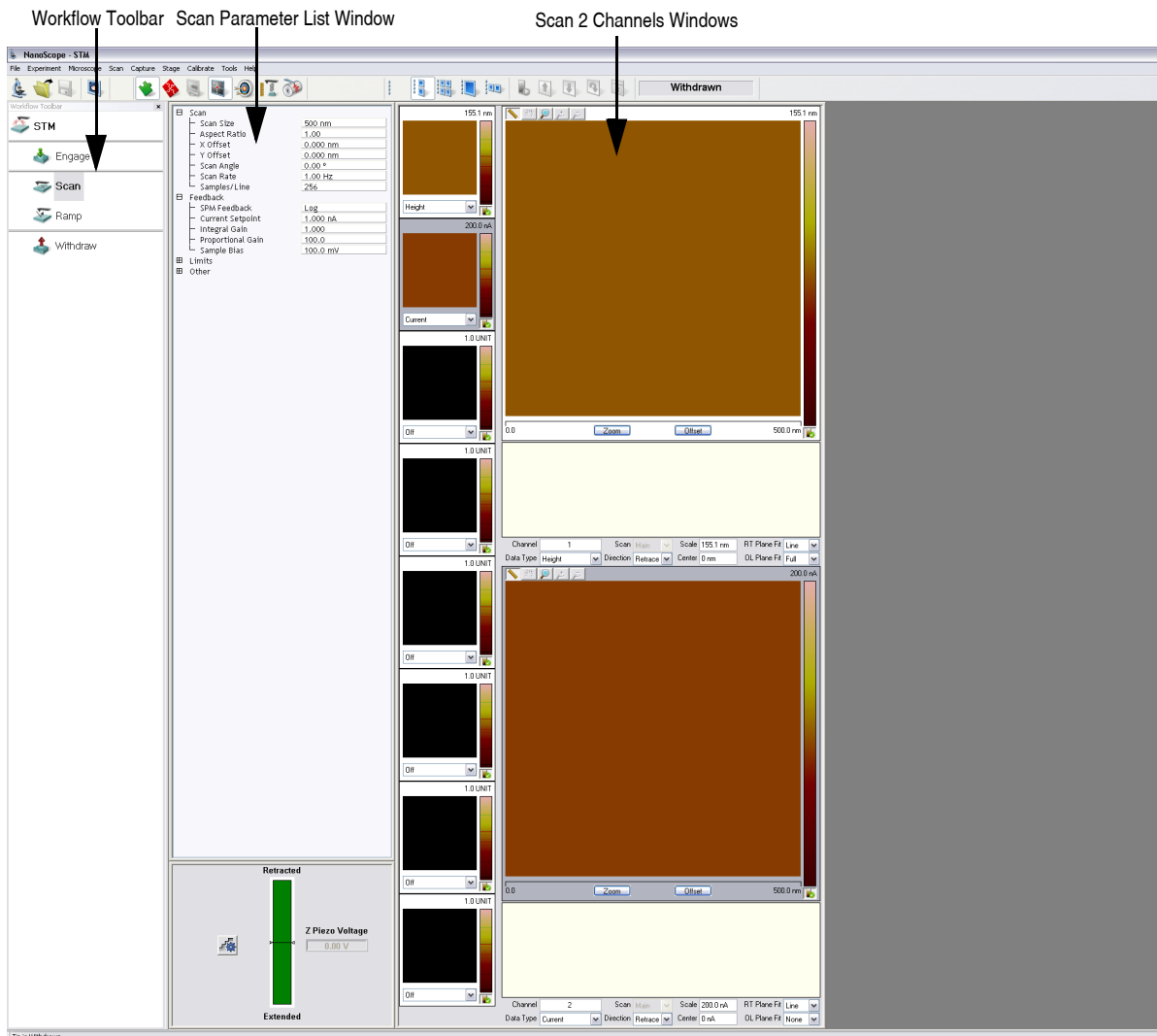
12.2.2 Prepare the sample

The STM bias voltage is supplied to the sample through the top of the scanner. The sample surface must be electrically connected to the scanner end cap. Attach the sample to a metal puck using an electrically conductive (e.g., silver-based) epoxy such as Dynaloy 325 or equivalent. It is not necessary to glue the metal puck to the scanner. The magnet that holds the metal puck to the scanner cap will provide a good electrical connection to the scanner cap.

> **Note:** If the sample you are going to measure is a conductive film on an insulative substrate, you need to bring the conductive path to the surface. Use additional conductive glue on the edge of the sample to ensure that the surface of the sample is connected to the metal puck.

12.2.3 Prepare the microscope

1. Insert a new STM probe in the STM converter head tipholder. The tips come in a small, plastic snap-box filled with foam. Grip the tip with tweezers near the sharp end and insert the blunt end of the tip into the tipholder tube until all but approximately 1mm of the tip protrudes from the holder. It is normal to feel a slight resistance as you insert the tip into the tube. The tip holder tube is slightly curved. The curve in the tube provides a good mechanical and electrical connection to the STM probe.

   > **Note:** For older tipholders (before 1995), it may be necessary to put a slight bend in the tip to help it stay in.

2. Gradually place the converter head atop the scanner, checking for clearance between the tip and the sample as the head is lowered. If the tip appears to be too low and threatens to crash into the sample, adjust the scanner screw(s) to obtain more clearance.

3. Secure the STM converter to the scanner by hooking the springs on the side of the scanner or MultiMode's base to the metal pins on the side of the converter. (The MultiMode head attaches in a similar way).

4. Verify that the mode switch on the MultiMode's base is on **STM**. The LED next to the STM setting on the base should glow and the LCD display on the front of the base will display "STM".

12.2.4 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 12.2c).

1. Set the **Scan Rate** to **1 Hz**.

2. Set the **Scan Size** to **500nm**.

3. Set the **Scan Angle** to **0** degrees.

Figure 12.2c   Suggested STM Settings

Feedback Panel

1. Set the **Integral gain** to **0.5** and the **Proportional gain** to **0.5** (see Figure 12.2c).

2. Set the **Sample Bias** to **100mV**.

3. Set the **Current Setpoint** to **1nA**.

Other Panel

1. Set the **Units** to **METRIC**.

Setpoint

1. Prior to engaging the microscope it is necessary to set the **Sample Bias** voltage and **Current Setpoint**. A good typical **Current Setpoint** is 1-2nA. A good recommended **Sample Bias** voltage is 20-50mv for highly conductive samples like graphite or gold. Try using **Sample Bias** voltages of 100-500mv for less conductive materials. Usually if **Scan Size** is greater than 1μm, altering the values of **Current** and **Bias** voltage do little. Varying the setpoint current on the bias voltage can help improve imaging quality on smaller scan sizes where atomic resolution is desired.

Channels 1 and 2

1. Set the **Channel 1 Data Type** to **HEIGHT** (see Figure 12.2d).

2. Set the **Channel 2 Data Type** to **CURRENT**.

3. Set **Data Scale** to a reasonable value for the sample.

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

4. Set **Line direction** to either **Trace** or **Retrace**.

5. Set the **Channel 3** through **Channel 8 Data Type** to **OFF**.

Figure 12.2d   Suggested Channel Settings

12.2.5 Engage

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

12.2.6   Adjust Scan Parameters

Once engaged, the scan parameters should be adjusted to obtain the best image. Observe the agreement between the Trace and Retrace lines, and adjust the **Setpoint**, **Gains** and **Scan Rate** to bring the Trace and Retrace lines into coincidence.

   Note: Generally, the **Data Type** field is set to **HEIGHT** whenever the MultiMode is used in STM mode to image sample surfaces with large fields of view.

Note: A poor tip, either because it has touched the sample, has contamination on the end, or because of its manufacturing, will render a poor image. In this case, there is little likelihood that the tip will recover, and the best procedure is to replace the tip. Click **Withdraw** to stop scanning, and retract the probe from the surface. After the tip is withdrawn, the WITHDRAWN message will appear in the status bar. Be certain there is sufficient clearance between tip and sample after installing the new tip—a new tip will extend a different distance out of the tip holder than the old tip.

The nominal Z position of the piezo provides useful diagnostic information. The real time status bar graph reflects the Z position of the piezo during the scan. Under most circumstances, the "Z center position" should be in the center of the graph, indicating that the Z voltage for the piezo is fluctuating around zero volts. If the sample is not level, or if there is drift in the mechanical system after the tip has engaged, the "Z center position" will fluctuate during the scan or drift off-scale.

Improving Image Quality

- Vary the **Scan Size** and the **X Offset** and **Y Offset**. Move to a new location on the sample by changing the Offset of the scan. The X and Y offset parameters define the center of the scan. Often, a better image can be obtained on a different portion of the sample. The X and Y offsets can be changed by altering the values in the control panel, or by using the **Offset** button below the image.

- Oscillate the tip by briefly setting the **Integral Gain** up to about 500. The oscillation will show up on the display as a grainy pattern of light and dark. Set the **Integral Gain** back down after about a second and check if the signal is quieter than before. Setting the **Integral Gain** high for a brief period is also useful for cleaning debris from the tip.

- Alternate the commands **Withdraw** and **Engage** a couple of times. This raises and lowers the tip and may eliminate contamination on the end of the tip.

- Use the **Step Motor** command to single-step the tip down a step or two. Be sure to minimize the **SPM step size** before stepping the motor down. Watch the **Z Center Position** scale to verify that the piezo has sufficient room to retract so the tip will not harm the surface of the sample. Stepping the tip down until the **Z Center Position** goes to the retracted end of the scale where the indicator turns red will usually destroy the tip.

- Vary the **Setpoint** current over the range 2 to 10nA. As a last resort, current may be increased up to 100nA for a very brief period of time.

- If none of the above procedures improves the quality of the signal, replace the tip and try again. If that doesn't work, call Bruker for technical assistance.

- Reduce the **LP Current BW**, available in Expanded Mode.

1. After a clean image has been obtained, saved it to disk with the **Capture** commands (or click a **Capture** icon). The **Capture** command saves the current scan image (or the next image if any changes have been made in the STM control panel) into the specified directory. The **Capture Filename** command can be used to define a custom filename or the captured file will be named with the concatenation of a date/time stamp. In either case, a three-digit suffix

which incrementally increases after each capture operation is appended to the filename. If the date/time stamp is used, a file captured at 3:32 p.m. on July 31 would be assigned the file name 07311532.001. A second file captured the same minute would be named 07311532.002.

2. Simple filtering operations can improve the look of the captured image. Double-click the image in the browse window.

3. Apply the LOWPASS filter by clicking the Lowpass icon on the toolbar and clicking EXECUTE. See the *NanoScope Software 8 User Guide*, Bruker p/n 004-994-000, for information about this command.

12.3   STM-Specific Information and Operations

The digital signal processor (DSP) calculates the desired separation of the tip from the sample by sensing the tunneling current flowing between the sample and the tip. The bias voltage applied to the sample encourages the tunneling current to flow. The DSP completes the digital feedback loop by outputting the desired voltage to the piezoelectric tube. The NanoScope STM can be operated in two modes. In one case the feedback gains are set high, the tunneling tip closely tracks the sample surface, and the variation in the tip height required to maintain constant tunneling current is measured by the change in the voltage applied to the piezo tube. In the other case, the feedback gains are set low, the tip remains at a nearly constant height as it sweeps over the sample surface, and the tunneling current is imaged. This section provides greater insight into the operation of the STM.

12.3.1   STM Hardware

Some individual STM components are described below:

1. Tips— The probes for the NanoScope STM must be less than 0.012" in diameter to fit into the tip holder. The two most commonly used tips are made from either a platinum iridium (PtIr) alloy or tungsten. The PtIr tips are mechanically formed and can be purchased directly from Bruker. At the end of this chapter, instructions are provided on how to etch tungsten tips from tungsten wire with an electrochemical process. In general, PtIr tips provide better atomic resolution than tungsten tips, but tungsten tips are more uniformly shaped. They may perform better on samples with steeply sloped features such as compact or optical disks.

2. Preamp—Mounted on the STM converter head is a circuit which contains the preamplifier for the tunneling current.

12.3.2   Fine Points of STM Operation

Getting the best images on NanoScope requires a good interface between sample and tip, reasonable vibration isolation, and proper settings in the Computer Workstation menus. This section provides greater detail on the operation of the STM.

12.3.3 STM Operating Modes

The two operating modes available on the NanoScope STM are selected with the **Data Type** option in the **Channel** panel (see Figure 12.3a). **Height** data reflects the change in tip position required to maintain a constant tunneling current. The DSP senses the tunneling current, calculates the difference from the desired tunneling current, and determines the voltage that must be applied to the piezo tube to keep the tunneling current constant; the change in voltage applied to the piezo tube translates directly to a change in distance. This distance data is recorded throughout the scan and displayed on the screen as the "height" of the sample.

Figure 12.3a   Typical STM Channel 1(above) and Channel 2 (below) Control Panel Parameters

Current data is a measure of the tunneling current at each point tested on the sample. The DSP measures the voltage drop across a resistor in series with the tip and calculates the tunneling current as the tip scans the surface of the sample. The tunneling current at each data point is recorded and displayed on the screen.

The two scan modes (height and current) require subtle changes in the menu parameters to operate effectively. The parameter changes also affect the application of the two modes. To operate effectively in collecting height data, the tip must closely track the sample surface. The gains must be maximized to force the piezo to respond quickly to the variations in the sample surface. The height mode is used for most applications. Conversely, the gains must be very low to keep the piezo from responding while collecting current data. After engaging, the tip scans the surface of the sample with very little variation in the piezo height. This constant height provides a reference from which to measure and record the fluctuations in the tunneling current. The current mode is most useful for imaging atoms with relatively small scan sizes.

12.3.4 STM-Specific Menu Parameters

In addition to the **DATA TYPE** discussed above, the **Feedback** control panel contains three items that are specific to the operation of the scanning tunneling microscope. The **SPM Feedback** (**Expanded Mode** only), **Sample Bias**, and **Current Setpoint** parameters pertain exclusively to the control of the STM.

The **SPM Feedback** parameters in the **Feedback** panel determine the transformation performed on the tunneling current prior to the feedback calculations. The two settings select **LINEAR** or **LOG** operations. Remember that tip-to-sample separation is proportional to the log of the tunneling current. The **Linear** selection causes the error signal for the feedback loop to be the difference between the instantaneous tunneling current and the setpoint current. The **Log** selection calculates the error signal as the difference between the log of the instantaneous tunneling current and the log of the setpoint current.

The **Bias** parameter controls the magnitude and sign of the bias voltage applied between the tip and the sample. A bias voltage encourages the tunneling current to flow. Although settings of 20 to 100 mV are typical for conductive samples, the allowable setting ranges from -10 to 10 volts. Positive settings of the bias voltage induce negative tunneling currents (i.e. electrons flowing from the tip to the sample).

Optimization of STM Scanning Parameters

The process of selecting and optimizing the scan parameters can be streamlined. In most cases the scan parameters are dictated by the sample. The **Data Type** is usually the first parameter set and the **Proportional** and **Integral gains** are directly related to the **Data type**. The **Scan Size** depends on the sample and the features of interest. The maximum **Scan Rate** is usually related to the **Scan Size**. The **Bias** voltage and the tunneling **Current Setpoint** depend on the sample. Usually, they are set at a standard value for engagement and fine-tuned along with the gains and filter to enhance the quality of the image.

As discussed above, a **Data Type** of CURRENT is the best for atomic-scale images. This mode is not practical for rough surfaces, because the tip will crash into the surface at low feedback gains. The HEIGHT **Data Type** is usually better for all but atomic-scale scans. In general, height data images are best at higher feedback gains and slower scan rates.

Settings for feedback gains depend on many factors, but perhaps the most important is the **Data Type**. If the **Data Type** is set to **Current**, the **Integral** and **Proportional Gains** should be set as close to zero as possible. The gains should be lowered for data captured using the LINEAR SPM **Feedback** type, especially with high **Setpoint** current levels. Large-scale images should be taken at increased gain. Experimentation may yield other values for your samples.

Often the best way to set the gains for the HEIGHT **Data Type** is to view the scope trace with the **Slow scan axis** disabled (Expanded Mode). This allows the feedback to be tuned while looking at a single scan line of data. First, increase the **Integral Gain** until oscillations start to appear, and then, back off a little. Next, adjust the **Proportional Gain**. High frequency noise will appear on the signal when the **Proportional Gain** is set too high. The **Setpoint** current and the **Bias** voltage can also be adjusted using the Scope display.

Bias voltages below 20mV usually provide the best quality images on samples with surface conductivities equal to or better than graphite, but there are exceptions. Resistivity across the surface of a sample can be measured with an ohmmeter. For samples with high resistivity (greater than 1 megohm/cm), bias voltages of 100mV or even higher may work best. For scans larger than 0.5µm, it is sometimes better to increase the bias voltage by 50mV to 100mV over the value for smaller scans. A higher bias keeps the tip further from the surface, giving the feedback loop greater tolerance in tracking the surface at high speeds.

Increasing the **Current Setpoint** can also be helpful for larger scans. This has the effect of raising the gain, but also brings the tip closer to the surface by a small amount. High setpoint currents of 6 nanoamps or more can also be useful in improving the signal-to-noise ratio for atomic images on some materials.

The **SPM Feedback** type can be set to either LOG or LINEAR input transformations. Because the tip-to-sample separation is proportional to the log of the tunneling current, the transformation performed on the tunneling current prior to the feedback calculation can have dramatic effects on

the performance of the feedback loop. **LINEAR** input is more protective of the tip, because the feedback error signal responds exponentially to tip-sample separation. When the tip-to-sample separation decreases, the error signal rises exponentially, quickly driving the tip away. However, the error signal is unsymmetrical. The same sample-to-tip separation change that caused the tip to move away so quickly will generate a small error signal when the tip is higher than it is supposed to be. This unsymmetrical response in the **LINEAR** mode will distort data. For this reason, the **LOG** mode (with ln(I) used in the feedback calculation) are preferable for most samples, because they respond in a more symmetrical fashion to positive and negative sample slopes. The log input has the advantage of having a gain which is insensitive to the value of the **Current Setpoint**.

Large scans cannot be taken at the same scan rate as small scans. When using **Scan Sizes** above a few microns, the **Scan Rate** should be lowered below 10Hz. Best results can be obtained at scan rates of 1 Hz or less, although image-taking is slow. At these scan rates, the 128 x 128 and 256 x 256 data formats are most useful, quadrupling and doubling the frame rate over the 512 x 512 format for a given scan rate. To be sure that there is no image degradation due to too high a scan rate, lower the rate and check for changes in the image. Check the **Scope** view to ensure that the scan is not slew-rate limited in Z, as evidenced by an artificial "sawtooth" appearance in the scope trace.

The **LP Current BW** (Expanded Mode) parameter provides the option of filtering the tunneling current signal. A lowpass filter with a cut-off frequency of 20kHz can be applied to the analog tunneling signal in hardware. Typically, the filter is selected for atomic-scale images; otherwise, no filtering should be selected.

Tunneling Tips

Although the microscope will accept any 0.010" diameter tip, tips made of platinum iridium (PtIr) and tungsten are used most often. Both are supplied by Bruker. In general, most of the discussion in this manual involving tips and noise reduction applies to both types of tips, but there are some applications which are tip specific.

PtIr tips seem to give better atomic resolution than tungsten in air and liquids, probably due to the lower reactivity of platinum. The PtIr tips are not as uniformly shaped as the tungsten tips, so freshly etched tungsten tips may provide cleaner data when scanning steeply sloped surfaces such as compact or optical disks. For tunneling on surfaces immersed in conductive liquids, coated tips have been used.

The quality of the mechanically formed PtIr tips will vary. Some give beautiful images as soon as tunneling starts; others start poorly but improve over time; while others start noisy and stay noisy.

Head Offset

The value of the head preamplifier offset can be measured with the **Offset** command in the **TOOLS** menu. The procedure is as follows:

- Withdraw the tip from the sample surface.

- Invoke the **TOOLS** > **OFFSET** command to observe the offset current of the preamplifier in nanoamps.

12.4 Spectroscopy with the STM

The NanoScope STM performs limited spectroscopic operations under the two scanning tunneling spectroscopy STS modes of operation. The variation of the tunneling current due to variations of the bias voltage or tip-to-sample separation can be tested and recorded at a single point using NanoScope's **Ramp** mode.

STM Ramps

In the STS ramp operation, the tip is positioned at a point on the surface, and a spectroscopic plot is acquired and displayed in a scope format. Between plots, the feedback is run to establish the tunneling current to the setpoint value. The different types of STS Plots that can be acquired are:

STS i(v) — To do this, select **Ramp** and choose SAMPLE BIAS for the **Ramp Output** in the **Ramp** panel of the **STM Ramp Parameter List**, shown in Figure 12.4a. Select CURRENT as the **Data Type** for **Channel 1** in the **Channel Selection** panel in the **STM Ramp Plots** window, shown in Figure 12.4b. The tunneling current as a function of the bias voltage is displayed. The tip height is held constant while the I-V plot is being acquired. In addition to I vs. V, F(d i,dv), F(d ln(i),dv), or F(d ln(i),d ln(v)) vs. V can be plotted.

Figure 12.4a STM Ramp Parameter List for STS i(V)

Figure 12.4b **Channel Selection** panel for STS i(V) in the **STM Ramp Plots** window.
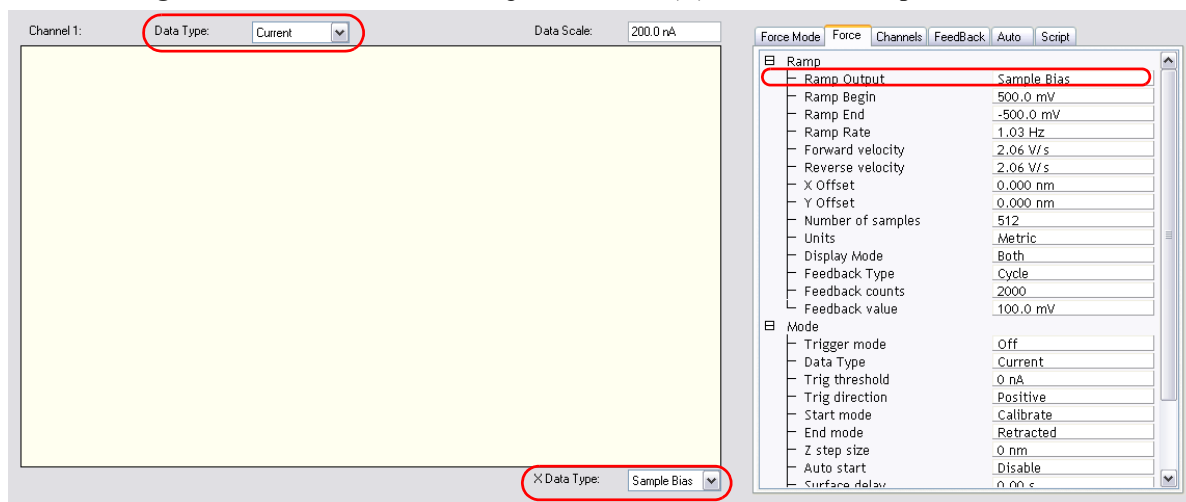
STS i(s) — To do this, select **Ramp** and choose **Z** for the **RAMP OUTPUT** in the **Ramp** panel of the **STM Ramp Parameter List**, shown in Figure 12.4c. Select **CURRENT** as the **DATA TYPE** for **Channel 1** in the **Channel Selection** panel in the **STM Ramp Plots** window, shown in Figure 12.4b. The tunneling current as a function of the tip height is displayed. The bias voltage is held constant while the I-S plot is being acquired. In addition to I vs. S, ln(I) vs. S can be plotted.

Figure 12.4c STM Ramp Parameter List for STS i(s)

12.4.1 Operation of STS

In the following sections, the operation of the spectroscopic functions of the NanoScope STM will be discussed. Additional information can be obtained from the *Command Reference Manual*.

STS Plot

There are several items that you should be aware of when using the NanoScope to acquire spectroscopic plots with the STS plot commands. The spectroscopic capabilities can provide information that can help distinguish different species although exact species identification is difficult, especially in air. The spectroscopic plots should aid in comparative studies between samples or between different regions on a sample, but they will not reveal the precise make-up of that sample.

A comparison to STM imaging reveals two somewhat conflicting requirements. As a good starting point, the sample and tip should produce consistent STM images. The images should repeat well from frame to frame and be fairly free of noise or areas on the surface that appear unstable. For current-versus-voltage type plots, the spectroscopic plots may be nice and smooth and repeat well, but switching back to the STM imaging mode reveals images which are quite noisy. This can be attributed to the fact that the quality and uniformity of the tip is probably more critical for imaging than for making the rather simple spectroscopic plots. This is generally not true for the I vs. S plots. Some general recommendations for acquiring spectroscopic plots are:

1. Low settings for the **Integral Gain** are preferred. Because the tip is not tracking topography, lower gains are acceptable and tend to make the plots more stable.

2. The maximum input range of ±100nA on the NanoScope with the standard preamps will restrict the current-versus-voltage plots.

12.5 Troubleshooting for STM

This section addresses errors or malfunctions encountered during the operation of the MultiMode as an STM. See Chapter 19 in this manual for additional troubleshooting tips.

12.5.1 Head and Microscope-Related Problems

This section deals with problems related to the scan heads or the microscope. If a problem exists with a scan head, try a second one under the same conditions, if possible. Otherwise, the following list of symptoms and cures may be helpful:

Head Engages Immediately

If the STM engages immediately after initiating the **Microscope** > **Engage** command, then one of the following probably occurred:

1. Tip On Surface—Make sure the tip is not touching the surface of the sample. Withdraw until the tip is far from the surface.

2. Controller Off—Verify that power to the controller is on and that the controller is connected to the computer workstation.

3. MultiMode base switch not set for STM—Verify that the switch on the left side of the MultiMode base is toggled to STM.

4. Disconnected—Verify that the 15-pin microconnector from the head is plugged into the microscope and the microscope is plugged into the controller.

Head Never Engages

If the scan head never engages, test for the following:

- Disconnected—Make sure the microscope is connected to the Controller.

- Binding on drive screw—Feel the rear, motor-drive adjustment screw during engagement to verify that it is rotating. If the motion is erratic, then the screw is fouled with debris and binding. Remove the screw, clean threads and re-grease per instructions in **Adjustment Screw Maintenance Procedure:** Section 19.2.

- Bias shorted—Measure the bias by using a voltmeter between the head and stage. If this is not in agreement with the settings in the *Bias* voltage item in the STM Parameter control panel and appears to be grounded, then check to see if anything is providing a conduction path between the Base and the Base Support or any other ground.

- Bias not applied to sample surface—In some cases the sample consists of a layer of electrically conductive material on top of an insulator. A conductive path must be provided between the sample puck and the sample surface. Carbon glue or silver epoxy can be used to connect the bias voltage to the sample surface. Check the bias voltage between the sample surface and chassis ground of the NanoScope V. Check jumper positions on the underside of the MultiMode base.

Tip Crashes

If the tip always crashes into the surface with the Z center either changing erratically or stuck in the fully retracted position, try the following:

- Check Sample Conductivity—There are two problems associated with sample conductivity. First, the bulk conductivity of the sample may make it difficult to image. If the resistance of the sample is greater than 1 $K\Omega$, higher bias voltages should be tried. If the resistance is greater than 1 $M\Omega$, bias voltages of 100 mV or more should be used. Samples with resistances 1 $M\Omega$ or greater will be difficult to image even with high bias voltages. Second, oxide and contamination layers on the sample surface can make imaging very difficult. Higher bias voltages are required for these types of samples.

Measuring the bulk conductivity of the sample with probes may not tell the whole story. Probes may easily penetrate oxide or contamination layers on the sample surface yielding reasonable resistance measurements. However, oxide and contamination layers on the sample surface can make imaging very difficult. Higher bias voltages are required for these types of samples.

Problems During Realtime Operation

This section deals with problems that are associated with the Realtime aspects of the NanoScope software although they often point to the scanning head or microscope.

Image goes out of Range

If the Z center position tends to go out of range at the edges, top and bottom or right and left of the scan, then try the following:

- Level Sample to Head—The Z center position is affected by sample tilt and the MultiMode head may be less tolerant of sample tilt due to its small vertical range compared to the large, lateral scan range. Try leveling the head relative to the sample by noting the trend in Z center position and adjusting the head tilt accordingly. Setting **Realtime Plane Fit** set to NONE or OFFSET can also be very helpful in eliminating tilt along the scan direction.

- Measure Coupling— A broken wire to one of the electrodes can cause this problem and will usually result in a large, monotonic increase (or decrease) in Z center position from the bottom of the scan to the top.

- Rotate the sample.

Z drift

If the Z center voltage tends to drift out of range rather quickly after the head is engaged, it may be a result of the following:

- Thermal Drift— Allow some time for the temperature to stabilize if the microscope and/ or scan head has been stored in a cold place overnight. Drift can be minimized by keeping the STM in a thermally stable environment.

- Tip not Tight—Make sure the tip is held tightly in the tip holder. Push and pull on the tip with the tweezers to see that it is not loose.

Realtime Image Hides Features

If the Realtime image appears to be flat, but captured images reveal detail, then try rotating the sample 90° in the microscope. The Realtime image leveling software tends to hide features that are parallel to the X-scan direction.

Image is Streaky or Wavy

If high-resolution images appear streaky or wavy, it may be a result of the following:

- Insufficient Vibration Isolation—Atomic-scale scans are the most susceptible to vibration in the acoustic and subacoustic frequencies. Verify that the MultiMode microscope is acoustically isolated with a functioning isolation table and, if required, an acoustic cover. Make certain that vibration tables are properly pressurized and adjusted.

- Bad Tip—Many strange effects have been pondered and hunted down for hours only to find that changing the tip fixes the problem. Try a different tip to see if the problem persists.

- Low-Scan Rates—The brute force method for reducing the effects of drift and even low frequency vibration is to simply raise the scan rate. Collect small-scale images at 156 Hz with 256 x 256 sample points.

Image is Triangular over Step-like Features

- The performance of the STM over large scans with high vertical features is very dependent on the ability of the feedback loop to force the tip to track the sample surface. The digital feedback used in the NanoScope has been designed to maximize performance on a variety of samples. Try the following to improve the image quality over step-like features:

- Log Feedback—The most dramatic increase in performance of the feedback over large scans is achieved using the **Log** feedback mode. These make the error signal symmetrical for conditions with the tip too far or too close to the surface.

- Gain—The settings of the **Integral Gain** term also tend to be critical for large scans. They should be high enough to produce a little fuzz on the image but not so high as to cause large oscillations.

- Scan Rate—The **Scan Rate** should be lowered for large scans, especially, if the sample surfaces are rough or contain large steps. Moving the tip quickly along the sample surface at high scan rates with large scan sizes will usually lead to a tip crash. Essentially, the **Scan Rate** should be inversely proportional to the **Scan Size**, since the tip must still be maintained roughly 1 nanometer above the surface.

- Setpoint Current—Raising the **Current Setpoint** will effectively raise the gain of the feedback loop which can be quite helpful for large scans. It will also bring the tip closer to the surface but only by a small amount ($i \approx e^{-S}$).

12.6 Low-Current STM

The following sections provide detailed instructions for performing low-current (sub-picoamp) STM using the Low Current STM Converter for MultiMode AFM (Model #MMSTMLC).

12.6.1 Description

Operation of conventional STMs with current (I_{tun}) in the nanoamp range is characterized by strong tip-sample force interactions. The forces applied to the sample during STM imaging in air actually exceed the forces in atomic force microscopy (AFM). The effects of high STM forces were found when imaging materials which included graphite [Figure 12.6b], inorganic layered compounds[Figure 12.6c], organic conducting crystals [Figure 12.6d] and organic adsorbates on conducting substrates [Figure 12.6e].

For metallic surfaces, the tip-sample gap resistance, $R_{\text{gap}} = V_{\text{bias}}/I_{\text{tun}}$, can be used as a qualitative measure of tip-sample separation. Where

$$V_{\text{bias}} = \text{ bias voltage}$$
$$I_{\text{tun}} = \text{ setpoint current}$$

In general, decreasing I_{tun} will increase the tip-sample separation and decrease the tip-sample force.

By operating at R_{gap} in the GΩ range, for example, $V_{\text{bias}} = 1.0$ V and $I_{\text{tun}} = 1$ pA the tip-sample distance increases enough to allow less-destructive imaging of a variety of surfaces.

Picoampere-level STM provides the opportunity to image semi-insulating samples deposited on conducting substrates. Indeed, it was shown that using STM at ultra high R_{gap} allows imaging of the topmost layers of alkanethiol adsorbates on gold surfaces and to reveal earlier unknown surface features.

12.6.2 Hardware Description

The Low Current STM head, which allows STM measurements with I_{tun} in the pA range is constructed for operation with our MultiMode and AFM bases.

Low-current STM operation also requires the Picoamp Boost Box, shown in Figure 12.6a, which is installed between the NanoScope V Controller and the MultiMode base.

Figure 12.6a Low-Current Converter components: MultiMode STM head and Picoamp Boost Box.
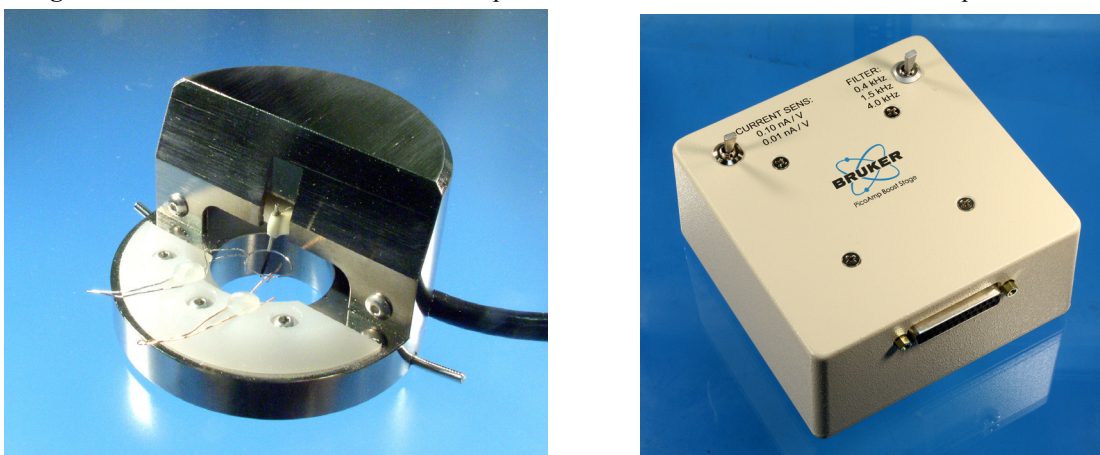
The gain is switchable between 10^{10} V/A or 10^{11} V/A and the bandwidth is switchable among 400Hz, 1.5kHz and 4kHz. Lower bandwidths allow for lower noise levels which are essential for operation at sub-picoampere I_{tun}.

IMPORTANT: The default calibration value of standard STM preamplifier electronics is 10 nA/V. This value **must** be changed to either 0.1 or 0.01nA/V for operation of the low-current head with gain factors of either 10^{10} and 10^{11} respectively. To changes this value, click **Calibrate** > **Scanner** > **Z**. The **Current Sensitivity** parameter is listed on the **Z Calibration** panel and can be changed by simply entering the new value. The current sensitivity setting is kept in the scanner calibration file, so it must be set separately for each scanner file in the **Equip** directory.

Preamp Setting	Current Sensitivity Setting
10^{10}	0.1nA/V
10^{11}	0.01nA/V

12.6.3 Precautions

Due to the low-current head's highly sensitive electronic components, special precautions must be taken.

1. The input stage operational amplifier is extremely vulnerable to electrostatic discharge (ESD) and is easily destroyed. It is recommended that the user be grounded with a wristband at all times that the low current head is handled, especially when tips are installed.

2. The feedback resistor and capacitor in the input stage are extremely vulnerable to contamination. At no time should these delicate components ever be touched with the hands.

3. Good grounding is essential for low-noise performance. A good contact between the SPM base and the metallic cover (can) included with the low current head is needed to reduce the electrical noise level. A simple grounding kit is included with the converter which includes a length of wire and connecting lugs. Make certain that the can cover and SPM base are electrically connected by the wire and that the can cover is in place. The can cover acts as a Faraday shield.

   Note: Cables connecting the SPM base, the Picoamp Boost Box and the NanoScope controller should not be lengthened.

CAUTION: Always disconnect the Picoamp Boost Box and Low-current STM Converter head before performing other types of imaging.

12.6.4 Installation

Generally, installation consists only of cable connections.

Table 12.6a   MultiMode Base Pico Amp Box Jumper Settings

Controller	Std.	Ext. or Torsion
NSV	EXT	Both

MultiMode SPM

1. Turn off all power to the SPM controller. Disconnect and remove the MultiMode head.

2. Connect the picoamp booster box (male receptacle) to the controller using the short 37-pin-to-25-pin connector cable.

3. Connect the PicoAmp booster to the SPM base using the 25-pin-to-25-pin cable connector.

4. Connect the low-current converter head to the MultiMode's support ring using the micro-D, 15-pin connector cable. Set switches on the PicoAmp booster to the desired current level and bandwidth.

5. Before repowering the system, double-check all cable connections. If atomic-level scans are intended, verify the cable between the SPM and picoamp booster box is unstrained (a taut cable will transmit vibrations). The cable should rest loosely. Before loading samples and running the SPM, attach the ESD wristband to prevent sudden electrical discharges.
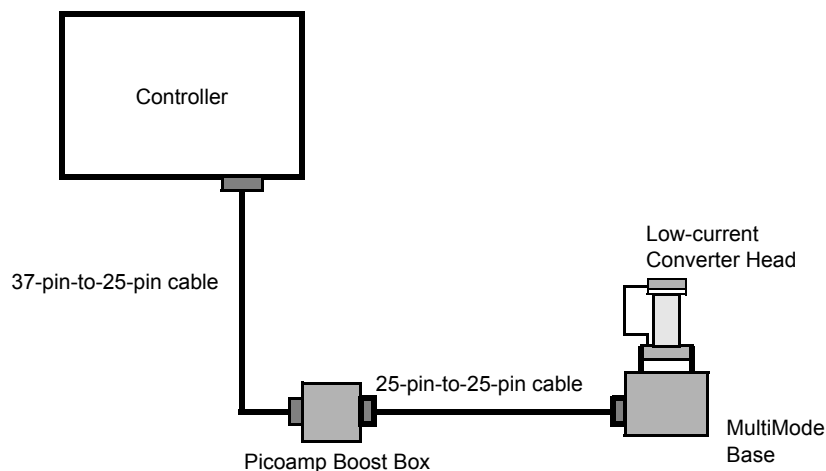
6. Verify that the current sensitivity settings in the software are set to the appropriate value:

IMPORTANT: The default calibration value of standard STM preamplifier electronics is 10 nA/V. This value **must** be changed to either 0.1 or 0.01nA/V for operation of the low-current head with

gain factors of either 10^{10} and 10^{11} respectively. To changes this value, click on **Calibrate** > **Scanner** > **Z**. The **Current Sensitivity** parameter is on the **Z Calibration** panel and can be changed by simply entering the new value.

Preamp Setting	Current Sensitivity Setting
10^{10}	0.1nA/V
10^{11}	0.01nA/V

12.6.5 Operation

The operation of the low-current STM head is typically checked in the atomic-scale imaging of graphite. The atomic-lattice of graphite is well resolved in the images obtained with I_{tun} in the 2-20pA and V_{bias} in the 20-100mV range. Mechanically-sharpened Pt/Ir tips are generally used for such measurements.

Peculiarities

The transition to low current brings some limitations, which are absent in STM measurements with standard heads. The bandwidth of the amplifier and, consequently, of feedback frequency response is substantially narrower than that used for standard STM measurements in the nA range (12kHz). This must be taken into account during imaging. The scan rate is typically reduced by at least half as compared to scan speeds normally used in the standard STM system.

However, the low current STM head can be used for imaging of atomically flat surfaces such as graphite and inorganic layered crystals at scan rates up to 50Hz and still achieve atomic-scale resolution in the Current data type.

Illustrative Examples

Several images obtained using low-current STM are presented below.

Figure 12.6b   STM current and height images of HOPG surface.
Scan size = 6.0nm, I_{tun} = 1.6pA, V_{bias} = 29mV.
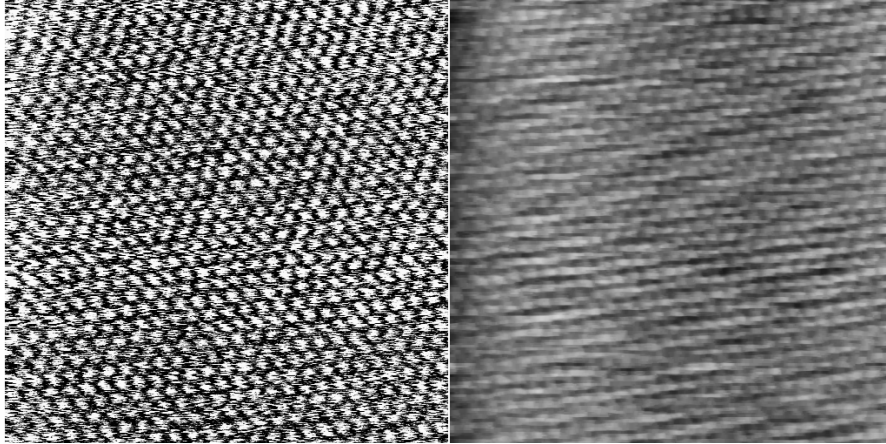
Figure 12.6c   STM current image of layered crystal α-RuCl$_3$.
Scan size = 4.48nm, I_{tun} = 1.5pA, V_{bias} = 42mV.
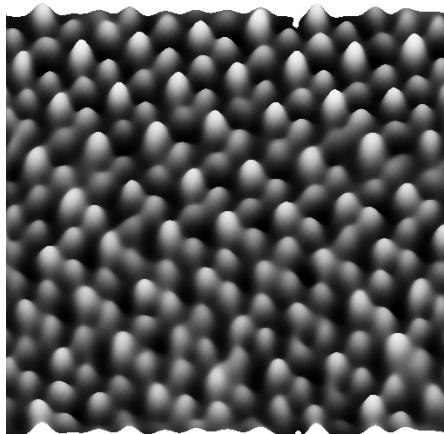
Figure 12.6d STM height image of alkanethiol layer on Au (111) substrate.
Scan size = 178.5nm, I_{tun} = 2pA, V_{bias} = 1V. (Courtesy of Dr. I. Tuzov, NCSU)
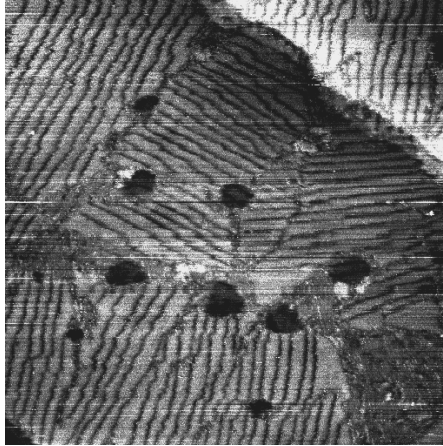
Figure 12.6e Molecular-scale STM current image of alkanethiol
layer on Au (111) substrate. Scan size = 10.0nm, I_{tun} = 13pA,
V_{bias} = 1V. (Courtesy of Dr. I. Tuzov, NCSU)
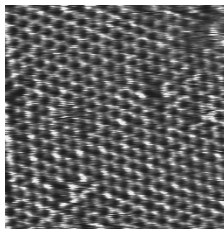
12.6.6 Servicing the Converter

The Low-current STM Converter features sensitive components which may be damaged if exposed to sudden voltage spikes. Spikes may be due to electrostatic discharge (ESD), or from external voltage sources. Operators should always wear an anti-static, grounding wristband (included in Low Current STM converter kit) while handling the head to protect against ESD.

The input op-amp is the most vulnerable component; therefore a spare is shipped with the unit. If the op-amp is damaged, it may be replaced by doing the following:

1. Turn off the microscope. Disconnect the Low-current STM Converter head by unplugging its cable from the support ring. Set the head on a workbench area grounded against ESD. Ensure that an ESD wristband is being worn.

2. Use the 0.050" allen wrench, included in the converter kit, to remove the two retaining screws which secure the head's cover. See Figure 12.6f.

Figure 12.6f Remove the cover on the STM head

Remove these two screws.
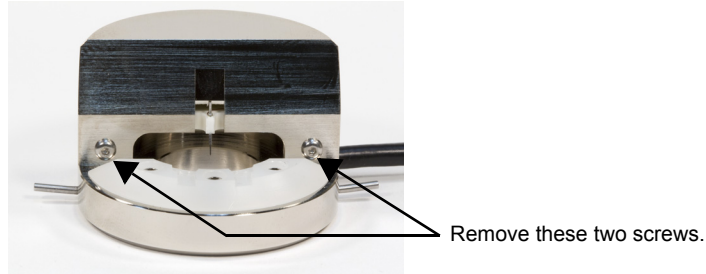
3. Pull the cover off to expose the PC board and electronics. It will appear as shown in Figure 12.6g.

Figure 12.6g STM PCB

Op-amp

Precision resistor and capacitor.
DO NOT TOUCH!
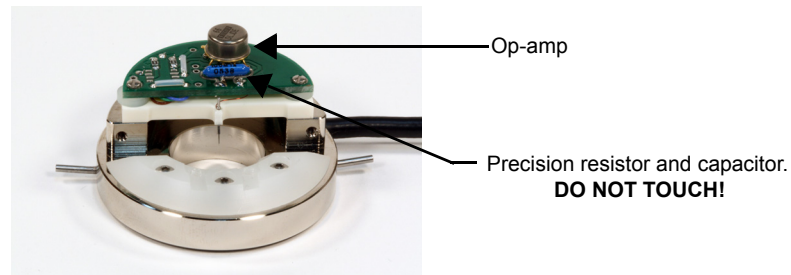
	CAUTION:	Throughout Step 4 - Step 6 below, avoid touching the precision resistor and capacitor. Any contaminant from the hands, including body oils, may cause excessive current flow, disabling the head. If these components are touched, they will have to be recleaned immediately using highly purified degreasing compounds.

4. Remove the op-amp by grasping it and pulling straight up. It should slide out of its socket.

5. Remove the new op-amp from its protective package. The op-amp is stored in conductive foam and its leads have been preformed to fit the socket. Be careful not to bend any of the leads. Orient the op-amp so that the metal tab on the edge of the can is in the "10:00" position as shown below. See Figure 12.6h.

Figure 12.6h Replace the op-amp

Metal tab at "10:00" position.

To remove, pull straight up.

To install, push straight down.
(Do not bend leads)
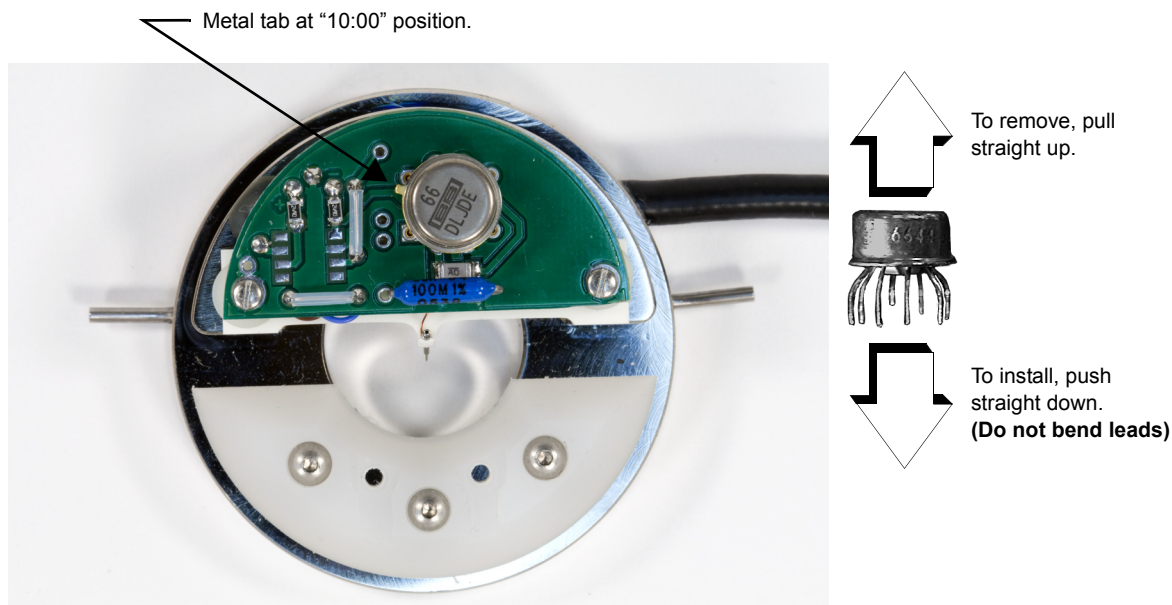
6. Verify that each of the op-amp's wire leads is properly aligned with the appropriate hole, then press the op-amp gently into its socket. Do not bend the wire leads.

7. Replace the metal cover on the head. Retighten retaining screws to secure. Do not overtighten.

12.7 Etching Tungsten Tips

You can purchase tungsten tips from Bruker or make them yourself. This section describes the process of etching tungsten tips.

Materials and Equipment Required:

- Variac auto transformer

- Optical microscope (20-100X)

- Sodium nitrite ($NaNO_2$)

- Distilled water

- Ethyl alcohol

- WD-40 (anti-oxidant)

- Two 50-ml beakers

- Tipholder (see below)

- Platinum wire

- Tungsten Wire, 0.010" Diameter

- Miscellaneous Wire/Clips

12.7.1 Procedure

1. Mix a 5 percent (by weight) solution of Sodium Nitrite in water.

2. Pour ∼ 40ml of the Sodium Nitrite solution into a beaker.

3. Pour ∼ 40ml of WD 40 into a beaker.

4. Construct an electrode out of the platinum wire and insert it into the beaker.

5. Adjust the variac for 30V, and with it Off, connect one output to the platinum electrode.

6. Cut 10 to 12 pieces of tungsten wire ∼ 1.25cm long. Before etching, check to make sure that at least one end of the wire has not split by inserting the ends into a tip holder. If an end has split, you will not be able to insert it into the tipholder. You can etch the end that has split, however, preserve the unspoiled end.

7. Place the tungsten tips into a holder. We like to use an IC socket (the low cost, edge-grip square contact type, not the machine-grip round contact type), with all the pins soldered together. The tips will then be held in place while inverting them over the solution. We also find it helpful to solder the IC socket to the back of a proto-board (perf-board). You can then invert the tips over a beaker with the proto-board sitting on the rim of the beaker.

8. Invert the tips over the Sodium Nitrite solution with ∼ 2mm of the tips' surface submerged. More than 2mm will cause excessive foaming of the solution during etching, and less than 2mm will result in tips that are too blunt.

9. Connect the other output of the variac to the common of all the tips.

10. Turn on the variac and etch the tips. While the tips are etching, the solution will foam, and the tips will start to glow. As the tips etch towards the surface, the foaming will be reduced. Continue to etch the tip until it stops.

11. Re-submerge the tips ∼ 1mm into the solution for ∼ 15 seconds at 30V. Turn on the variac and re-etch the tips. There should be only slight bubbling from the tip and it should not glow.

> **Note:** It is possible to vary the tip shape at this point by lowering the voltage on the variac and increasing or decreasing the amount of time the tips are submerged.

> **Note:** Longer time gives blunter tips.

12. Dip the tips into ethyl alcohol to clean them. If you plan to keep the tips around for more than a day, then dip them into the WD 40 after cleaning. Dispose of unwanted tips in an appropriately labelled solvent-contaminated waste container.

13. Examine the tips under the optical microscope. Tips that are too long, too blunt, or split at the end will hardly ever be good tips and can be thrown out at this time. Of course, this is a subjective process. As your experience in etching grows, you will get better at throwing out bad tips.

14. Repeat the etching procedure. Replace the etching solution when a fairly large amount of residue is present. Typically, you can etch 60 to 80 tips in a 40ml solution.

Chapter 13 Lateral Force Mode

The MultiMode SPM is capable of measuring frictional forces on the surfaces of samples using a special measurement known as lateral force microscopy (LFM). The name derives from the fact that cantilevers scanning laterally (perpendicular to their lengths) are torqued more as they transit high-friction sites; low-friction sites tend to torque cantilevers less. The relative measure of lateral forces encountered along a surface yields a map of high- and low-friction sites.

After obtaining a good topographical image in Contact Mode, it is relatively easy to use LFM to view and acquire lateral force data. It is important to obtain a good image in Contact mode before measuring LFM data. The NanoScope system will continue to run the feedback based on measuring the vertical deflection signal and feedback gains in the control panel while LFM data is acquired and displayed.

The following sections are included in this chapter:

- **Basic LFM Operation:** Section 13.1

- **Advanced LFM Operation:** Section 13.2

  - **Scan Direction:** Section 13.2.1

  - **Probe selection:** Section 13.2.2

  - **Understanding the LFM Signal:** Section 13.2.3

  - **Understanding the Color Scale:** Section 13.2.4

  - **Enhancing the LFM Data by Subtracting Two Images:** Section 13.2.5

  - **Height Artifacts in the Signal:** Section 13.2.6

13.1 Basic LFM Operation

13.1.1 Select Mode of Operation

1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 13.1a.

Figure 13.1a   The Contact Mode LFM **Select Experiment** window
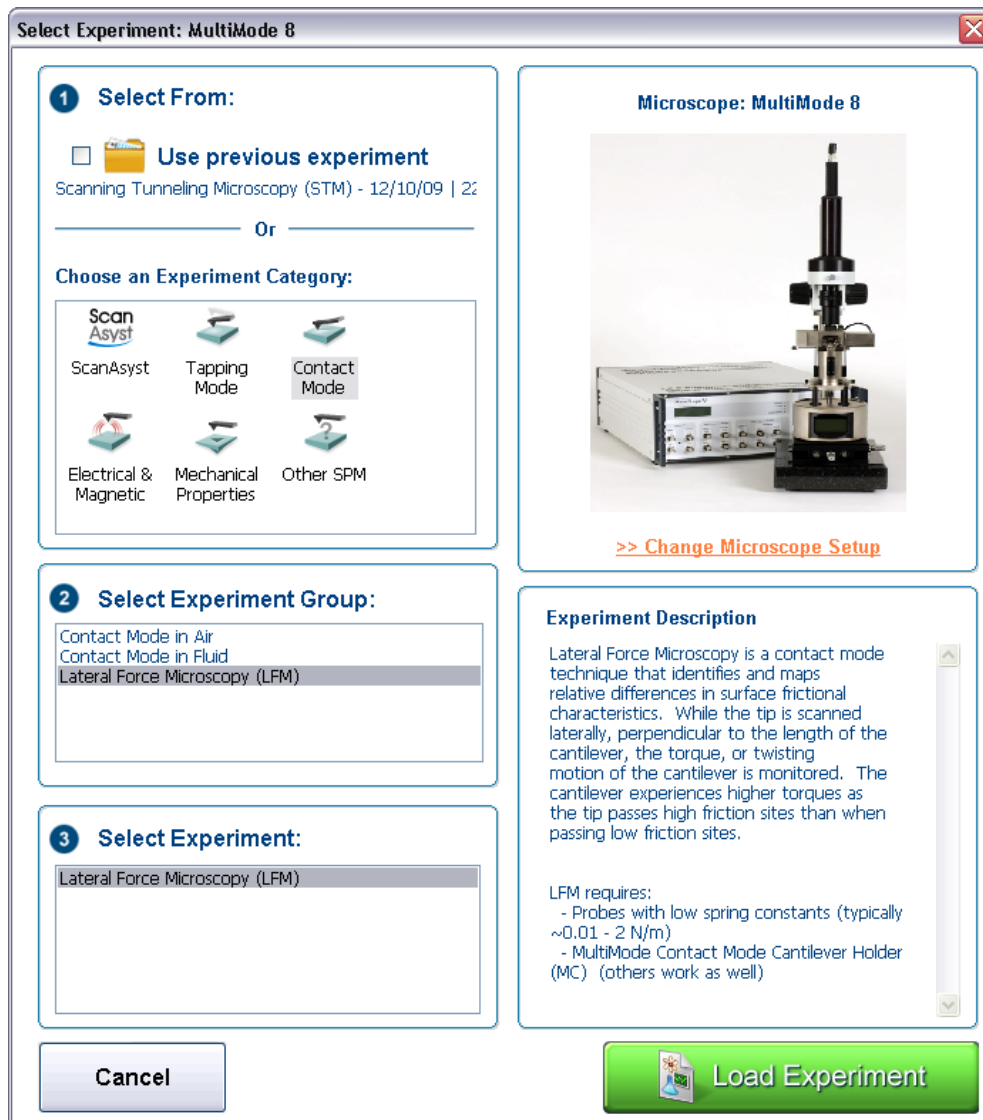
2. Select **CONTACT MODE** in the **Choose an Experiment Catagory** panel.

3. Select **LATERAL FORCE MICROSCOPY (LFM)** in the **Select Experiment Group** panel.

4. Select **LATERAL FORCE MICROSCOPY (LFM)** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. This opens the **Workflow Toolbar**, the **Scan 4 Channels** windows and the **Scan Parameters List** window, shown in Figure 13.1b.

Figure 13.1b   LFM (Simple Mode) configuration
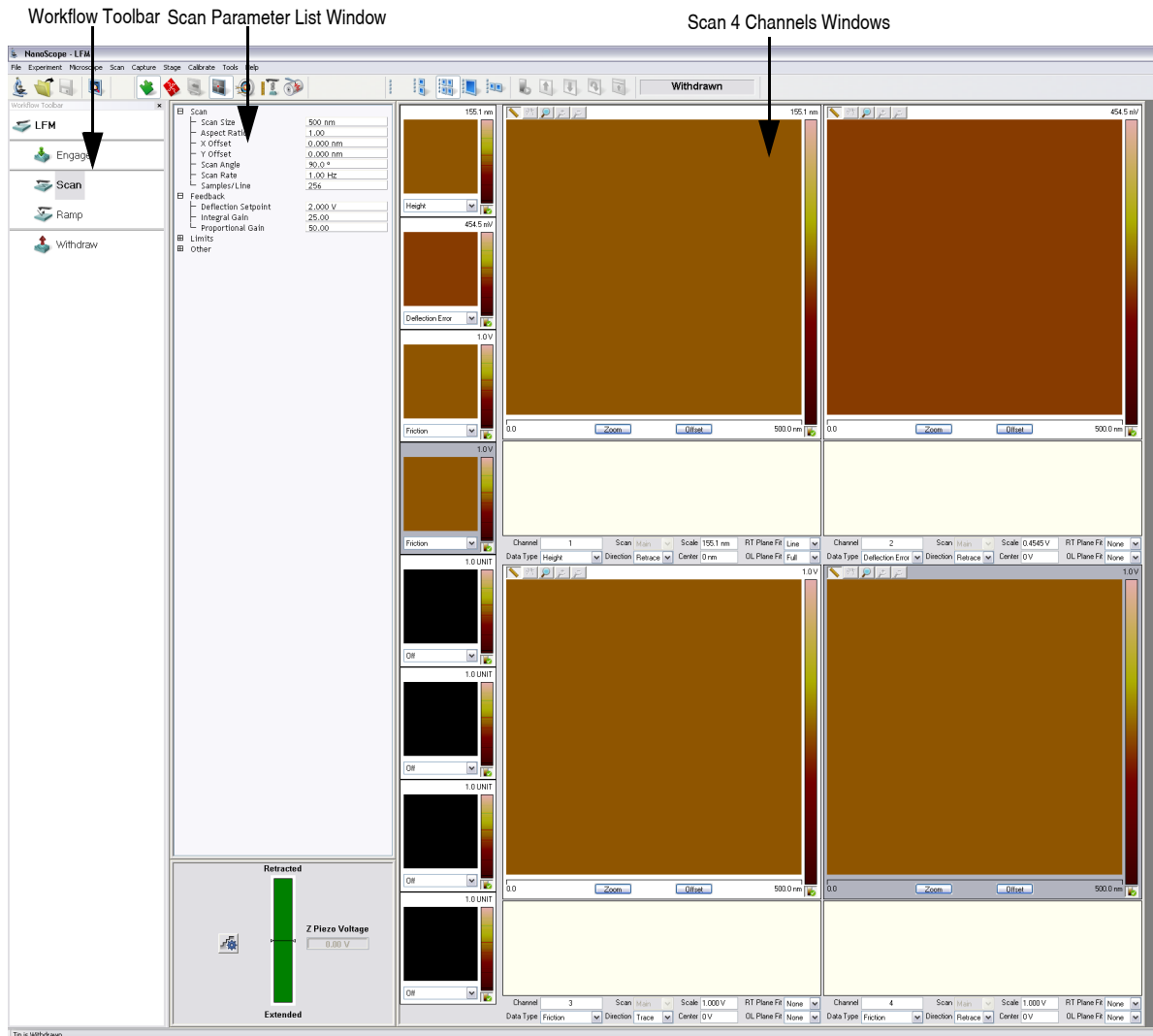
13.1.2 Head, Cantilever and Sample Preparation

1. Install a silicon nitride tip onto an AFM probe holder. See **Load Probe in Probe holder:** Section 7.1.3.

2. Install the probe holder in the MultiMode head. See **Install the Probe holder:** Section 7.1.4.

3. Align the laser on the cantilever. See **Laser Alignment:** Section 7.2.

4. Adjust the photo detector. See **Maximize the SUM Signal:** Section 7.2.3.

13.1.3 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 13.1c).

1. Set the **Scan Rate** to **1 Hz**.

2. Set the **Scan Size** to **500nm**.

3. Set the **Scan Angle** to **90.0** degrees.

Figure 13.1c Suggested Contact Settings

⊟ Scan	
├ Scan Size	500 nm
├ Aspect Ratio	1.00
├ X Offset	0.000 nm
├ Y Offset	0.000 nm
├ Scan Angle	90.0 °
├ Scan Rate	1.00 Hz
└ Samples/Line	256
⊟ Feedback	
├ Deflection Setpoint	0.5000 V
├ Integral Gain	2.000
└ Proportional Gain	5.000
⊟ Limits	
└ Z Limit	5.500 um
⊟ Other	
└ Units	Metric

Feedback Panel

1. Set the **Integral gain** to **2** and the **Proportional gain** to **5** (see Figure 13.1c).

2. Set the **Deflection Setpoint** to **0.5V**.

Other Panel

1. Set the **Units** to METRIC.

Channels 1 and 2

1. Set the **Channel 1 Data Type** to HEIGHT (see Figure 13.1d).

2. Set the **Channel 2 Data Type** to FRICTION.

3. Set the **Channel 3 Data Type** to DEFLECTION ERROR.

4. Set **Data Scale** to a reasonable value for the sample.

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

5. Set **Channel 2 Line direction** to TRACE.

   Note: If the **Line Direction** parameter is changed to **Retrace**, the plus-minus sign of the lateral force changes and low lateral force will be displayed on the top of the scope display. Setting the scan angle to 270 degrees will invert the polarity of **Trace** and **Retrace**.

Figure 13.1d   Suggested LFM **Channel 1**, **Channel 2** and **Channel 3** Settings

6. Set the **Channel 4** through **Channel 8 Data Type** to OFF.

7. Optimize the scan parameters in AFM mode for **Channel 1**.

   Note: LFM is a qualitative measurement, not quantitative. NanoScope software does not provide a way to calibrate the torsional spring constant of a cantilever.

   Note: The trace and retrace LFM signals may not be the same magnitude. The tip will never be exactly perpendicular to the sample. In most cases, internal stress in the material of the cantilever will cause the cantilever to have a natural bias in some direction.

13.2 Advanced LFM Operation

13.2.1 Scan Direction

The cantilever is most susceptible to frictional effects when the scan direction runs perpendicular to the major axis of the cantilever as shown in Figure 13.2a. The **Scan angle** parameter in the **Scan Controls** panel must be set to **90°** or **270°** to produce this scan direction.

Figure 13.2a   Scan Angle Selection
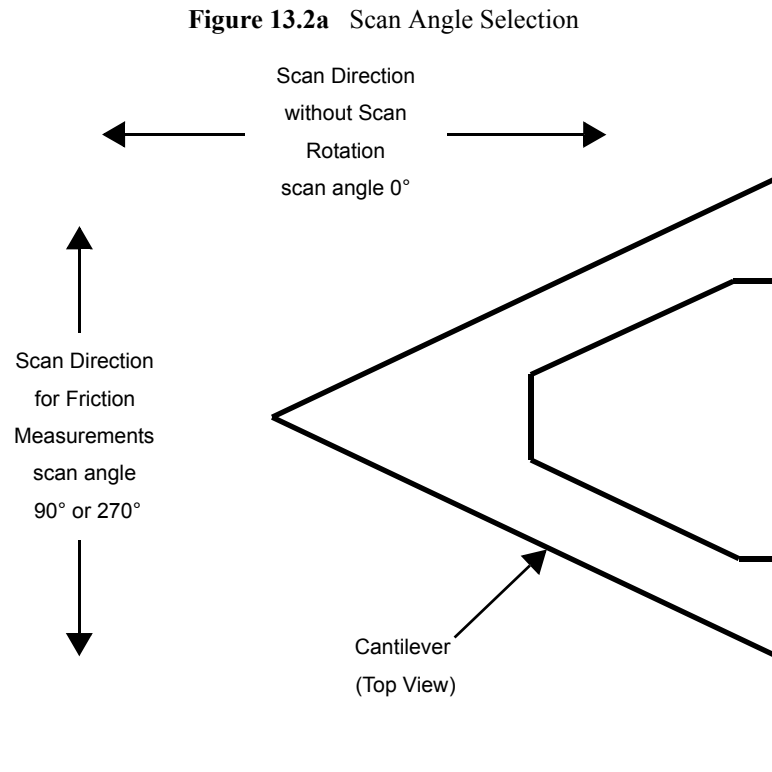
The contact mode setpoint voltage will slightly adjust the gain of the lateral force signal. By increasing the setpoint, the contact force applied will increase, and so will the frictional or torsional forces in an approximately linear fashion. If the frictional effects are far too large or too small, it will be necessary to resort to changing the cantilever probe used, but if the value is near the dynamic range desired, adjustment of the contact force will produce modest changes in the lateral force or frictional signal.

13.2.2 Probe selection

The analog-to-digital converter on the auxiliary input channel which is used for LFM data has a maximum input range of ±10V. This, and the anticipated interaction between tip and sample define the selection of the cantilever to be used for the measurement.

A 200μm cantilever with wide legs provides a good starting point for frictional measurements. It is flexible enough to provide reasonable signal levels on samples with moderate friction. If the signal exceeds $\pm$ 10.00V with the 200μm wide-legged cantilever, one of the stiffer 100μm cantilevers should be used. If the signal level is too small, the narrow-legged 200μm cantilever will provide a larger signal.

13.2.3 Understanding the LFM Signal

To understand LFM, examine a sample's trace and retrace in the scope window. Figure 13.2b below illustrates the movement of the tip as it encounters friction during a scan.

Figure 13.2b   Example of Frictional Forces on Tip During Scan (Front View)
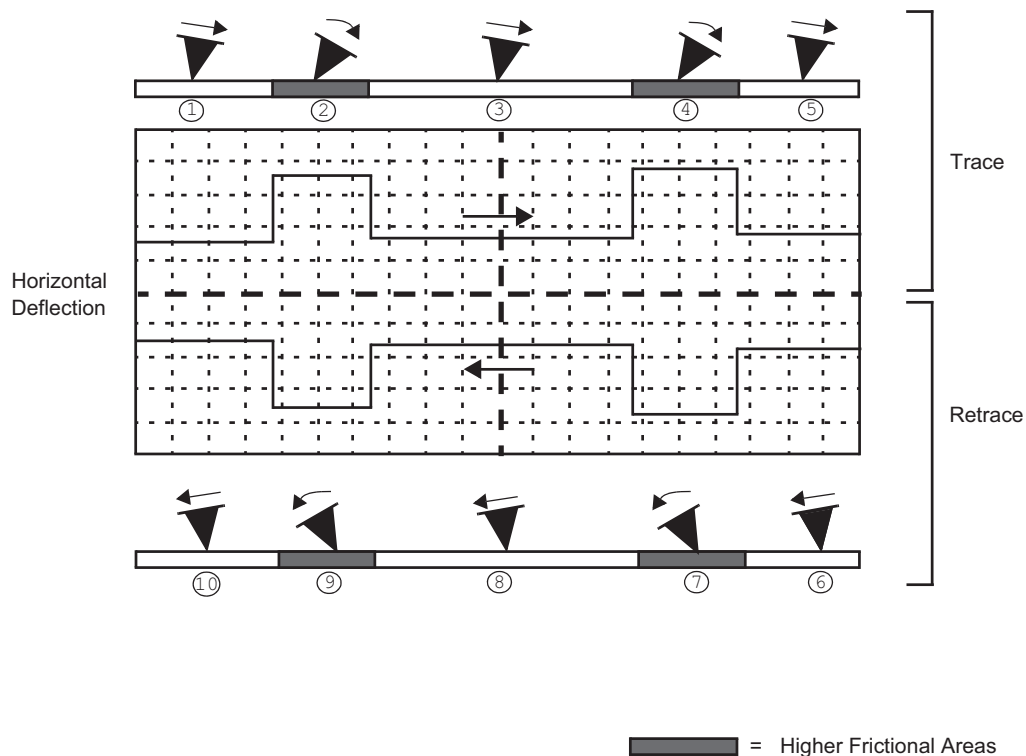
Table 13.2a Key for LFM Example Above

Number	Trace or Retrace?	Tip Movement	Level of Friction	Photo Diode Voltage
1	trace	slightly right	slight	slightly positive
2	"	severely right	higher	increases positive
3	"	straightens	slight	returns to slightly positive
4	"	severely right	higher	increases positive
5	"	straightens	slight	returns to slightly positive
6	retrace	slightly left	slight	slightly negative
7	"	severely left	higher	increases negative
8	"	straightens	slight	returns to slightly negative
9	"	severely left	higher	increases negative
10	"	straightens	slight	returns to slightly negative

13.2.4 Understanding the Color Scale

LFM data is typically monitored in the trace direction with the scan angle set to 90 degrees. The data then follows a convention where lighter colors mean higher friction. Darker colors mean less friction. The sign of the color is inverted if Retrace is monitored (see Figure 13.2c).

Figure 13.2c Friction Data
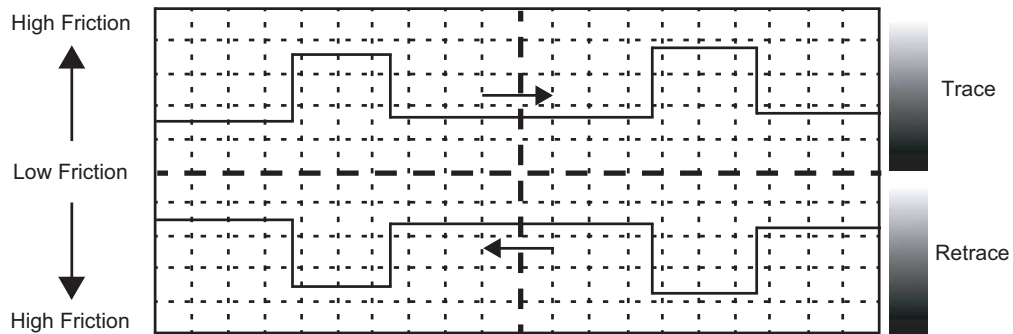
13.2.5 Enhancing the LFM Data by Subtracting Two Images

It is possible to enhance the magnitude of the LFM data by subtracting the trace from the retrace. This is done by collecting three data channels with one scan.

1. Set **Channel 1** to HEIGHT.

2. Set **Channel 2 Data Type** to FRICTION, **Line Direction** to TRACE, **Offline Plane Fit** to NONE.

3. Set **Channel 3 Data Type** to FRICTION, **Line Direction** to RETRACE, **Offline Plane Fit** to NONE.

4. Collect a scan at **90** degree **Scan Angle** and save it.

5. Load the file and use the SUBTRACT IMAGE subtract feature to subtract RETRACE from TRACE. The resulting data will show double the LFM data and half the background noise caused by tracking error. Information about the Subtract Image function can be found the *NanoScope Software 8 User Guide*, Bruker p/n 004-994-000.

> **Note:** The X-Y motion of an open loop scanner in Trace and Retrace does not overlap exactly. You can still use image subtraction, but some erroneous height data might be added to the LFM data in the final image. This is not an issue for closed loop scanners.

13.2.6 Height Artifacts in the Signal

LFM is subject to height artifacts due to coupling with surface topography. Delay in the feedback loop causes the tip to momentarily twist as it climbs up an edge. This will be visible in the friction data if it's severe enough.

To distinguish real friction data from height artifacts, remember that friction causes the cantilever to twist in the opposite direction as it travels. Actual LFM information will always show up as a mirror image in the trace and retrace directions in the scope display. Height artifacts will be the same direction in the scope display (see Figure 13.2d).

Figure 13.2d   Height Artifacts in LFM Data

0 = No torsion
1 = Slight torsion
3 = High torsion
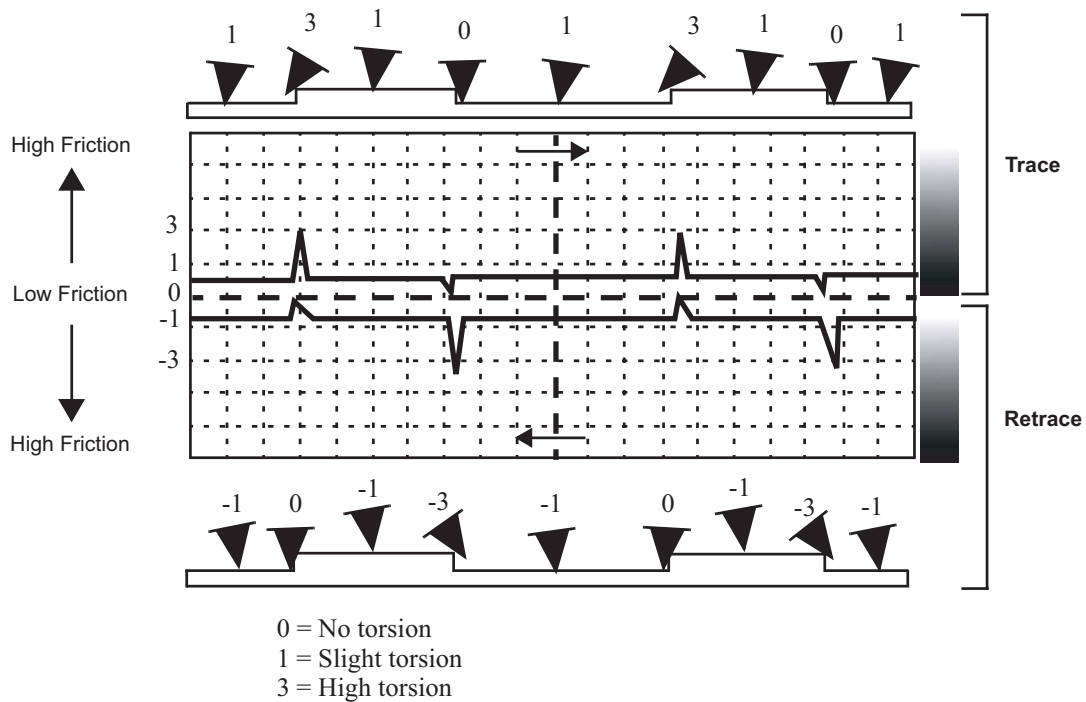
Chapter 14 Force Imaging

Force plots measure tip-sample interactions and determine optimal setpoints. More recently, microscopists have plotted force measurements across entire surfaces to reveal new information about the sample. This area of scanning probe microscopy promises to open new chapters in materials science, biology and other investigative areas.

Specifically, this chapter details the following topics:

- **Force Calibration (TappingMode):**

  - **Force Plots:**

  - **Obtaining a Force Plot (TappingMode):**

  - **High Contact Force:**

  - **Probe Selection:**

- **Force Modulation:**

  - **Introduction:**

  - **Selecting a Force Modulation Probe:**

  - **Operating Principle:**

  - **Force Modulation Procedure:**

  - **Select Mode of Operation:**

  - **Head, Cantilever and Sample Preparation:**

  - **Align Laser:**

  - **Adjust the Setpoint:**

  - **Adjust Photodetector:**

  - **Find the Bimorph Resonant Frequency::**

  - **Set Initial Scan Parameters:**

  - **Engage:**

  - **Optimize Scan Parameters:**

  - **Notes About Artifacts:**

- **Force Modulation with 'Negative LiftMode':**

  - **Set Interleave Controls:**

  - **Obtain a TappingMode Image:**

  - **Obtain a Negative LiftMode Force Modulation Image:**

- **Force Volume:**

14.1 Force Plots–An Analogy

A force plot is an observation of tip-sample interactions which yields information regarding the sample and tip. By way of analogy, suppose a materials researcher must determine how powerful two different types of magnets are. One magnet is made of iron, the other is a stronger, so-called rare earth magnet.

A simple way of measuring each magnet's power would be to determine its pull upon a steel plate. For example, the researcher could hang each magnet from a simple spring scale, "zero" the scale, then lower the magnet toward a heavy steel plate. At regular distances from the plate, the amount of pull shown on the scale is recorded. At some unique height above the plate, each magnet is attracted strongly enough to attach itself to the plate. A plot of height, H, versus magnetic pull gives a comparative index of each magnet's power. Similarly, after attachment the researcher could pull each magnet away from the plate and measure the pulling force at regular intervals until the magnet breaks free. The pull-off point of each magnet gives an additional index of its holding power.[1] A representation of this setup during a lowering cycle of one magnet is illustrated in Figure 14.1a.

Figure 14.1a   Comparative Index of Pulling Forces
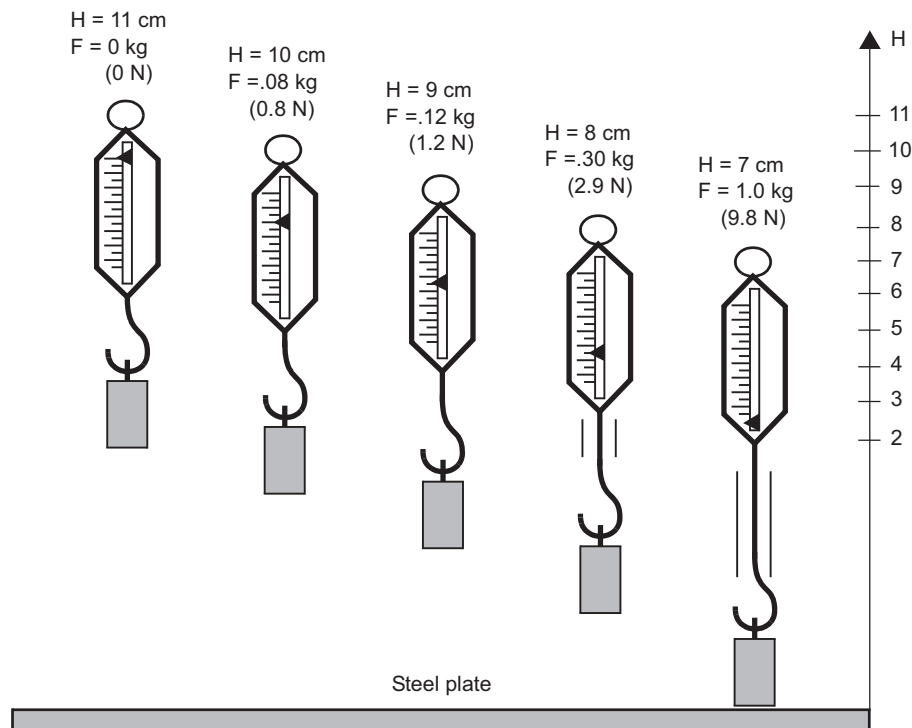
The pulling force is measured at 1 cm height intervals while the scale and magnet lower and lift in a controlled cycle. Figure 14.1b depicts a plot of this experiment using two magnets. The plot depicts each magnet's attraction as it approaches the plate, and its tenacity when pulled off the plate. Assuming both magnets are the same size, this reveals information about each magnet's

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1. For the sake of simplicity, forces are represented here using a common scale in kilogram units; however, force is properly measured in Newtons (1 kg = 9.8 N).

strength. First, magnet #1 is weaker, attaching to the steel plate with 7N of pulling force at 6cm, and magnet #2 is stronger, attaching at 7cm with a force of 10N.

Figure 14.1b   Pulling Forces Graph
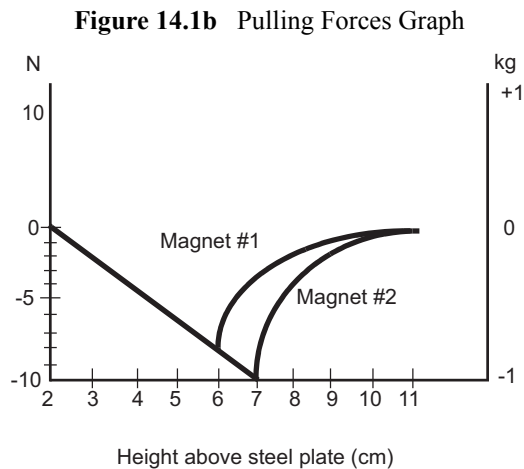
This oversimplified model depicts activity between SPM tips and various materials. In reality, SPM force plots reveal far more. For example, by combining force curves at regularly spaced intervals across the sample, you may generate a force map of the sample's electric properties, elastic modulus, and chemical bonding strengths.

14.2 Force Calibration Mode

The **Continuous ramping** command in the **Ramp** menu allows you to quickly check the interaction between the cantilever and the sample surface. In the **Continuous ramping** mode, the X and Y voltages applied to the piezo tube are held constant and a triangular waveform similar to the one depicted in Figure 14.2a is applied to the Z electrodes of the piezo tube.

Figure 14.2a   Continuous ramping Z Waveform

$$\text{Scan period (sec)} = \frac{1}{\text{Z scan rate (Hz)}}$$
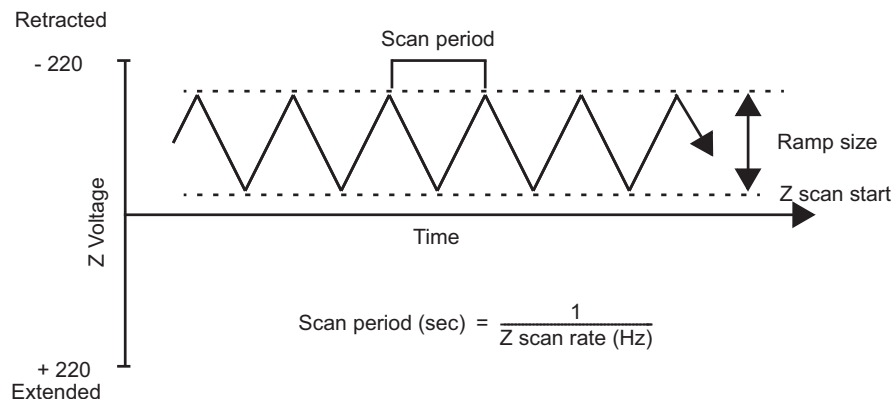
As a result of the applied voltage, the sample moves up and down relative to the stationary cantilever tip as shown in Figure 14.2b. The **Z scan start** parameter, available through the **Ramp Parameter List**, sets the offset of the piezo travel, while the **Ramp size** parameter defines the total travel distance of the piezo. Therefore, you can obtain the maximum travel distance by setting the **Z scan start** to **+220V**, with the **Ramp size** set to **440V**.

Figure 14.2b   Piezo Travel in Continuous ramp Mode
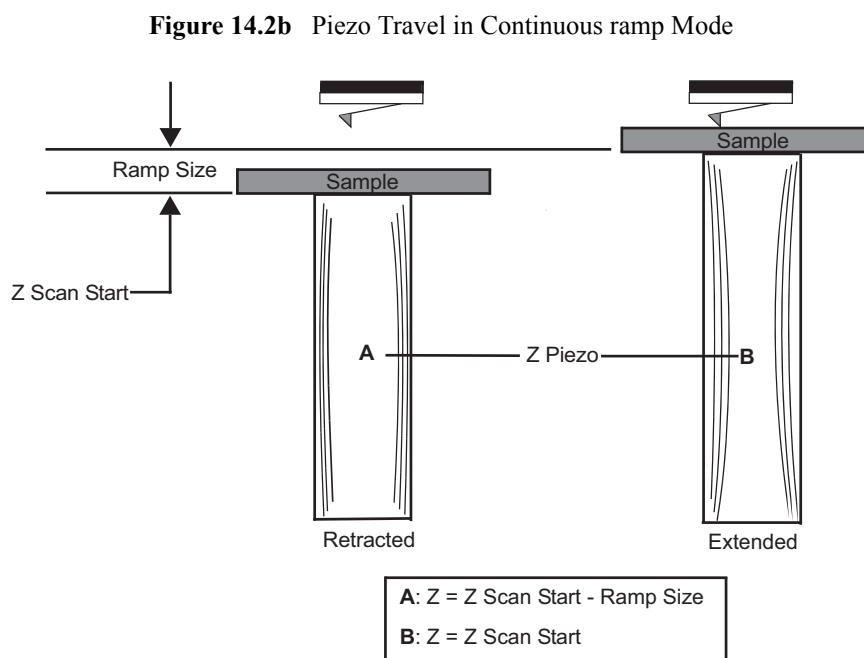
As the piezo moves the sample up and down, the cantilever-deflection signal from the photodiode is monitored. The force curve, a plot of the cantilever deflection signal as a function of the voltage applied to the piezo tube, displays in the **Force Plots** window. The controls detailing parameters for controlling the microscope in **Continuous ramping** mode are available in the **Force Plots** window and the **Ramp Parameters List** window.

Continuous ramp mode is frequently used to adjust and calculate contact forces between the cantilever and the sample. Other uses of **Continuous ramp** mode include characterization of the forces on the cantilever tip, diagnosing AFM performance, and determination of the sensitivity of the cantilever deflection voltage in terms of voltage applied to the piezo. If used correctly, **Continuous ramp** mode provides a variety of useful information.

14.2.1 Example Force Plot

A Contact Mode AFM force plot using a silicon nitride probe is the most simple SPM force plot. Because of the lower spring constant of silicon nitride probes, they are very sensitive to attractive and repulsive forces. A force plot in Contact Mode AFM is shown in Figure 14.2d.

Figure 14.2c compares portions of the force curve shown in Figure 14.2d to relative positions of the tip and sample at seven points. The force curve represents the deflection signal for each complete extension-retraction cycle of the piezo (Figure 14.2a). The **Z scan rate** parameter in the **Ramp Parameter List** defines the rate at which the piezo completes an extension-retraction cycle (and therefore the rate at which new curves are displayed).

Figure 14.2c   Tip-Sample Interaction During a Force Plot
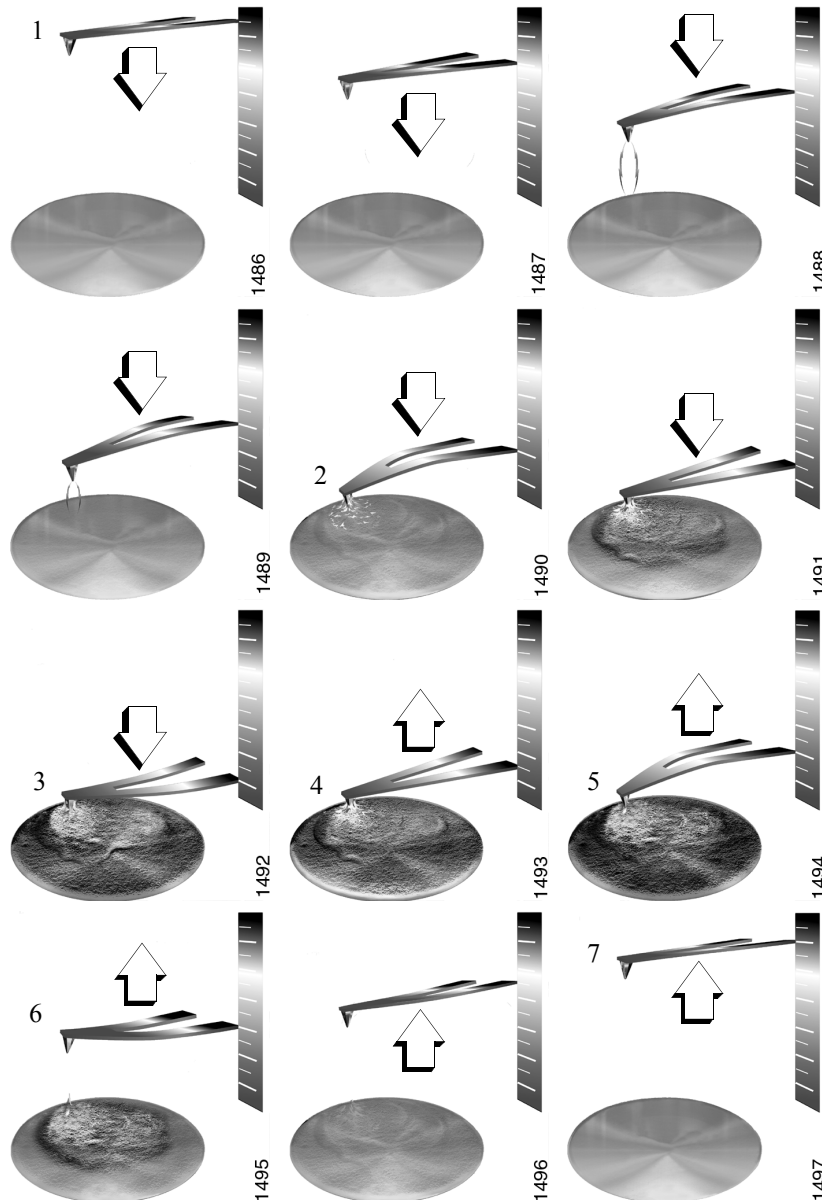
Figure 14.2d   Anatomy of a Force Curve
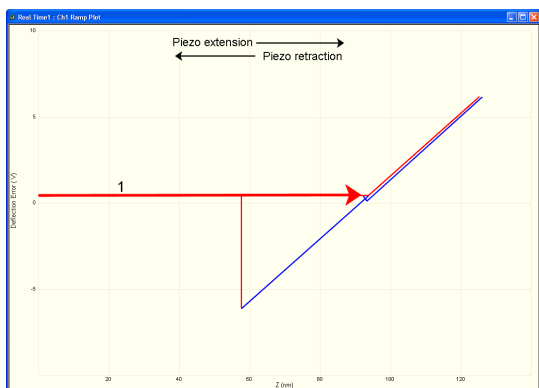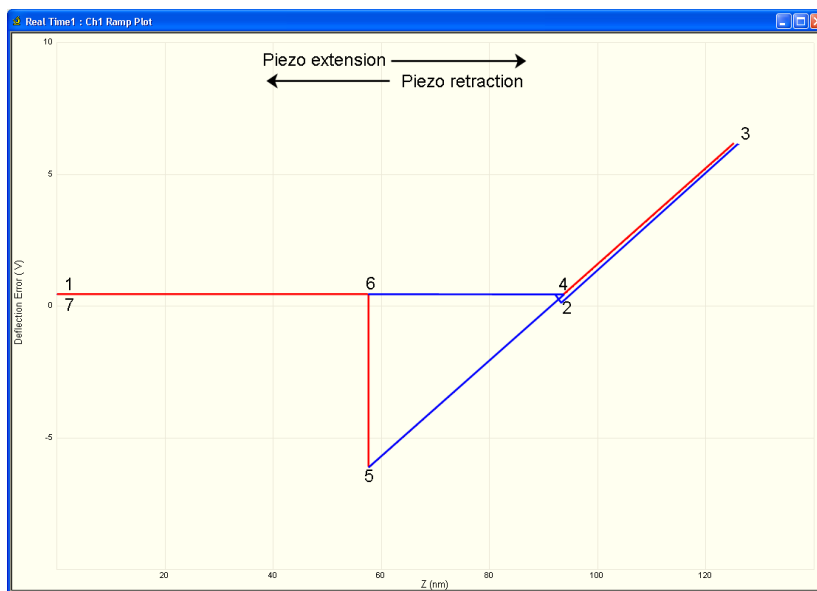
Piezo extends; tip descends.
No contact with surface yet.
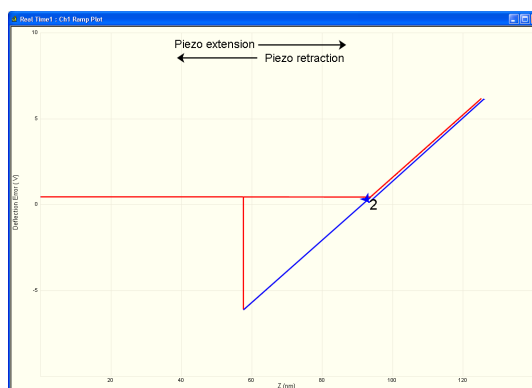
Attractive forces near surface
pull tip down.
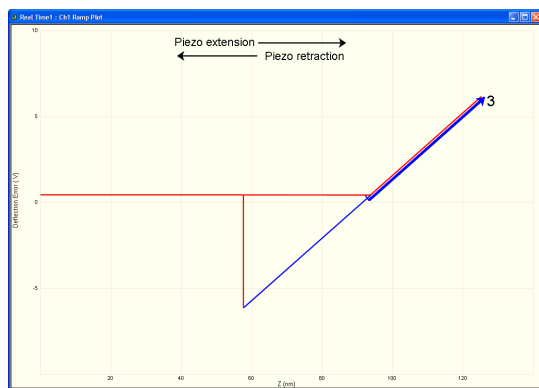
As tip presses into the surface,
cantilever bends upward.
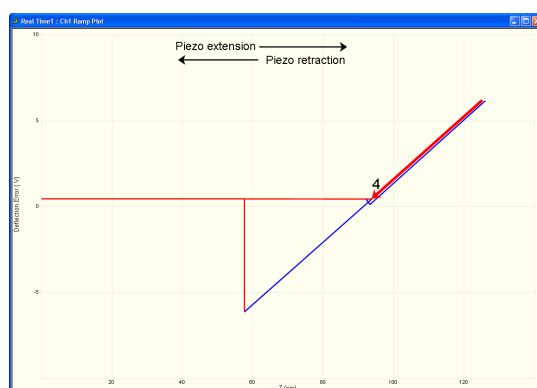
Piezo retracts; tip ascends until forces are in
equilibrium with surface forces.
Cantilever relaxes downward.

Figure 14.2e Anatomy of a Force Curve (continued)
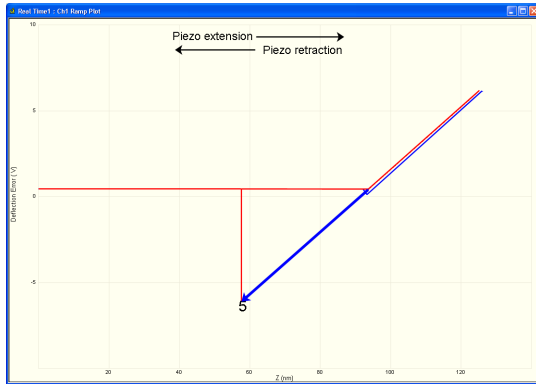
Piezo continues retraction; tip ascends further. Cantilever bends downward as surface attraction holds onto the tip.
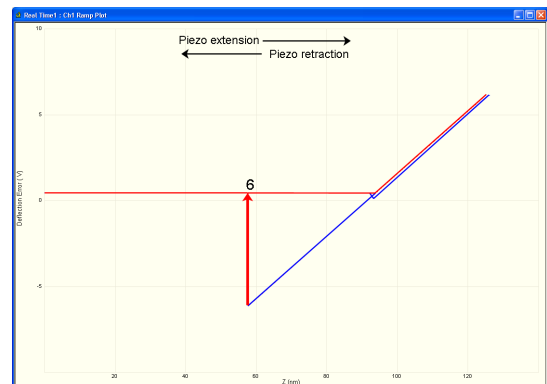
As tip continues its ascent, tip finally breaks free of surface attraction. Cantilever rebounds sharply upward.
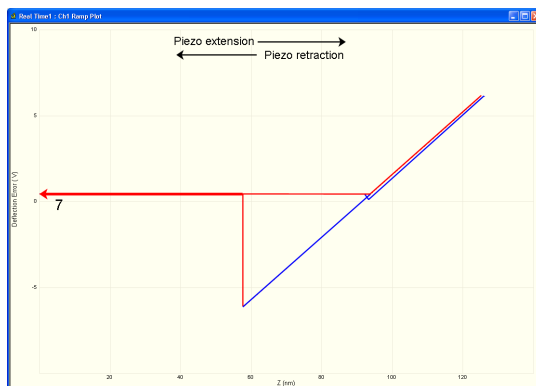
As piezo continues retracting, tip continues its ascent. No further contact with surface this cycle.

The horizontal axis plots the tip movement relative to the sample. By extending the Z-axis piezo crystal, the tip descends toward the sample and the tip-sample distance decreases. The descent plots from left-to-right in red in the NanoScope **Force Plots** window. By retracting the Z-axis piezo crystal, the tip ascends away from the sample and the tip-sample distance increases. The ascent plots from right-to-left in blue on the NanoScope **Force Plots** window.

Cantilever deflection plots on the vertical axis of the graph. When the cantilever deflects downward, it plots on the graph's downward vertical; when the cantilever deflects upward, it plots on the graph's upward vertical.

You can use force plots to adjust the setpoint so that minimal force is applied to the sample. Although attractive forces appear small, the tip is extremely sharp. Because only a few nanometers of the tip actually touch the sample, even minute forces add up quickly when distributed over an exceedingly small area. The tip can easily dent many materials under such conditions.

The graph detailed in Figure 14.2d reveals the following types of information:

Tip-Sample Attraction

As the tip approaches the sample, various attractive forces reach out and grab the tip. This is evident at point 2 (slight dip) in the graph above. The tip plunges toward the sample during its descent. This is also referred to as the "jump-to-contact" point and is usually due to electrostatic attraction or surface tension (capillary) forces.

Attraction is also evident between points 4 and 5 (sloped line) as the cantilever pulls away from the sample. If attractive forces are strong enough, the tip clings to the sample surface as it pulls clear. Eventually, the sample retracts and the tip rebounds sharply upward (white line between points 5 and 6). You can measure attractive forces of tip-sample interactions if you know the spring constant.

Material Elasticity

It is possible to extract information regarding the elasticity of the material by studying force curves. In the graph above, the tip is in constant contact with the sample between points 2 and 4. As the tip presses further into the sample material, the cantilever flexes. The amount of cantilever flexion for a given amount of downward tip movement indicates the material's elasticity.

For example, if the material is extremely hard, pressing the tip downward results in a relatively large amount of cantilever flexion. On the other hand, if the material is soft, the cantilever will flex less during its descent. The shape and slope of the contacted portion of the force curve gives detailed information about surface elasticity. It is possible to obtain quantitative measurements of sample elasticity. (See Radmacher, *et al*. 1994. *Science*, Vol. 265:1577-1579).

Two imaging techniques measure and display elasticity at multiple points on a sample surface: **force modulation** and **force volume imaging**.

14.3 Ramp Parameter List

The **Ramp Parameter List** window, shown in Figure 14.3a, manipulates the microscope in **Ramp** mode. The parameters control the rate, start position and amplitude of the triangle wave applied to the Z piezo. You can also adjust the **Setpoint** value of the cantilever deflection voltage used in the feedback loop during imaging. The **Capture** button stores the force curve for **Offline** viewing. Some parameters influence the operation of the microscope during imaging; most of the menu parameters affect only **Ramp** mode.

Figure 14.3a   Ramp Parameter List (Contact Mode AFM) Expanded Mode

To view the **Ramp Parameter List** click the RAMP icon in the **Workflow Toolbar**.

Items in the **Ramp Parameter List** window are discussed individually below.

14.3.1 Ramp Controls

Ramp Output

This parameter specifies which output you will ramp. To collect force plots, this parameter should be set to **Z**. For STM IV curves, **Bias** is typically selected.

Ramp Size

As shown in Figure 14.3a, this parameter defines the amplitude of the triangular waveform applied to the Z piezo. The units of this item are volts or nanometers, depending on the setting of the **Units** parameter. Regardless of the size of the scan, the entire scan is shown in the force curve; therefore, increasing the value of this parameter increases the horizontal axis scale on the force plot.

Z scan start

This parameter defines the maximum voltage applied to the Z electrodes of the piezo during force calibration operation. The triangular waveform shown in Figure 14.2a displaces the piezo up and down in relation to the value of **Z scan start**. Increasing the value of the **Z scan start** parameter moves the cantilever closer to the sample by extending the piezo tube. The units of this item are volts or nanometers, depending on the setting of the **Units** parameter. The initial value of **Z scan start** is equal to the average Z-center voltage defined by the feedback just prior to entering the **Ramp Parameter List**. Decreasing the value of this parameter shifts the force curve on the display to the right, while increasing the parameter shifts the curve to the left.

Scan Rate

This parameter defines the rate that force plots are collected (typically ~1Hz). The ramp size, forward velocity and reverse velocity determine the scan rate.

Forward Velocity

This parameter defines the piezo speed to bring together the tip and sample.

Reverse Velocity

This parameter defines the piezo speed to separate the tip and sample.

X Offset, Y Offset

These parameters define the X and Y positions where the force plot will be collected, offset from the image center.

Number of samples

This parameter defines the number of data points captured during each extend and retract operation of the Z piezo during a ramp. This parameter does not affect the number of samples used in **Image Mode**.

Spring Constant

The parameter defines the spring constant of the cantilever. The spring constant will be saved with captured force plots. The spring constant can be evaluated using the Thermal Tune method. Refer to the *NanoScope V Controller Manual NanoScope Software v 8* (Bruker p/n 004-1030) for details.

Plot Units

This item selects the units, either metric lengths or volts on the piezo, that define the parameters and the axes of the graph. Force may be displayed in nN of deflection force. This uses the spring constant. Changing this item in **Ramp** mode also changes it in the **RealTime** imaging mode.

Display Mode

This parameter defines the display as either **EXTEND**, **RETRACT**, or **BOTH**.

X Rotate

X Rotate allows you to move the tip in the X direction during ramping. This is useful because the cantilever is at an angle relative to the surface. **X Rotate** prevents the cantilever from plowing the surface laterally, typically along the X direction, while it indents in the sample surface (in the Z direction). Plowing occurs when the cantilever bends during indentation or with X movement caused by coupling of the Z and X axes of the piezo scanner. **X Rotate** moves the scanner in the opposite direction in which the cantilever points. Without **X Rotate** control, the tip moves in the direction of the cantilever during indentation. Normally, it is set to about **22.0 degrees**. This parameter typically ranges between **12** and **25 degrees**.

14.3.2 Channel 1, 2, 3 Panels

Data Type

This parameter allows you to select the type of data you want to display on the vertical axis. **Data Type**: Channels may be assigned to any input signal available including: **Deflection**, **Amplitude**, **Phase**, **Friction** and so on. Any channel may be switched to Off, however, *at least one channel* is always on.

Data Scale

This parameter sets the vertical scale in the force plot. Increasing this parameter expands the range of the display about the centerline causing more of the force curve to fall on the graph. Initially, set this parameter to its maximum value, then gradually reduce.

Data Center

This parameter shifts the displayed force plot in the vertical axis.

> **Note:** For **Data type** = **Deflection** in Contact Mode or **Data type** = **Amplitude** in TappingMode, the data is also shifted by the setpoint.(i.e., if the **Data type** = **Deflection** and **Data Center** = **0**, then the setpoint is at the center of the vertical scale.

TM Deflection Sensitivity (TappingMode)

TM Deflection Sensitivity relates the vibrational amplitude of the cantilever (in nm) to the amplitude signal (in V) from the controller. To calculate the **TM Deflection Sensitivity**, measure the slope of the **RMS amplitude** versus the **Z piezo position** when the tip is in contact with a very hard, stiff sample. The NanoScope software automatically calculates and enters the value from the graph. You must properly calibrate the **TM Deflection Sensitivity** before amplitude data can be correctly displayed in nm.

14.3.3 Feedback Panel

All of the parameters in the **Feedback** panel also affect **Image** mode.

Deflection Setpoint (Contact Mode)

By changing the deflection setpoint, you can adjust the cantilever deflection voltage maintained by the feedback loop in **Image** mode. This parameter defines the center of the range of Cantilever Deflection Voltages that can be collected. If the **Data Center** = **0**, then the setpoint is at the center line of the force plot. Changing the setpoint shifts the force calibration curve on the graph. For example, if the setpoint is set to -3.0V and the data center is 0, the cantilever deflection axis of the graph centers around -3.0V. Raising the setpoint to -2.0V shifts the force calibration curve down by one volt so the graph centers at -2.0V. Changing the value of this parameter also changes it in **Image** mode.

Amplitude Setpoint (TappingMode)

The RMS value of the cantilever deflection voltage maintained by the feedback loop in **Image** mode can be adjusted by changing **Amplitude Setpoint**. In a TappingMode force plot mode, the setpoint defines the centerline of the vertical, "Amplitude" axis of the amplitude calibration plot (the Data Center must be 0 for that Channel). Changing the **Amplitude Setpoint** shifts the

amplitude curve on the graph. For example, if the setpoint is at 3.0V, the RMS amplitude axis of the graph will be centered around 3.0V; raising the setpoint to 4.0V will shift the amplitude curve down by one volt, so the graph will be centered at 4.0V. Changing the value of this parameter in the **Amplitude Calibration** mode also changes the setpoint parameter in **Image** mode.

Z Limit

This parameter changes the gain applied to the input signal. You can reduce the input signal to see more of the **Force Calibration** plot deflection or amplitude. This parameter also affects **Image** mode and the resolution of the system; therefore, change the value back to the **Image** value before returning to imaging.

Drive Frequency (TappingMode)

This parameter defines the frequency at which the cantilever is vibrated. This frequency should be close to the resonant frequency of the cantilever. Changing the value of this parameter in the **Force Plot** menu also changes the **Drive Frequency** parameter in the **Image** mode.

Drive Amplitude (TappingMode AFM)

This parameter defines the amplitude of the voltage applied to the piezo system which drives the cantilever vibration. Changing the value of this parameter in **Force Plot** mode also changes the **Drive Amplitude** parameter in the **Image** mode.

14.3.4 Mode Panel

Trigger Mode

The **Mode** panel allows you to use various triggers when obtaining **Force Plot** and **Force Volume** plots. The idea of triggering is simple: it limits the total amount of force exerted by the tip upon the sample. Depending upon which trigger you use and how it is set, you may operate the trigger independent of drift (**Relative**) or at some arbitrarily fixed point (**Absolute**).

Figure 14.3b Absolute and Relative Triggers
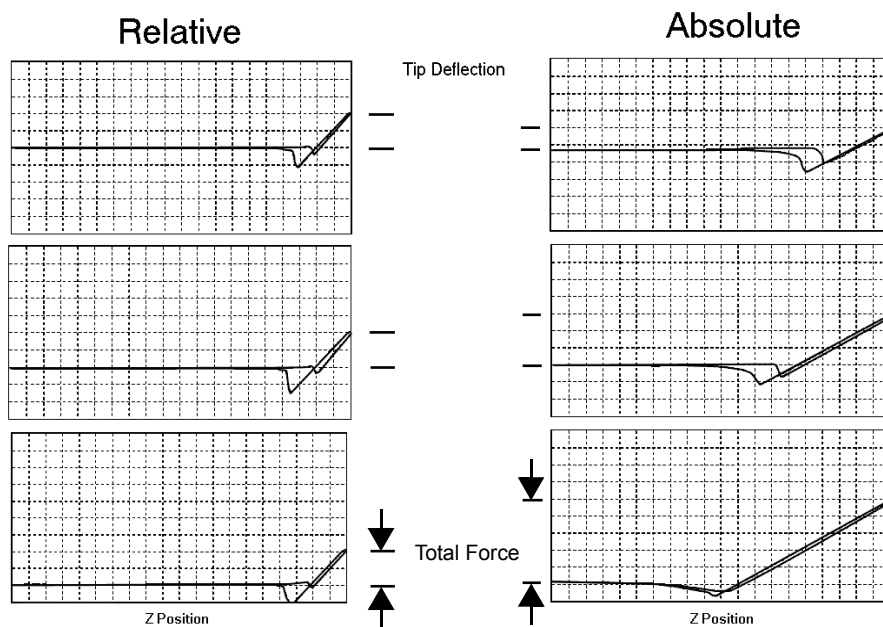
The plots in Figure 14.3b show the effect of drift on each of the two trigger types. The plot series shown on the left side of Figure 14.3b utilizes a **RELATIVE** trigger and maintains force at a constant level defined by the **Trig threshold** parameter. The **RELATIVE** trigger threshold is relative to the data at the right of the plot where the cantilever is typically undeflected. As illustrated, the total force is maintained, even as the system drifts.

The plot series on the right side of Figure 14.3b utilizes an **ABSOLUTE** trigger. Notice that as the system drifts (plot moves slowly downward), total force increases. Drift may be due to mechanical causes, or due to thermal effects on the cantilever. An **ABSOLUTE** trigger permits the total force to be set using **Setpoint** values.

Data Type

This parameter defines the Data Type to watch to determine when to reverse the ramp.

Trigger Threshold

This parameter as well as the **Trigger direction** and **Trigger mode** define the level at which triggering occurs.

Trigger Direction

The **Trigger Direction**s are defined as follows:

- **POSITIVE**: Trigger occurs on positive values only (**Trigger Threshold** must be positive)

- **NEGATIVE**: Trigger occurs on negative values only (**Trigger Threshold** must be negative)

- **ABSOLUTE**: Sign of **Trigger Threshold** is ignored (trigger may occur on positive or negative values).

Start Mode

The Start Mode parameters are defined as follows:

- **CALIBRATE** and **STEP**: Normal ramping (no stepping) unless you use **RAMP** > **RAMP SINGLE** or **RAMP CONTINUOUS**.

- **MOTOR STEP**: Uses the stepper motor to offset ramp instead of the piezo.

End Mode

The **End mode** parameters specify the location of the tip when the mode is switched back to Image mode. **RETRACTED** is the typical setting.

	CAUTION:	Use caution when using the **EXTENDED End mode** parameter; you could damage the tip.

Z Step Size

This parameter defines the size of Z step for **Step** or **Motor Step** modes. For **Step**, it is the amount to move the piezo; for **Motor Step** it is the amount to move the motor.

Auto start

When **Enabled** this parameter starts taking force curves upon entering Force Calibrate Mode.

Surface Delay

This parameter sets the amount of time to wait with the piezo extended before retracting.

Retracted Delay

This parameter sets a delay to occur each time the piezo is retracted while continuously ramping.

14.3.5 Menu Bar Commands

Capture

The **CAPTURE** button stores the force plot for **Offline** viewing. Use other options in the **Capture** menu to change a capture filename and for **CONTINUOUS CAPTURE** of all the force plots collected.

Probe Menu Commands

These commands allow for highly controlled tip movement designed to prevent damage to the tip. Each of these commands has an icon to access directly from the menu bar.

- **RAMP CONTINUOUS**: The tip is continuously lowered and raised by a distance equal to the Z scan size. This is the normal, default motion during **Force Calibrate**.

- **RAMP SINGLE**: Lowers and raises tip *once* by a distance equal to the Z scan size, then halts.

- **STOP**: Halts all tip movement.

Step Motor

The **Microscope > Step Motor** menu allows you to withdraw or manually control the tip position using the stepper motor. Selecting **Step Motor** opens a dialog box containing the following buttons:

- **Tip Up**: This command moves the tip up by the **SPM step size** displayed inside the window.

- **Tip Down**: This command moves the tip down by the **SPM step size** displayed inside the window.

14.4 Force Calibration (Contact Mode AFM)

14.4.1 Obtaining a Good Force Curve

Figure 14.4a   Typical Force Calibration Curve
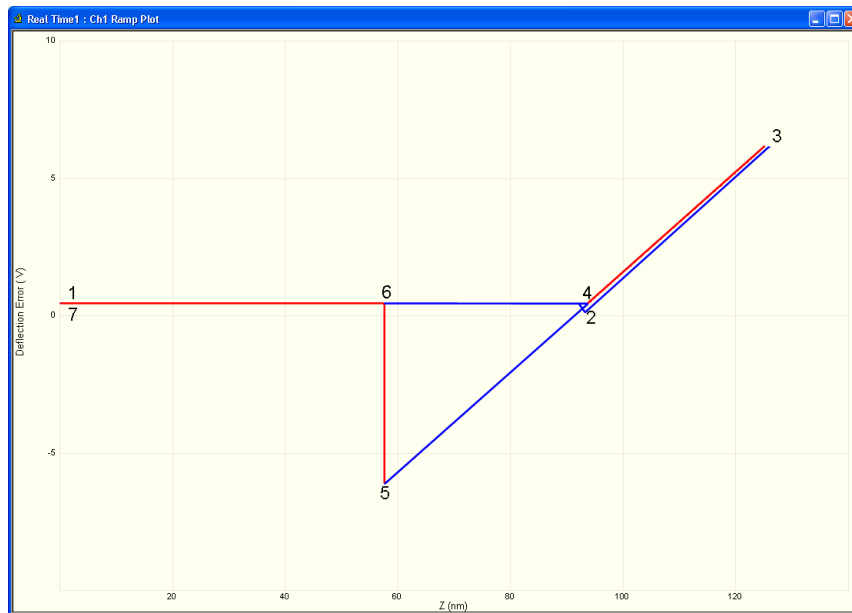
Figure 14.4b   Piezo Positions for Typical Force Curve
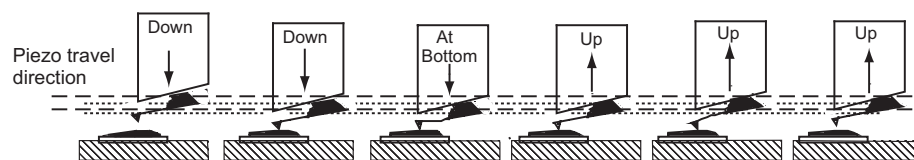
To minimize or calculate the contact force between the tip and sample, obtain a good force curve which shows the typical features displayed in the example curve in Figure 14.4b. However, the force curve rarely looks "typical" immediately after invoking the **Ramp Continuous** mode. This section discusses general approach adjustments to improve force curves obtained after engaging the microscope.

The basic approach to obtaining a good force curve entails adjusting the Z motion of the piezo relative to the sample (with the **Z scan start** and **Ramp size** parameters) and shifting the graph (with the **Setpoint** parameter) so the pull-off point of the tip displays on the graph. In general, use the following steps to obtain a good force curve:

1. **Engage** and verify the tip is tracking the surface. Then switch to **Ramp Continuous** mode.

2. Maximize the **Z Limit** parameter in the **Feedback** panel.

3. Select DEFLECTION ERROR as the **Data Type** for **Channel 1**.

4. Maximize the **Data Scale** parameter.

5. Adjust the **Ramp size** parameter to about **1µm**.

6. If the tip does not reach the sample surface (for example, see Figure 14.4a, between points 2 and 3), slowly increase the **Z scan start** value.

7. As the **Z scan start** increases, the traces on the force curve move to the right.

8. Adjust the **Setpoint** parameter.

   Note: Adjusting the **Setpoint** value moves traces up and down on the graph. Decreasing the value moves the curve up while increasing the value moves the curve down. (Remember that the red horizontal centerline of the graph is equal to the **Setpoint** if the **Data Center** = **0**, so that changing the **Setpoint** actually shifts the graph up and down.) Typically, changing the **Setpoint** value to **-2 volts** shows the desired features of the force curve. If it does not, decrease the **Setpoint** value further.

   Note: Sometimes the entire ramp will occur in the contact portion of the curve (see Figure 14.4a, between points 3 and 5). If this occurs, increase the **Ramp size** and/or decrease the **Z scan start**.

14.4.2 Helpful Suggestions

To minimize or calculate the contact force between the tip and sample, it is important to obtain a good force curve; however, there will be situations where you will not obtain a good force curve no matter how much you adjust the **Ramp size**, **Z scan start** and **Setpoint**. The following sections discuss a few such situations. Suggestions on working in **Ramp Continuous** mode are included.

False Engagement

Figure 14.4c illustrates a force curve resulting from a falsely engaged tip. The photodiode receives light reflected off the sample, causing an increase in the deflection signal until the signal equals the setpoint and the system "engages" (even though the tip is not on the surface). Interference in the reflected light causes the hump-shaped waveform.

The easiest way to correct a false engagement is to withdraw the tip, adjust the photodetector positioner to make the top/bottom differential voltage more negative, then re-engage the tip. This compensates for the slight slope in Figure 14.4c).

Figure 14.4c   False Engagement
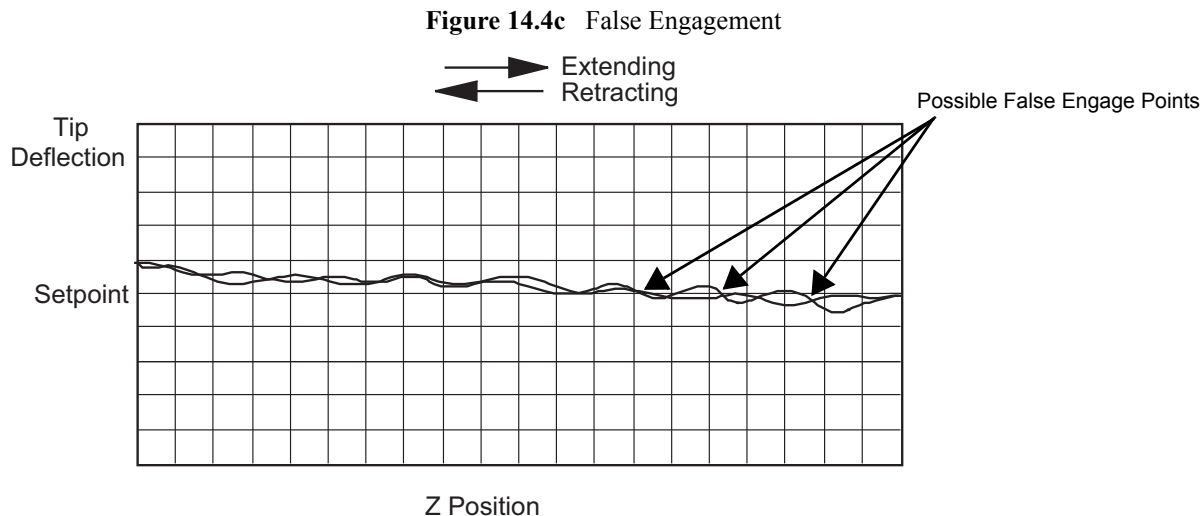
Motor Control

The **Microscope > Step Motor** > **Tip Up** and **Tip Down** buttons provide coarse adjustment of **Z center** voltage. These buttons move the SPM head vertically. The feedback loop causes the Z piezo to adjust to compensate for the head movement. If you use **Tip Up**, the Z piezo extends. If you use **Tip Down**, the Z piezo retracts.

> Note: The **Step Motor** function is generally used only when the scanning range of the Z piezo is exceeded or when it is necessary to position a force measurement in the center of the scanner range. Because the Z-axis leadscrew has some backlash, it may be necessary to rotate the leadscrew several turns by clicking on the **Tip Up** or **Tip Down** buttons before obtaining movement. A more effective method is to increase the motor step size temporarily until the backlash is gone, then reduce it for fine control.

Adjust Photodiode

In an analogous manner, you can use the photodiode positioner as a coarse adjustment for **Setpoint** voltage. Changing the laser beam position on the photodiode by rotating the photodiode adjustment knobs shifts the force curve on the graph. Rotating the top photodiode adjustment knob counterclockwise shifts the curve up, just as decreasing the **Setpoint** parameter shifts the curve up. Conversely, rotating the top photodiode adjustment knob counterclockwise moves the curve down.

14.4.3 Advanced Techniques

Sensitivity Determination

The **Deflection Sensitivity** allows conversion from the raw photodiode signal (in Volts) to deflection of the cantilever (in nm), and is normally set from the **Ramp Continuous** mode. The sensitivity must be calibrated before accurate deflection data can be obtained. Sensitivity is equal to the inverse of the slope of the force curve while the cantilever is in contact with a hard sample surface. Complete the following steps to calculate the sensitivity:

1. Obtain a good force curve using a hard sample so that the sample does not deform.

2. Drag vertical cursors from the plot edge to contact.

3. Click **RAMP > UPDATE SENSITIVITY** to calculate the cantilever sensitivity (see Figure 14.4d). Click **YES** to accept the computed sensitivity.

Figure 14.4d Set the Sensitivity Parameter
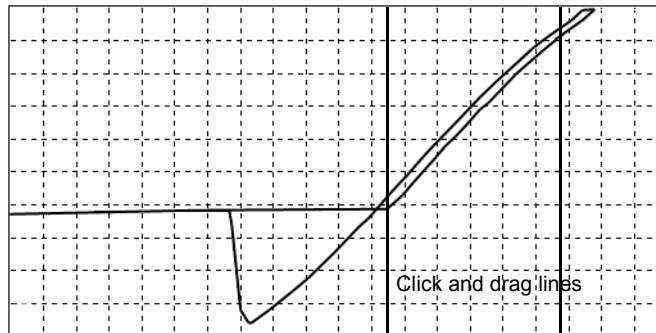
If **Sensitivity** is calibrated on a material much stiffer than the cantilever, it measures the inverse of the value of the AFM's optical lever sensitivity; i.e., how many volts of deflection signal are produced by a given deflection of the cantilever tip. The sensitivity will change for different cantilever lengths and styles (shorter cantilevers give lower **Sensitivities**). **Sensitivity** will also change with the position of the laser on the cantilever and the quality of the laser beam reflection from the cantilever.

> **Note:** It is important to calibrate the **Sensitivity** parameter on a hard substrate as described here BEFORE using the force curve's vertical scale for quantitative measurements.

> **Note:** **Deflection Sensitivity** can be expressed in terms of the photodiode voltage versus the distance traveled by the piezo, or the photodiode voltage versus the voltage applied to the piezo, depending on the setting of the **Units** parameter.

Force Minimization

The **Ramp Continuous** mode allows minimization of the contact force of the cantilever on the sample surface. The force curve clearly illustrates the relationship between the **Setpoint** and the cantilever deflection voltage when the cantilever is off the sample surface. You can adjust the **Setpoint** to set the normal deflection of the cantilever and, therefore, the normal force applied by the cantilever during data collection.

You can run the microscope below the point of zero deflection of the cantilever to minimize the contact force of the cantilever on the sample. It is possible to get a negative deflection whenever the cantilever sticks to the surface. To engage, you must the set **Setpoint** greater than the deflection voltage when the tip is not interacting with the surface. However, you can change the setpoint after entering **Ramp Continuous** mode; you can lower the setpoint to the zero cantilever-deflection point and beyond, while viewing the force curve. You can adjust the setpoint (most often made more negative) so that it lies between the flat segment of the force curve which corresponds to the zero deflection point, and the tip of the retraction scan where the cantilever pulls off the sample surface V_{CSmin} (see Figure 14.4e). The contact force is at its minimum when V_{CSmin} is on the centerline of the deflection-signal axis (**Setpoint** = V_{CSmin}).

In practice, V_{CSmin} must be a little below the centerline because V_{CSmin} is the point where the cantilever pulls off the surface and operation at this deflection is unstable. Changing the **Setpoint** option in the **Feedback** panel changes the setting of the **Setpoint** parameter in its **Image Mode** counterpart when you exit the **Ramp Continuous** mode. After exiting, if the image looks good, you can decrease the force further by lowering the **Setpoint** in small increments until the cantilever pulls off the sample surface. Resetting the **Setpoint** to a value higher than the voltage when the tip is not interacting with the surface, recaptures the cantilever. (Slowly adjust to a more positive value until the tip is back on the surface.) Adjusting the **Setpoint** a few tenths of a volt above the point where the cantilever pulled off provides a low contact force.

If a high initial contact force adversely affects the sample, engage the cantilever with a very small scan size. Then, minimize the force while the tip is confined to a small area of the sample where it experiences the relatively high initial engagement force. Once the force is minimized, increase the **Ramp size** or offset the scan to a different area of the sample. However, keep in mind that if the force is minimized in a smooth area of the sample, the cantilever may pull off when it translates to a rougher part of the sample.

Calculating Contact Force

The force curve clearly shows the relationship between the setpoint and the deflection of the cantilever. Because the setpoint defines the value of the deflection signal maintained by the feedback loop, the force curve can be used to calculate the contact force of the tip on the sample if the spring-constant, k , of the cantilever is known. The contact force is defined by the equation:

$$F = k \times d$$

where " d " is the deflection measured from the setpoint to V_{CSmin} in nanometers. An example of how to compute the contact force from the **Force Plot** graph is shown in Figure 14.4e.

Figure 14.4e Computing Contact Force
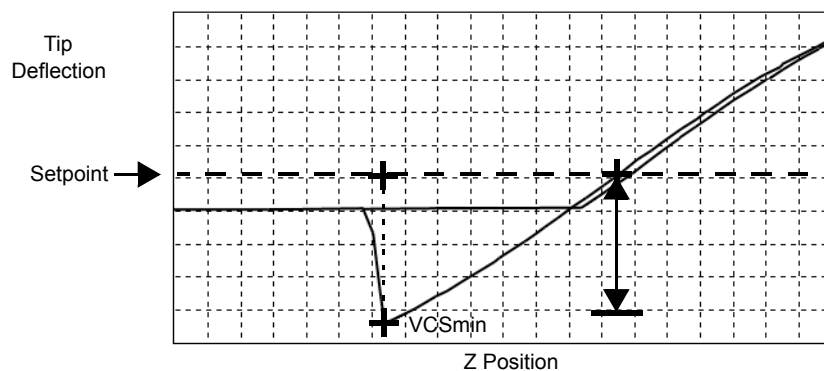
Recalling that contact force F = kd, we can calculate the contact force from the sample plot above. Let us assume, for example, that the spring constant of the cantilever is $k = \dfrac{0.6N}{m}$ and that deflection sensitivity $= \dfrac{70nm}{V}$. The plot above may be measured at the points where the retract

portion of the curve intersects the setpoint to the pull-off (point). The distance is then multiplied times the deflection sensitivity to obtain d. In this example:

$$d = (4.5 \text{ div}) \, (1\text{V/div}) \, (70\text{nm/v}) = 315\text{nm}$$

Therefore, the contact force is calculated as:

$$F = (0.6\text{N/m}) \, (315\text{nm})$$
$$= 189\text{nN}$$

When the **Data Type** is set to **Height** with the feedback gains set high, the tip tracks the sample surface with nearly constant deflection of the cantilever. When the cantilever deflection is constant, the force is constant and the force calculation above determines the contact force between the tip and the surface over the entire scan area.

Force calculations are not as straightforward on images captured with the **Data Type** set to **Deflection Error**. When collecting deflection data, the feedback gains are ideally set low so the sample stays at a nearly constant position relative to the cantilever holder. In this case, the cantilever deflection (and therefore the force applied to the sample) varies as features on the surface are encountered. The contact force can be calculated by adding the deflection data from the image to d before multiplying by the spring constant: F = k (deflection image data + d). Note that the **Sensitivity** parameter must be accurate—that is, previously determined and entered—before the deflection data will be accurate.

A simple alternative to calculating force is to set the units to force.

14.4.4 Interpreting Force Curves

An examination of force curves can prove useful in determining adhesion, hardness, and elastic characteristics of samples. The examples in Figure 14.4f represent some of the general variations in force curves. For more information regarding force imaging, refer to Bruker's application note *Probing Nano-Scale Forces with the Atomic Force Microscope*.

Figure 14.4f Force Curve Examples
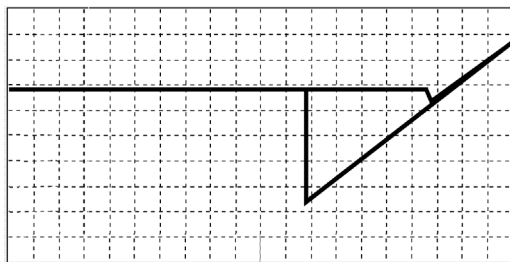
Large adhesion
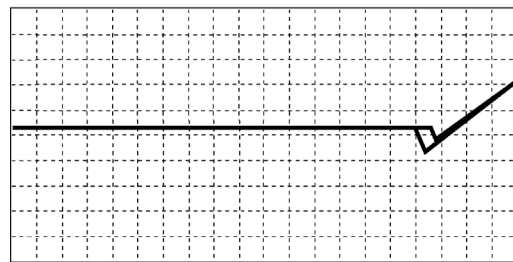
Small adhesion
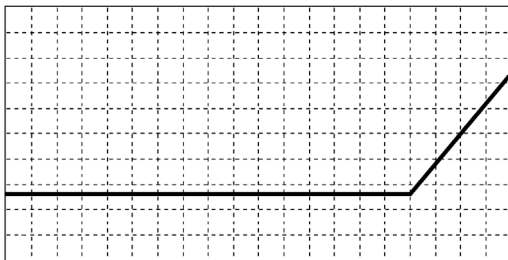
Stiff sample
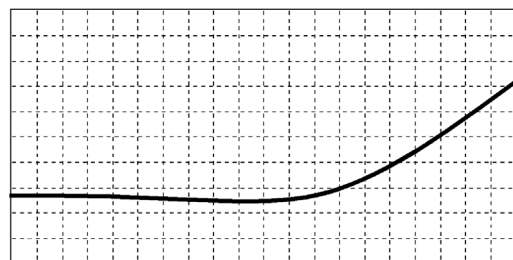
Soft sample
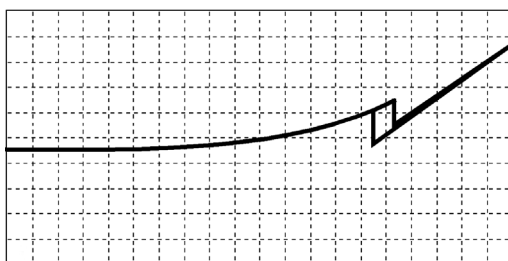
Long-range repulsion
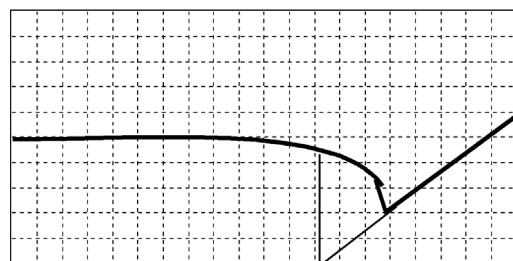
Long-range attraction
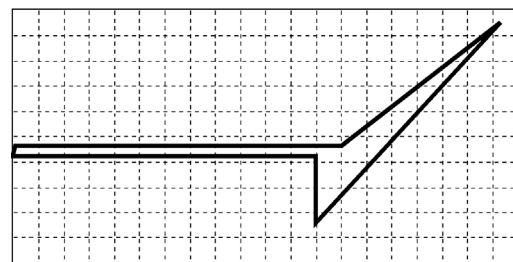
Plastic Deformation

14.5 Force Calibration (TappingMode)

> ⚠ **CAUTION:** Because TappingMode cantilevers are relatively stiff, **Force Mode** can potentially damage the tip and/or surface. Before using Force Calibration, read and understand the following section.

Force Mode allows the imaging of forces between the tip and surface, including chemical bonds, electrostatic forces, surface tension and magnetic forces. In TappingMode, you can observe forces by measuring changes in tip RMS amplitude, phase, or TM deflection. The user may collect force plots in one of two forms: **Force Plot** and **Force Volume**. The two forms are similar, with **Force Volume** generating a map of many individual force plots. To produce high-quality force plots, it is necessary to precisely control tip position relative to the surface.

14.5.1 Force Plots

When performing **Force Plot** in TappingMode, the piezo moves to the center of the current X-Y scan, then turns off the X-Y scan motion. Next, a triangular waveform is applied to the Z electrodes of the piezo tube resulting in the oscillating tip moving up and down relative to the sample. The same Z-axis piezo motion occurs in Contact Mode AFM force plots. However, in TappingMode, the force plot is a graph of the piezo's extension versus oscillating tip amplitude, phase or TM deflection, instead of deflection or friction.

Uses of **Force Calibration** in TappingMode include characterizing forces on the cantilever tip, diagnosing SPM performance, calibrating the RMS amplitude or TM deflection, and configuring **Lift Mode**. For example, as the oscillating tip is brought closer to the surface, tip motion is dampened, which shows as an immediate drop in amplitude. When plotted, the graph resembles Figure 14.5a.

Figure 14.5a   Piezo Extension Versus RMS Amplitude and Deflection
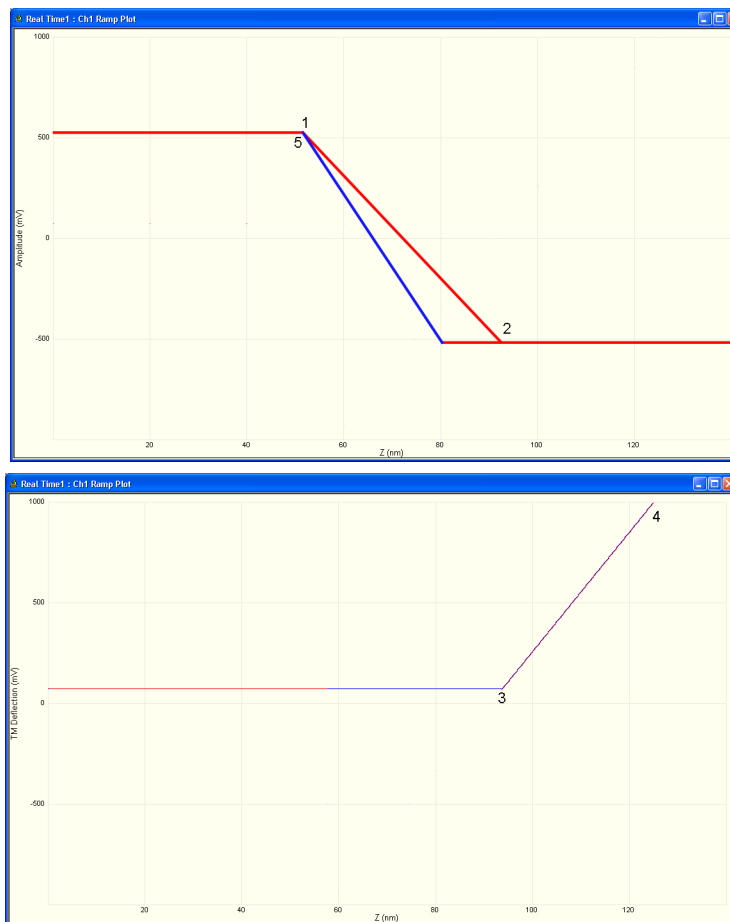
Figure 14.5a illustrates a two-channel TappingMode force plot. The vertical axes of the graphs represent the amplitude (top) and TM deflection signal (bottom) of the cantilever. The position of the Z piezo plots along the horizontal axis. **Channel 1** (top) demonstrates how the cantilever amplitude decreases as the tip moves closer to the sample. The plot represents the amplitude for one complete extension-retraction cycle of the piezo. The **Scan Rate** parameter in the **Ramp** panel defines the rate at which the piezo completes an extension-retraction cycle; therefore, the rate at which new curves display. At point 1, the tip encounters the sample surface. Upon encountering the surface, the tip oscillation amplitude decreases, dropping off as the tip moves closer to the sample surface. Between points 1 and 2, voltage to the Z piezo is increased, bringing the tip about 50 nm closer to the surface. Over the same interval, the cantilever amplitude diminishes about 2 volts due to dampening effects.

Collecting a force plot on a hard sample and dividing the change in amplitude by the change in Z piezo position gives the responsiveness of the electronics that measure the amplitude. The **Amplitude Sensitivity** value in the **Ramp** menu is the inverse of the responsiveness. You can determine this value by using the mouse to drag vertical markers from the edge (between points 1 and 2 where the tip amplitude dampens) and then selecting **RAMP > UPDATE SENSITIVITY**. Tip damping occurs as a result of mechanical-acoustic coupling between the tip and sample. As the z piezo is extended further, oscillation eventually ceases and the amplitude drops to zero (point 2).

For **TappingMode** in air on a hard sample, each nanometer decrease in the cantilever position decreases the peak-to-peak vibration of the cantilever by two nanometers. Once the tip encounters the sample surface, the oscillation amplitude of the cantilever decreases as the piezo is extended. When the piezo turns around and begins to retract, the oscillation amplitude of the cantilever increases until the tip is free of the surface, leveling off at the free-air amplitude (point 5).

Channel 2 (bottom) in Figure 14.5a plots average cantilever deflection (TM deflection) versus piezo extension. The deflection signal is low-pass filtered to eliminate the high-frequency TappingMode oscillation. Even as tip RMS amplitude dampens during its encounters with the sample surface, the average deflection is unchanged. This condition changes once the tip is so close to the sample that all oscillation ceases. Extending the piezo still further causes the average deflection to increase, applying an increasing force to the sample (point 2).

At point 3 in Figure 14.5a, the cantilever begins to deflect. The region between points 3 and 4 may be hazardous to the tip, because the tip is pressed firmly against the sample surface. Most single crystal silicon TappingMode tips rapidly become dull in this region, depending upon the hardness of the sample.

14.5.2 Obtaining a Force Plot (TappingMode)

⚠️	**CAUTION:** Use **Ramp Continuous** with caution or when it is important to obtain experimental information shown in **Force Calibration.** When using stiff TappingMode cantilevers, it is easy to blunt the tip with excess contact force during **Force Calibration** measurements.

When obtaining force plots in TappingMode, set up scan parameters so that the reduction of amplitude is minimal (approximately 25 percent of the free air amplitude). If the amplitude is reduced to zero, the tip and sample may sustain damage.

To generate a TappingMode force plot of a silicon calibration reference, complete the following steps:

1. Verify the cantilever holder is loaded with a TappingMode tip.

2. Mount the calibration reference on the SPM stage.

3. Click the **SELECT EXPERIMENT** icon.

4. Select **TAPPINGMODE MODE AFM** in the **Experiment Category** panel.

5. Select **TAPPINGMODE IN AIR** in the **Experiment** panel and click **START EXPERIMENT**.

6. Tune the cantilever.

7. Engage. and obtain a TappingMode image.

   Note: You are now in **Image mode**.

8. To switch to **Force Mode**, click the RAMP button in the **Workflow Toolbar**. Two panels should be visible:

   • **Force Plots**

   • **Ramp Parameter List**

9. Set the **Ramp Output** to **Z**. This is the default in the **Simple Mode**.

10. Set the **Channel 1 Data Type** to **TM D**EFLECTION.

11. Set the **Channel 1 X Data Type** to **Z**.

12. Adjust the **Z scan start** parameter to obtain a satisfactory force plot using the left-right arrow keys.

14.5.3 High Contact Force

Figure 14.5b shows a curve produced when the tip pushes too far into the sample. The flat portion on the right side of the amplitude curve in Figure 14.5b occurs because the tip is so close to the surface that it no longer vibrates. As the piezo extends the tip further, the amplitude of vibration does not change because the tip is always in contact with the sample surface. The contact force is very high due to the stiffness of the TappingMode cantilevers. The tip may shatter if deflection continues.

Figure 14.5b   Amplitude Force Plot with High Contact Force
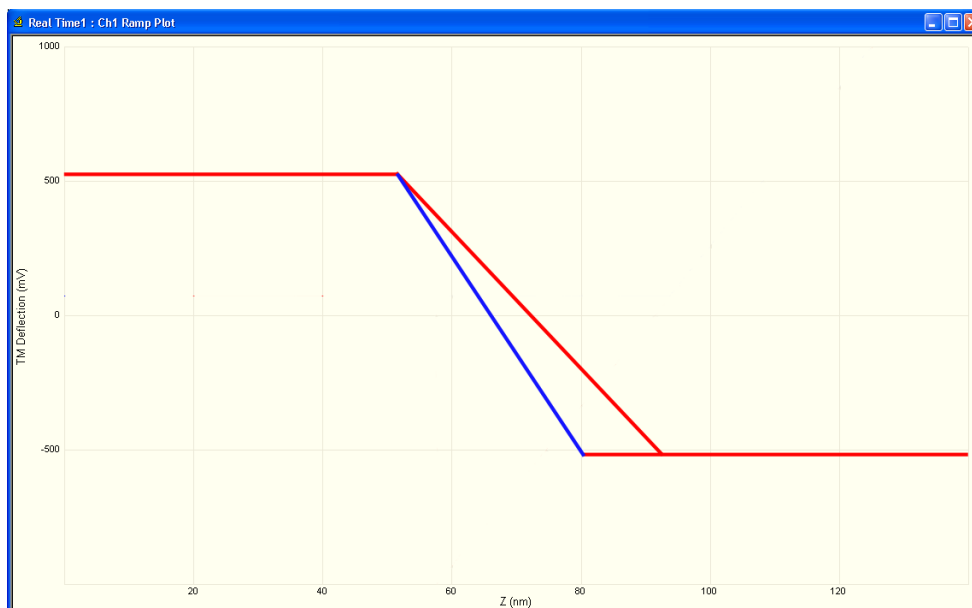
You can avoid this situation by using triggers (see Section 14.3.4) or by reducing the value of **Z scan start** until there is no flat portion on the right side of the curve. Rapidly increasing the value of **Z scan start** is dangerous because the total oscillation amplitude of the cantilever is small relative to the total Z travel of the long-range scanner.

14.5.4 Probe Selection

You may use virtually any TappingMode tip to obtain TappingMode force plots; however, the ultimate choice depends upon the delicacy of the sample and the magnitude of the forces to be gauged. Longer cantilevers have lower spring constants (i.e., they are more pliant) and therefore offer greater sensitivity for most samples. Shorter cantilevers afford better control when gauging strong attractive forces and are less prone to entrapment by surface tension forces. Sharper tips are more likely to plastically deform the sample, and are less sensitive to adhesive forces, than tips that are more blunt. Experiment to determine the tip that best meets your needs.

14.6 Force Modulation

14.6.1 Introduction

This section describes the operation of force modulation mode, which you can use to image local sample stiffness or elasticity. This method is useful for imaging composite materials or soft samples on hard substrates where you can obtain contrast between regions of different elasticity. This section assumes knowledge of operation of Contact Mode AFM in air (see Chapter 9). It is useful, but not essential, to have experience operating in TappingMode (see Chapter 10).

Force modulation measures local sample elasticity by oscillating a cantilever such that the tip indents slightly into a sample. The tip indents soft materials more easily than harder materials. The amount of cantilever deflection is inversely related to the amount of indentation. For example, a soft sample allows the tip to indent more deeply into the surface, resulting in a very small deflection of the cantilever. A hard sample allows less indentation, with the cantilever deflected by a larger amount. To measure the relative elasticity of the sample the system records the amplitude of the tip deflection versus position over the sample as depicted in Figure 14.6a.

Figure 14.6a   Contrast Generation in Force Modulation Mode

Small Cantilever
Response on Soft Material

Large Cantilever Response
on Hard Material

1510

Soft Material
Large Indentation

Hard Material
Small Indentation
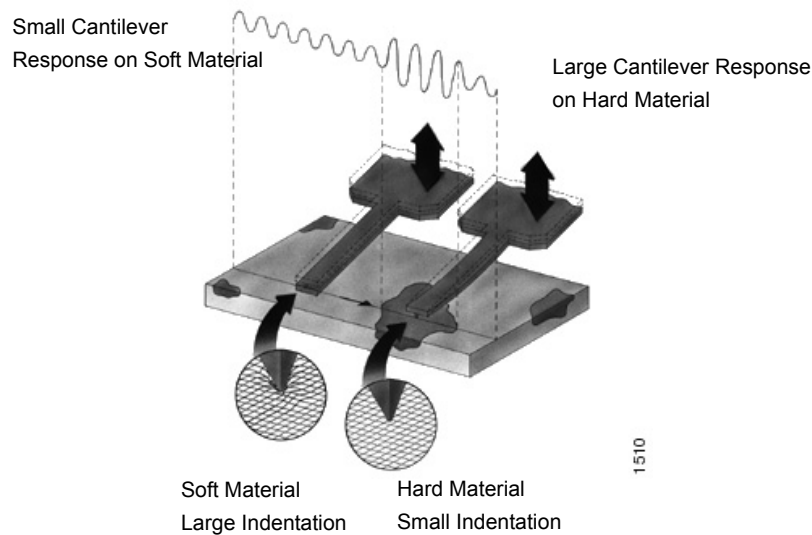
Force modulation requires the use of a special optional cantilever holder, shown in Figure 14.6b. This cantilever holder uses a piezoelectric bimorph to oscillate the cantilever against a sample surface. The force modulation cantilever holder is similar to the standard tapping cantilever holder; however, its piezo stack is much larger, allowing larger driving amplitudes.

Figure 14.6b   Force Modulation Cantilever Holder
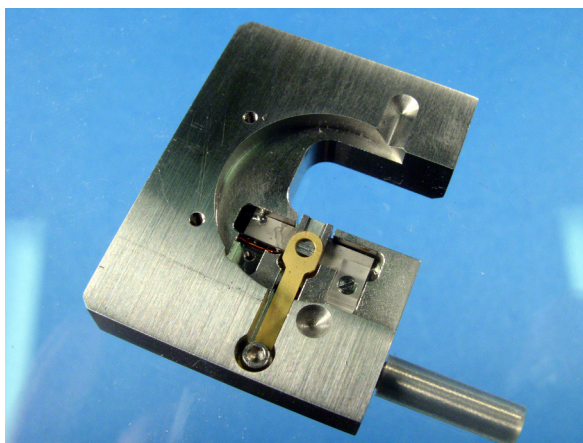
14.6.2 Selecting a Force Modulation Probe

The key consideration when selecting a force modulation cantilever is its spring constant. Ideally, the cantilever must have a spring constant which complements the pliancy of the two contrasting materials (or close to the pliancy of one, but not the other). This way, the tip indents into one material more than the other providing good force modulation image contrast. If the tip is so stiff that it indents equally into both materials, or so soft that it indents neither material, then you will not see contrast in the force modulation image. Instead, the image will consist primarily of edge and frictional artifacts. It may take experimentation to find a cantilever that matches the sample's requirements. For rubber and plastic samples Bruker recommends using 225µm long force modulation (Model # FESP) silicon cantilevers. For more delicate, samples, use 450µm long silicon cantilevers or silicon nitride cantilevers. For hard materials, use stiffer tips like the 125µm single crystal silicon TappingMode tips.

Bruker offers cantilevers with a wide range of spring constants (see Table 14.6a). Choosing a probe depends upon how stiff the sample is For samples of unknown hardness, start with a force modulation cantilever (Model FESP) and determine whether the probe is sufficiently stiff then adjust accordingly.

Table 14.6a   Force Modulation Probes

Cantilever	Model No.	Cantilever Length	Spring Constant	
Standard Silicon Nitride	NP, DNP	100—200μm	0.01—0.6 N/m	SOFTER
Oxide-sharpened Silicon Nitride	NP-S, DNP-S	100—200μm	0.01—0.6 N/m	
Contact AFM Etched Silicon	ESP	450μm	0.02—0.1 N/m	
Force Modulation Etched Silicon	FESP	225μm	1—5 N/m	
TappingMode Etched Silicon	LTESP	225μm	20—70 N/m	HARDER
TappingMode Etched Silicon	TESP	125μm	20—100 N/m	

14.6.3 Operating Principle

Force modulation mode is very similar to Contact Mode AFM. The NanoScope system scans the sample under the probe while trying to keep the cantilever deflection constant. The deflection setpoint determines the average deflection during operation. In addition, the cantilever is oscillated up and down by a piezoelectric bimorph in the probe holder so that the tip indents slightly into the sample surface as it is scanned across the surface. The NanoScope system records the amplitude of the cantilever motion, which indicates the relative indentation of the tip into the surface. For softer samples the tip penetrates further into the surface resulting in a smaller change in the angle of the cantilever. A small change in angle results in a small measured amplitude which displays as a bright area on the image. For harder samples the tip penetrates less into the surface resulting in a larger change in angle of the cantilever. A large change in angle causes a large measured amplitude which displays as a dark area on the image.

14.6.4 Force Modulation Procedure

This section gives instructions for operating in **Force Modulation** mode.

14.6.5 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 14.6c.

Figure 14.6c The **Select Experiment** window
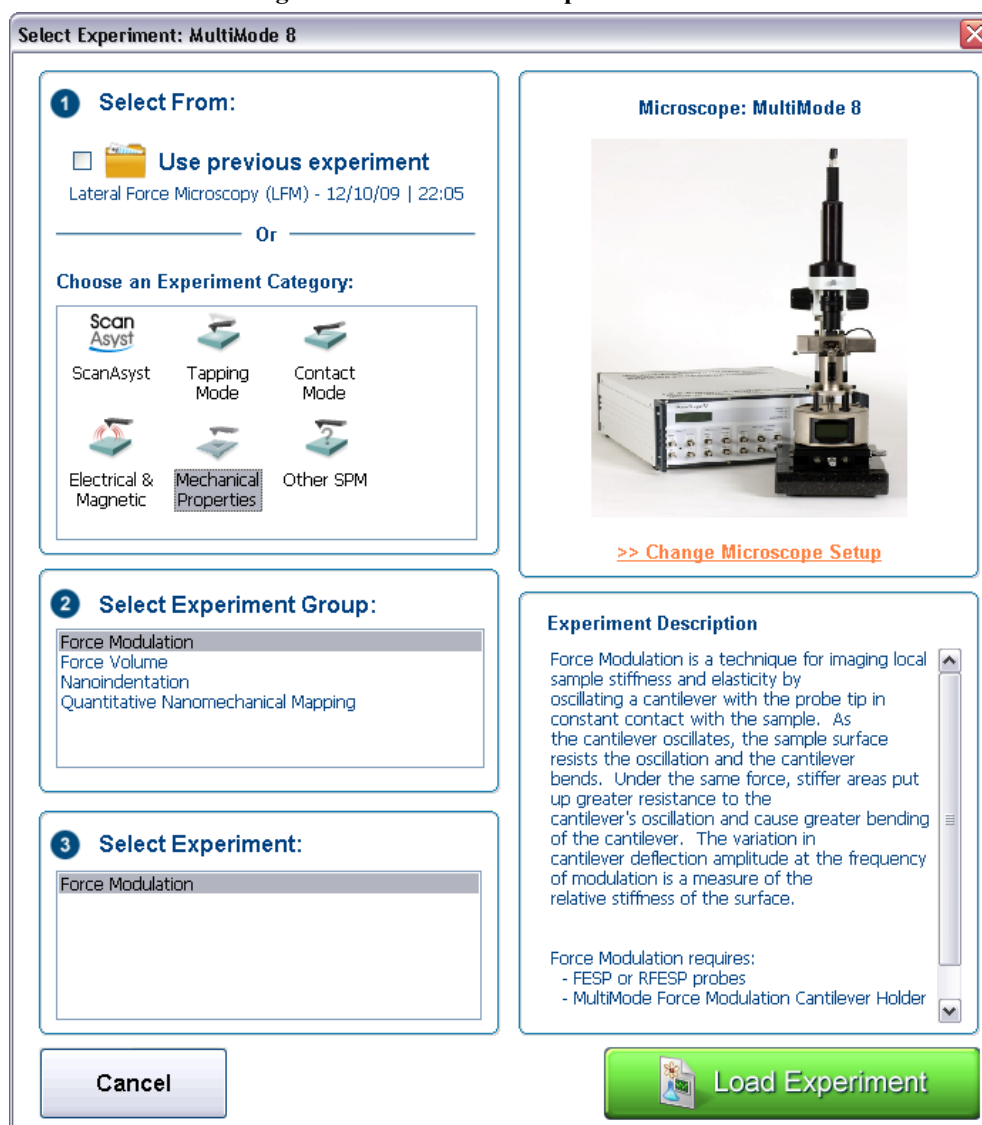
2. Select MECHANICAL PROPERTIES in the **Choose an Experiment Catagory** panel.

3. Select FORCE MODULATION in the **Select Experiment Group** panel.

4. Select FORCE MODULATION in the **Select Experiment** panel and click LOAD EXPERIMENT.

5. This opens the **Workflow Toolbar**, the **Scan 4 Channels** windows and the **Scan Parameters List** window, shown in Figure 14.6d.

Figure 14.6d   Force Modulation (Simple Mode) configuration
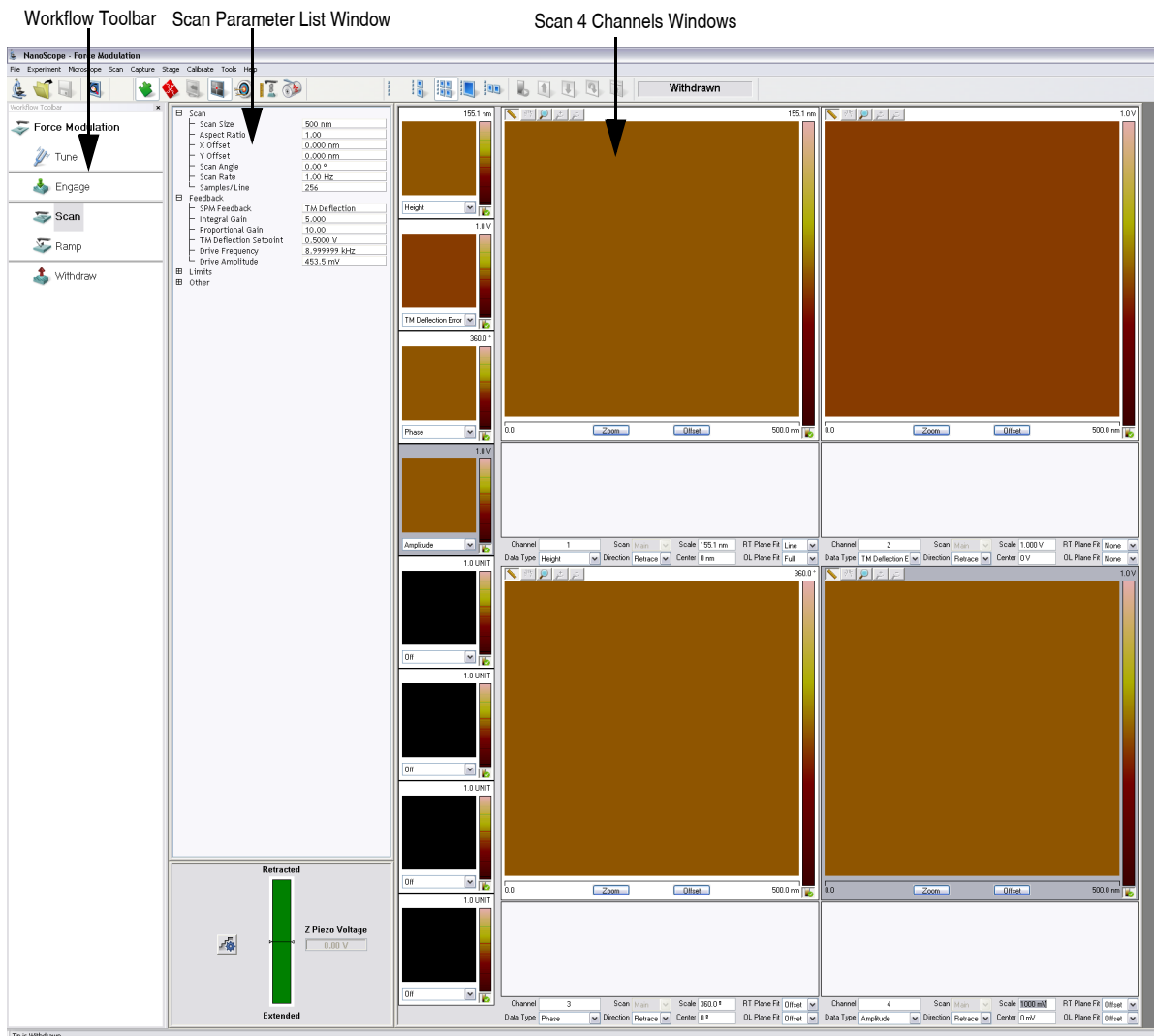
14.6.6 Head, Cantilever and Sample Preparation

- Load the special force modulation cantilever holder with a cantilever (see Chapter 7).

   Note: Refer to the section on Probe Selection earlier in this chapter to help you choose an appropriate cantilever.

- Load the cantilever holder with installed tip into the MultiMode SPM head. See **Initial Preparation for Contact AFM Imaging:** Section 7.1.

14.6.7 Align Laser

Align the laser using the laser control knobs. See **Laser Alignment:** Section 7.2. for more detailed instructions.

14.6.8 Adjust the Setpoint

Begin with a **Setpoint** of **0V**. Recall that higher setpoints cause the probe to push harder on the surface during scanning.

14.6.9 Adjust Photodetector

1. Turn the photodetector adjustment knobs to center the laser spot on the laser detector. Adjust the **Vertical Deflection** to approximately -**0.5V** for FESP or TESP cantilevers, -**2V** for NP cantilevers. See **Maximize the SUM Signal:** Section 7.2.3. for more detailed instructions.

   Note: The force the cantilever applies to the surface is related to the difference between the **Vertical Deflection** and the **Setpoint** and the spring constant of the cantilever used. If the sample is very delicate, set the **Vertical Deflection** value closer to the chosen **Setpoint**.

2. Set the **Vertical Deflection** to **0V**.

14.6.10 Find the Bimorph Resonant Frequency:

The cantilever is oscillated by a small piezoelectric bimorph mounted in the cantilever holder. For Force Modulation, oscillate the bimorph at or near its resonant frequency. The bimorph resonance frequency is usually the largest peak in the 5-30kHz range. This ensures the cantilever moves with sufficient amplitude to produce elasticity contrast.

   Note: You need to find the bimorph's resonant frequency only once. (Resonant frequencies are unique to each force modulation cantilever holder.) Once you find the resonant frequency, write it down for future use. This **Drive**

Frequency may be used as a starting place each time you perform force modulation imaging; however, recheck the bimorph's resonant frequency in free air using **Cantilever Tune** each time you install a new cantilever.

1. Use a FESP or TESP cantilever. NP probes have confusing low frequency resonances.

2. Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window, shown in Figure 14.6e.

Figure 14.6e   The **Cantilever Tune** window.
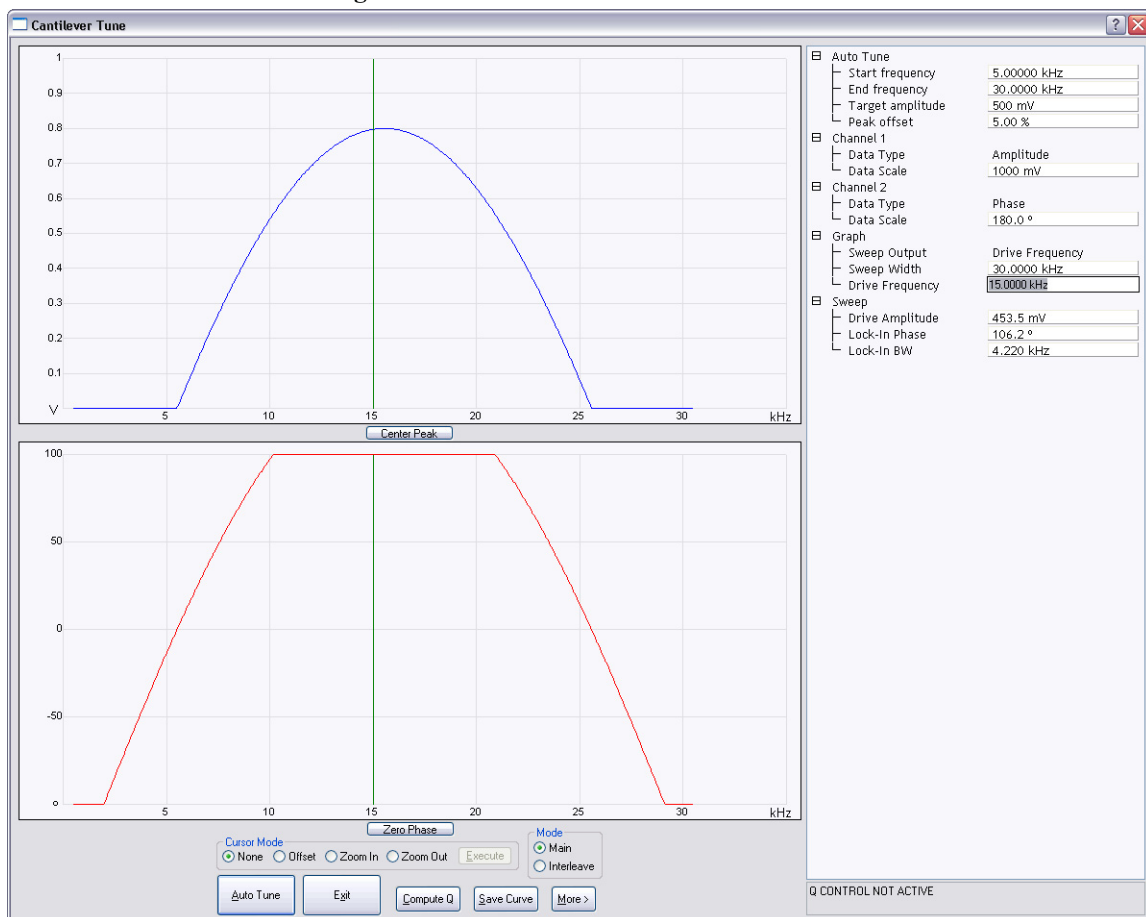
The graphs plot the **Frequency Sweep**, showing cantilever oscillation amplitude and phase versus frequency. To set the parameters controlling the **Frequency Sweep** plot, use the parameters in the **Sweep** panel.

3. Set the **Drive frequency** to **15kHz** and the **Sweep width** to **30kHz**.

4. Set the **Drive amplitude** at **1000mV** to start. You can readjust this value later.

Note: The large drive amplitude is necessary because peaks are smaller than normally
        seen during TappingMode operation. This is because the cantilever is not at
        resonance; therefore, its motion is mostly vertical. Vertical motion is not
        amplified by the beam deflection detection technique which is sensitive
        primarily to changes in cantilever angle.

5.   Adjust the **Sweep Width, Data Scale** and the **Drive Amplitude** until you can clearly see
     peaks. Bimorph resonances typically occur between **8-20kHz**.

6.   Click the AUTO TUNE button, shown in Figure 14.6e, to begin optimizing the cantilever tune
     to the above parameters. The computer automatically changes the **Drive Frequency** so that
     the peak is centered.

Once you choose the desired frequency, the **Frequency Sweep** plot should be recentered similarly
to the plot shown in (see Figure 14.6f).

Figure 14.6f   Correctly Tuned Force Modulation Frequency
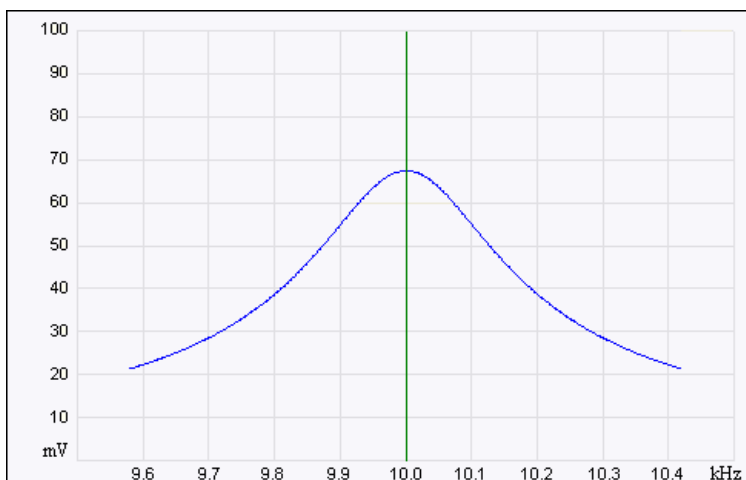
Note: Bimorph resonance should be between **5 kHz** and **30 kHz**. If peaks are found at
        a much higher frequency, these are cantilever resonances, *not* bimorph
        resonances.

7.   Record the **Drive Frequency**. This is the resonant frequency of your force modulation
     bimorph.

14.6.11   Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 14.6g).

1.   Set the **Scan Rate** to **1 Hz**.

2.   Set the **Scan Size** to **500nm**.

3.   Set the **Scan Angle** to **0** degrees.

Figure 14.6g   Suggested Force Modulation Settings

Feedback Panel

1.   Set the **SPM Feedback** to **TM DEFLECTION**.

2.   Set the **Integral gain** to **0.5** and the **Proportional gain** to **5** (see Figure 14.6g).

3.   Set the **TM Deflection Setpoint** to **0V**. Recall that higher setpoints cause the cantilever to push harder on the surface during scanning.

Other Panel

1.   Set the **Units** to **METRIC**.

Channels

1. Set the **Channel 1 Data Type** to HEIGHT (see Figure 14.6h).

2. Set the **Channel 2 Data Type** to TM DEFLECTION ERROR.

3. Set the **Channel 3 Data Type** to PHASE.

4. Set the **Channel 4 Data Type** to AMPLITUDE.

5. Set **Data Scale** to a reasonable value for the sample.

    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**.

7. Set the **Channel 5** through **Channel 8 Data Type** to OFF.

The multi-image capability views both the topography data and the force modulation (elasticity) data at the same time.

Figure 14.6h   Suggested Force Modulation Channel Settings

14.6.12 Engage

1. Reduce the **Drive Amplitude** to **0** before engaging.

2. Select **RealTime** > **Engage** or click the ENGAGE icon on the toolbar. A pre-engage check begins, followed by Z-stage motor motion.

14.6.13   Optimize Scan Parameters

1. Adjust the **Integral Gain**, **Proportional Gain**, **Setpoint,** and **Scan Speed** to obtain a good topography (**Height**) image. For force modulation operation, set the **Integral Gain** and **Proportional Gain** to values of **0.5-10** and set the **Setpoint** as low as possible using the cursor keys (or by typing in new **Setpoint** values) until the cantilever pulls off the surface and the Z-center voltage jumps to **-220V**.

2. Record the **Setpoint** value where the cantilever pulls off the surface (the "pull-off value").

3. Increase the **Setpoint** until the cantilever touches the surface, and an image appears. Then decrease the setpoint to a value just above the value where the pull-off occurred.

   Note: If you adjust the **Setpoint** very close to the pull-off value, imaging perform with the smallest and least damaging force. However, in this condition imaging may be more unstable, as the cantilever may pull off the surface unexpectedly. The setpoint value affects the force modulation contrast. Examine the contrast of the force modulation image.

4. Optimize **Drive Amplitude** for Force Modulation imaging. The amount of contrast and the quality of both **Height** and **Amplitude** images depends on the **Drive Amplitude**. In general, increasing the **Drive Amplitude** provides greater contrast in the force modulation image.

   Note: It is possible to set the **Drive Amplitude** too high. If this occurs, the **Drive Amplitude** no longer increases the contrast in the amplitude image. Instead, the overall contrast in the force modulation image remains roughly constant, but you will observe more artifacts in the image. For example, if the drive amplitude is too high, the force modulation image becomes contaminated by "edge effects" or "friction effects." Operate at the lowest drive amplitude that gives sufficient contrast to examine the sample. Low drive amplitudes also help extend the life of the cantilever tip and reduce sample damage.

Procedure for Optimizing Drive Amplitude

1. Start with a small **Drive Amplitude** value of **50mV**.

2. Increase the **Drive Amplitude** with the right arrow keys or type in new values. The contrast of the **Amplitude** (force modulation) image increases.

3. Continue increasing the **Drive Amplitude** until sufficient contrast appears in the **Amplitude** image. If the **Drive Amplitude** increases to the point where contrast is no longer improving, reduce the **Drive Amplitude** slightly.

   Note: Force modulation contrast also depends on the **Setpoint**. In general, if using a larger **Setpoint** (larger tracking force) use a smaller **Drive Amplitude** to obtain good force modulation images without artifacts. If it is difficult to obtain a clear force modulation image that is free of artifacts, try reducing or increasing the **Setpoint** and then optimizing the **Drive Amplitude**.

4. Readjust gains (if necessary).

5. The value of the **Drive Amplitude** may also affect the Contact Mode AFM image, causing the system to go into unwanted oscillations. If the **Drive Amplitude** changes by a large amount, readjust the **Integral Gain** and **Proportional Gain.** Set the gains as high as possible to track the sample topography, but not so high that they cause oscillation due to the bimorph oscillation.

Sometimes an oscillation that appears in the data will be due to "aliasing" as described in the next section. If you cannot adjust gains to eliminate unwanted oscillations without compromising the height image's quality, see **Notes About Artifacts:** Section 14.6.14.

14.6.14 Notes About Artifacts

It is possible to see artifacts in force modulation images that are not due to differences in elasticity. Some artifacts to look for are outlined below:

Aliasing

Under some conditions, unwanted oscillations appear in the data due to aliasing of the **Drive Frequency** with the image pixel rate. This problem can be eliminated by changing the **Drive Frequency** by small increments. Use the arrow keys to change the **Drive Frequency** up or down very slightly until the oscillation disappears. On some materials, shifting the **Drive Frequency** slightly (1-3Hz) below or above a resonant peak value may improve image contrast. Operators are encouraged to experiment.

Edge Effects

Sometimes force modulation images show changes in amplitude at the edge of topographic features like steps or bumps. These artifacts typically look like the derivative of the sample topography.

To see if a feature is an edge effect, try reversing the **Line Direction** from **Trace** to **Retrace,** for example. If the contrast reverses or the amplitude change now appears on the other side of the topographic feature, then the amplitude change is likely due to the topographic edge, not differences in elasticity. To minimize edge effects, reduce **Drive Amplitude, Setpoint** or **Scan Speed.** Set the **Integral Gain** and **Proportional Gain** as high as possible without causing unwanted oscillations.

Frictional Effects

Because the cantilever is held at an angle to the sample surface, the cantilever tip will slide laterally ("skate") as the tip pushes into the sample (see Figure 14.6i). The amplitude of cantilever motion is affected by differences in friction between two different materials. For this reason, force modulation images may contain information about differences in local frictional forces. To reduce the influence of frictional effects, use a smaller **Setpoint** or **Drive Amplitude**. If you are using "Negative LiftMode" (see Section 14.7), use a more positive **Lift Height**.

Figure 14.6i   Friction on Force Modulation Images

Effect of friction on force modulation images
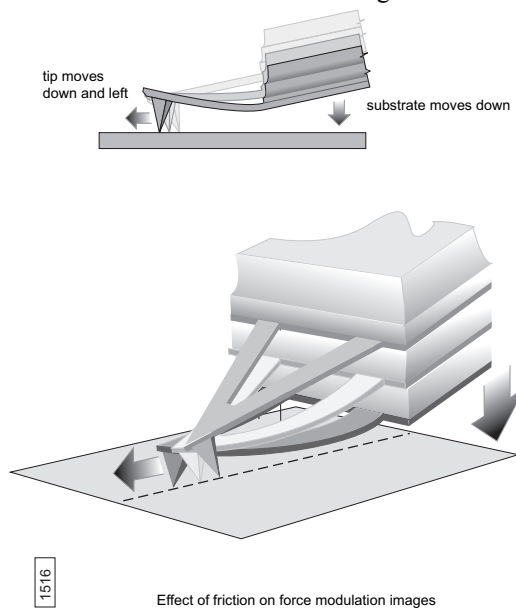
Tip Shape

The amount of indentation into a surface for a given applied force depends on the shape of the cantilever tip. For the same **Drive Amplitude** a sharper tip indents deeper than a dull tip. Because it is possible for the tip to dull during imaging, replace the cantilever if the force modulation contrast deteriorates over time. Also, note that it will be difficult to obtain reproducible, quantitative elasticity measurements between different samples and different cantilevers because the tip shapes may be different. In general, the force modulation technique is a qualitative tool for identifying regions of harder and softer material on a sample, rather than a tool for quantitative analysis.

Not all samples lend themselves to standard force modulation imaging. Even samples with excellent elasticity contrast may not show up in force modulation if their absolute elasticity is out of instrumental range. "Negative LiftMode" may prove useful for imaging otherwise difficult materials (see Section 14.7).

14.7 Force Modulation with 'Negative LiftMode'

A new form of force modulation imaging utilizing TappingMode and LiftMode operation known as "Negative LiftMode," allows imaging of certain materials previously not visible with Contact Mode AFM force modulation. This method is especially suited for softer materials, yielding higher resolution. Best results using negative LiftMode are obtained on relatively smooth samples (< 500nm vertical features); however, Bruker encourages experimenting with this technique on rougher surfaces as well. A general procedure for Force Modulation with "Negative LiftMode" is described in Section 14.7.3.

> **Note:**   Force modulation contrast is very sensitive to the spring constant of the probe, which varies according to the length and thickness of the cantilever. You may perform TappingMode easily with either force modulation or TappingMode cantilevers, but you may have difficulty with Contact Mode AFM silicon and silicon nitride cantilevers. (see Table 14.6a).

1. Verify the probe is withdrawn from the sample surface.

2. Switch to the **Expanded Mode**.

3. Verify in the **Feedback** panel that **SPM Feedback** = **TM DEFLECTION**.

14.7.1 Set Interleave Controls

In the **Interleave** panel, shown in Figure 14.7a, set the following

1. Click the **Drive Frequency** and **Drive Amplitude** parameters; when enabled (decoupled from the main scan), the buttons appear green.

2. Set the **Drive Amplitude** on the **Interleave** panel to **200-400mV**.

3. Set the **Drive Frequency** on the **Interleave** panel to the bimorph resonant frequency found in Section 14.6.10.

4. Set **Lift Start Height** and **Lift Scan Height** to **0.00nm**.

5. For now, set the **Interleave Mode** parameter to **DISABLED**.

Figure 14.7a Suggested Force Modulation Interleave Parameters

14.7.2 Obtain a TappingMode Image

While negative LiftMode force modulation data is imaged using **Channel 2**, height data is obtained using TappingMode on **Channel 1**. You must obtain a satisfactory TappingMode image to generate good data.

1. Verify that the **Interleave Mode** parameter on the **Interleave** panel is **DISABLED**.

2. Verify that the **Microscope Mode** parameter in the **Other** panel is set to **TAPPING**.

3. Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window, shown in Figure 14.6e.

4. Enter a **Start frequency** value of **5kHz** and **End frequency** value of **100kHz** for FESP tips.

5. Set **Target amplitude** to **0.5V**, and click the **AUTO TUNE** button.

6. Click the **Interleave Controls** button to verify that the bimorph drive parameters are correct.

7. Exit the **Cantilever Tune** dialog box.

8. Verify that all **Scan** and **Feedback** parameters are set for obtaining a TappingMode image.

9. Set the **Channel 1** panel **Data Type** parameter to **HEIGHT**.

10. Set the **Line Direction** to **RETRACE** and enter an appropriate **Data Scale** value for your sample.

11. Engage the tip on the surface and obtain a good TappingMode image.

14.7.3 Obtain a Negative LiftMode Force Modulation Image

1. Set the **Data Type** parameter in the **Channel 2** panel to **AMPLITUDE**. Set the **Line Direction** parameter to **RETRACE**.

2. Switch the **Interleave Mode** parameter in the **Interleave** panel to **LIFT**. Negative LiftMode imaging is now in effect.

3. Using the left arrow key, optimize the negative LiftMode image by slowly decreasing the **Lift Scan Height** parameter in the **Interleave** panel from zero until you reach the surface. In most cases, you should not go below -**60.0nm**.

> **Note:** When the interleaved scan is enabled, the tip's TappingMode height above the sample reduces by the **Lift Scan Height** amount. This places the oscillating tip in contact with the sample as surface features are profiled. The contrast between light and dark reveals areas of high and low elasticity, with the dark area indicating harder material and the lighter areas indicating softer material.

> **Note:** If the **Lift Scan Height** is too large, the interleave may be tapping, and
> therefore not in the continuous contact required for force modulation.

4. Adjust the interleaved **Drive Amplitude** and **Lift Scan Height** until the force modulation
   image is optimized. This may require some experimentation.

> **Note:** If you see a lot of contrast in the amplitude image before reaching the surface,
> try reducing the **Integral** and **Proportional Gains** in the **Feedback** panel.
> Adjust gains slightly lower than when performing normal TappingMode
> imagery. Verify by setting a **Lift Scan Height** of **100nm**, then adjusting the
> gains (and possibly **Drive Amplitude**) until you see the minimum amount of
> contrast in the amplitude image. Once you minimize contrast, enter a **Lift Scan
> Height** of **0.0nm** and approach the surface.

14.8 Force Volume

Force volume imaging with the atomic force microscope (AFM) combines force measurement and
topographic imaging capabilities. A force volume data set can be used to map in two or three
dimensions the interaction forces between a sample and the AFM tip and correlate the force data
with topographic information. Possible applications include elasticity, adhesion, electrostatic,
magnetic, and binding studies. Advantages of force volume imaging include the ability to collect
distributions of forces at various Z-positions and at thousands of XY positions during a single
image scan, correlation of surface topography to interaction force, better quantization of the
interaction force, and new methods of analysis. For detailed information regarding force volume
imaging, refer to the *NanoScope Software 8 User Guide*, Bruker p/n 004-1025-000.

Chapter 15   Interleave Scanning

This chapter provides general information regarding the **Interleave** procedure and commands, with emphasis on **LiftMode**. Specifically, this chapter details the following topics:

- **Preface: Interleave Scanning & LiftMode:**

- **Interleave Mode Description:**

- **LiftMode Description:**

- **Operation of Interleave Scanning / LiftMode:**

- **Use of LiftMode with TappingMode:**

  - **Main Drive Amplitude and Frequency selection:**

  - **Setpoint Selection:**

  - **Interleave Drive Amplitude and Frequency Selection:**

  - **Amplitude Data Interpretation:**

  - **Cantilever Oscillation Amplitude:**

15.1　Preface: Interleave Scanning & LiftMode

Interleave is an advanced feature of NanoScope software which allows the simultaneous acquisition of two data types. Enabling **Interleave** alters the scan pattern of the piezo. After each main scan line trace and retrace (in which topography is typically measured), a second (**Interleave**) trace and retrace is made with data acquired to produce an image concurrently with the main scan.

Typical applications of interleave scanning include MFM (magnetic force microscopy) and EFM (electric force microscopy) measurements; Chapter 16 provides detailed instructions for obtaining MFM images of a standard magnetic sample. Enabling **Interleave** with the mode set to **LIFT** enacts **LiftMode**. During the interleave scan, the feedback is turned off and the tip is lifted to a user-selected height above the surface to perform far field measurements such as MFM and EFM. By recording the cantilever deflection or resonance shifts caused by the magnetic or electric forces on the tip, a magnetic force or electric force image is produced; see Chapter 16 and Chapter 17. **LiftMode** was developed to isolate purely MFM and EFM data from topographic data.

INTERLEAVE can also be used in **Interleave Mode**. In this mode, the feedback is kept on while additional topography, phase lateral force, or force modulation data is acquired.

The **Interleave** commands utilize a set of **Interleave** controls which allow several scan controls (Drive amplitude, Setpoint, and various gains) to be set independently of those in the main scan controls.

This chapter provides a general discussion of the **Interleave** action and commands, with emphasis on **LiftMode**. Chapter 16 and Chapter 17 provide step-by-step instructions for using **Interleave** scanning and **LiftMode** for obtaining magnetic and electric force data. For purposes of learning to use **Interleave** scanning, this section may be useful even to those users whose end application is not magnetic force microscopy.

15.2 Interleave Mode Description

Enabling **Interleave** changes the scan pattern of the tip relative to the imaged area. With **Interleave** mode disabled, the tip scans back and forth in the fast scan direction while slowly moving in the orthogonal direction as shown on the left of Figure 15.2a. This is the standard scan pattern of the NanoScope.

Figure 15.2a X-Y Scan Pattern

INTERLEAVE
DISABLED
(standard)

four scan lines
(trace/retrace pairs)
shown

slow scan
direction

fast scan
direction

INTERLEAVE
ENABLED

four Main and four Interleave
scan lines shown

INTERLEAVE
trace and retrace

MAIN trace
and retrace

With INTERLEAVE enables, scanner executes
trace/retrace pairs, alternately performing a
MAIN pair, then an INTERLEAVE pair.
Vertical Scan speed is 1/2 normal rate with
twice the number of lines.

With **Interleave** mode enabled, the system first performs a standard trace and retrace with the main **Feedback** controls in effect. The tip moves at half the normal rate in the slow scan direction. As shown on the right of Figure 15.2a, an additional trace and retrace are then performed with the **Interleave** feedback controls enacted. The frame rate halves because twice as many scan lines are performed for the same scan rate.

Two modes are possible for **Interleave** scan: **INTERLEAVE** and **LIFT**. With **INTERLEAVE** selected, the feedback remains on during the interleave pass with the values under **Interleave** feedback controls (Setpoint, Gains, etc.) in effect. In **LIFT** mode, the feedback is instead turned off, and the tip lifted off the surface and scanned at a user-selected height. Topography data recorded during the main pass is used to keep the tip a constant distance from the surface during the **Interleave** trace and retrace.

15.3 LiftMode Description

With the **Interleave** scan option set to **LIFT**, the motion of the tip during the **Interleave** trace and retrace is as shown in Figure 15.3a.

Figure 15.3a LiftMode Profiles
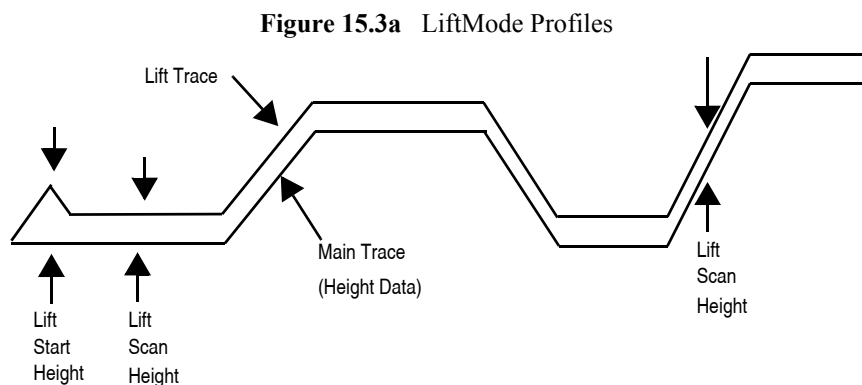
The tip first moves to the **Lift Start Height**, then to the **Lift Scan Height**. A large **Lift Start Height** can be used to pull the tip from the surface and eliminate sticking. The **Lift Scan Height** is the final tip height. This value is added point-by-point to the height data obtained during the Main topography trace and retrace. Values can be positive or negative.

15.4 Operation of Interleave Scanning / LiftMode

These instructions apply to STM, contact AFM, or TappingMode AFM modes. It is assumed the user is familiar with TappingMode or contact AFM to obtain good images of surface topography. Use of interleave scanning requires the steps below; see Chapter 16 for specific examples using MFM.

1. Obtain a topography scan using the appropriate (usually Contact or TappingMode) method: When using **LiftMode**, it is important that the gains and setpoint under **Feedback** controls be adjusted to give a faithful image of the surface. Because the height data is used in the lift pass to trace the topography, a poor measurement of surface height may give inaccurate measurement during the lift pass or cause the tip to strike the surface. Typically, the height data is displayed on Channel 1.

2. Adjust the **Interleave** controls panel to desired settings: Choose the **Interleave Mode** (**INTERLEAVE** or **LIFT**) appropriate for the measurements to be performed. Set the **Interleave** controls as desired. When using TappingMode, the **Drive Amplitude**, **Drive Frequency**, **Gains**, and **Amplitude Setpoint** can be set differently in the **Interleave** panel than in the main **Feedback** panel; see Section 15.5. However, it is often convenient to begin with the main and interleave values set equal; this can be done by toggling the parameters of the appropriate **Interleave** parameters to an "off" (grayed) condition. The values can be changed once engaged. If using **LIFT**, set the **Lift Start Height** and **Lift Scan height**. If using **INTERLEAVE** mode, set the **Gains** and **Amplitude Setpoint**.

Note that certain constraints are imposed: scan sizes, offsets, angles, rates and numbers of samples per scan line are the same for the main and interleave data, and the imaging mode (contact, TappingMode, or force modulation) must also match.

3. Choose the **Interleave Data Type**: Depending on the type of microscope, **Interleave** mode allows the options of amplitude, phase, frequency, potential, input potential, or data types for doing far-field (MFM or EFM) imaging; see Chapter 16 for detailed examples. Auxiliary channels are also available for some applications.

Once the **Interleave Scan Line** is chosen, **Interleave** mode is automatically enabled, triggering interleave scanning. Interleave data typically displays as the second image. Note that the scan rate in the slow direction is halved.

Figure 15.4a Interleave Scan Line
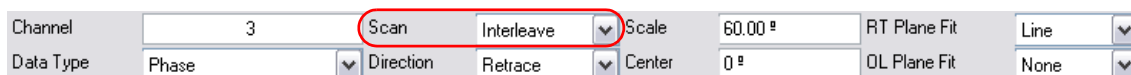
4. Display the interleave data by switching **Scan Line** (in the **Channel** panels) to **Interleave**. See Figure 15.4a.

- **Lift Scan Height**: The lateral and vertical resolutions of the LIFT data depend on the distance between tip and sample: the lower the tip, the higher the resolution. However, the **Lift Scan Height** must be high enough that the tip does not contact the sample during the LIFT trace and retrace.

- **Tip Shape**: As shown in Figure 15.3a, the tip separation in the **LiftMode** is defined in terms of the Z direction only. The **Lift Scan Height** is added to the height values taken from previous scan lines point-by-point. However, the tip may be closer to the sample than the Z separation indicates. On features with steep edges, the tip may get very close to the sample even though the Z separation is constant; see Figure 15.3a.

- Scan **Line Direction**: The **Line Direction** should be set to RETRACE for both the main and interleaved scans. If it is set instead to Trace, a band may appear along the left side of the images due to the ramp between the surface and the **Lift Scan Height**.

15.5   Use of LiftMode with TappingMode

Additional considerations when using **LiftMode** with TappingMode are discussed below.

15.5.1   Main Drive Amplitude and Frequency selection

As usual, these parameters are set in Cantilever Tune before engaging. It is helpful to keep in mind the measurements to be done in **LiftMode** when setting these values. For example, if **Amplitude** data will be monitored during the Lift scan for magnetic force imaging, the **Drive Frequency** should be set to the side of the resonance; see Chapter 16. (However, certain parameters can be set independently for the interleave scan; see below.)

15.5.2 Setpoint Selection

When the main and interleave **Drive Amplitudes** and **Drive Frequencies** are equal (parameters in the **Interleave** panel grayed out), the cantilever oscillation amplitude increases to the free oscillation amplitude when the tip is lifted off the surface in **LiftMode**. If a small setpoint value forces the oscillation amplitude low while the feedback is running, the amplitude can grow considerably when the tip is lifted free of the sample surface. The change can also be large if the main **Drive Amplitude** was increased or the main **Drive Frequency** altered after the tip was engaged. (The vibration amplitude remains at the setpoint during the main scan even if these parameters are changed.) This could make the tip hit the surface in the lift scan for small **Lift Scan Heights**.

15.5.3 Interleave Drive Amplitude and Frequency Selection

The cantilever drive amplitude can be set differently in the Lift scan as compared to the main scan by clicking on the parameter of the corresponding parameter in the **Interleave** panel to "on" (green) and adjusting the value. This allows the tuning of a measurement in the Lift scan lines without disturbing the topography data acquired during the Main scan lines. The **Interleave Drive Amplitude** must be set low enough that the tip does not strike the surface during the Lift pass.

⚠️	**WARNING:**	Before enabling the **Interleave Drive Amplitude**, check that its value is not much larger than the main Drive Amplitude value to prevent possible damage to the tip.
	AVERTISSEMENT:	Lors d'un travail en mode intercalé (**Interleave Mode**), vérifier que la tension appliquée pour osciller le bras de levier lorsqu'il est en positon haute n'est pas beaucoup plus importante que la tension appliquée lorsque ce même bras de levier est en position basse (**Main Drive Amplitude**). Le non respect de cette procédure peut entraîner la destruction de la pointe.
	WARNUNG:	Bevor Sie im **Interleave-Mode** die "**Interleave Drive-Amplitude**" einschalten, vergewissern Sie sich bitte, daß der dort eingetragene Wert nicht wesentlich größer ist, als der Wert der "Main Drive-Amplitude", um evtl. Beschädigungen der Spitze vorzubeugen.

The **Interleave Drive Frequency** can also be adjusted, which may be useful if acquiring amplitude data in **LiftMode**; see Chapter 16.

15.5.4 Amplitude Data Interpretation

When monitoring amplitude data in **LiftMode**, brighter regions correspond to smaller amplitude, and darker regions to larger amplitude.

15.5.5 Cantilever Oscillation Amplitude

The selection of the oscillation amplitude in **LiftMode** depends on the quantity to be measured. For force gradients which are small in magnitude but occur over relatively large distances (sometimes hundreds of nm, as with magnetic or electric forces), the oscillation amplitude can be large, which for some applications may be beneficial. The **Lift Scan Height** must be correspondingly large so that the tip does not strike the surface. However, the lateral resolution of far field (MFM or EFM) measurements decreases with distance from the surface. Typically, the resolution is limited to a value (in nm) roughly equal to the Lift scan height; see Chapter 16.

Small amplitudes must be used to sense force gradients, such as Van der Waals forces, which occur over short distances (typically a few nm). As much of the cantilever travel as possible should be within the range of the force gradient.

Chapter 16　　Magnetic Force Microscopy

This chapter describes how to perform Magnetic Force Microscopy (MFM) using the Interleave and **LiftMode** procedures discussed in . Please review those sections prior to attempting MFM. These hardware units allows phase detection and frequency modulation for optimal MFM imaging.

Specifically, this chapter discusses the following topics:

16.1   Magnetic Force Microscopy

In MFM, a tapping cantilever equipped with a special tip first scans over the surface of the sample to obtain topographic information. Using **LiftMode** as shown in Figure 16.1a, the tip then raises just above the sample surface. The surface topography from the initial scan is added to the lift height to maintain constant separation during the lifted scan. The influence of magnetic force is measured using the principle of force gradient detection.

16.1.1   Force Gradient Detection

In the absence of magnetic forces, the cantilever has a resonant frequency f_0. This frequency is shifted by an amount Δf proportional to vertical gradients in the magnetic forces on the tip. The shifts in resonant frequency tend to be very small, typically in the range 1-50 Hz for cantilevers having a resonant frequency $f_0 \sim 100$ kHz. These frequency shifts can be detected three ways: **phase detection** which measures the cantilever's phase of oscillation relative to the piezo drive; **amplitude detection** which tracks variations in oscillation amplitude; and **frequency modulation** which directly tracks shifts in resonant frequency. **Phase detection** and **frequency modulation** produce results that are generally superior to **amplitude detection**.

Figure 16.1a   MFM LiftMode Principles

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Magnetic Fields
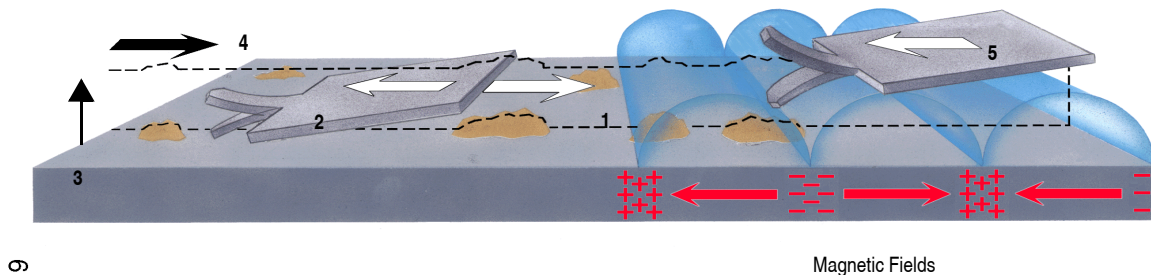
1. Cantilever traces surface topography on first trace.

2. Cantilever retraces surface topography on first retrace.

3. Cantilever ascends to Lift scan height.

4. Lifted cantilever profiles topography while responding to magnetic influences (second trace).

5. Lifted cantilever reprofiles topography while responding to magnetic influences (second retrace).

16.1.2   Amplitude Detection Techniques

Amplitude detection has largely been superseded by frequency modulation and phase detection.

16.2 Basic MFM Operation

In the instructions below, steps specific to phase and amplitude imaging are described independently.

16.2.1 MFM Using LiftMode

LiftMode allows imaging of relatively weak but long-range magnetic interactions while minimizing the influence of topography (see Figure 16.1a). Measurements are taken in two passes across each scan line; each pass consists of one trace and one retrace. In the first pass, topographical data is taken in TappingMode. The tip is then raised to the lift scan height and a second trace and retrace performed while maintaining a constant separation between the tip and local surface topography. Magnetic interactions are detected during this second pass. In LiftMode, topographical features are virtually absent from the MFM image (see Figure 16.2f).

This section provides instructions for using the **LiftMode** of Interleave Scanning to obtain MFM images. These guidelines will help you obtain an MFM image of a standard magnetic sample (metal-evaporated video tape). Standard tape samples are provided with the MFM starter kit, and can be obtained from Bruker. Other samples can also be used; however, you will not have the benefit of comparing your results with the images shown here. Obtaining a good image of the tape sample will familiarize you with Interleave and MFM techniques and provide a check that the system is correctly tuned to image magnetic samples. Many of the principles discussed here also apply to Electric Force Microscopy (EFM).

As mentioned above, NanoScope controllers use force gradient detection for MFM imaging. Within this general technique, there are three possible schemes, known as frequency modulation, phase detection, and amplitude detection. Phase detection and frequency modulation detection are superior methods for magnetic force imaging, offering greater ease of use, better signal-to-noise ratios, and reduced artifact content as compared to amplitude detection.

For MFM procedures, magnetic-coated tips are required. Various kinds of MFM probes are available for specific applications; contact Bruker for more information. For specific information regarding TappingMode and Interleave Scanning please refer to Chapter 10 and Chapter 15 respectively.

The procedure below suggests parameter values that should work well for most applications. Further adjustment, in some cases, will improve the quality of MFM scans. Some experimentation may be needed to optimize the imaging of specific samples. See the suggestions at the end of this section.

16.2.2 Magnetic Force Microscopy Procedure

1. Magnetize a NanoProbe magnetic probe with a strong permanent magnet before installing the probe holder on the AFM head.

   Note: Tips are usually magnetized with the field aligned along the tip axis (perpendicular to the sample surface). The MFM then senses force gradients from the perpendicular component of the samples's stray field. Tip magnetizers can be purchased from Bruker.

2. Mount a NanoProbe magnetic probe on the scanner or tip holder.

3. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 16.2a.

Figure 16.2a The ELECTRICAL & MAGNETIC AFM Select Experiment window
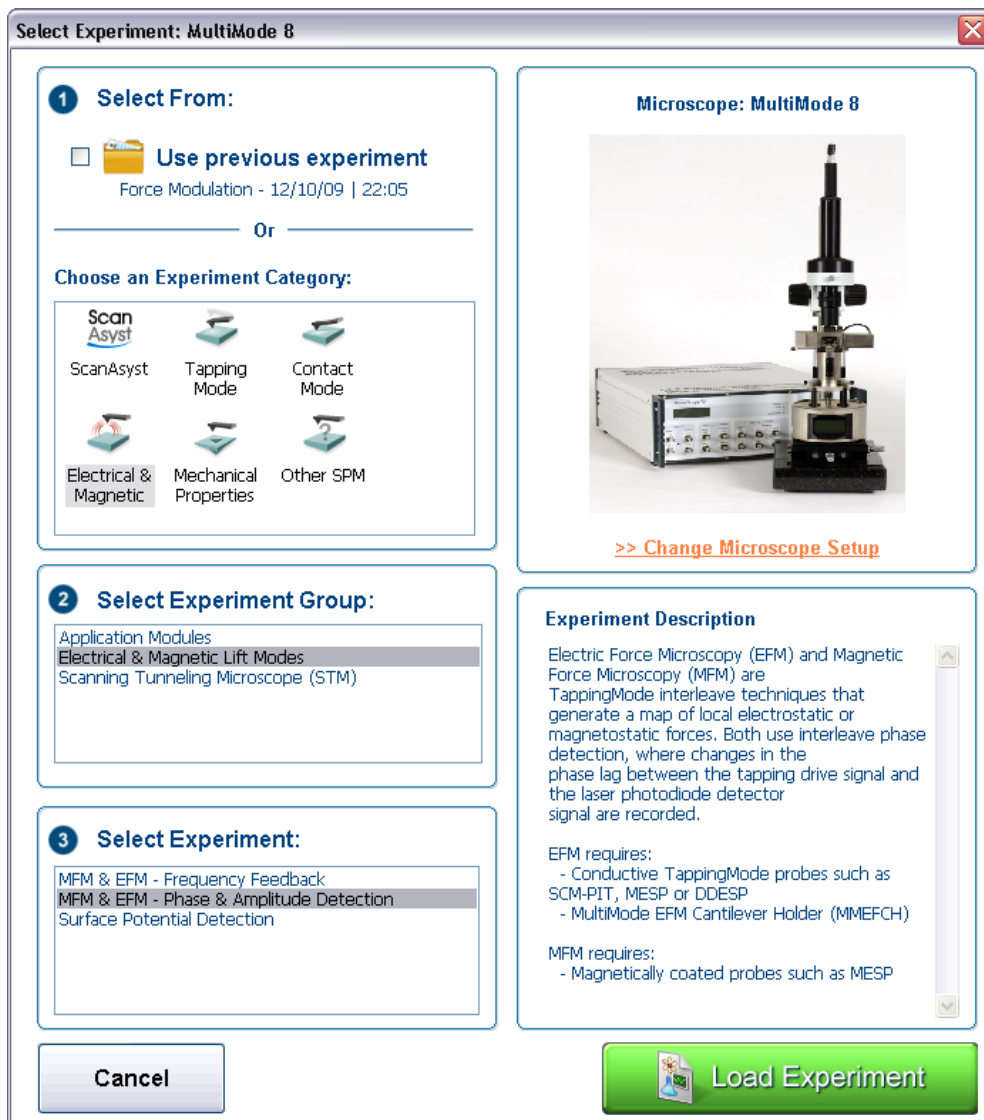
4. Select ELECTRICAL & MAGNETIC in the **Choose an Experiment Catagory** panel.

5. Select ELECTRICAL & MAGNETIC LIFT MODES in the **Select Experiment Group** panel.

6. Select MFM & EFM - PHASE & AMPLITUDE DETECTION in the **Select Experiment** panel and click LOAD EXPERIMENT.

7. Set up the AFM for TappingMode operation (see Chapter 10).

8. Tune the cantilever **Drive Frequency**.

> **Note:** The procedure to tune the cantilever drive amplitude depends on whether you are using phase detection or amplitude detection. Both cases rely on automatic **Cantilever tune** just as when preparing for TappingMode (see Chapter 10). MFM cantilevers have resonant frequencies between 50 and 100 kHz. If using the **AutoTune** feature, these values can be used as bounds for the frequency sweep.

Setting a Drive Frequency for Phase Detection

The Drive frequency should be set to the center of the cantilever resonance, as shown in Figure 16.2b. This occurs automatically if using **AutoTune.**

Figure 16.2b   Cantilever Tune for Phase Detection and Frequency Modulation
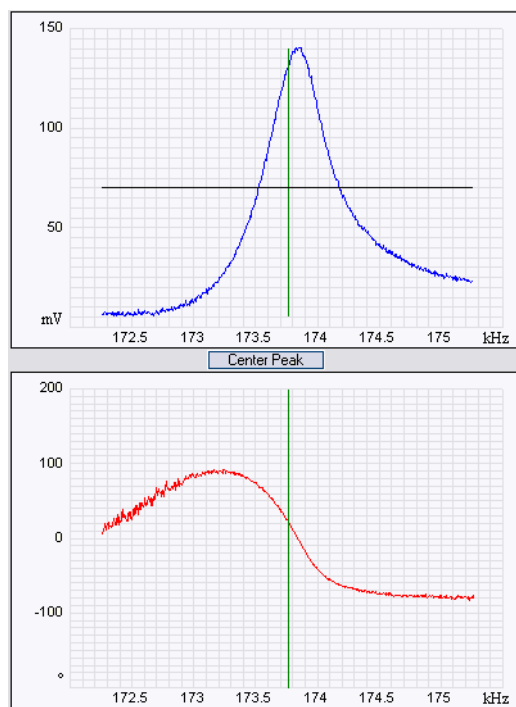
To correctly track the cantilever phase, the **Phase offset** parameter must be adjusted. This automatically occurs in **AutoTune**; alternatively, **Zero Phase** can be selected from the **Channel 2** panel. The phase curve should appear as in Figure 16.2b, decreasing with increasing frequency and crossing the center line (corresponding to a 90° phase lag) at the peak frequency. The phase curve then measures the phase lag between the drive voltage and the cantilever response. Again, vertical gradients in the magnetic force cause a shift Δf_0 in the resonance frequency. In this case, resonance shifts give rise to phase shifts $\Delta\phi$ which then give an image of the magnetic force gradients (see Figure 16.2c**).**

Figure 16.2c Shift In Phase at Fixed Drive Frequency
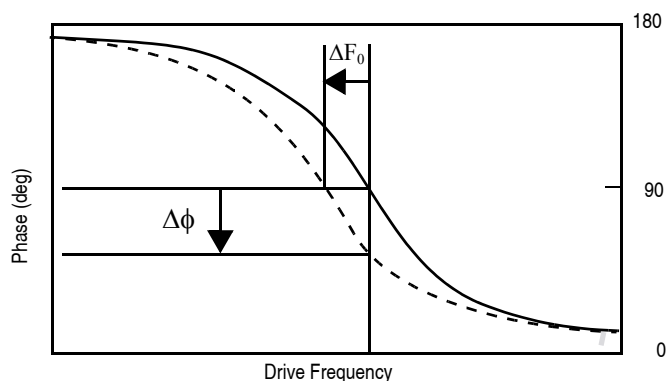
Setting a Drive Frequency for Amplitude Detection

1. Use the **AutoTune** feature to find the resonance peak.

2. Select the **Offset** function in the **Cantilever Tune** window to manually move the drive frequency to the side of the resonance (see Figure 16.2d).

3. Set the **Drive Frequency** to the steepest part of the resonance curve for maximum sensitivity.

> **Note:** As the tip oscillates above the sample, a gradient in the magnetic force will shift the resonance frequency f_0 (see Figure 16.2e). Tracking the variations in oscillation amplitude while in LiftMode yields an image of the magnetic force gradients.

Figure 16.2d Cantilever Tune for Amplitude Detection
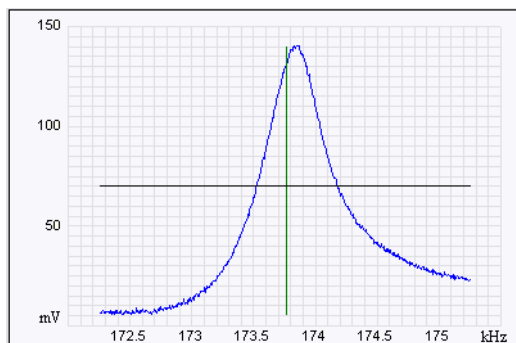
Figure 16.2e   Shift in Amplitude at Fixed Drive Frequency
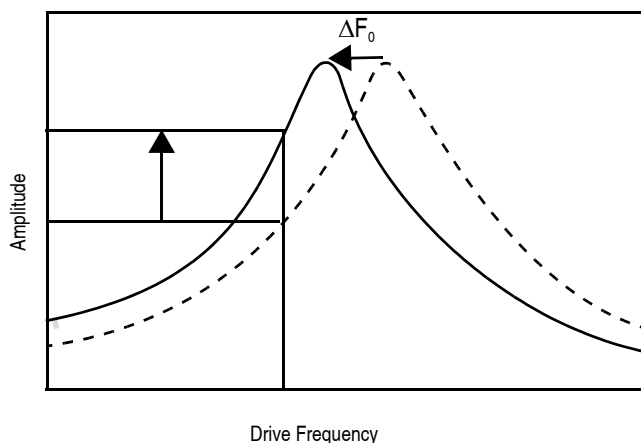
4. Select an appropriate **Target Amplitude** (approximately 560mV) using **Auto Tune** to adjust the **Drive Amplitude** so that the RMS voltage response of the photodetector is approximately 560mV before tuning. Or, exit **Cantilever Tune** and manually adjust the **Drive Amplitude** parameter in the **Feedback** panel.

> **Note:**   Somewhat larger values may be beneficial if using amplitude detection.

5. Exit **Cantilever Tune** and return to **Image Mode**.

6. Under **Interleave** panel set the **Lift Start Height** to **0nm**, and **Lift Scan Height** to **100nm**.

> **Note:**   The lift height can be optimized later.

7. Set the remaining Interleave parameters (**Setpoint**, **Drive Amplitude**, **Drive Frequency**, **Gains**) to the **Main** values by setting the flags left of the **Interleave** column to **Off**.

8. Under **Scan** controls, set the **Scan size** to **5μm** and **Scan rate** to **1–2Hz**.

9. Set the **Channel 1 Data Type** to **HEIGHT**, the **Data Scale** to **75nm** and the **Line Direction** to **RETRACE**.

10. Engage the AFM and make the necessary adjustments to obtain a good topographical image while displaying height data.

	CAUTION:	Use the highest possible **Setpoint** to ensure that the tip is contacting the surface only lightly. The image should be similar to the topographic image shown on the left of Figure 16.2f. The surface is fairly flat with lubrication nodules of various sizes. A good image of the nodules indicates that the tip is sharp.

Figure 16.2f Topographic (left) and Magnetic Force Gradient Image (right)

TMAFM Images
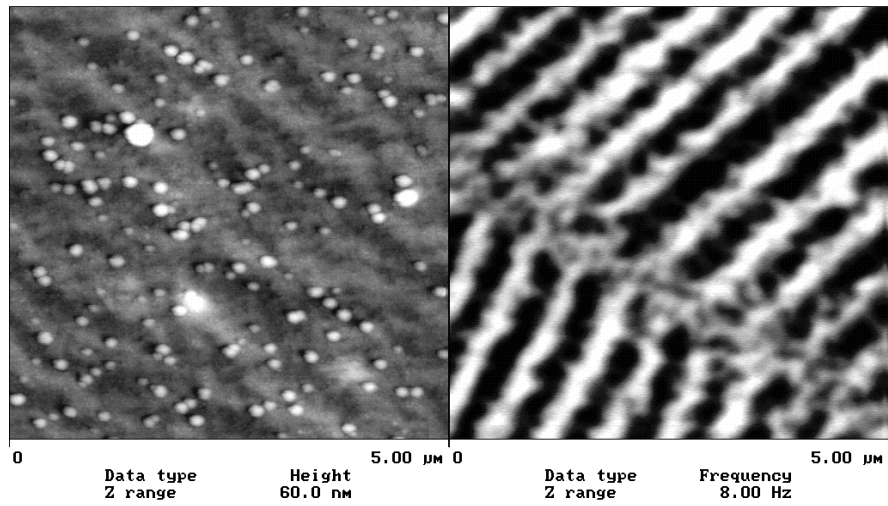
Phase Detection (Recommended)

Set the **Channel 3** image **Data Type** to PHASE, **Scan Line** to INTERLEAVE, **Z range** to **3 degrees**; and **Line Direction** to RETRACE.

> **Note:** It is important that the **Line Direction** be set to RETRACE for both the main and interleave scans. If instead it is set to TRACE, a band may appear along the left side of the images due to the time taken for the tip to move between the surface and the lift scan height.

Amplitude Detection

1. Set the **Channel 3** image **Data type** to AMPLITUDE, **Z range** to **1nm**, and **Line direction** to **Retrace**.

2. Change **Interleave mode** to LIFT to invoke **LiftMode**.

3. Set the **Channel 3 Scan line** to **Interleave** to display the interleaved data.

> **Note:** This can only be done after **Interleave mode** is enabled. A magnetic force gradient image similar to that shown on the right of Figure 16.2f should appear as the Channel 3 image. The alternating dark and light stripes represent the recorded magnetic information, signifying a varying resonant frequency and magnetic force gradient on the tip.

> **Note:** Keep the **Setpoint** as large as possible while consistent with a good image. Wider scans ($> 25\mu m$) will reveal separate tracks in which the magnetic stripes are at different angles

Frequency Modulation

It may be desirable to use frequency modulation. This activates a feedback loop which modulates the **Drive Frequency** to keep the cantilever phase lag at 90 degrees relative to the drive, corresponding to resonance. The frequency **Data Type** displays the resulting shift in **Drive Frequency** in *Hz*, and gives the most direct, quantitative image of force gradients.

To enable frequency modulation, follow the procedure above for obtaining an MFM image with phase detection with the following modifications:

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 16.2g.

Figure 16.2g   The ELECTRICAL & MAGNETIC AFM **Select Experiment** window
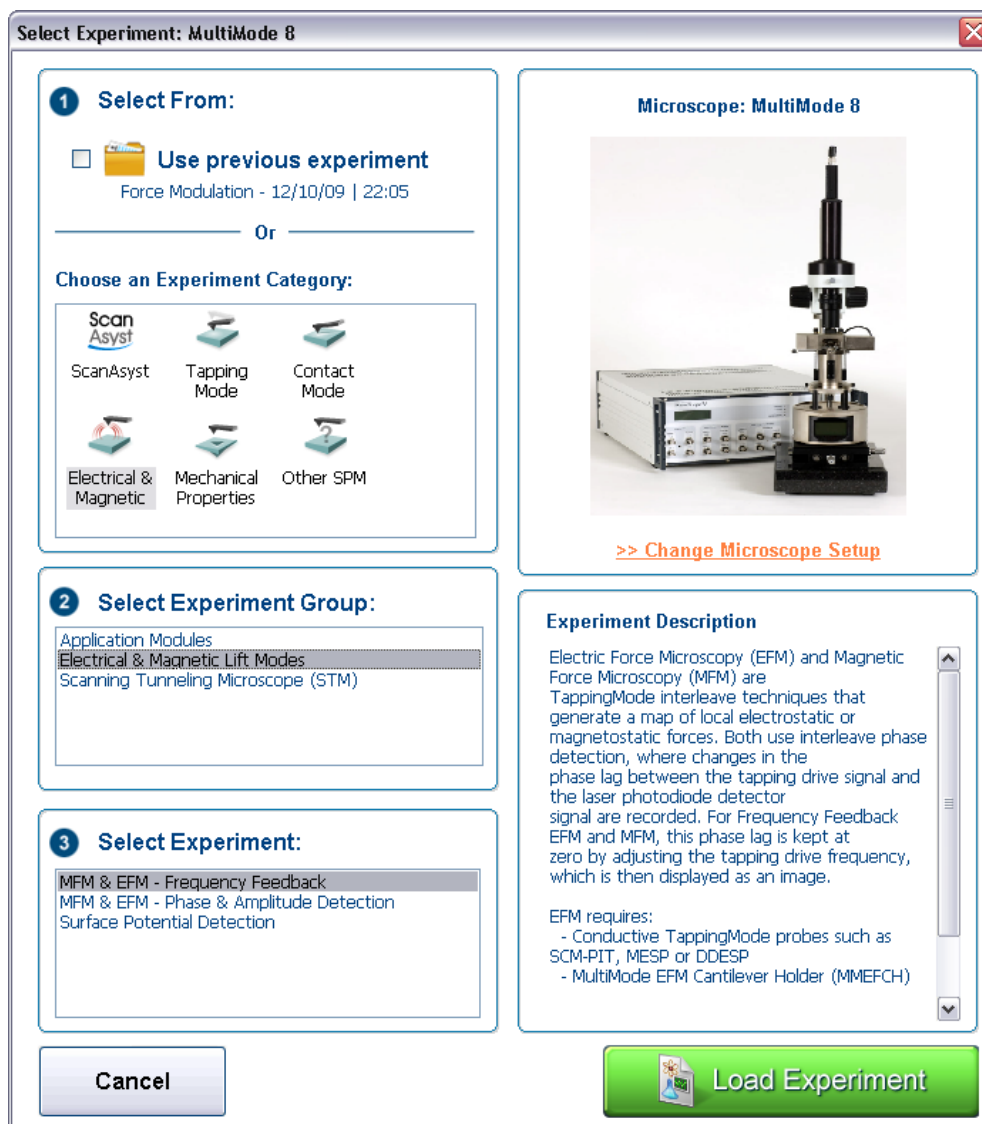
2. Select ELECTRICAL & MAGNETIC in the **Choose an Experiment Catagory** panel.

3. Select ELECTRICAL & MAGNETIC LIFT MODES in the **Select Experiment Group** panel.

4. Select **MFM & EFM - PHASE & FREQUENCY FEEDBACK** in the **Select Experiment** panel and click LOAD EXPERIMENT.

5. In the **Interleave** panel, set the **Input Feedback** to FREQUENCY.

6. Switch the Channel 3 image **Data type** to **Frequency**. Try a **Data Scale** (frequency shift) of approximately **10 Hz**.

7. If necessary, adjust the frequency modulation gains in the **Interleave** panel.

As with topography gains, the scan can be optimized by increasing the gains to maximize feedback response, but not so high that oscillation sets in.

16.3   Advanced MFM Operation

16.3.1 Lift Scan Height and Magnetic Imaging Resolution

The most important parameter affecting imaging resolution is **Lift Scan Height**. The range of 10–200nm is most useful. In general, MFM resolution is roughly equal to the lift height. Smaller **Lift Scan Heights** give better resolution; magnetic features smaller than the **Lift Scan Height** may not be resolved. The tip also experiences stronger fields close to the surface, giving improved signal-to-noise ratios.

For example, the image of metal-evaporated tape in Figure 16.2f has a resolution limited by the 100 nm **Lift Scan Height**. To improve the resolution, try reducing the **Lift Scan Height** to ~ **25nm**. Ensure that the tip does not strike the surface on the low point of its swing in the Lift image. Tip strikes appear as black or white spots, or even noisy, high-contrast streaks crossing the image. If the tip begins to strike the surface, reduce the **Interleave Drive Amplitude**. (In general, MFM tips are not damaged by intermittent tip strikes in **LiftMode**, except in extreme cases of very large amplitude and small lift heights.) An example of an image of the metal-evaporated tape taken with a **Lift Scan Height** of **30nm** is shown in Figure 16.3a. Note the fine magnetic structure that is not visible in Figure 16.2f. When imaging a sample for the first time, begin with moderate **Lift Scan Heights** (**50nm or greater**), then adjust downward. On relatively smooth samples (e.g., hard disks), lift heights down to 0nm can be used, as long as the drive amplitude is adjusted accordingly. (Lift scan heights of 0nm still correspond to a non-zero mean tip-sample distance. See the section on **Setpoint** below.) It is usually not beneficial to use **Lift Scan Heights** much smaller than the surface roughness. Users are encouraged to experiment for the best images on their samples.

The ultimate lateral resolution of MFM is near 20nm. Resolution is affected by properties of the tip, including mechanical sharpness and magnetic structure. When in good condition, magnetically-coated tips routinely give 50nm resolution, and many achieve 30nm or better.

Figure 16.3a   High-resolution Magnetic Force Gradient Image
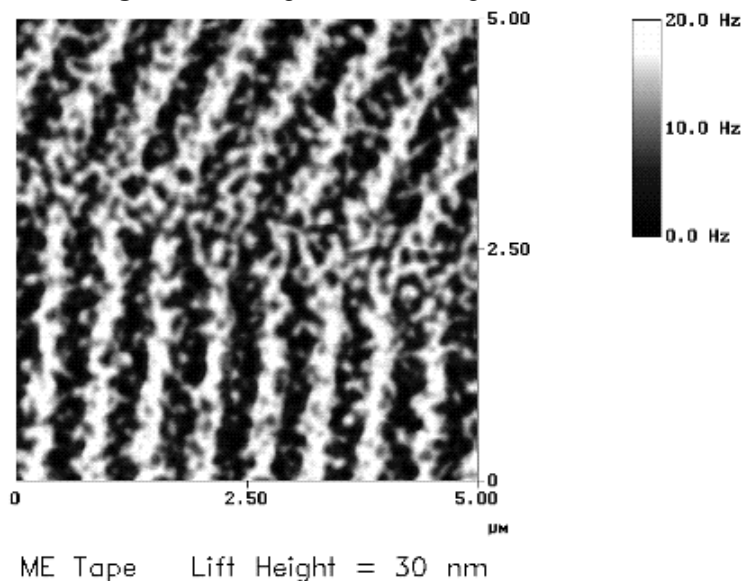
16.3.2 Fine Tuning Interleave Controls

Certain scanning parameters found under **Interleave** can be set to values in the Interleave (Lift) scan that differ from the values in the main scan. These parameters are enabled by clicking on the desired Interleave control, toggling its state from **Off** (gray) to **On** (green). When the flag is set to **Off**, the main scan control parameter setting takes precedence. When the flag is set to **On** the displayed Interleave scan value is active, overriding the main scan value.

16.3.3 Drive Amplitude

For MFM, of particular use is the Interleave **Drive Amplitude**. This parameter can affect a magnetic force image in a variety of ways.

- Increasing the **Drive Amplitude** can improve the signal-to-noise ratio when using phase detection or frequency modulation. This is because intrinsic, low-level noise interferes less when measuring the phase of a larger cantilever oscillation amplitude and hence stronger photodetector output. As an illustration, try setting the Interleave **Drive Amplitude** to **0**; the resulting phase image will be pure noise because one cannot measure the phase of a non-oscillating cantilever.

- In **LiftMode**, the Interleave **Drive Amplitude** can often be set to a value larger than in the main scan, thus giving optimal signal-to-noise. In some cases this is beneficial as long as the **Drive Amplitude** is not increased to the extent that the tip strikes the surface on the low point of its swing. The signatures of tip-sample contact are white and black spots in the image, or, in extreme cases, noisy, high-contrast streaks across the whole image. It is usually safe to increase the **Drive Amplitude** until the first signs of tip strike are noticed, then reduce the amplitude slightly.

• Before enabling Interleave **Drive Amplitude**, check that its value is not much larger than the main **Drive Amplitude** value. This prevents the cantilever oscillation from jumping to a very large amplitude when the parameter is enabled, possibly damaging the tip. The **Drive Amplitude** can be adjusted even when the parameter is disabled (grayed).

⚠	**CAUTION:**	Before enabling the Interleave **Drive Amplitude**, check that its value is not much larger than the main **Drive Amplitude** value to prevent possible damage to the tip.
	ATTENTION:	Lors d'un travail enmode intercalé (Interleave Mode) vérifier que la valeur de tension appliquée à l'oscillateur piézo-électrique est inférieure à celle appliquée à l'oscillateur en mode imagerie (Main Drive Amplitude). Le non respect de cette procédure peut entraîner la destruction de la pointe.
	VORSICHT:	Um mögliche Beschädigung der Meßspitze zu vermeiden, vergewissern Sie sich, daß der Wert der Interleave Drive Amplitude nicht wesentlich größer ist, als der Main Drive Amplitude, ehe Sie den Interleave Mode aktivieren.

• When using **Amplitude Detection**, variations in **Drive Amplitude** affect sensitivity and image contrast as well as signal-to-noise ratio. This is because changes in the oscillation amplitude change the slope of the amplitude vs. frequency curve, and hence the effective sensitivity; see Figure 16.3b. With phase detection and frequency modulation, changes in amplitude produce no change in contrast, and results are thus more reproducible than with amplitude detection.

Setpoint

For the most reproducible results, it is best to use a consistent setpoint. In **LiftMode**, the total tip-sample distance h_{tot} is the sum of the average tip-sample distance in TappingMode h_T, and the lift scan height h_{lift} (see Figure 16.3b). In **TappingMode**, the average tip-sample distance h_T is equal to the oscillation amplitude, which is determined by the setpoint and the amplitude sensitivity of the tip. MESPs typically have an amplitude sensitivity of approximately 25nm/V.

Large variations in setpoint can change the total tip-sample distance in **LiftMode**, sometimes with visible results in the magnetic image. For this reason, reproducible results are most easily obtained by using consistent setpoints. Note that a lift scan height of 0nm still gives a mean tip-sample distance of h_T in **LiftMode**.

Figure 16.3b   Tip Height and Oscillation Amplitudes (TappingMode & LiftMode)
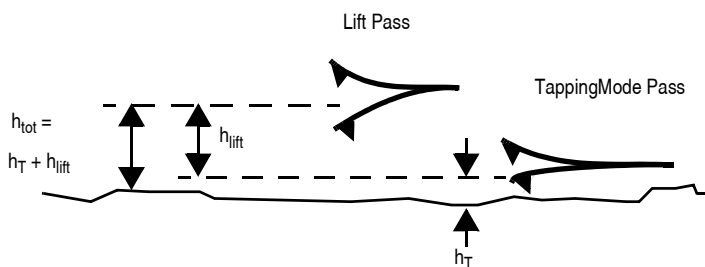
The relationship between setpoint voltage and oscillation amplitude is known as the sensitivity. Its value can be determined with **Force Calibration** (see Chapter 14 for details). For 225µm MFM cantilevers, the sensitivity is typically in the range 20-25nm/V. The exact value depends on the position of the laser spot on the cantilever, the cantilever geometry, and the particular instrument. A 1V **Setpoint** typically corresponds to $h_T \sim 14\text{-}18\text{nm}$.

16.4   Troubleshooting

16.4.1   MFM Image Verification

The procedure described above should produce a good magnetic force gradient image of the videotape sample. If there is a problem, check that the **Interleave** mode is set to **LIFT** and that the **Scan Line** is set to **INTERLEAVE**. Check also that the **Interleave** values of **Drive Amplitude** and **Drive Frequency** are initially set equal to the main **Scan** values.

16.4.2 Saturation in Amplitude Detection

If using amplitude detection, the magnetic force image can saturate (appear completely featureless) if the **Interleave Drive Amplitude** is significantly different than the **Drive Amplitude** in the main scan. Adjust the **Interleave Setpoint** to restore the image.

> **Note:** The **Interleave Setpoint** has no physical effect in **LiftMode** since there is no surface feedback during the lift pass.

16.4.3 Optical Interference

Optical interference may sometimes appear in the Lift (magnetic force gradient) image when imaging highly reflective samples. Optical interference appears as evenly spaced, sometimes wavy lines with ~1–2μm spacing superimposed on the lift image. This occurs when ambient laser light (i.e., light passing around or through the cantilever, then reflecting off the sample) interferes with laser light reflecting from the cantilever. Interference can be alleviated by moving the beam spot up the cantilever away from the tip; one-third of the cantilever length works well. The adjustment can be refined by carefully moving the beam spot laterally a small distance on the cantilever while scanning until interference fringes are minimized. Be careful not to move the beam off the cantilever or feedback may be lost.

Chapter 17   Electric Techniques

This chapter describes how to perform two electric techniques: Electric Force Microscopy (EFM) and Surface Potential. EFM is similar to Magnetic Force Microscopy (MFM) and shares many of the same procedural techniques. Both modes utilize the Interleave and LiftMode procedures discussed in previous chapters. Please read those chapters before attempting electric force measurements.
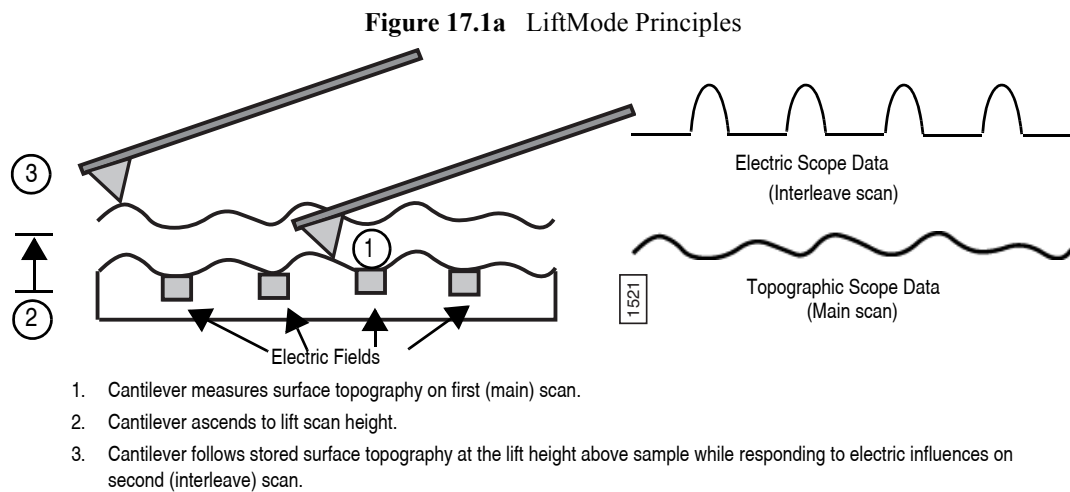
Specifically, this chapter includes:

17.1   Electric Techniques Overview

There are two types of electric techniques used with MultiMode microscopes: **Electric Force Microscopy** (EFM) and **Surface Potential Detection**.

Electric techniques are similar to magnetic force microscopy (MFM) and share many of the same procedural techniques. Electric techniques and MFM both use the **Interleave** and **LiftMode** procedures. The two-pass LiftMode measurement allows the imaging of relatively weak but long-range magnetic and electrostatic interactions while minimizing the influence of topography (see Figure 17.1a). LiftMode records measurements in two passes, each consisting of one trace and one retrace, across each scanline. First, LiftMode records topographical data in TappingMode on one trace and retrace. Then, the tip raises to the **Lift Scan Height**, and performs a second trace and retrace while maintaining a constant separation between the tip and local surface topography.

Figure 17.1a   LiftMode Principles

1. Cantilever measures surface topography on first (main) scan.
2. Cantilever ascends to lift scan height.
3. Cantilever follows stored surface topography at the lift height above sample while responding to electric influences on second (interleave) scan.

17.1.1 Electric Force Microscopy Overview

Electric Force Microscopy measures variations in the electric field gradient above a sample. The sample may be conducting, nonconducting, or mixed. Because the surface topography (e.g. sharp points on the surface concentrate the field gradient) shapes the electric field gradient, large differences in topography make it difficult to distinguish electric field variations due to topography or due to a true variation in the field source. The best samples for EFM are samples with fairly smooth topography. The field source includes trapped charges, applied voltage, etc. Samples with insulating layers (passivation) on top of conducting regions are also good candidates for EFM.

17.1.2 Surface Potential Imaging Overview

Surface potential imaging measures the effective surface voltage of the sample by adjusting the voltage on the tip so that it feels a minimum electric force from the sample. (In this state, the voltage on the tip and sample is the same.) Samples for surface potential measurements should have an equivalent surface voltage of less than ±10V, and operation is easiest for voltage ranges of ±5V. The noise level of this technique is typically 10mV. Samples may consist of conducting and nonconducting regions, but the conducting regions should not be passivated. Samples with regions of different materials will also show contrast due to contact potential differences. Quantitative voltage measurements can be made of the relative voltages within a single image.

17.2   Electric Force Microscopy

17.2.1 Electric Force Microscopy Theory

Electric Force Microscopy is analogous to standard MFM, except that gradients being sensed are due to electrostatic forces. In this method, the cantilever is vibrated by a small piezoelectric element near its resonant frequency. The cantilever's resonant frequency changes in response to any additional force gradient. Attractive forces make the cantilever effectively "softer," reducing the cantilever resonant frequency. Conversely, repulsive forces make the cantilever effectively "stiffer," increasing the resonant frequency. A comparison of these force additives is shown in Figure 17.2a.

Figure 17.2a   Comparison of Attractive and Repulsive Forces

Attractive gradient equivalent to additional spring in tension attached
to tip, reducing the cantilever resonance frequency.

Repulsive gradient equivalent to additional spring in compression attached
to tip, increasing the cantilever resonance frequency.
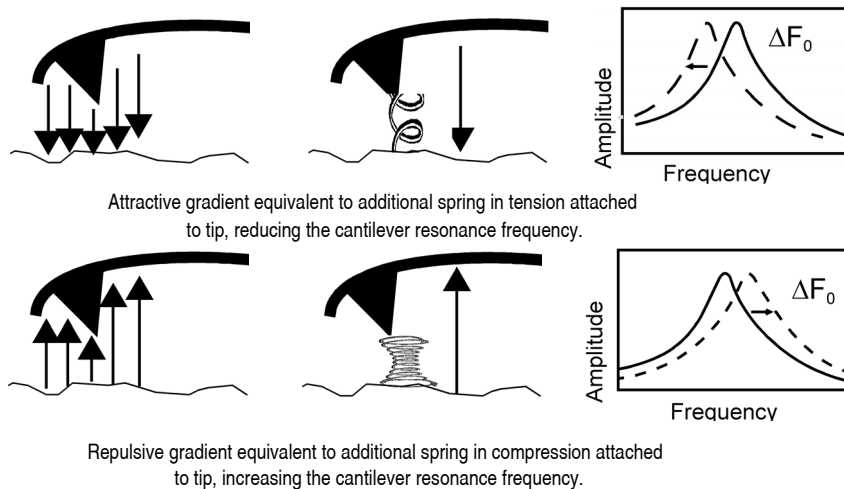
Changes in cantilever resonant frequency are detected in one of the following ways:

- Phase detection

- Frequency modulation

- Amplitude detection (not recommended due to artifacts)

All of the above methods rely on the change in resonant frequency of the cantilever due to vertical force gradients from the sample. Figure 17.2b shows a diagram of how the NanoScope V provides signal enhancement and feedback allowing gradient detection. The best candidates for electric field gradient imaging are samples that have large contrasts in the electric force gradient due to material differences or regions at substantially different potentials. For other samples having rough surface topography or small voltage variations, this technique may be undesirable because topographic features appear in the **LiftMode** image.

Figure 17.2b Block Diagram of NanoScope V for EFM
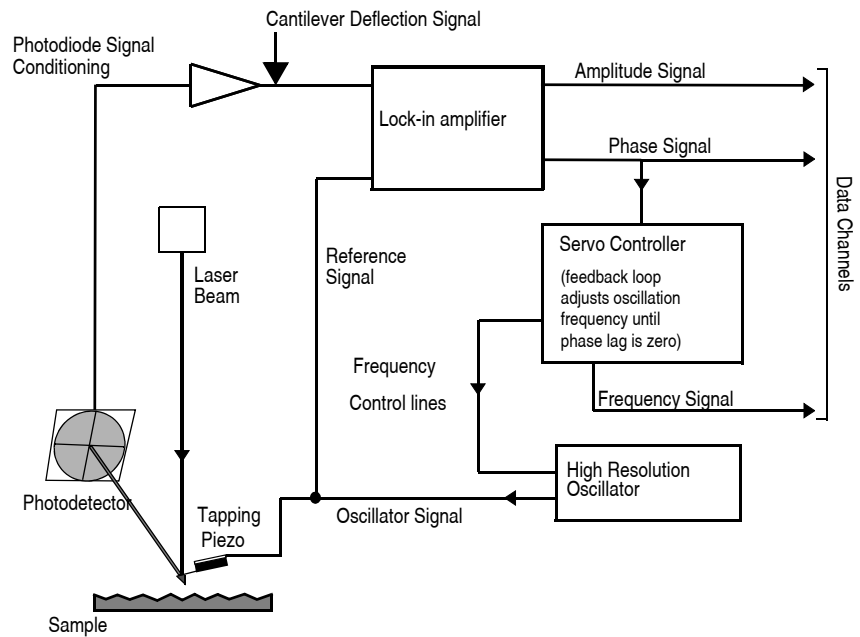
In many cases, you must apply a voltage to the tip or sample to achieve a high-quality image. Various methods for applying voltages to the tip and sample are included in the sections that follow. Samples with permanent electric fields may not require voltage application.

17.2.2 Electric Force Microscopy Preparation

This section explains how to apply a voltage to the tip or sample to generate electric fields. If the sample has a permanent electric field which does not require the external application of voltage, the steps below are not required and you can proceed to Section 17.2.3.

Enabling Tip/Sample in the Software

If you have chosen a configuration where a bias is applied to the tip or sample, you must enable **Tip Bias Control** and/or **Sample Bias Control** in the software. These parameters appear in the **Other** panel, shown in Figure 17.2c. Select **TIP BIAS** and **SAMPLE BIAS** to enable software control of tip and sample voltages.

Figure 17.2c   EFM Scan Parameters

Setting Tip/Sample Bias in the Software

Set the **Tip** and/or **Sample Bias** parameters in the **Feedback** panel, shown in Figure 17.2c, after you have enabled them. The **TIP BIAS** can be set within the range of -12V to +12V. The **SAMPLE BIAS** can be set within the range of -10V to +10V.

17.2.3 Electric Force Microscopy Procedures

1. Verify that **Tip** and **Sample Bias** have been properly set.

2. Electrically connect the sample by mounting it to a standard sample disk or stage using conducting epoxy or silver paint.

3. Verify the connection is good (a poor connection introduces noise).

   Note: If an external power supply connects directly to leads on the sample itself, it is important to electrically isolate the sample from the stage. A piece of Kapton tape covering the bottom of a sample puck works well.

4. Mount a metal-coated NanoProbe cantilever into the cantilever holder.

   Note: MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz, models MESP and SCM-PIT) usually work well. It is also possible to deposit custom coatings on model FESP silicon TappingMode cantilevers. Ensure that any deposited metal you use adheres strongly to the silicon cantilever.

5. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 17.2d.

Figure 17.2d   The ELECTRICAL & MAGNETIC AFM **Select Experiment** window
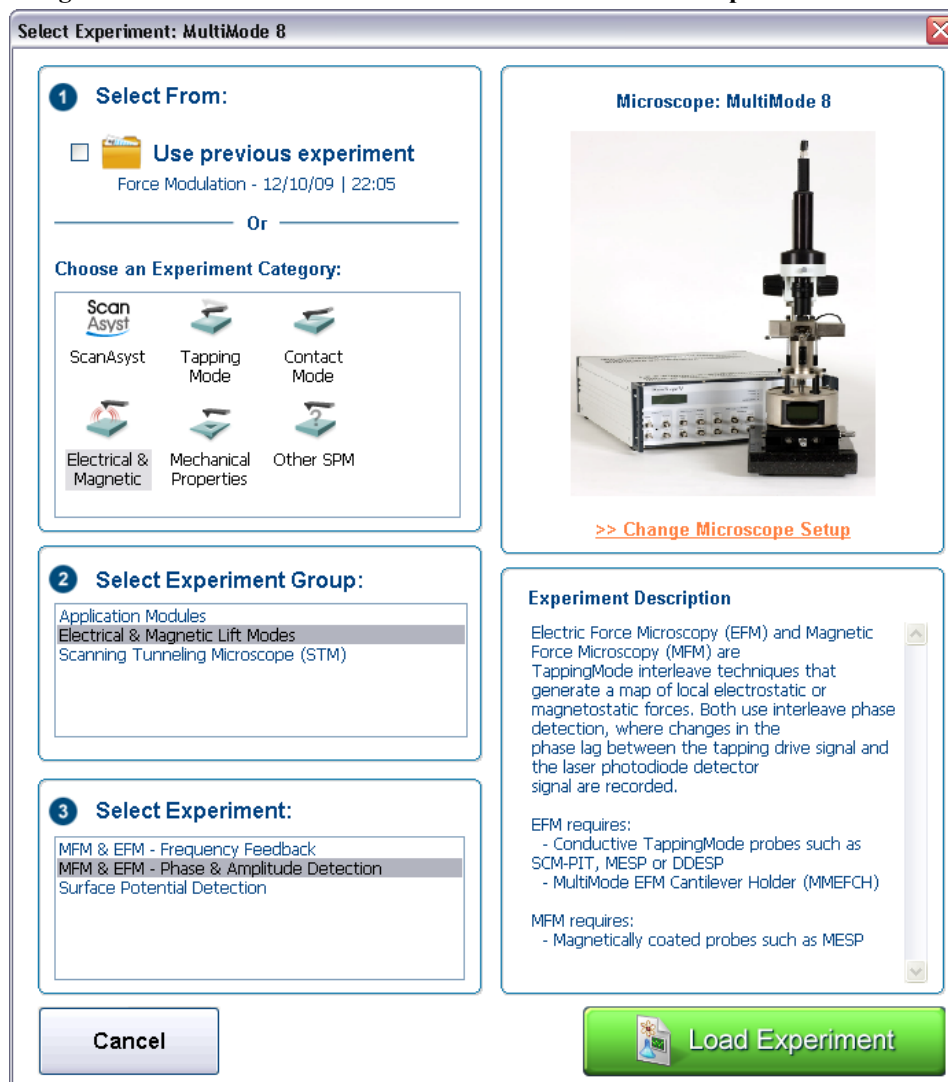
6. Select ELECTRICAL & MAGNETIC in the **Choose an Experiment Catagory** panel.

7. Select ELECTRICAL & MAGNETIC LIFT MODES in the **Select Experiment Group** panel.

8. Select **MFM & EFM - PHASE & AMPLITUDE DETECTION** in the **Select Experiment** panel and click LOAD EXPERIMENT.

9. Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window.

10. Follow the procedure below for the type of electric force imaging desired, **Phase Detection**, **Frequency Modulation** or **Amplitude Detection** (see sections 17.2.4 through 17.2.6).

17.2.4 Phase Detection

1. In the **Auto Tune** panel, set **Start frequency** and **End frequency** to appropriate values for your cantilever (e.g., for 225μm MFM cantilevers, set **Start frequency** to 40kHz and **End frequency** to 100 kHz).

2. Select **Auto Tune**.

    Note: Two curves appear on the Cantilever Tune graph (see Figure 17.2e): the *amplitude* curve on top (the blue line in Figure 17.2e), and the *phase* curve below.

Figure 17.2e   Phase Detection Cantilever Tune
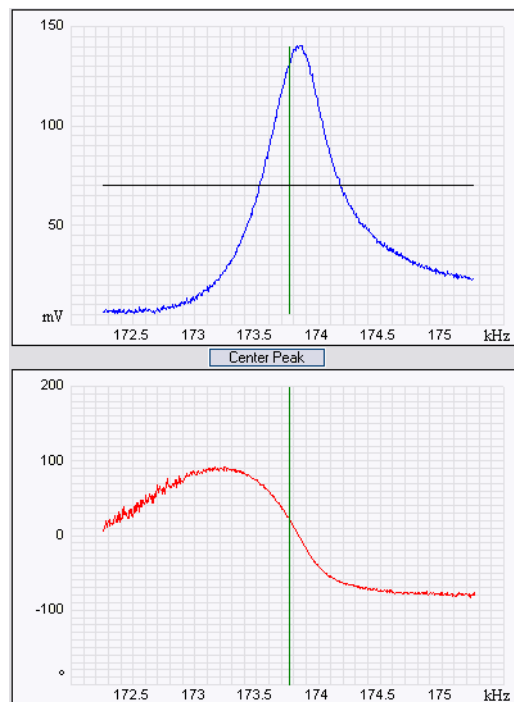
Note: The phase should decrease with increasing frequency and cross the center line (0° point) at the peak frequency. The phase curve then correctly reflects the phase lag between the drive and the cantilever response. Gradients in the electric force cause a shift ΔF_0 in the resonance frequency. Resonance shifts also give rise to phase shifts $\Delta\phi$ used to generate an image of the electric force gradients; see Figure 17.2f.

Figure 17.2f Shift In Phase at Fixed Drive Frequency
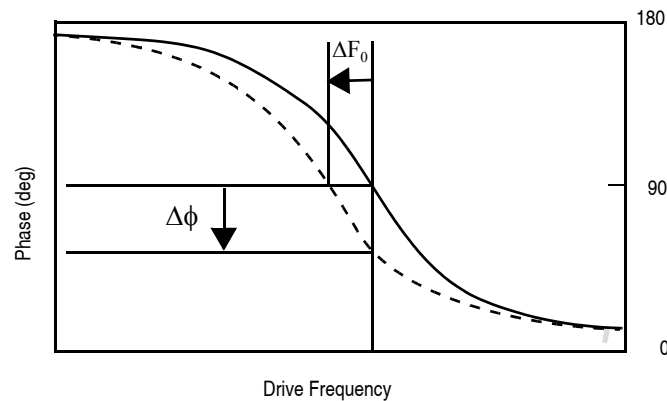
3. **Exit Auto Tune**.

4. Engage the AFM and make the necessary adjustments to obtain a good topography (**Height**) image on Channel 1.

5. In the **Interleave** panel, shown in Figure 17.2g, set the **Interleave Mode** to **Lift**.

6. In the **Interleave** panel, set the **Lift Start Height** to 0nm (default setting), and **Lift Scan Height** to 100 nm. (The lift height can later be optimized).

Figure 17.2g EFM Interleave Scan Parameters (Expanded Mode)

7. Set the remaining Interleave parameters (**Setpoint**, **Drive Amplitude**, **Drive Frequency**, **Gains**) to the main Feedback values.

  Note: This can be done by setting the flags to the left of each parameter to "**Off**" (grayed).

8. Set the **Channel 3 Data Type** to PHASE and choose RETRACE for the scan **Line Direction** on both Channel 1 and 3 images. See Figure 17.2h.

Figure 17.2h   EFM Channel 3 Setup

9. Set the **Channel 3 Scan Line** to INTERLEAVE to display interleave data. This screen should now display the cantilever phase change due to electrical force gradients from the sample in the right image and topography in the left image.

> **Note:**   If a voltage is being applied to the tip or sample, it is recommended to apply it only during the **Interleave line** if feasible. Set TIP BIAS to the desired voltage in the **Interleave** panel. In the **Feedback** panel set TIP BIAS to **0V**.

10. Optimize the **Lift Scan Height**. For high-resolution, make the **Lift Scan Height** as small as possible without crashing the tip into the surface.

17.2.5 Frequency Modulation

For more quantitative results, use frequency modulation (FM). This technique provides a direct measure of change in resonant frequency sensed by the cantilever.

1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 17.2i.

Figure 17.2i The **ELECTRICAL & MAGNETIC AFM Select Experiment** window
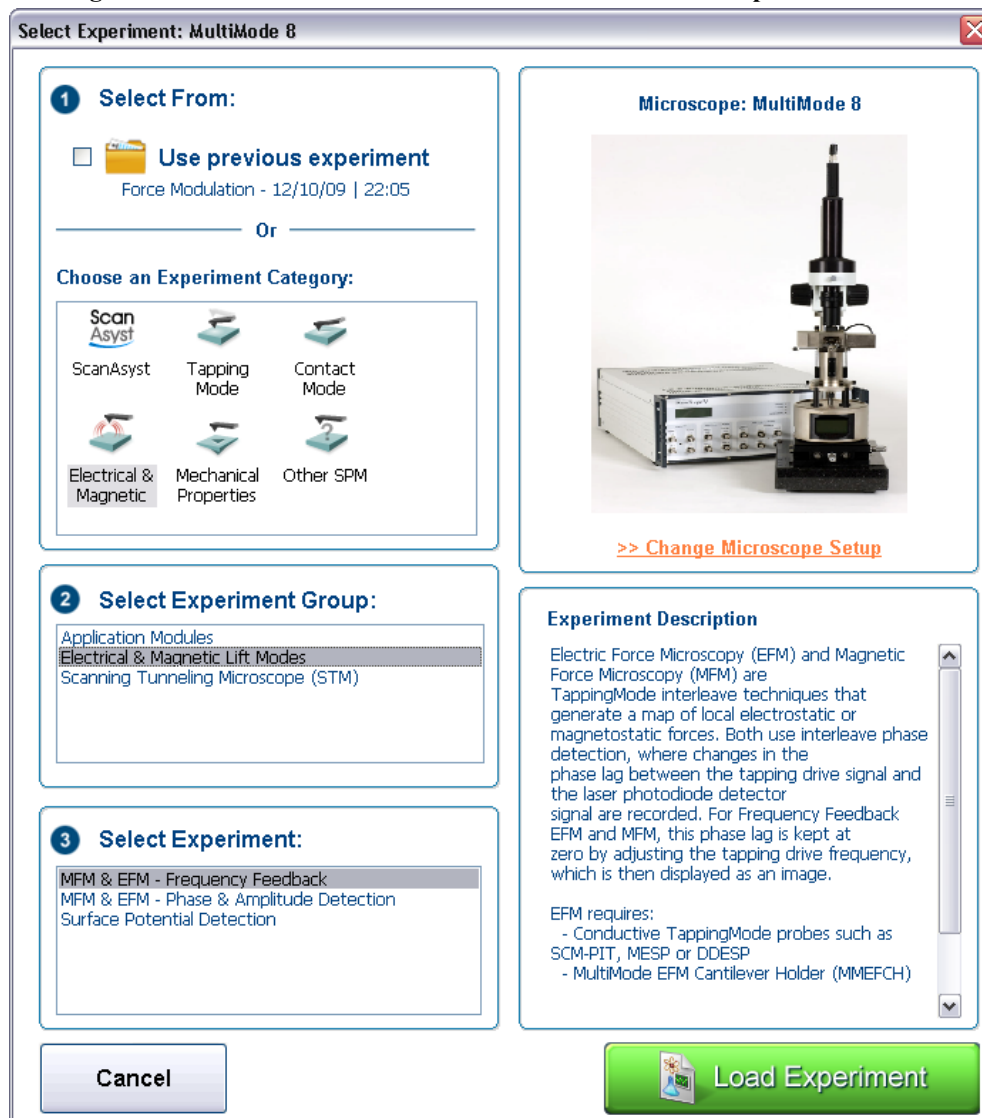
2. Select **ELECTRICAL & MAGNETIC** in the **Choose an Experiment Catagory** panel.

3. Select **ELECTRICAL & MAGNETIC LIFT MODES** in the **Select Experiment Group** panel.

4. Select **MFM & EFM - PHASE & FREQUENCY FEEDBACK** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. In the **Interleave** panel, set the **Input Feedback** to **FREQUENCY** (the default).

6. Switch the Channel 3 image **Data type** to **Frequency**. Try a **Data Scale** (frequency shift) of approximately **10 Hz**.

> **Note:** It may be necessary to optimize the FM gains in the **Interleave** panel to properly track the shifts in resonant frequency (starting values: **Input Igain** = **40** and **Input Pgain** = **60**).

17.2.6 Amplitude Detection

Amplitude detection is inferior to the phase and frequency detection methods described previously and is not the recommended technique.

This section describes the differences in set up for amplitude detection. Changes in the cantilever amplitude provide an *indirect* measure of shifts in the cantilever resonance frequency as shown in Figure 17.2j.

Figure 17.2j Shift In Amplitude at Fixed Drive Frequency
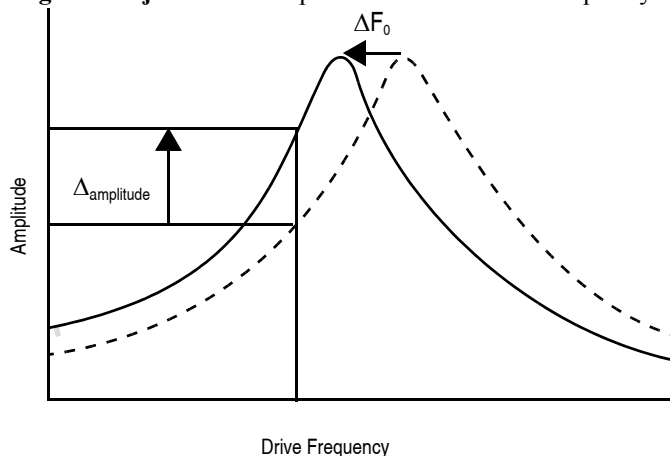
1. Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 17.2k.

Figure 17.2k   Amplitude Detection Cantilever Tune
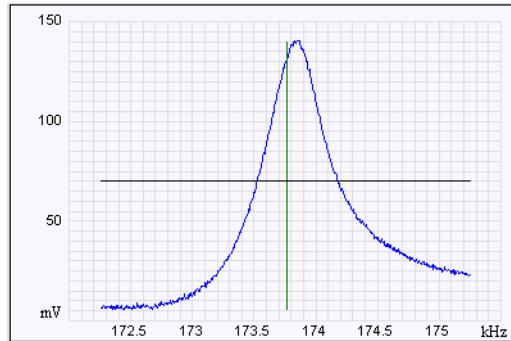
2. For maximum sensitivity, set the **Drive Frequency** to the steepest part of the resonance curve.

> **Note:** As the tip oscillates above the sample, a gradient in the electric force shifts the resonance frequency F_0 (see Figure 17.2j). Tracking the variations in oscillation amplitude while in LiftMode yields an image of the electric force gradients. You may use either side of the resonance, though we have obtained slightly better results on the low side, as shown in Figure 17.2j.

17.3   EFM Troubleshooting/Pointers

17.3.1   Use Low Setpoint When Tapping in Electric Field

If a voltage is applied during the main TappingMode line (as well as the interleave line) ensure proper operation since it is possible to enter a mode in which the tip is never actually touching the surface, even though the amplitude setpoint is being met. As the feedback moves the tip closer to the sample surface, the resonant frequency of the tip is shifting (see Figure 17.2j). The closer the tip is to the surface, the larger the force gradient and the more the resonant frequency shifts. It is possible for the resonant frequency to shift enough so that the amplitude setpoint is met without the tip touching the surface. The tip can be a significant distance away from the sample. The height image has comparatively poor resolution as does the corresponding electric field gradient image. A significantly lower amplitude setpoint is needed to really tap the surface in the presence of a strong electric field. The probability of this problem can be reduced by applying the **TIP** or **SAMPLE BIAS** only during the interleave scan line.

17.3.2   Verify Electric Field at Surface

If applying a **TIP** or **SAMPLE BIAS** to generate an E-field, set the phase channel **Realtime Plane Fit** to **NONE**. While viewing the scope trace, vary the **Interleave TIP** or **SAMPLE BIAS** value and verify that **PHASE** signal shifts accordingly.

17.3.3   Fine Tune Lift Scan Height

Set the lift height to be as small as possible without hitting the surface, because lateral resolution of EFM improves with decreased tip/sample separation. The minimum lift height depends on the roughness of the sample, the difference between the amplitude setpoint and free air amplitude, and the quality of the height image. Hitting the surface usually produces phase data with extremely high contrast (either black or white pixels).

17.3.4   Fine Tune Interleave Drive Amplitude

Decrease the drive amplitude in the interleave line to further minimize the lift height. Be aware that the S/N ratio also decreases with decreasing drive, so there are diminishing returns after a point.

17.3.5 Optimize Tune in Vicinity of Surface

Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window while engaged. A tip offset of 30nm usually works well for smooth surfaces (if the tune has a noisy straight area in the middle instead of a smooth peak, the tip is hitting the surface and a larger tip offset is needed typically for rough surfaces). Adjust the **Drive Frequency** to center of peak or just to the left. If phase data is to be collected during the main line, zero the phase at the drive frequency.

17.3.6 Optimize Tune in Interleave

Find optimal lift height. Choose a **Tip Offset** equal to the **Lift Height**. Make any changes to the **Drive Frequency** and **Drive Phase** to adjust the main tune (tip #3). Activate (turn green by clicking) the **Drive Frequency** and **Phase** in the **Interleave** panel.

17.3.7 If Voltage is Needed, Use NanoScope When Possible

It is often very difficult to get a high quality TappingMode image with an electric field present. The NanoScope Controller has an important advantage over an external power supply – it can be used to create an E field only in interleave while set to 0V in the main line. Its range is limited to +/- 12V.

17.3.8 Try Uncoated Si Tip

EFM has been successful with a variety of tips, including standard Si tips with no metal coating (fesp, tesp, ltesp). The Si is highly n-doped and is often conductive enough for EFM measurements. Metal coatings make the tip less sharp, decreasing lateral resolution. The standard tips also seem to give cleaner images over longer periods of time. There is likely a reduction in sensitivity to small E fields with the decreased conductivity.

17.3.9 Minimize Optical Interference

Optical interference may sometimes appear in the lift (electric force) image when imaging highly reflective samples. Optical interference appears as evenly spaced, sometimes wavy lines with about 1–2µm spacing superimposed on the lift image. This occurs when ambient laser light (i.e., light passing around or through the cantilever, then reflecting off the sample) interferes with laser light reflecting from the cantilever. Interference can be alleviated by moving the beam spot up a little along the cantilever away from the tip; about one-third of the cantilever length from the tip usually works well. On the MultiMode head, the adjustment can be refined by carefully moving the beam spot laterally on the cantilever while scanning until interference fringes are minimized.

17.4 Surface Potential Detection

17.4.1 Surface Potential Detection Overview

Surface potential detection measures the effective surface voltage of the sample by adjusting the voltage on the tip to match that of the surface, thereby minimizing the electric force from the sample. Samples for surface potential measurements must have a surface voltage in the range [-10,+10 volts], and operation is easiest for voltages in the range of [-5,+5V]. The noise level with this technique is typically 10mV. Samples may include both conducting and nonconducting regions. Samples with regions of different metals will also show contrast due to contact potential differences. Quantitative voltage measurements are made of the relative voltages within a single image.

17.4.2 Surface Potential Detection—Theory

Theory Overview

Surface potential detection is a two-pass procedure where the surface topography is obtained by standard TappingMode in the first pass and the surface potential is measured on the second pass (see Figure 17.4a). The two measurements are interleaved: that is, they are each measured one line at a time with both images displayed on the screen simultaneously. A block diagram of the Surface Potential measurement system is shown in Figure 17.4b. On the first pass, in TappingMode, the cantilever is mechanically vibrated near its resonant frequency by a small piezoelectric element. On the second pass, the tapping drive piezo is turned off and an oscillating voltage $V_{AC} \sin \omega t$ is applied directly to the probe tip. If there is a DC voltage difference between the tip and sample, then there will be an oscillating electric force on the cantilever at the frequency ω. This causes the cantilever to vibrate, and an amplitude can be detected.

Figure 17.4a LiftMode Principles Used in Surface Potential Detection

Surface Potential Scope Data
(Interleave scan)

Topographic Scope Data
(Main scan)

Electric Field Sources
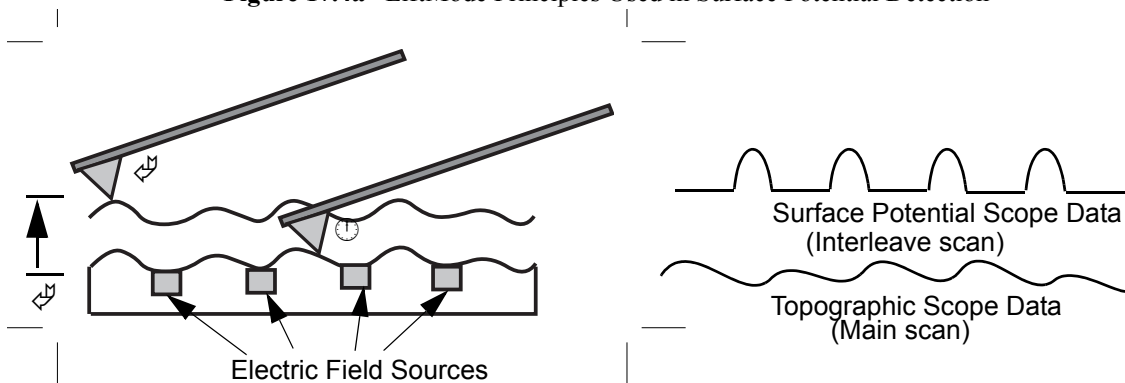
1. Cantilever measures surface topography on the first (main) scan.
2. Cantilever ascends to lift scan height.
3. Cantilever follows stored surface topography at the lift height above sample while responding to electric field influences on the second (interleave) scan.

If the tip and sample are at the same DC voltage, there is no force on the cantilever at ω and the cantilever amplitude will go to zero. Local surface potential is determined by adjusting the DC voltage on the tip, V_{tip}, until the oscillation amplitude becomes zero and the tip voltage is the same as the surface potential. The voltage applied to the probe tip is recorded by the NanoScope Controller to construct a voltage map of the surface.

Figure 17.4b Simplified Block Diagram of Surface Potential Detection
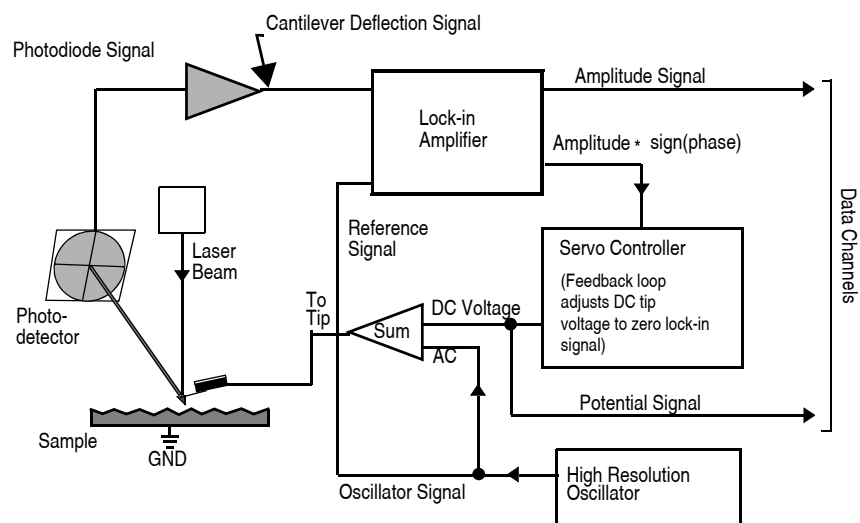
Theory Details

Surface potential microscopy can be modeled as a parallel plate capacitor. When two materials with different work functions are brought together, electrons in the material with the lower work function flow to the material with the higher work function. See Figure 17.4c. If these materials are charged, the system can be thought of as a parallel plate capacitor with equal and opposite surface charges on each side. The voltage developed over this capacitor is called the contact potential. Measuring the contact potential is done by applying an external backing potential to the capacitor until the surface charges disappear. At that point the backing potential will equal the contact potential. In surface potential microscopy (scanning Kelvin probe force microscopy), this "zero-charge" point is determined by adjusting the tip voltage so the electrical force felt by the AFM cantilever is "0".

Figure 17.4c   The electric energy level diagram for two conducting specimens, where ϕ_1 and ϕ_2 are the respective work functions. E_{F1} and E_{F2} are the respective Fermi energies.
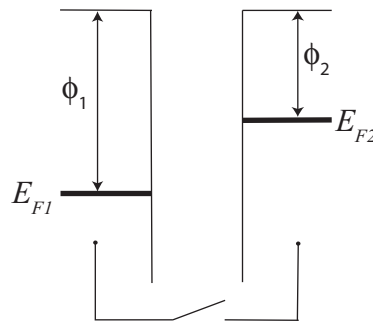
If an external electrical contact is made between the two electrodes, their Fermi levels equalize and the resulting flow of charge (in the direction indicated in Figure 17.4d) produces a potential gradient, termed the contact potential V_c, between the plates. The two surfaces become equally and oppositely charged.

Figure 17.4d   Electrical contact between the two specimens
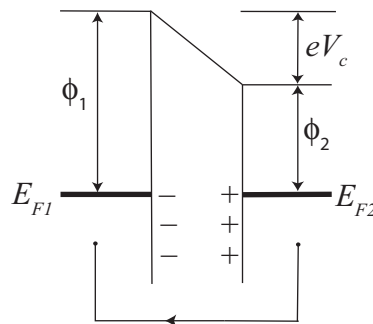
Inclusion of a variable backing potential, V_b, in the external circuit permits biasing of one electrode with respect to the other. When $V_b = V_c = (\phi_1 - \phi_2)/e$ — the average — the electric field between the plates vanishes. See Figure 17.4e.

Figure 17.4e Variable backing potential, V_b, added
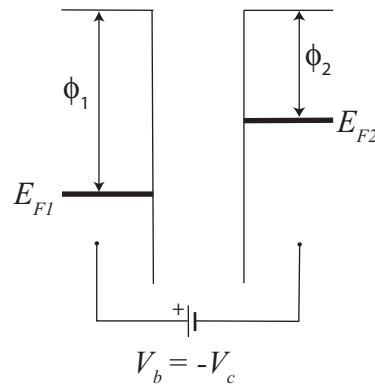
A good way to understand the response of the cantilever during Surface Potential operation is to start with the energy in a parallel plate capacitor, $U = \frac{1}{2}C(\Delta V)^2$, where C is the local capacitance between the AFM tip and the sample and ΔV is the voltage difference between the two. The force on the tip and sample is the rate of change of the energy with separation distance:

$$F = -\frac{dU}{dZ} = -\frac{1}{2}\frac{dC}{dZ}(\Delta V)^2$$

Figure 17.4f Force as a Function of Voltage
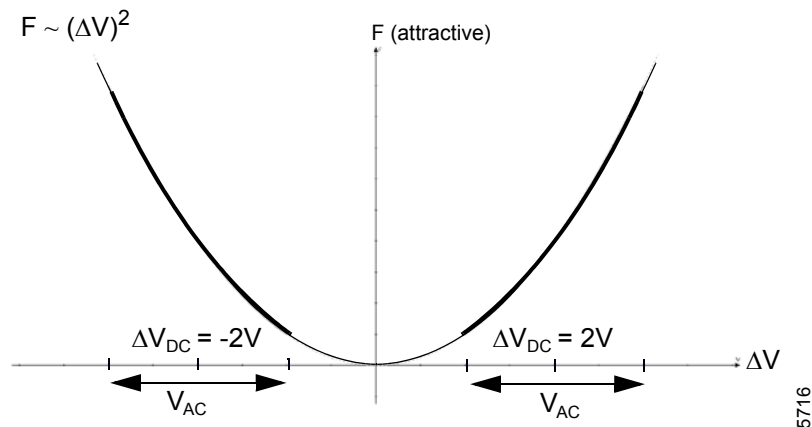
The voltage difference, ΔV, in Surface Potential operation consists of both a DC and an AC component. The AC component is applied from the oscillator, $V_{AC}\sin\omega t$, where ω is the resonant frequency of the cantilever.

$$\Delta V = \Delta V_{DC} + V_{AC} \sin\omega t$$

ΔV_{DC} includes applied DC voltages (from the feedback loop), work function differences, surface charge effects, etc. Squaring ΔV and using the relation $2\sin^2 x = 1 - \cos(2x)$ produces:

$$F = -\frac{1}{2}\frac{dC}{dZ}(\Delta V_{DC}^2 + \frac{1}{2}V_{AC}^2) - \frac{dC}{dZ}\Delta V_{DC} V_{AC} \sin \omega t + \frac{1}{4}\frac{dC}{dZ}V_{AC}^2 \cos(2\omega t)$$

$$\underbrace{\phantom{-\frac{1}{2}\frac{dC}{dZ}(\Delta V_{DC}^2 + \frac{1}{2}V_{AC}^2)}}_{\text{DC term}} \quad \underbrace{\phantom{\frac{dC}{dZ}\Delta V_{DC} V_{AC} \sin \omega t}}_{\omega \text{ term}} \quad \underbrace{\phantom{\frac{1}{4}\frac{dC}{dZ}V_{AC}^2 \cos(2\omega t)}}_{2\omega \text{ term}}$$

The oscillating electric force at ω acts as a sinusoidal driving force that can excite motion in the cantilever. The cantilever responds only to forces at or very near its resonance, so the DC and 2ω terms do not cause any significant oscillation of the cantilever. In regular TappingMode, the cantilever response (RMS amplitude) is directly proportional to the drive amplitude of the tapping piezo. Here the response is directly proportional to the amplitude of the F_ω drive term:

$$\textit{amplitude of } F_\omega = \frac{dC}{dZ}\Delta V_{DC}V_{AC}$$

The goal of the Surface Potential feedback loop is to adjust the voltage on the tip until it equals the voltage of the sample ($\Delta V_{DC}=0$), at which point the cantilever amplitude should be zero ($F_\omega=0$).

The larger the DC voltage difference between the tip and sample, the larger the driving force and resulting amplitude will be. But the F_ω amplitude alone is not enough information to adjust the voltage on the tip. The driving force generated from a 2V difference between the tip and sample is the same as from a -2V difference (see Figure 17.4f).

What differentiates these states is the phase. The phase relationship between the AC voltage and the force it generates is different for positive and negative DC voltages (see Figure 17.4g through Figure 17.4j).

In the case where $\Delta V_{DC} = 2V$, the force is in phase with V_{AC}. When $\Delta V_{DC} = -2V$, the force is out of phase with V_{AC}. Thus, the cantilever oscillation will have a different phase, relative to the reference signal V_{AC}, depending on whether the tip voltage is larger or smaller than the sample voltage. Both the cantilever amplitude and phase are needed for the feedback loop to correctly adjust the tip voltage. The input signal to the Surface Potential feedback loop is the cantilever amplitude multiplied by the sign of its phase (i.e., amplitude for phase ≥ 0 degrees, -amplitude for phase <0 degrees). This signal can be accessed in the software by selecting **POTENTIAL INPUT** (interleave scan line) in one of the channel panels.

If $\Delta V_{DC} = 0$, the electric drive force is at the frequency 2ω. The component of the force at ω is zero so the cantilever does not oscillate (see Figure 17.4k and Figure 17.4l). The Surface Potential feedback loop adjusts the applied DC potential on the tip, V_{tip}, until the cantilever's response is zero. V_{tip} is the Potential data that is used to generate a voltage map of the surface.

Figure 17.4g V_{AC} at ω, $\Delta V_{DC} = 2V$
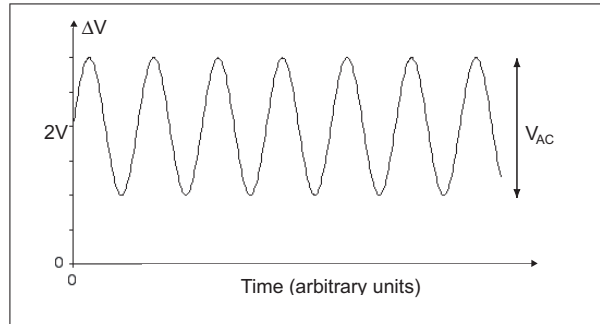
Figure 17.4h Major Force Component in Phase with V_{AC} at Frequency ω, $\Delta V_{DC} = 2V$
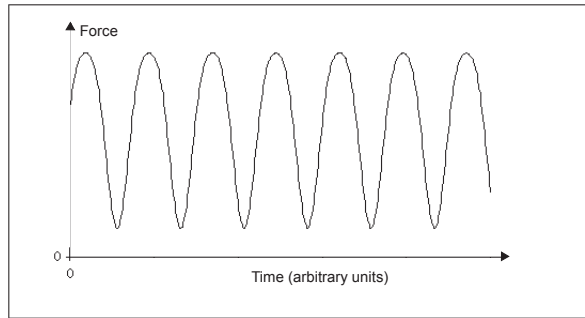
Figure 17.4i V_{AC} at ω, $\Delta V_{DC} = -2V$
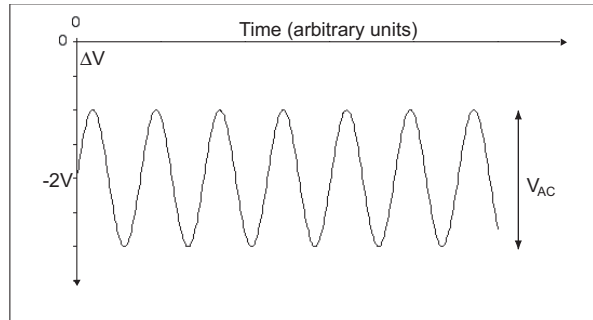
Figure 17.4j Major Force Component 180° Out of Phase with V_{AC} at Frequency ω, $\Delta V_{DC} = -2V$
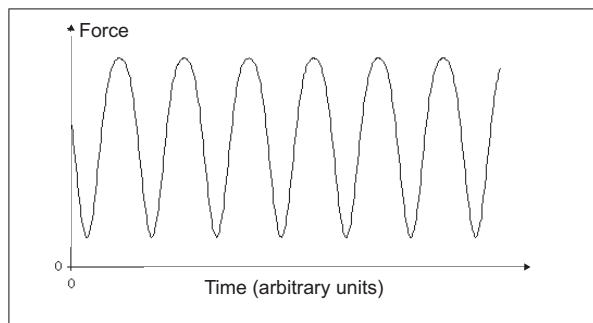
Figure 17.4k V_{AC} at ω, $\Delta V_{DC}= 0V$
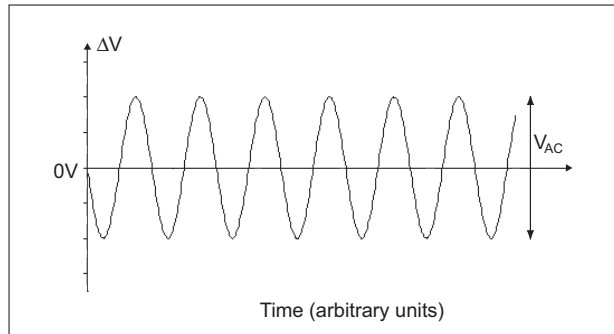
Figure 17.4l   Force at frequency of 2ω, $\Delta V_{DC} = 0V$
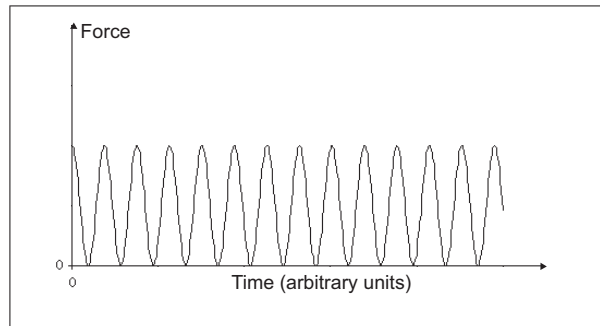
17.5   Surface Potential Detection—Preparation

The sample should be electrically connected directly to the chuck, so that it can be held at ground potential (normal operation) or biased through the chuck, or a standard sample puck using conductive epoxy or silver paint as shown below:

Conductive Epoxy
or Paint

Sample

Sample Chuck
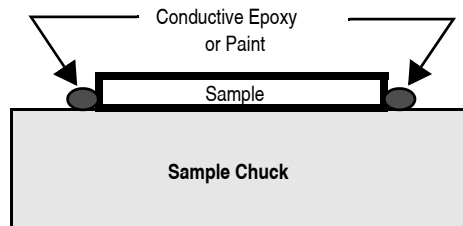
17.6   Surface Potential Imaging—Procedure

1. Mount a sample onto the sample holder.

2. Mount a metal-coated cantilever into the standard probe holder. MFM-style cantilevers (MESP, 225µm long, with resonant frequencies around 70kHz) usually work well. It is also possible to deposit custom coatings on model FESP silicon TappingMode cantilevers. Verify that all deposited metal adheres strongly to the silicon cantilever. SCM-PIT and OSCM-PT also work well for surface potential. Uncoated highly doped Si probes such as FESP and LTESP, which provide higher resolution, can also be used.

3. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 17.6a.

Figure 17.6a   The **ELECTRICAL & MAGNETIC AFM Select Experiment** window
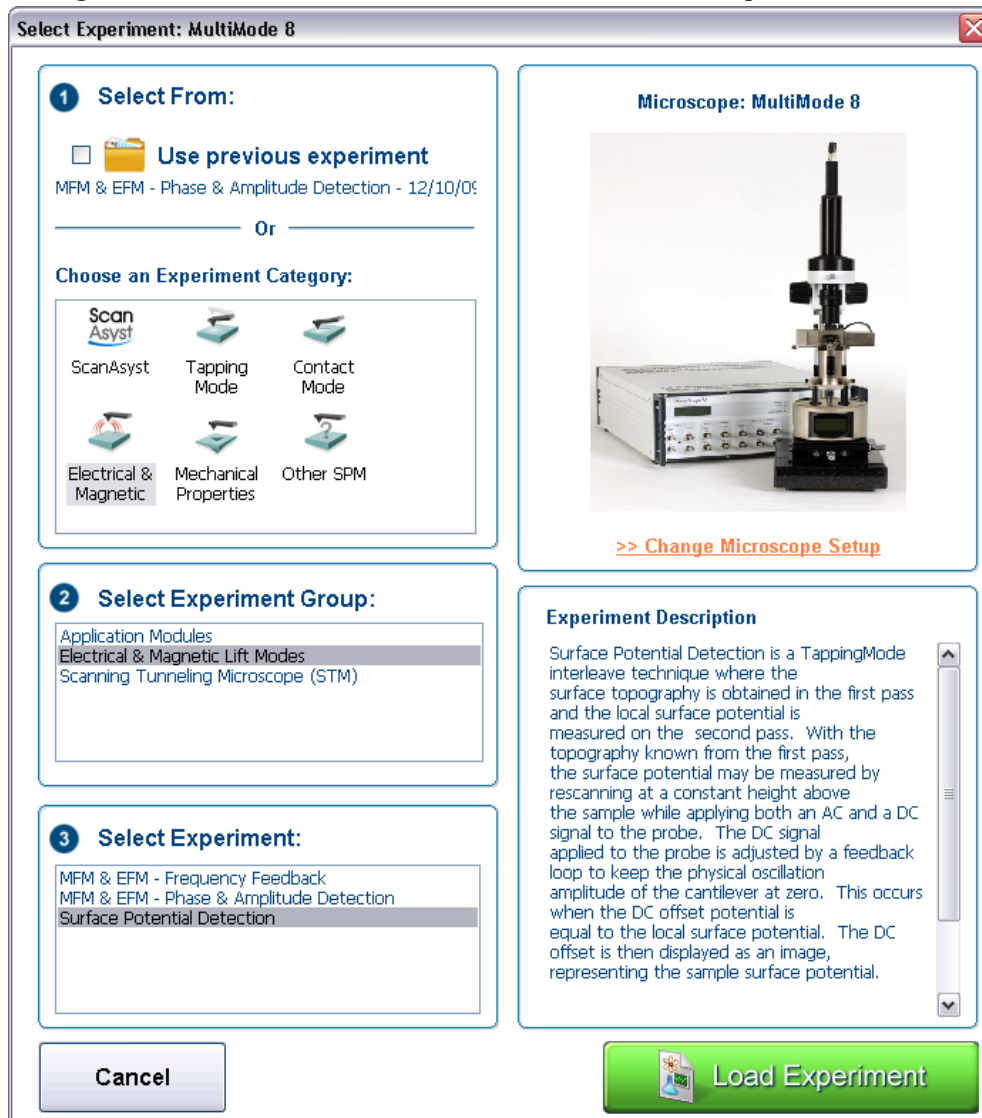
4. Select **ELECTRICAL & MAGNETIC** in the **Choose an Experiment Catagory** panel.

5. Select **ELECTRICAL & MAGNETIC LIFT MODES** in the **Select Experiment Group** panel.

6. Select **SURFACE POTENTIAL DETECTION** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

7. In the **Interleave** panel, set the **Input Feedback** to **POTENTIAL** (the default).

8. Switch the Channel 3 image **Data type** to **POTENTIAL**.

9. Use **Cantilever Tune**: **AutoTune** to locate the cantilever's resonant peak.

In this case, two curves should appear in the **Cantilever Tune** window: the *amplitude* curve and the *phase* curve. In the event you find more than one resonance, select a resonance that is sharp and clearly defined, but not necessarily the largest. It is also helpful to select a resonant peak where the lock-in phase also changes very sharply across the peak. Multiple peaks can often be eliminated by making sure the cantilever holder is clean and the cantilever is tightly secured.

10. Engage the AFM and make the necessary adjustments for a good TappingMode image while displaying height data.

11. Set the **Interleave** gains:

    a. **Input Igain = 1**

    b. **Input Pgain = 5**

12. In the **Interleave** panel, shown in Figure 17.6b, set the **Interleave Mode** to **LIFT**.

13. In the **Interleave** panel, set the **Lift Start Height** to 0nm, and **Lift Scan Height** to 100 nm. (The lift height can later be optimized).

14. Leave the **Interleave Drive Frequency** at the main **Feedback** value (gray).

15. Set the **Input Feedback** to **POTENTIAL**.

Figure 17.6b Surface Potential Interleave Scan Parameters (Expanded Mode)

16. Enter an Interleave **Drive Amplitude**. This is the ac voltage that is applied to the AFM tip. Higher **Drive Amplitude** produces a larger electrostatic force on the cantilever and this makes for more sensitive potential measurements. Conversely, the maximum total voltage (ac + dc) that may be applied to the tip is ±10V. So a large **Drive Amplitude** reduces the range of the DC voltage that can be applied to the cantilever. If the sample surface potentials to be measured are very large, it is necessary to choose a small **Drive Amplitude**, while small surface potentials can be imaged more successfully with large **Drive Amplitudes**. To start choose a **Drive Amplitude** of **500mV**.

17. Set the Channel 3 image **Data Type** to **POTENTIAL**. Set the scan **Line Direction** for the main and interleave scans to **RETRACE**. Remember to choose the **RETRACE** direction because the lift step occurs on the trace scan and can cause artifacts in the data. Set the **Channel 4 Scan Line** to **INTERLEAVE**.

18. Adjust the Input gains. As with the topography gains, the scan can be optimized by increasing the gains to maximize feedback response, but not so high that oscillation sets in. More information on tuning the feedback loop is given in Section 17.6.1, below.

19. Optimize the lift heights. Set the **Lift Scan Height** at the smallest value possible that does not make the Potential feedback loop unstable or cause the tip to crash into the sample surface. When the tip crashes into the surface during the Potential measurement, dark or light streaks appear in the Potential image. In this case, increase the **Lift Scan Height** until these streaks are minimized.

20. Optimize the drive phase. **Lock-In Phase** adjusts the phase of the reference signal to the lock-in amplifier. As discussed in **Surface Potential Detection—Theory:** Section 17.4.2, the correct phase relationship must exist between the reference and the input signals to the lock-in for the potential feedback loop to perform correctly. **Lock-In Phase** depends on the mechanical properties of the cantilever. For cantilevers with resonant frequencies from 60-80kHz (such as MESP, SCM-PIT, and FESP), use an interleave **Lock-In Phase** of 170 degrees. For cantilevers with higher resonant frequencies, increased electronics phase lag must be compensated. For cantilevers with resonant frequencies around 300kHz (such as TESP, RTESP) an interleave **Lock-In Phase** near 130 degrees often works well. Follow the procedure detailed in **Determination of Lock-in Phase:** Section 17.7 to more accurately determine the **Lock-In Phase**.

21. For large sample voltages or qualitative work, select a **Data Type** = **PHASE** in addition to **POTENTIAL**. When the controller has been configured for surface potential measurements, the "phase" signal is actually the cantilever amplitude signal, as measured by a lock-in amplifier. If the feedback loop is not enabled by selecting the **Data Type** = **POTENTIAL**, the lock-in cantilever amplitude depends on the voltage difference between the tip and sample in a roughly linear fashion. (The lock-in amplifier produces a voltage that is proportional to the cantilever amplitude.) Qualitative surface potential images can be collected using this lock-in signal. Also, if the sample has a surface potential that exceeds ±10V (greater than the range of the "Potential" signal), it is possible to use the lock-in signal to provide qualitative images that reflect the sample surface potential. To view the lock-in signal with the reconfigured controller, select the **Data type** = **PHASE**.

17.6.1 Troubleshooting the Surface Potential Feedback Loop

The surface potential signal feedback loop can be unstable. This instability can cause the potential signal to oscillate or become stuck at either +10V or -10V. Here are some tips to see if the feedback loop is working properly with no oscillation:

- Look at the scope display for the potential signal. If oscillation noise is evident in the signal, reduce the gains. If oscillations persist even at very low gains, try increasing the **Lift Scan Height** and/or reducing the **Drive Amplitude** until oscillation stops. If the tip crashes into the surface the Lock-in signal becomes unstable and can cause the feedback loop to malfunction. Increasing the **Lift Height** and reducing the **Drive Amplitude** can prevent this problem. Once oscillation stops, the gains may be increased for improved performance.

If the Potential signal is perfectly flat in the **Scope** panel and shows no noise even with a small Z-range, the feedback loop is probably stuck at ±10V. (You can verify this by changing the value of **Realtime Plane Fit** to NONE in the **Channel 1** panel.) Reduce the **Scan Rate** and watch the scope trace which indicates the cantilever amplitude. On the topographic trace, the voltage displayed should be the setpoint selected for the Main scan. On the Potential trace, this voltage drops close to zero if the cantilever oscillation is being successfully reduced. If the value instead goes to a large nonzero value, the feedback loop is probably not working properly. In this case, try reducing the **Drive Amplitude** and increasing the **Lift Scan Height** or optimizing the Lock-In Phase (see Section 17.7). It may also be helpful to momentarily turn the **Interleave Mode** to DISABLED, then back to INTERLEAVE. Also try reducing any external voltage that is being applied to the sample to stabilize the feedback loop, then turn the voltage back up.

17.7 Determination of Lock-in Phase

For surface potential microscopy (also called scanning Kelvin probe force microscopy-SKPFM) measurements, the feedback adjusts the DC voltage applied between the tip and sample to compensate their intrinsic potential difference. The DC voltage map across the surface thus reflects surface potential variations of the sample surface.

When the difference is perfectly compensated, the tapping oscillation amplitude of the metal coated AFM cantilever (excited by an AC bias field) approaches "0", the very reason this technique is called a nulling technique. In doing so, the feedback uses the tapping amplitude and phase to determine whether to increase or decrease the DC voltage. Phase is the one parameter that users may have difficulty selecting. The following sections outline a procedure to address this matter using **Generic Sweep** available in NanoScope software.

While the procedure itself is simple, plots are shown for better understanding of the measurement technique.

Generic Sweeps are taken on **Interleave**. In some cases, it is helpful to switch back and forth between MAIN and INTERLEAVE for the sweep parameters to take effect. Some inconsistency may occur, for instance, potential feedback may not actually be on when **Input Igain** and **Input Pgain** are non-zero.

17.7.1 Interleave Tuning Curve - Phase Lag

Refer to Figure 17.7a. A phase difference, here –90° (in the bottom plot), is usually seen between tip oscillation signal and the AC bias driving signal. This phase lag varies from tip to tip, with operating frequency, and the potential difference between the tip and the spot on the sample.

Figure 17.7a   Interleave Tuning Curve - Phase Lag
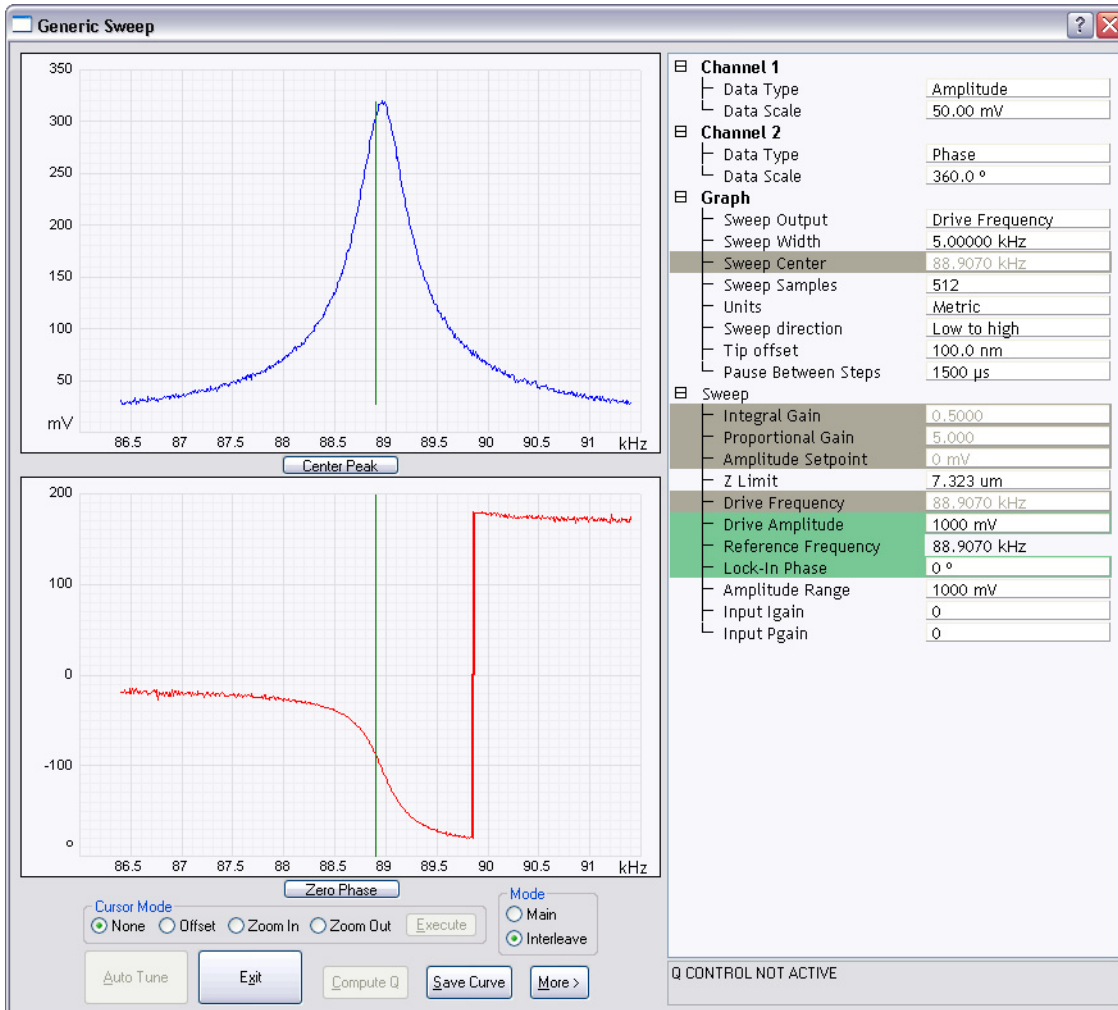
17.7.2 Amplitude and Phase vs. Sample Bias—Feedback Off

Figure 17.7b shows a **Generic Sweep** of SAMPLE BIAS while the potential feedback is turned off. Note that **Input Igain** and **Input Pgain** are 0.

The amplitude, shown in the upper plot, dips to 0 when the sample is biased to match the tip potential; as one moves away from that point, the amplitude increases. Potential feedback seeks that zero point. Because amplitude alone can not determine whether one is on the left side or right side of that zero amplitude point, the feedback relies on the phase to determine which direction to move the DC bias.

Figure 17.7b shows the phase (bottom plot) changing by $180°$ across "0" amplitude. It is desirable to choose a **Lock-In Phase** to offset the oscillation phase, so that the phase is negative when the tip voltage is positive relative to the sample. Figure 17.7b shows that when the sample bias is -2V (tip is positive), the output **Lock-In Phase** is $90°$. The **Interleave Lock-In Phase** then needs to be set to $-180°$ to make it $-90°$.

Figure 17.7b   Amplitude and Phase vs. Sample Bias. Feedback Off
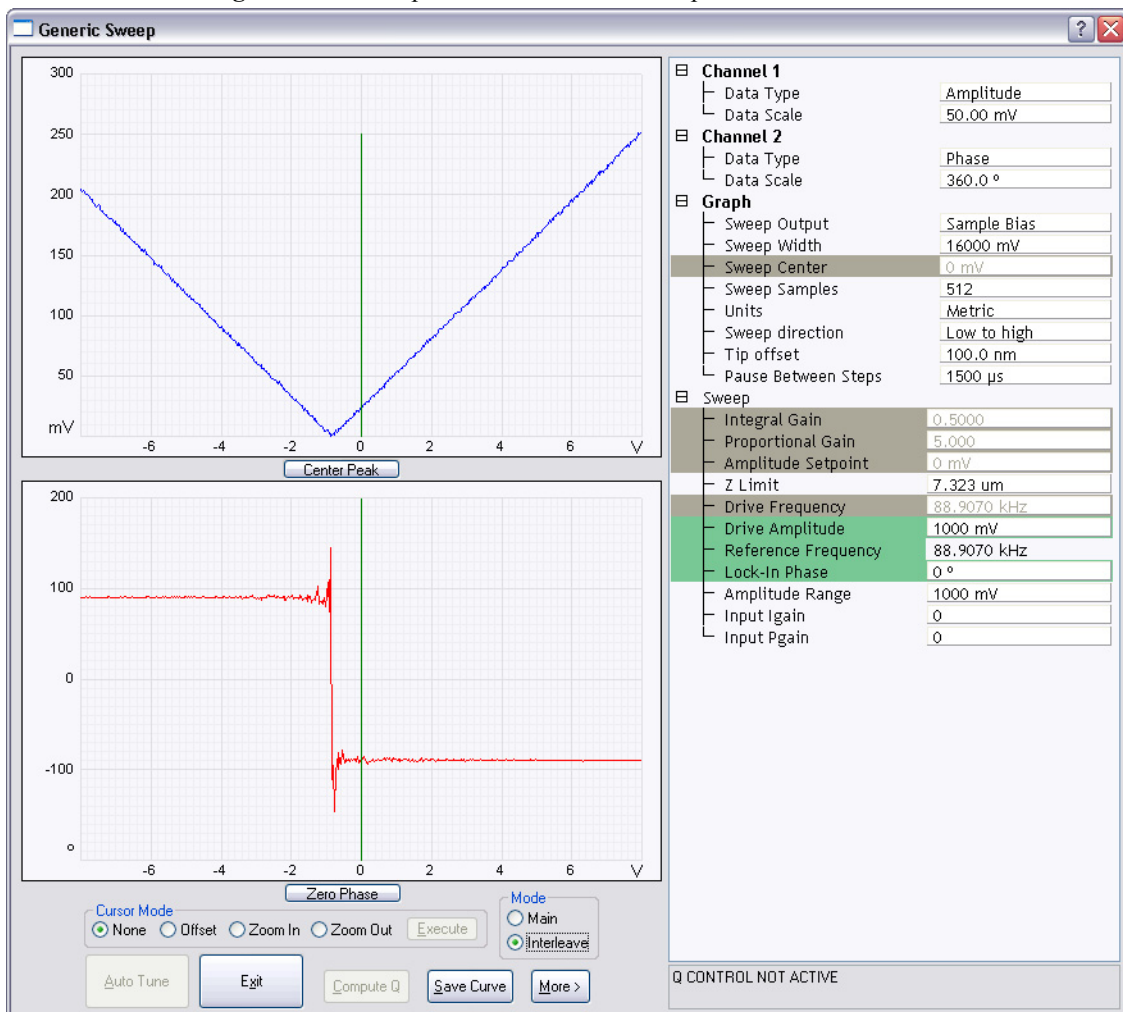
17.7.3 Phase vs. Lock-In Phase, Feedback On—The Straightforward Approach

Refer to Figure 17.7c. It is logical to sweep the lock-in phase in a full circle to determine the phase range where feedback is working while the feedback is turned on (**Input Igain** and **Input Pgain** are non-zero).

The top plot shows how the amplitude changes while lock-in phase is swept. In the mid-range of the phase sweep (approximately -100° to $+100^\circ$), the amplitude stays non-zero, indicating that the phase is not correct for the feedback to work properly. On both ends, the amplitude remains around "0", implying that feedback is working properly in "nulling" the amplitude. In principle, it is fine to choose any lock-in phase in these two regions $100^\circ \sim 180^\circ$ and $-180^\circ \sim -90^\circ$ (equivalent to $180^\circ \sim 270^\circ$); actually a combined region of $100^\circ \sim 270^\circ$. It makes practical sense, however, to choose a value somewhere in the middle to have a good margin, e.g. -170°.

The lower plot shows phase changing linearly in the middle of the sweep range, while jumping around when feedback is working on either end. This is explained in **Amplitude and Phase vs. Sample Bias—Feedback Off:** Section 17.7.2.

Figure 17.7c   Phase vs. Lock-In Phase Feedback On—the Straightforward Approach
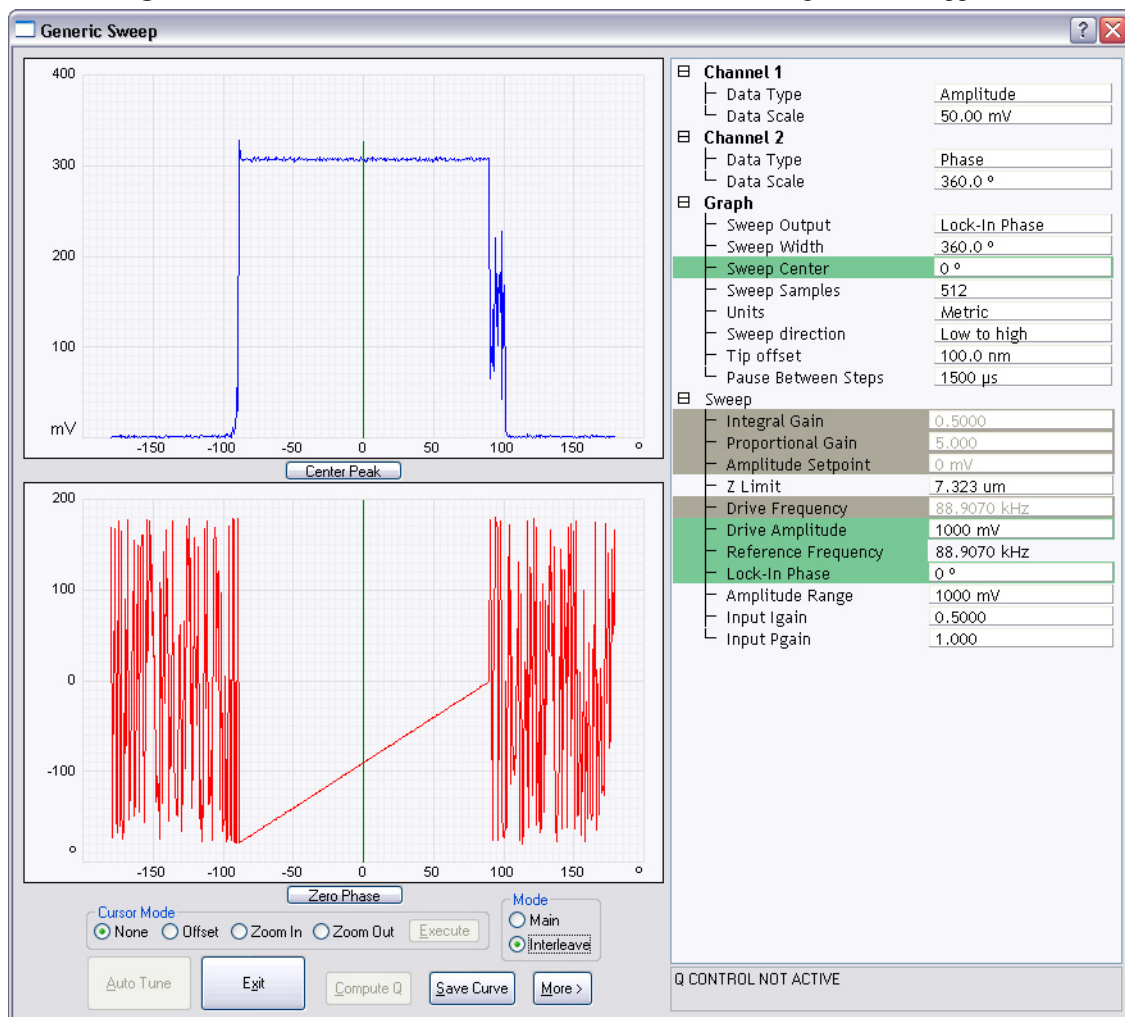
17.7.4 Phase vs. Lock-In Phase Feedback Off—An Alternative Approach

Refer to Figure 17.7d, Generic Sweep of Lock-in phase while potential feedback is **O**ff (Input Igain and Input Pgain are "0").

The top plot shows the amplitude remaining unchanged while the **Lock-In Phase** is swept.

The bottom plot shows the phase changing linearly as the lock-in phase is swept. There is one exception where the phase jumps from $180°$ to $-180°$, so-called phase wrapping.

In case the plots in the previous section were not obtained (due to software inconsistency), do the following. From the left bottom plot, choose a lock-in phase where oscillation phase falls within $-180°\sim0°$ (or $0\sim180°$), e.g. $10°$ ($-170°$), if this does not work, add/subtract $180°$, that is $-170°$ ($10°$).

Of course, one can simply pick any lock-in phase, if it works, fine. Otherwise, change the phase by 180°. The difference is that with the above outlined approach you know what kind of margin you have.

Figure 17.7d   Phase vs. Lock-In Phase, Feedback Off—an Alternative Approach
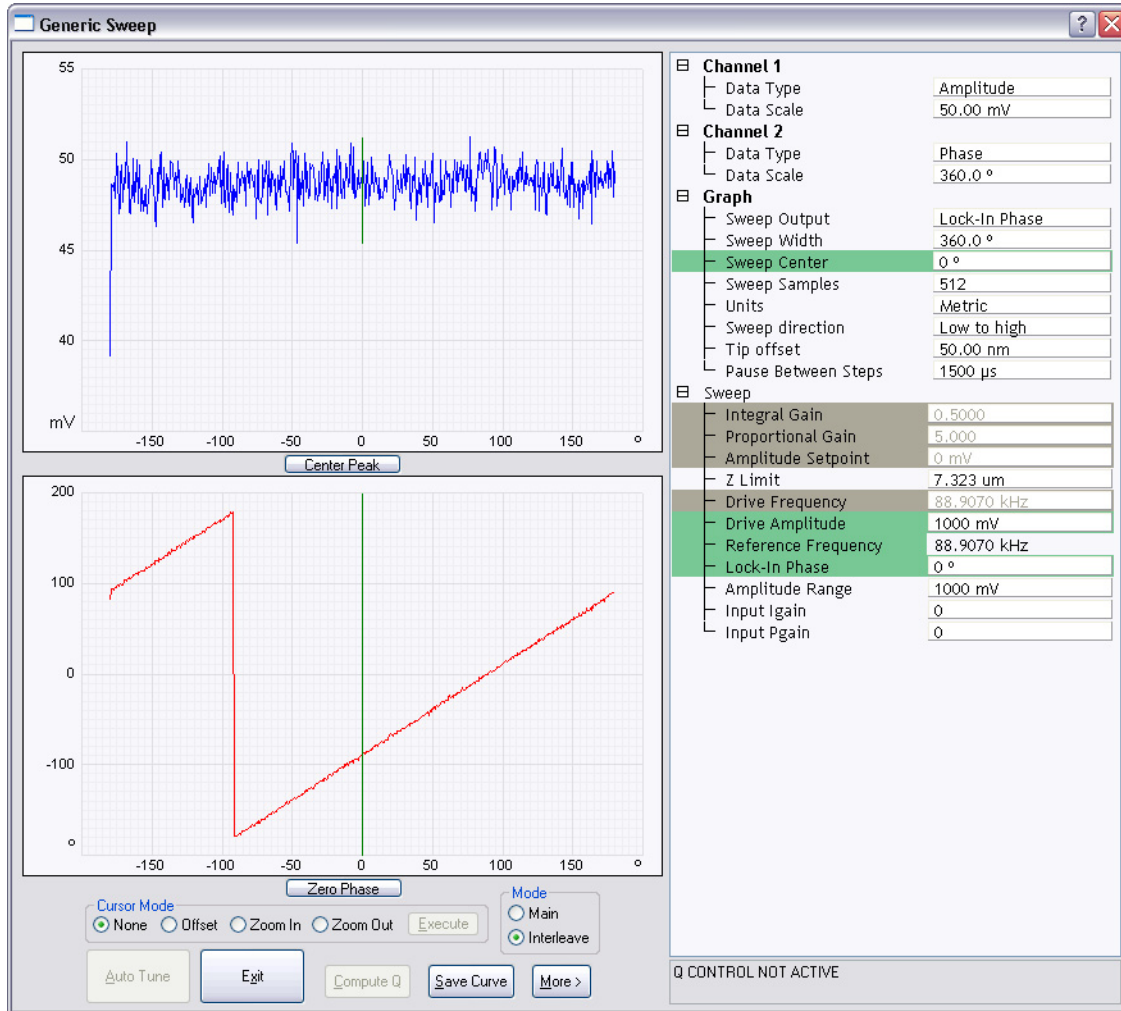
Chapter 18   Calibration

This chapter provides detailed instructions for the fine calibration of MultiMode SPMs. Specifically this chapter includes the following topics:

- **Fine-tuning for X-Y Calibration:** Section 18.7

  - **Prepare System for Fine-tuning:** Section 18.7.1

  - **Measure Horizontally at 440V Scan Size:** Section 18.7.2

  - **Measure Vertically at 440V Scan Size:** Section 18.7.3

  - **Measure Horizontally at 150 V Scan Size:** Section 18.7.4

  - **Measure Vertically at 150V Scan Size:** Section 18.7.5

  - **Change Scan angle and Repeat Calibration Routines:** Section 18.7.6

- **Calibrating Z:** Section 18.8

  - **Engage:** Section 18.8.1

  - **Capture and Correct an Image:** Section 18.8.2

  - **Measure Vertical Features:** Section 18.8.3

  - **Correct Z Sensitivity:** Section 18.8.4

  - **Recheck Z-axis Measuring Accuracy:** Section 18.8.5

  - **Calculate Retracted and Extended Offset Deratings:** Section 18.8.6

18.1 SPM Calibration Overview

Bruker employs a software-guided calibration procedure for all microscopes. The procedural details of how calibration is executed using NanoScope software are beyond the scope of this document and include proprietary methods exclusive to Bruker. A brief overview of the theory is available in Section 18.1.1. The calibration is summarized in four basic steps outlined below.

- **Orthogonality Adjustments**: The orthogonality adjustment compensates for a possible non-square scan pattern.

- **Scanner Linearization**: Adjust parameters for correcting the capacitive nature of the piezo material and force the X-Y motion of the piezo to move at a constant velocity.

- **X, Y Calibration**: Designate the software conversion values using one or both of the following methods.

  - **Capture Calibration and Auto calibration**: A built-in software routine automatically controls the scanner while capturing a series of data files. The data files are then used to "teach" the software the actual distance that the scanner moved. This process creates a customized set of conversion values that will be used to convert the known signals to a metric scale.

    -or-

  - **Fine Tuning**: Manually test and adjust X-Y sensitivity and derating values to within 1% accuracy. The conversion values that scale the scanner motion are confirmed, or are adjusted if necessary to within 1%. These same values should be adjusted by the customer as the scanner ages due to use. A scanner will typically lose 10-20% of its scan range over the first year of typical use. The scanner will then become stable as long as it is not damaged.

- **Calibrate Z**: Calibrate the Z scan tube to within 2%. A separate calibration "look-up" table stores the conversion values for converting the control voltages applied to the Z tube. These values are determined by scanning over a sample with a known depth.

Bruker recommends that you adhere to the Calibration schedule shown in Table 18.1a. After initial installation, perform the **Fine-Tuning X-Y Calibration** (see Section 18.7) per the following time schedule. If you find that the calibration measurements are more than 10% off at any point during Fine-Tuning Calibration, stop and perform the **Full X-Y Calibration Routine** (see Section 18.2). For most applications it is sufficient to perform the Z calibration with the same frequency. For critical height measurements we recommend monthly Z calibration.

<div align="center">**Table 18.1a**   Calibration Schedule</div>

Calibration Routine	**Time Frame**	**Frequency**
Fine-Tuning Calibration	First Year	Every 3 months
(or Full X-Y Calibration if required)	Subsequent Years	Every 6 months
Z Calibration	First Year	Every 3 months
(for General Applications)	Subsequent Years	Every 6 months
Critical Height Measurements	All Years	Monthly

> **Note:**   **Small Scan Size Calibration**: If using scan sizes of 5µm or smaller, Bruker recommends calibrating the scanner for small scan sizes. Contact Bruker for further instructions.

18.1.1 Theory Behind Calibration

Scanners typically consist of a hollow tube made of piezoelectric material such as PZT (lead zirconium titanate). Piezo materials contract and elongate when voltage is applied, according to whether the voltage is negative or positive, and depending upon the orientation of the material's polarized grain structure. Scanners are used to precisely manipulate sample-tip movement in order to scan the sample surface. In Dimension SPMs, the sample is stationary while the scanner moves the tip.

Not all scanners react exactly the same to a voltage. Because of slight variations in the orientation and size of the piezoelectric granular structure (polarity), material thickness, etc., each scanner has a unique "personality." This personality is conveniently measured in terms of *sensitivity*, a ratio of piezo movement-to-piezo voltage. Sensitivity is not a linear relationship, however. Because piezo scanners exhibit more sensitivity (i.e., more movement per volt) at higher voltages than they do at lower voltages, the sensitivity curve is just that—curved. This non-linear relationship is determined for each scanner crystal and follows it for the life of the scanner. As the scanner ages, its sensitivity will decrease somewhat, necessitating periodic recalibration.

The diagram below depicts scanner crystal voltage versus photodiode voltage (see Figure 18.1a). In this instance, detector sensitivity is given as volt per volt.

Figure 18.1a Scanner Crystal Voltage and Photodiode Voltage
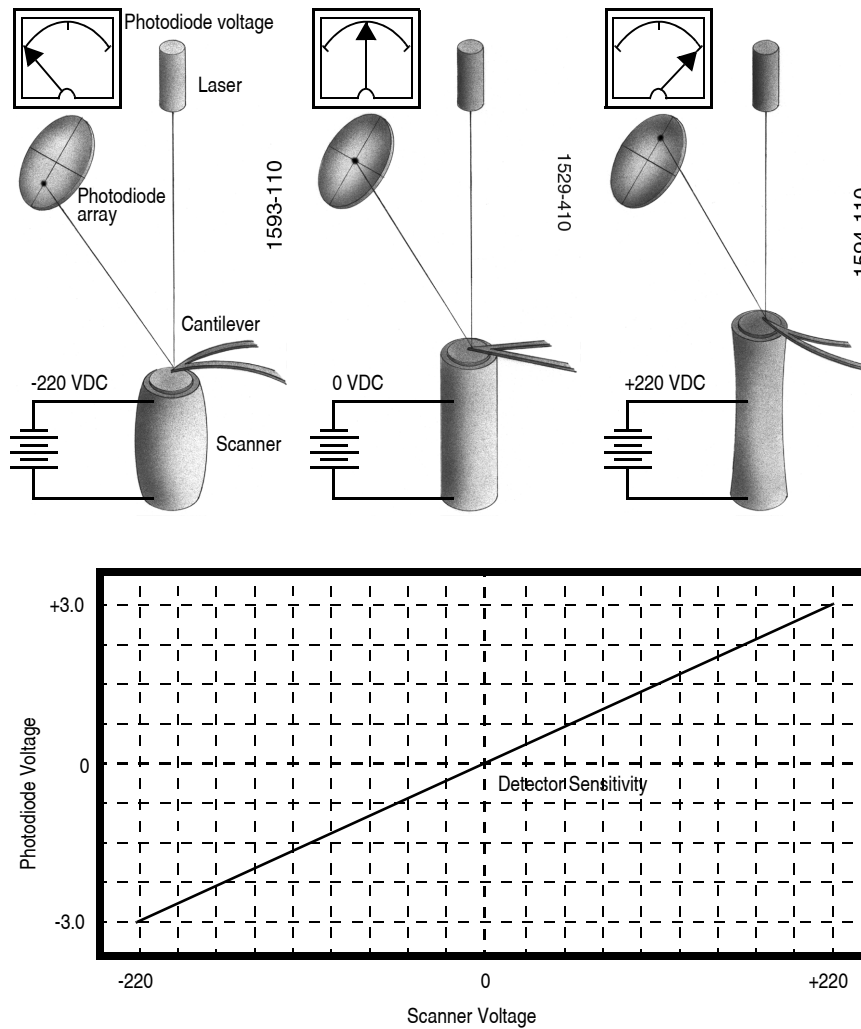
The **Calibrate** > **Scanner > XY...** function displays the **Scanner Calibration** dialog box, allowing users to enter the sensitivity of their scanner's X-Y axes. Sensitivity is measured in terms of lateral displacement for a given voltage (nm/volt). In addition, the NanoScope software employs various derating and coupling parameters to model scanners' nonlinear characteristics. By precisely determining points along the scanner's sensitivity curve, then applying a rigorous mathematical model, full-range measuring capabilities can be achieved with better than 1 percent accuracy. Consider the sensitivity curve represented in Figure 18.1b.
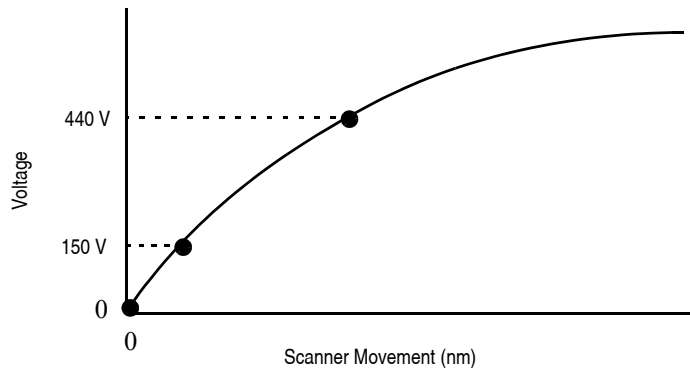
This curve typifies scanner sensitivity across the full range of movement. The vertical axis denotes voltage applied to the scanner. The horizontal axis denotes scanner movement. At higher voltages, the scanner's sensitivity increases (i.e., more movement per voltage applied). At zero volts, the scanner is "motionless." Plotting each point along the curve describes a second-order, exponential relationship which provides a rough approximation of scanner sensitivity.

However, because piezo materials exhibit hysteresis, their response to increasing voltage is not the same as their response to decreasing voltage. That is, piezo materials exhibit "memory," which causes the scanner to behave differently as voltages recede toward zero. Figure 18.1c below represents this relationship.

Figure 18.1c Effect of Nonlinearity and Hysteresis
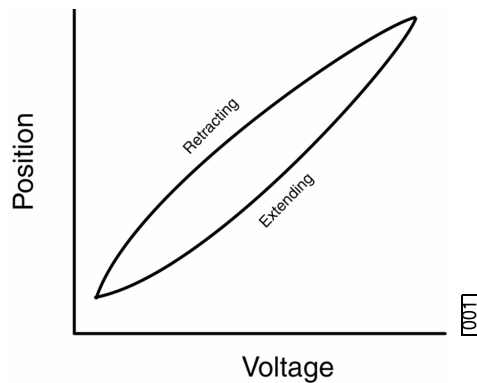
To produce the sharp, linear movements (triangular waveform) required for accurate back-and-forth scanning, it is necessary to shape the applied voltage as shown on the top graph in Figure 18.1d. Moreover, the applied voltage must compensate for scan rate and scan size. As scan rate slows, the applied voltage must compensate for increased memory effects in the piezo material. As scan size is decreased, the piezo exhibits more linearity. These effects are further complicated by X-Y-Z coupling effects (the tendency for one axis to affect movement in other axes).

Figure 18.1d Scanner Voltage and Movement
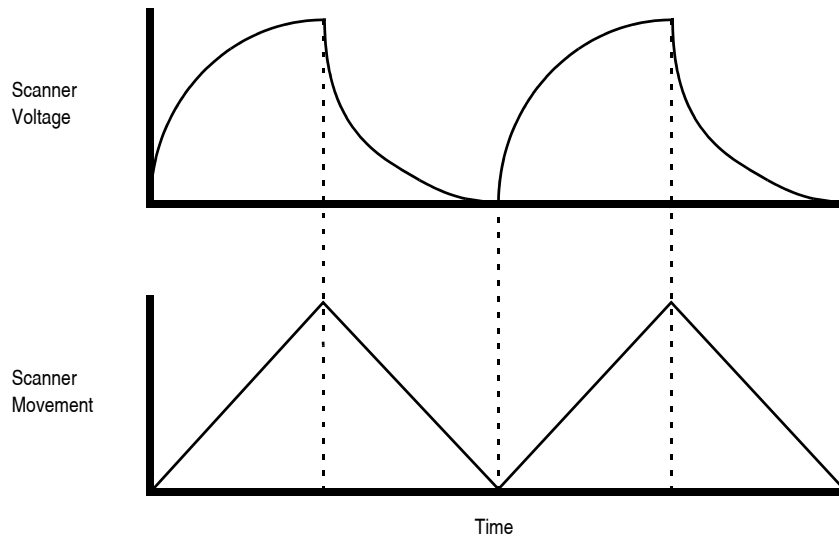
Through rigorous quality control of its scanner piezos, Bruker has achieved excellent modeling of scanner characteristics. Two calibration points are typically used for fine-tuning: 150 and 440 volts. A third point is assumed at 0 volts. These three points yield a second-order sensitivity curve to ensure accurate measurements throughout a broad range of scanner movements.

Because scanner sensitivities vary according to how much voltage is applied to them, the reference must be thoroughly scanned at a variety of sizes and angles. The user dictates, via the software, the distance between known features on the reference's surface and a parameter is recorded to compensate for the scanner's characteristics. The X, Y and Z axes may be calibrated in any sequential order; however, the linearization adjustments must be performed before any calibrations are attempted (see **Linearity Correction:** Section 18.4). Otherwise, calibrations will be undone by the linearity adjustments.

> **Note:** Check the SPM's measuring accuracy periodically to ensure that images are dimensionally represented within acceptable limits of error. If measuring accuracy is critical, or if environmental factors (e.g., humidity, temperature) impact the SPM significantly, this may require a quick check at the start of each imaging session. Establish a three-month service schedule for maintenance and calibration.

18.1.2 Calibration References

As described above, each scanner exhibits its own unique sensitivities; therefore, it is necessary to precisely measure these sensitivities, then establish software parameters for controlling the scanner. This task is accomplished with the use of a calibration reference (see Figure 18.1e).

Figure 18.1e   Silicon Calibration Reference
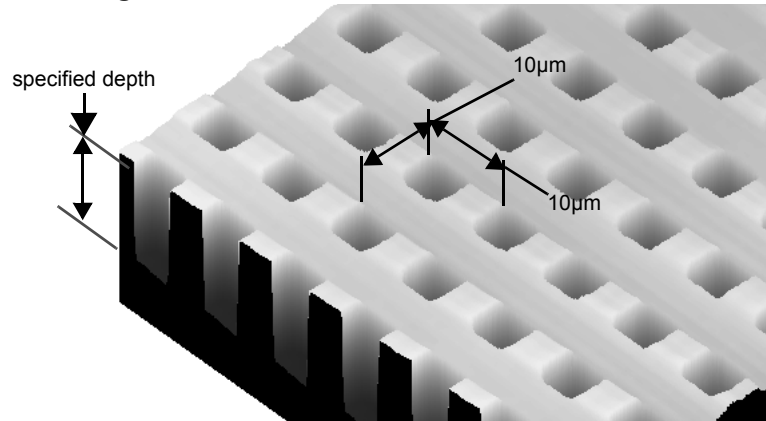
A calibration reference consists of a silicon substrate having a regular series of pits, at a depth specified on the container (typically 180 or 200nm), which is plated with platinum. Pits are spaced apart on 10μm centers. Other similar surfaces are available with different dimensions. Atomic-scale calibrations are generally carried out with mica or graphite, which exhibit very regular atomic lattices. Calibration references serve as the primary tool by which SPMs are calibrated. They serve as measuring sticks with which to gauge scanner displacement for a given voltage.

The SPM should be capable of measuring a calibration reference with an accuracy of 2 percent or better while scanning at the maximum **Scan size** setting. Using fine calibration techniques, it is possible to calibrate the SPM with even greater accuracy.

18.2   Calibration Setup

### 18.2.1 Check Scanner Parameter Values

1. If the system's original scanner parameters are deleted, copy the scanner parameters from the Equip.bak directory located in D:\DI Service. Individually purchased scanners are shipped with a head/scanner disk containing backup files or a hard copy of the scanner parameters.

2. In the event that files are not found, contact Bruker for scanner calibration records.

18.2.2 Align Calibration Reference

1. Load the silicon calibration reference into the SPM.

2. Align the reference with the microscope scanner so that the tip scans parallel to the reference's features with the **Scan angle** set at **0** degrees (see Figure 18.2a).

Figure 18.2a 0 Degrees Scan Angle
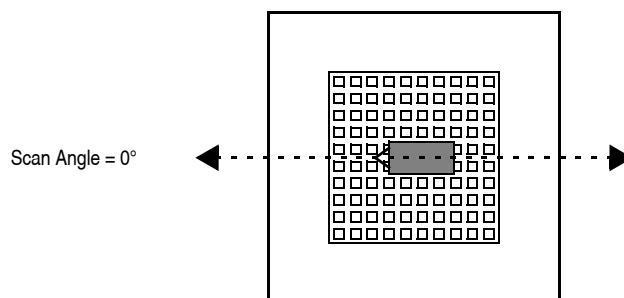
3. Align the reference within approximately 2º of perpendicularity to the scan axes.

18.2.3 Set Realtime Parameters

1. Set parameters in the control panels to the following values:

Panel	Parameter	Setting
Scan Controls	Scan Size	440 V
	X Offset	0.00 nm
	Y Offset	0.00 nm
	Scan Angle	0.00 deg
	Scan Rate	2.44 Hz
	Lines	256
	Slow scan axis	Enabled
	Z limit	330 V
Other Controls	Units	Volts
Channel 1	Data Type	Height
	Data Scale	~ 20 V[a]

a. Adjust the **Data Scale** parameter to obtain the best contrast.

18.2.4 Set Up Contact AFM

1. Set the **Microscope Mode** to **Contact**.

   > **Note:** The microscope can be calibrated using STM; however, this example utilizes contact AFM.

2. Set the **Scan Angle** to **0** degrees.

3. Adjust scan parameters to obtain a good-quality, maximum **Scan Size** image (**440V**). Set the **Scan Rate** to **2.44 Hz** and the **Lines** parameter to **256**.

4. With the sample engaged, check the scanning line relative to the reference features; the scan should be orthogonal to the pits on the reference.

5. If the reference requires rotation, **WITHDRAW** and rotate the sample to improve orthogonality between sample and scan line.

6. Repeat until features are oriented orthogonally with the scan frame.

18.3   Check Sample Orthogonality

Check the sample scan for orthogonality along both the X- and Y-axes. If the scan is aligned along one axis of the scan but not another, it may be necessary to adjust the microscope's **Orthogonality** parameter in the **Scanner Calibration** panel.

18.3.1 Measure Orthogonality

1. To measure a captured image's orthogonality, draw a horizontal line along an edge near the image center as shown in Figure 18.3a. Note the displayed angle.

Figure 18.3a   Horizontal Reference Line

Draw a horizontal line.

53.83 µm 0.1°
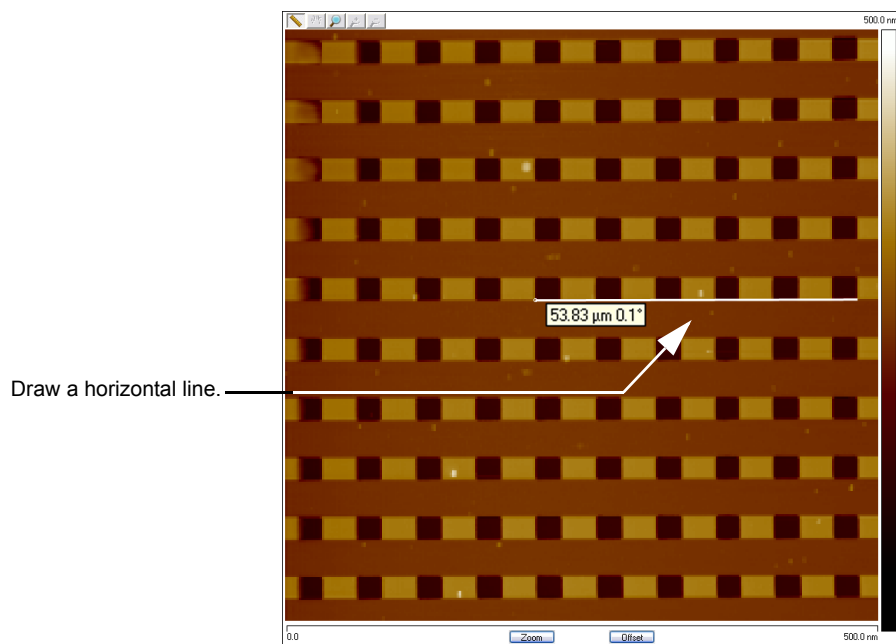
2. Draw a vertical line along an edge near the image center as shown in Figure 18.3b. Note the displayed angle.

Figure 18.3b Vertical Reference Line

Draw a vertical line.
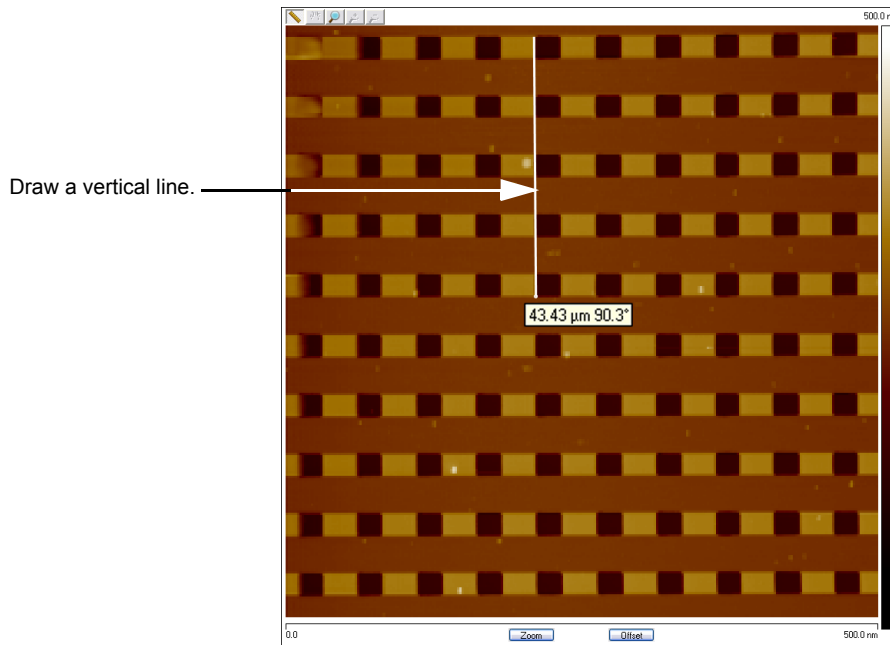
3. Perform the following calculation:

old **ORTHOGONALITY AT (0,0) OFFSET**
+ 90º
- (vertical angle - horizontal angle)

= new **ORTHOGONALITY AT (0,0) OFFSET**

4. Enter this value into **ORTHOGONALITY** in the **Scanner Calibration** dialog box.

5. Click **OK** to exit the **Scanner Calibration** panel.

6. Repeat correction of **Orthogonality** until the scanned image shows less than 0.5º of error.

Note: After a major change to the orthogonality parameter, you may need to physically realign the calibration standard to the image frame.

Figure 18.3c Non-Orthogonal and Corrected, Orthogonal Image
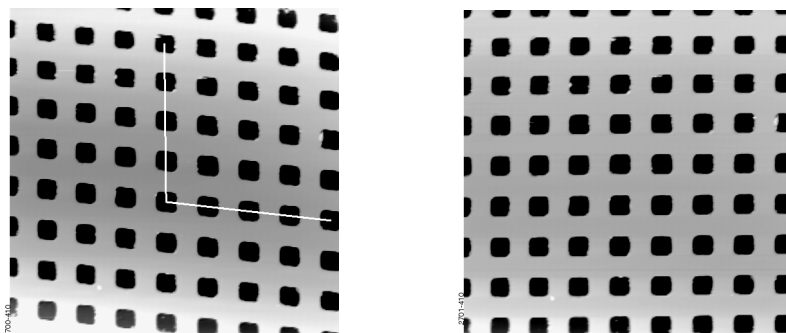
Note: In Figure 18.3c, pits align with the vertical (slow) axis but skew with the horizontal (fast) axis. The angle should be measured with the vertex near the center of the image and the vertices in the upper-right or lower-left quadrant.

Note: Apparent otrthogonality will change slightly between TRACE and RETRACE or UP and DOWN scans due to the software displaying perfectly horizontal lines while the scanner is actually moving at a slight angle during each scan line.

18.4   Linearity Correction

For applications which demand good linearity, the following procedure can be used to optimize the linearity correction parameters for individual scanners. As discussed previously, linearity correction is especially important for long-range scanners.

18.4.1 Adjust Mag0 and Arg

Set the **mag0** and **arg** values while noting the scan beginning and ending.

> **Note:**   When **Line direction** is set to TRACE, the beginning of the fast scan is on the left, as indicated by the arrow base. Because the monitor screen itself may be nonlinear, use the Realtime **Zoom** box to check **mag0** and **arg**.

> **Note:**   When viewing up or down scans, check the image against itself, not the residual image from the previous scan. Be careful to compare only the part of the scan drawn since the last parameter change.

Adjust Fast Mag0

1. Mount a calibration reference in the SPM and begin imaging. This may consist of a generic (e.g., 10 micron, silicon) reference, or a sample having features of known dimensions (e.g., grating, etc.).

2. Verify that the SCAN ANGLE is set to `0.00` degrees.

3. Engage and begin scanning in Contact Mode.

4. Optimize the image quality.

5. Move the mouse cursor to the scan window, click the right mouse button and select **BOX** then draw a box while depressing the left mouse button.

6. Adjust the box until it is about one-third the size of the scan.

7. Click **Calibrate** > **Scanner** > **X-Y...** to open the open loop **Scanner Calibration** window, shown in Figure 18.4a. As parameters values are changed, the effects will be seen on the monitor.

Figure 18.4a   The Open Loop Scanner Calibration window
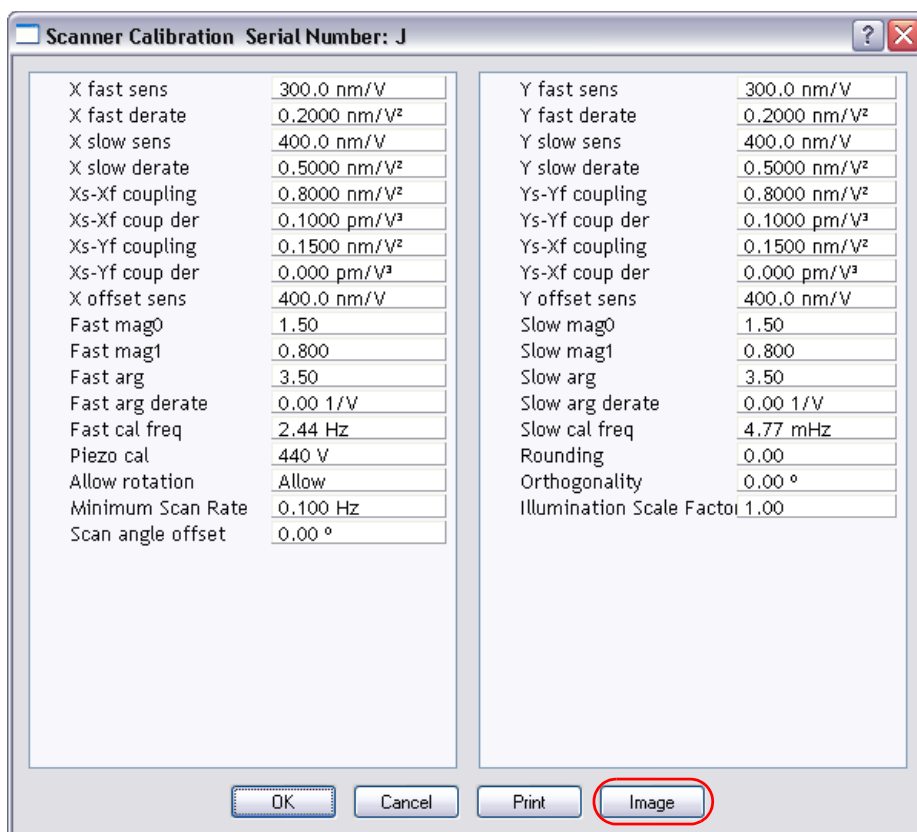
8. Click **IMAGE**, circled in Figure 18.4a, to make a measurement while in the **Scanner Calibration** window.

9. Select **Fast mag0,** the first scanner parameter to modify.

10. Move the zoom box to the start of the fast scan (on the left if the **Line direction** is set to **Trace**).

11. Move and size the zoom box until the beginning third of the scan's features are exactly aligned with the zoom box.

       Note: The beginning third of the scan is the standard for judging almost all of the linearity values. Ignore the set of features near the edges of the scan since these may be distorted slightly.

12. Move the zoom box to the end third of the scan.

13. Align one side of the zoom box with desired features and observe how the other side aligns with the features under it.

14. If the features are too large to fit an equal number inside the zoom box, decrease the **Fast mag0** value. If the features are too small, increase the **Fast mag0** value. Change **Fast mag0** by about 0.1 to 0.3 units at a time.

15. The entire scan axis is affected each time a parameter changes, so after every change, resize the zoom box at the beginning third of the scan and compare again with the end third.

Figure 18.4b   Fast Scan Linearization: Mag0

Mag0 Too Large   Mag0 Too Small
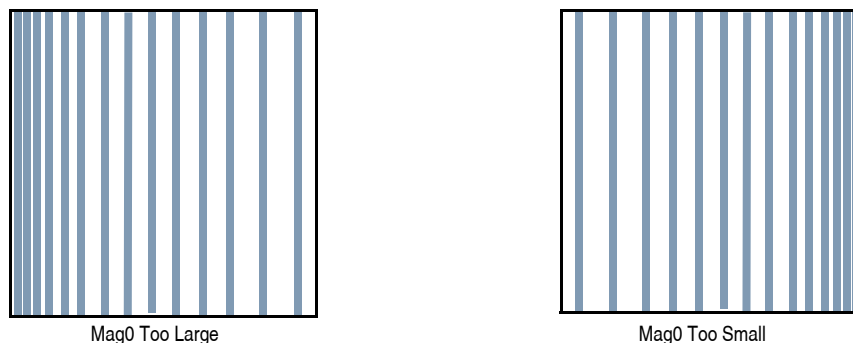
Adjusting Fast arg

1. Once the beginning third of the scan is equal to the end third, check to see if the center needs adjusting.

2. If the center features are too large for the box, decrease the **Fast arg** value. If the center features are too small, increase the **Fast arg** value. Change **arg**s by 0.2 to 0.5 units at a time.

3. Changes affect the entire scan, so continue to resize the zoom box after each change.

4. After an adjustment in **Fast arg** is made, **Fast mag0** may need readjusting. Repeat **Fast mag0** adjustment procedure until the rulings are evenly spaced across the Fast-axis.

Figure 18.4c   X Scan Linearization

Center Compressed   Center Expanded
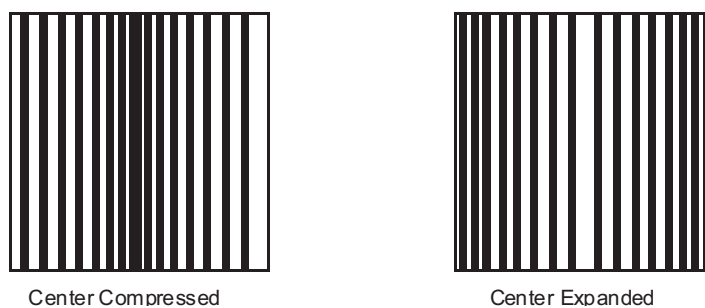
5. After setting **Fast mag0** and **Fast arg**, insert the values for **Slow mag0** and **Slow arg**. These values serve as close starting points before adjusting the slow linearities.

Adjusting Slow Mag0

1. Follow the same instructions as for **Fast mag0**.

2. After a parameter change, wait until a new third of a scan begins before setting new parameters and resizing the zoom box.

    Note: Setting the slow linearities requires a lot of time.

3. Move the resized box to the end of the scan and prepare to measure the end promptly.

    Note: You may type in a new parameter value before the scan starts again.

    Note: Be careful not to confuse scan top and bottom with beginning and ending, as the scan direction alternates.

4. Adjust the zoom box to fit the beginning third of the scan and check against the end third.

5. If the features of the end third are too large for the box, decrease the parameter. If the features are too small, increase the parameter.

    Note: Compare only parts of the current scan, not the previous scan.

Figure 18.4d   Y Scan Linearization

Center Compressed   Center Expanded
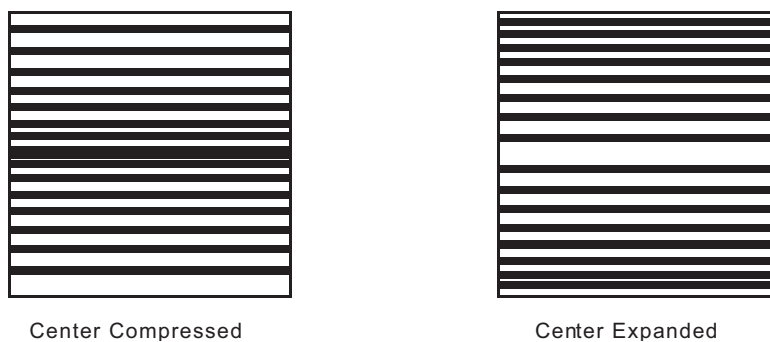
Adjusting Slow arg

1. Follow the same instructions for **Fast arg**.

2. Adjust the zoom box to fit the beginning and ending of the scan, then check the center.

3. If the features in the center are too large, reduce the **Slow arg** value. If the features in the center are too small, increase the **Slow arg** value.

4. After adjusting **Slow arg** values, check if **Slow mag0** requires readjusting.

5. Repeat the procedure for adjusting **Slow mag0** until the rulings are evenly spaced along the slow axis.

18.4.2 Adjust Fast mag1

Initial Adjustment

1. For initial adjustment, click **OK** to close the **Scanner Calibration** window.

   Note: Selecting **Cancel** resets parameters to default values when the box is opened.

2. Change the **Scan size** to **150V**.

3. If the two scope traces do not overlap, **Fast mag1** needs adjusting.

4. On the **Scan** panel, select **Slow Scan Axis**. When tall features appear on the scope trace, press the keyboard right or left arrow key to switch the **Slow Scan Axis** to **Disabled**.

5. Select **Calibrate** > **Scanner** > **X-Y...** to open the **Scanner Calibration** box.

6. Select **Fast mag1**.

7. Change the value until the two traces align.

   Note: The retrace line will shift to the right as the value is increased.

8. When done, click **OK** to close the **Scanner Calibration** window.

9. Set **Slow scan axi**s back to **Enabled**.

10. Select **Image** mode.

Fine Adjustment

Initial adjustment is usually adequate; however, if more precision is desired, use the following fine adjustment techniques to adjust **Fast mag1**.

1. Use the same procedure for adjusting **FAST MAG0**.

2. As before, set the **Zoom** box for the beginning of the scan and then check the ending. Because the scan is small, use a **Zoom** box up to one-half as large as the scan.

3. If the end of the scan is larger than the beginning, reduce the **Fast mag1** value. If the end is too small, increase the value of **Fast mag1**.

18.4.3 Adjust Slow mag1

1. With the **Scan size** set to **150V**, select **Calibrate** > **Scanner** > **X-Y...**

2. Select **Slow mag1** and input the value from **Fast mag1**.

    Note: For medium-sized scanners (C to F), further adjustments should not be necessary. The **Slow mag1** value is usually 100-120 percent of the **Fast mag1** value.

3. If more precision is desired, adjust **Slow mag1** using the same procedure for adjusting **Slow mag0**.

4. Set the **Zoom** box for the beginning of the scan and then check against the ending. Because the scan is small, use a **Zoom** box up to one-half as large as the scan.

5. If the end of the scan is larger than the beginning, decrease the **Slow mag1** value. If the end is too small, increase the value of **Slow mag1**.

6. Wait one complete frame with the new value before readjusting the **Slow mag1** value.

7. Check the final result by capturing an image and checking it with the **Crop and Split** function. The scanner is now ready for calibration of the X and Y parameters.

18.5   X-Y Calibration using Capture Calibration and Autocalibration

18.5.1   Capture Calibration

Scanners are calibrated by setting parameters in the NanoScope software using the **Capture Calibration** command. The basic calibration procedure using a 10μm pitch calibration reference (see Figure 18.1e) is described below.

1. With the **Scan Rate** set to **2.44Hz** and **Samples/Line** parameter to **256**, a full **Capture Calibration** requires approximately 60 minutes.

    Note: Increasing the **Samples/Line** or decreasing the **Scan Rate** significantly increases the required time but should be considered if your typical measurements are at higher resolution or slower scan rates.

2. Using the mouse, click on **Capture** > **Capture Calibration**. The **Capture Calibration** dialog box lists twelve parameters used in the calibration procedure (see Figure 18.5a).

**Figure 18.5a**   Capture Calibration Prompt
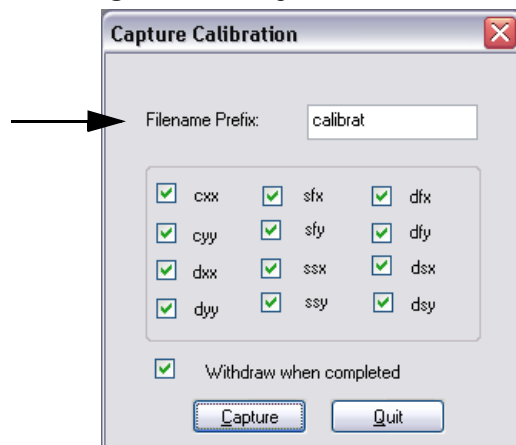
3. If this is a first time calibration, or if the microscope's calibration has not been checked within the last three months, verify that all parameters are selected. Otherwise, select those you need.

4. Change the **Filename Prefix** if desired.

5. Click **CAPTURE** to initiate the automatic calibration routine.

    Note: The microscope begins an automatic series of scans on the reference which require approximately one hour to complete. During each scan, the scanner moves the piezo using carefully calculated movements. Many of these movements are unusual, giving rise to a variety of images which do not resemble the normal reference. For example, pits may resemble trenches and features may be presented at various angles.

6. As each routine is executed, adjust the scan slightly to optimize the calibration image using the **Capture Control** dialog box displayed on the control monitor throughout the calibration routines (see Figure 18.5b).

Figure 18.5b Capture Control Prompt
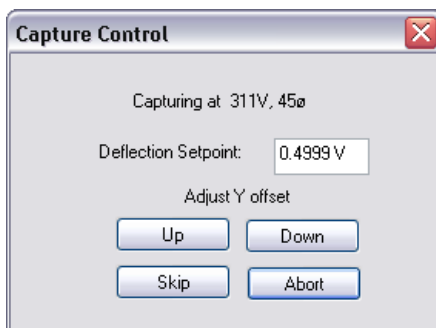
Note: The capture status will begin at skip 2. The program skips the current scan plus one more before capturing an image for later calibration. This allows hysteresis and drift to settle out when the scan changes direction and size between images.

7. For cxx, cyy, dxx, dyy images, if portions of features are missing, or if the image is blank, click repeatedly on **Up, Down**, **Left** or **Right** to adjust the scan until more of the features are imaged. Features should extend across as much of the displayed image as possible (see Figure 18.5c).

Figure 18.5c Improved Calibration Image

Partial Calibration Image   Improved Calibration Image
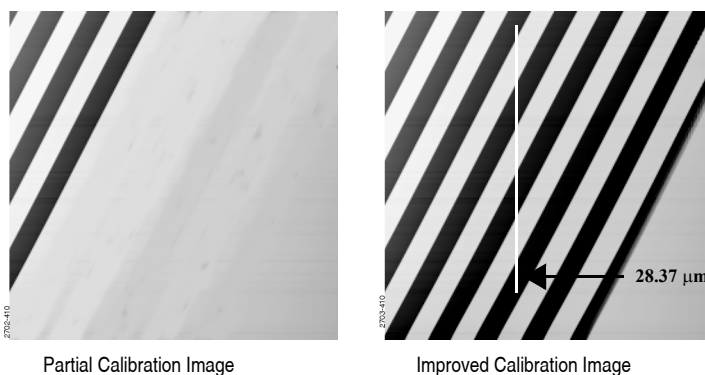
Note: After the first four images with the diagonal stripe pattern are captured, you can leave the system unattended while the program continues to completion. Some of the following images appear stretched in one dimension; however, this is normal.

8. If the scan has not stabilized by the time the capture status changes to **Forced**, click **SKIP** to increment the capture to skip the current scan.

> **Note:** Do not click ABORT unless you want to stop the entire **Capture Calibration** program.

9. Once the image is optimized, allow the software to capture the entire image without disturbing it. The software automatically indexes to the next image. Once all calibration images are obtained, the software prompts the user that it is finished.

10. Go to the directory where all **Capture Calibration** files are saved.

11. Select the stored images in the **Browse** window to review all **Capture Calibration** files.

12. Verify that all calibration images contain features spanning the full width and height of the image frame (see the right image in Figure 18.5c).

13. Recapture all images unsuitable for calibration. Record the file name extensions for all unusable files (e.g., .cxy, .dyy), then delete the files.

14. Re-engage on the reference surface and select **Capture Calibration**.

15. Verify that the file name prefix is identical to that of the usable files. Remove the "x" from all file name extensions except for the unusable file names recorded in Step 13.

16. Finally, click **Capture** to recapture the selected files.

18.6   Auto Calibration

After the **Capture Calibration** routine is completed, the user measures surface features contained within each image and enters their dimensions into the software. The software compares its estimates with the actual (user-entered) dimensions to make final corrections. This portion of calibration is carried out using the **CALIBRATE** > **SCANNER** > **AUTO CALIBRATION** command.

1. The system will prompt the user to select the calibration image, shown in Figure 18.6a. Click **OK** then do the following:
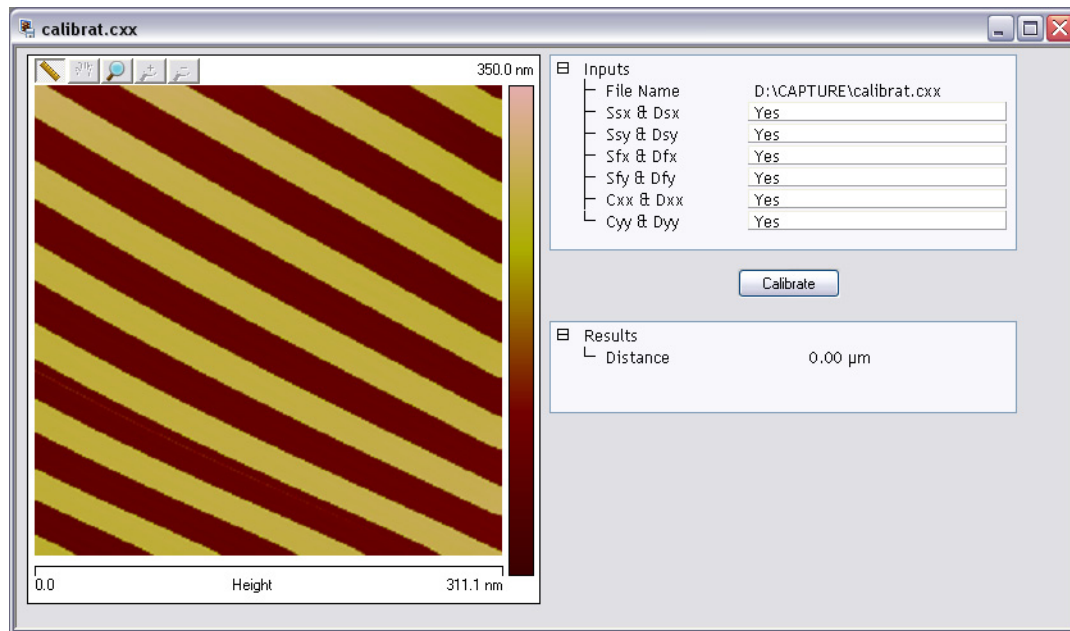
Figure 18.6a   Select the calibration image
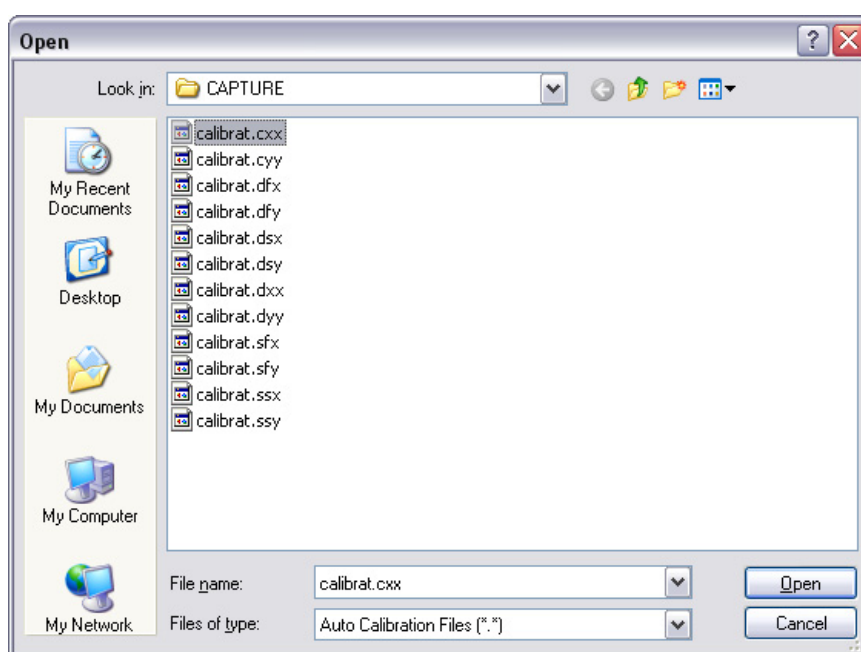
2. Select any of the desired captured calibration images. and click **Open**.

3. The system will open a user interface, shown in Figure 18.6b. In the right upper **Input** panel, select those you wish to calibrate as **YES** and those you wish to skip as **NO**. For normal calibration, verify that all parameters are selected. Click the **CALIBRATE** button to start the calibration.

Figure 18.6b X-Y Piezo Calibration Selection Prompt

4. Depending on what **Inputs** are selected in Step 3, the software will sequentially present various calibration images. You will be asked to draw either a vertical (shown in Figure 18.6c) or horizontal (shown in Figure 18.6d) line on those images. The software displays the span of that drawn line initially in the **New Distance** field (e.g. 105µm in Figure 18.6c) and in the **Results** / **Distance** /**Value** field. This value is the software's estimate of the length of the line based on the current calibration values. Input the known value (e.g. 100um in Figure 18.6c) in the **New Distance** field to reflect the correct line length.

5. Click the **APPLY** button. The software will finish this image and continue to the next image.

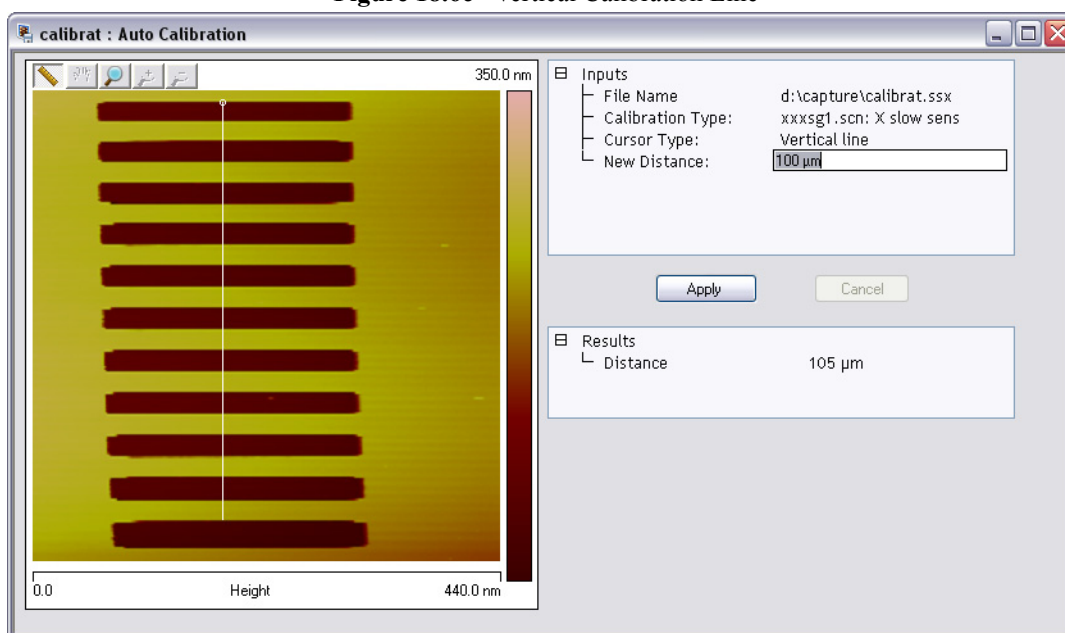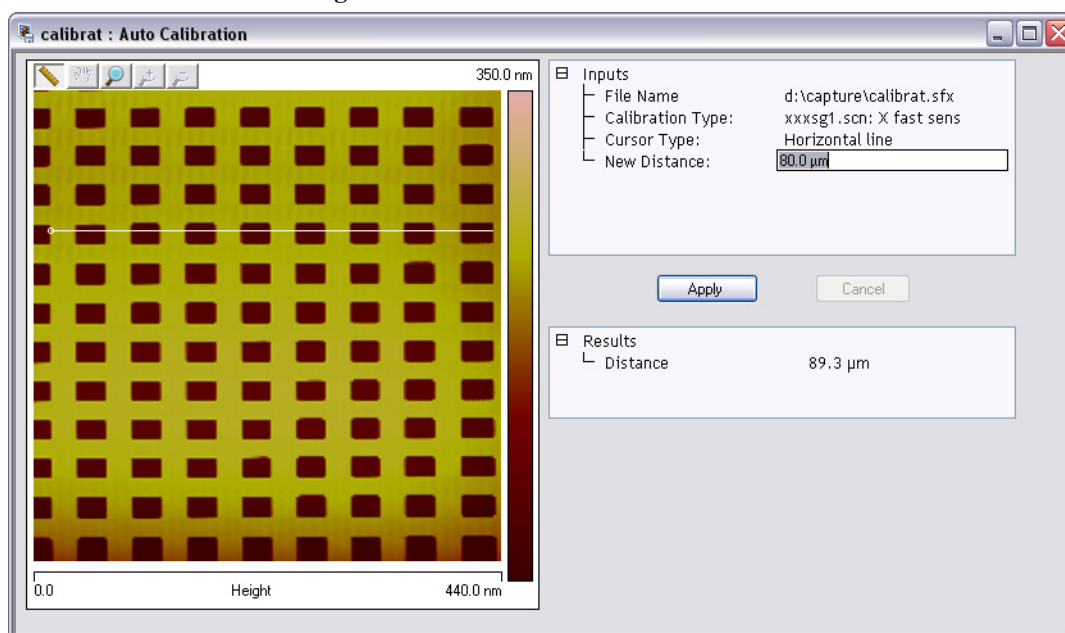**Figure 18.6c** Vertical Calibration Line

Figure 18.6d Horizontal Calibration Line

Note: When using the mouse to draw a line on the image, draw it to span as many features as possible, connecting similar edges (see Figure 18.6c and Figure 18.6d). Measure features without regard to how they appear in the calibration images. Features may be represented with stretched, distorted, or angled appearances due to the unusual movements employed during capture calibration scanning. Regardless, features are separated by the same spacing.

6. Continue drawing lines and entering measured distances until all capture calibration images are measured. When the software is finished, it will prompt the user that it is done. Click **OK** to acknowledge this message.

7. The software prompts the user that "The current workspace must be closed. Would you like to save and reload the current workspace." Click either **YES** or **NO**. The software will then close the current workspace and start a new one for the newly calibrated parameters to take effect.

If the **Capture Calibration** and **Auto Calibration** routines are completed correctly, the SPM will be calibrated within 1-2 percent accuracy over most of the scanner's measuring range. To obtain still better accuracy, the SPM can be fine-tuned to obtain maximum measurement accuracy. This is accomplished through the use of calibration parameters discussed in Section 18.7.

18.7 Fine-tuning for X-Y Calibration

Fine-tuning is usually performed at two **Scan size** settings: **150V** and **440V**. Both horizontal and vertical measurements of sample features are made, then compared with actual distances. Based upon this comparison, computer parameters are fine-tuned. To fine-tune your SPM for maximum X-Y measuring accuracy, review the procedure below.

18.7.1 Prepare System for Fine-tuning

1. Set the SCAN SIZE parameter on the **Scan Control**s panel to the maximum value (**440V**).

2. Verify that the SCAN ANGLE is set to **0.00** degrees.

3. Mount a calibration reference in the SPM and begin imaging. This may consist of a generic (e.g., 10 micron, silicon) reference, or a sample having features of known dimensions (e.g., grating, etc.).

4. Optimize the image quality.

> **Note:** Your calibration and fine-tuning procedures are no better than the procedures and references used. **Choose both carefully**.

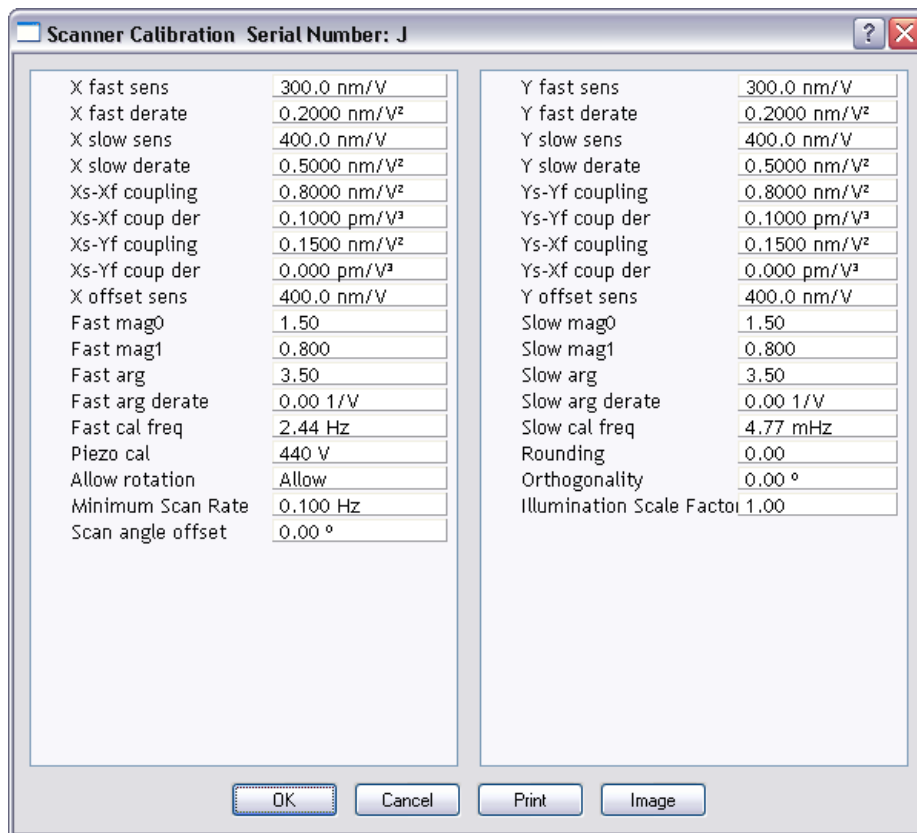### 18.7.2 Measure Horizontally at 440V Scan Size

1. Set the **Scan size** parameter on the **Scan** panel to the maximum value (**440V**).

2. Verify that the **Scan angle** is set to **0.00** degrees.

3. **Engage** the surface.

> **Note:** Contact mode is preferred for this procedure because the scan rate is faster for contact mode than tapping mode.

4. After you have completed a scan pass, click **CALIBRATE** > **SCANNER** > **XY**. The **Scanner Calibration** dialog box displays (see Figure 18.7a).

Figure 18.7a   Scanner Calibration Dialog Box

X fast sens	300.0 nm/V	Y fast sens	300.0 nm/V
X fast derate	0.2000 nm/V²	Y fast derate	0.2000 nm/V²
X slow sens	400.0 nm/V	Y slow sens	400.0 nm/V
X slow derate	0.5000 nm/V²	Y slow derate	0.5000 nm/V²
Xs-Xf coupling	0.8000 nm/V²	Ys-Yf coupling	0.8000 nm/V²
Xs-Xf coup der	0.1000 pm/V³	Ys-Yf coup der	0.1000 pm/V³
Xs-Yf coupling	0.1500 nm/V²	Ys-Xf coupling	0.1500 nm/V²
Xs-Yf coup der	0.000 pm/V³	Ys-Xf coup der	0.000 pm/V³
X offset sens	400.0 nm/V	Y offset sens	400.0 nm/V
Fast mag0	1.50	Slow mag0	1.50
Fast mag1	0.800	Slow mag1	0.800
Fast arg	3.50	Slow arg	3.50
Fast arg derate	0.00 1/V	Slow arg derate	0.00 1/V
Fast cal freq	2.44 Hz	Slow cal freq	4.77 mHz
Piezo cal	440 V	Rounding	0.00
Allow rotation	Allow	Orthogonality	0.00 °
Minimum Scan Rate	0.100 Hz	Illumination Scale Factor	1.00
Scan angle offset	0.00 °		

5. Click **IMAGE** to view the image while in the **Scanner Calibration** window, shown in Figure 18.7b.

6. Select two widely-spaced features on the sample image of known separation. Use the mouse to draw a horizontal line between them (see Figure 18.7b). (For example, on a 10µm silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The screen will display the measured distance between pits next to the line.

Figure 18.7b Calibration Horizontal Reference

Draw a horizontal line.
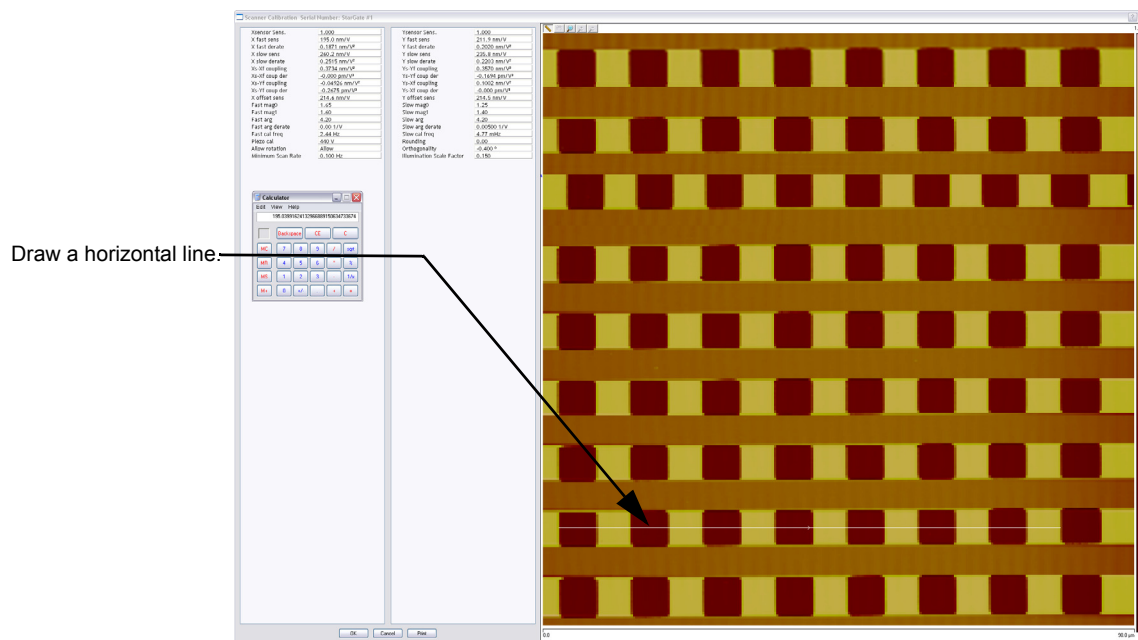
7. Verify that the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to **Measure Vertically at 440V Scan Size:** Section 18.7.3.

8. Based upon the results in the above step, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

9. Multiply the quotient obtained above by the **X fast sens** value shown on the **Scanner Calibration** panel.

10. Enter the new value. The new value adjusts the scanner's fast axis to more closely match calculated distances with actual feature distances. The new sensitivity setting takes effect as soon as it is entered.

11. Repeat Step 6 to check your change.

12. To save new values to the computer's hard disk, click on **OK**. This closes the **Scanner Calibration** panel.

18.7.3   Measure Vertically at 440V Scan Size

1. Return to the image of the calibration reference.

2. Select the **CALIBRATE** > **SCANNER** > **XY** function to display the **Scanner Calibration** dialog box.

3. Click **IMAGE** to view the image while in the **Scanner Calibration** window.

4. Wait for at least three full scans to allow the piezo to stabilize then select two widely spaced features and then draw a vertical line connecting like portions of features (top edge-to-top edge, etc.). See Figure 18.7c.The SPM displays the calculated distance between features.

Figure 18.7c Calibration Vertical Reference

Draw a vertical line.
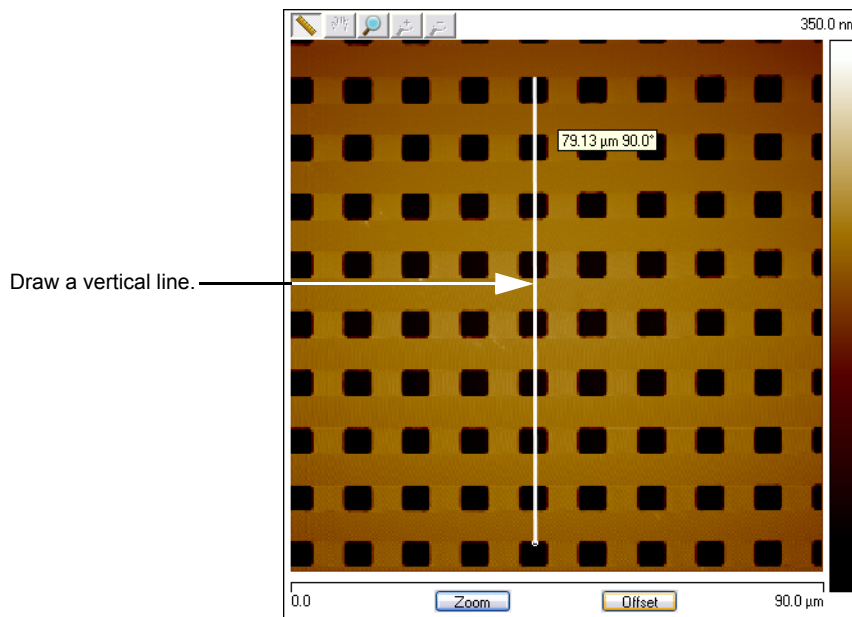
5. Verify that the microscope's calculated distance agrees with the known vertical distance. If there is significant disagreement between the two, fine tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to **Measure Horizontally at 150 V Scan Size:** Section 18.7.4.

6. Using the results from the above step, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

7. Select the **Y slow sens** parameter.

8. Multiply the quotient obtained earlier by the **Y SLOW SENS** value shown on the **Scanner Calibration** dialog box.

9. Enter the new value to adjust the scanner's slow axis to more closely match calculated distances with actual feature distances.

10. To save the new parameter value, click **OK**.

11. Repeat Step 4 to check your change.

18.7.4 Measure Horizontally at 150 V Scan Size

1. Verify that the **Scan angle** is set to **0.00** degrees, and that **Units** (**Other** panel) is set to **Volts**.

2. Set the **Scan size** parameter on the **Scan** panel to one-third the maximum (**150V**).

3. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a horizontal line between them.

   Note: For example, on a 10μm, silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible. The microscope displays the measured distance next to the line.

4. Verify that the microscope's measured distance agrees with the known horizontal distance.

5. If there is significant disagreement between the two, fine-tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to **Measure Vertically at 150V Scan Size:** Section 18.7.5.

6. Perform fine-tuning adjustments using either trial and error or calculate the precise correction (see Calculation Method).

Trial and Error Method

1. Select the **CALIBRATE > SCANNER > X-Y** function to display the **Scanner Calibration** dialog box.

2. Click **IMAGE** to make a measurement while in the **Scanner Calibration** window.

3. Select the **X fast derate** parameter or **Y slow derate** for Y-axis adjustment.

4. If the measured distance is less than the actual distance, decrease the **X fast derate** parameter slightly or **Y slow derate** for Y-axis adjustment and re-measure image features.

5. Adjust deratings up or down until measurements accord with known feature distances.

Calculation Method

1. Select **CALIBRATE > SCANNER > X-Y** to display the **Scanner Calibration** dialog box.

2. Click **IMAGE** to make a measurement while in the **Scanner Calibration** window.

3. Record the **X fast derate** or **Y slow derate** value.

4. Perform the following calculation:

$$\frac{s - \left(\frac{a}{m} \bullet [s - d(440 - v)] \right)}{440 - v}$$

where
s is the **X FAST SENS** or **Y SLOW SENS** value;
a is the actual distance;
d is the **X FAST DERATE** or **Y SLOW DERATE** value you recorded in the above step;
m is the measured distance;
and v is the **SCAN SIZE** in volts.

5. Return to the **Scanner Calibration** dialog box.

6. Enter the new **X FAST DERATE** value calculated in Step 4. This adjusts the scanner's fast axis to more closely match calculated distances with actual feature distances.

7. To set the new parameter value, click **OK**.

8. Measure horizontally again to verify that the measurement is now in agreement with the known distance.

18.7.5 Measure Vertically at 150V Scan Size

1. Select two widely-spaced features on the sample image of known separation.

2. Use the mouse to draw a vertical line between them.

> **Note:** For example, on a 10μm, silicon reference, draw the line from the top edge of one pit to the top edge of another pit as far away as possible. The microscope displays the measured distance next to the line.

3. Verify that the microscope's measured distance agrees with the known vertical distance. If there is significant disagreement between the two, execute the fine tuning procedure; go to the next step. If the displayed distance agrees with the known distance, no further calibration is required.

4. Adjust the **Yslow derate** value by using one of two methods: 1) the Trial and Error Method or 2) the Calculation Method described in **Measure Horizontally at 150 V Scan Size: Section 18.7.4** above.

18.7.6 Change Scan angle and Repeat Calibration Routines

• Change the **Scan angle** on the **Scan Controls** panel to **90º**.

• Repeat steps above for the following parameters: **Y FAST SENS, X SLOW SENS, Y FAST DER, AND X SLOW DER** to ensure the scanner is calibrated properly along the X- and Y-axis for scanning at 90º.

18.8   Calibrating Z

In terms of obtaining accurate Z-axis measurements, it is generally not difficult to obtain accurate X-Y calibration references; however, it is much more difficult to obtain accurate Z-axis results. Z-axis calibration is very sample-dependent. It is difficult to control Z piezo dynamics because the Z-axis does not move at a constant rate as the X- and Y-axes do during scanning. Furthermore, offsets affect the piezo over a period of minutes. The silicon calibration references distributed by Bruker have specified (on the calibration container) vertical features accurate to within ± 3 percent. The calibration reference is referred to throughout the examples provided in this section. If you require greater accuracy, you must select an appropriate calibration standard, and a closed loop head employed with a Bruker Dimension Series microscope.

> **Note:**   Refer to the label on your calibration reference sample to verify the measurement.

18.8.1 Engage

1. Set up the microscope for **TappingMode** imaging.

2. Select **ENGAGE** under the **Microscope** drop-down menu or click the **ENGAGE** icon in the **Workflow Toolbar**.

3. Set **Channel 1** to **HEIGHT** and **Scale** to **300NM** (See Figure 18.8a). Ensure that the probe tip is tracking well.

Figure 18.8a   Set **Channel 1** to **HEIGHT** and **Scale** to **300NM**

4. Find a square pit and center the pit in the image using a **Scan size** of approximately **10μM**.

5. Set the Aspect Ratio to at least 4 to speed up imaging of a horizontal band of data across the pit.

6. Verify that the **Z Center Position** value shown next to the image display is close to **0 volts** (**±5 VOLTS**), shown in Figure 18.8b.

Figure 18.8b   Z Piezo Voltage (position)
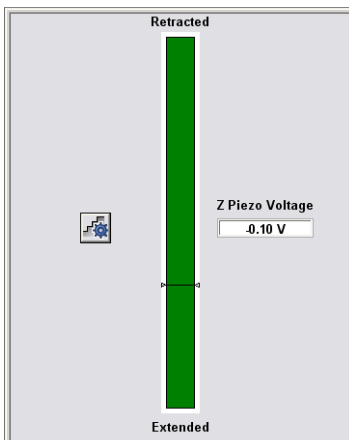
7. If the **Z Center Position** value is not close to zero, use the **Microscope** > **Step Motor** > **TIP UP** and **TIP DOWN** buttons to adjust (see Figure 18.8c).

Figure 18.8c   Step Motor
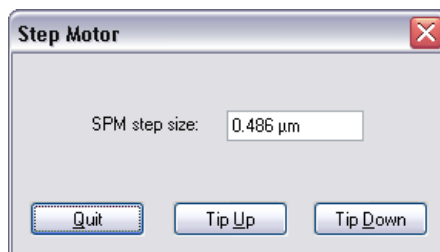
18.8.2 Capture and Correct an Image

1. Set **CHANNEL 1 DATA TYPE** to **HEIGHT**.

2. Capture an image by selecting **Capture** from the NanoScope tool bar, or click the **CAPTURE** icon.

3. When the image is captured, load the image by double clicking it in the browser bar.

4. Click the height image (**CHANNEL 1**) to select it.

5. Remove all tilt and scan line errata from the image by selecting **Modify** > **Flatten** or click the **FLATTEN** icon on the NanoScope tool bar.

   a. Set the **FLATTEN ORDER** parameter in the input box to **1**.

   b. Go to the display window and draw a stopband over the pit as shown in Figure 18.8d.

Figure 18.8d   Draw a Stopband
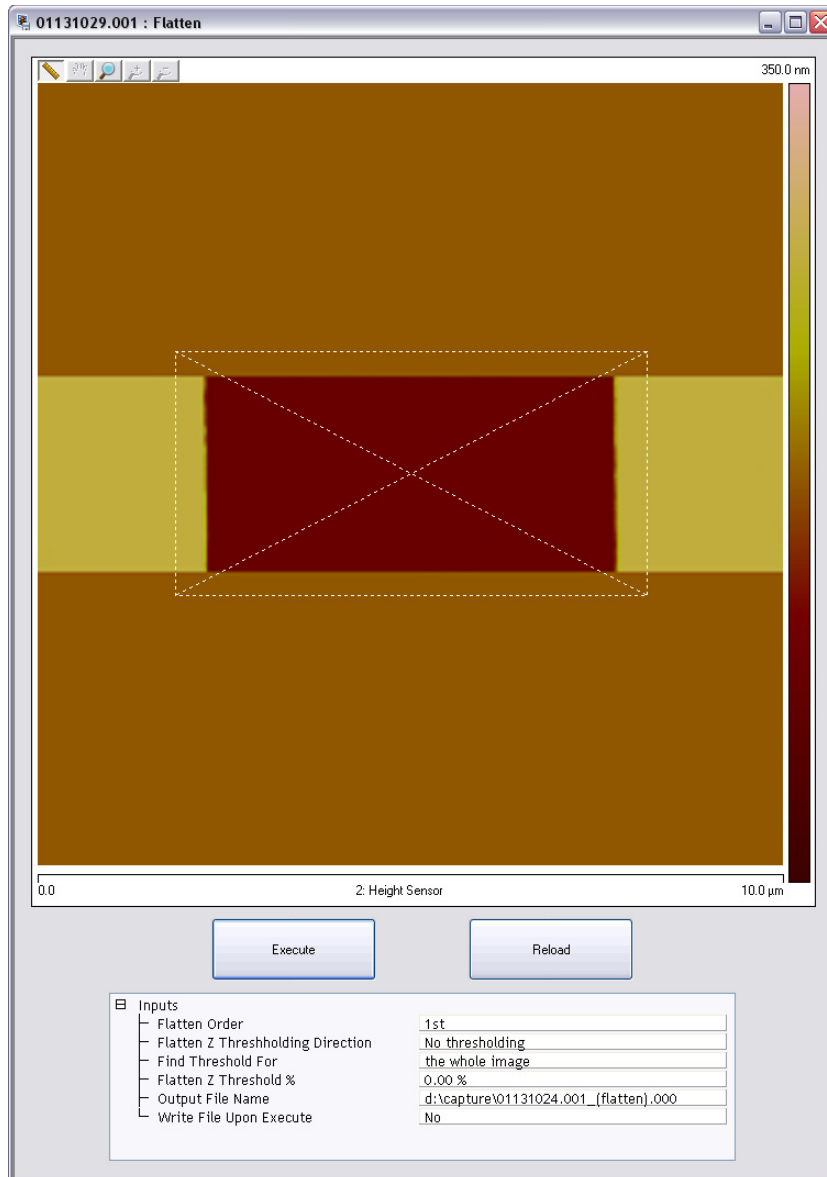
   c. Click **EXECUTE** to complete the flattening procedure.

   d. Right click the image, select **CHANNEL 1** (**HEIGHT**) and click **EXECUTE** again to flatten **CHANNEL 1**.

18.8.3 Measure Vertical Features

With the image corrected, its vertical features may now be measured. This is performed using
Depth analysis to utilize more data points.

1. Select the **Analysis** > **Depth** command or click the **DEPTH** icon on the NanoScope tool bar. This loads the **Depth** analysis screen shown in Figure 18.8e.

2. Right click the image and select **CHANNEL 1**.

Figure 18.8e   Depth Analysis Screen
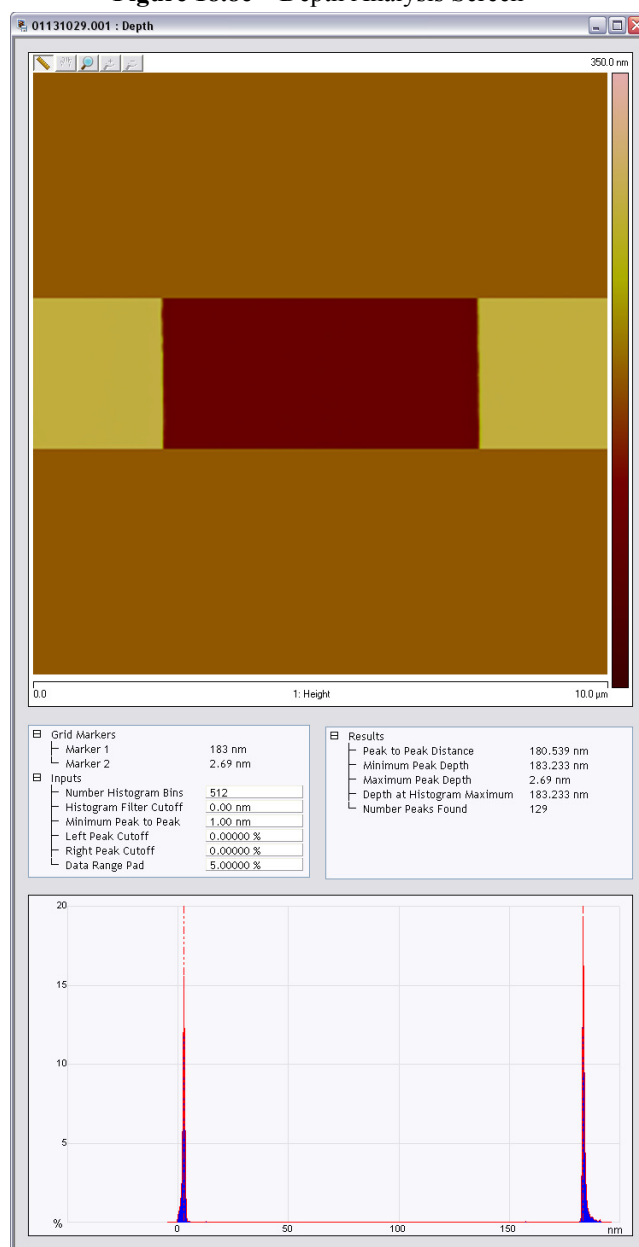
3. If desired, part of the image may be selected for analysis by drawing a cursor box surrounding that region. Otherwise, the whole image will be used (see Figure 18.8e).

> **Note:** A histogram of the height data within the drawn cursor box displays in the window, showing two prominent peaks. These peaks correspond to two elevations on the surface: the bottom of the pit and the top surface. There should be a line cursor on each peak.

4. If the two peaks do not appear in the display, increase the **HISTOGRAM FILTER CUT OFF** in the **Inputs** dialog box to **2-10ɴᴍ**. If the two peaks are too close together, increase the **MIN PEAK TO PEAK** in the **Inputs** dialog box.

5. In the **Results** dialog box in the **Depth** panel, check the **Peak to Peak distance** and record it for future reference.

18.8.4 Correct Z Sensitivity

If the depth of the pit on the 10μm silicon calibration reference deviates significantly from the actual depth, correct the **ZSCAN SENS.** parameter in the **Z Calibration** dialog box.

1. Open the **Z Calibration** dialog box by selecting **Calibrate** > **Z**.

2. Divide the actual depth of features by the measured depth (indicated in **Depth** analysis by the **PEAK TO PEAK DISTANCE**):

$$\frac{\text{actual depth}}{\text{Peak to peak distance}}$$

3. Multiply this quotient by the **ZSCAN SENS.** value in the **Z Calibration** dialog box, and replace the former **ZSCAN SENS.** value with the new result.

4. Click **OK** to enter the new **ZSCAN SENS.** value.

18.8.5 Recheck Z-axis Measuring Accuracy

After executing the steps above, recheck the Z-axis measuring accuracy of the SPM by repeating the steps outlined above until you obtain accuracy of 1 to 2%.

18.8.6 Calculate Retracted and Extended Offset Deratings

Piezoelectric materials exhibit greater sensitivity at higher voltages. In the steps outlined above, the Z-axis was calibrated while scanning near the middle of the piezo voltage range (i.e., **Z Center Position** ~ **0V**). In this section, you will calibrate the Z-axis piezo while extended and retracted to determine how much the Z sensitivity varies with **Z Center Position**.

1. If you are not already Engaged, select **ENGAGE** under the **Microscope** drop-down menu or click the **ENGAGE** icon in the **Main** panel. See **Engage:** Section 18.8.1.

2. Use the **Microscope** > **Step Motor** > **TIP UP** button until the **Z Center Position** reads **100V** (±10V).

    Note: By using the motor to move the tip up, the feedback loop forces the Z-axis piezo to extend to continue its tracking of the surface.

3. Refer to the steps above (**Capture and Correct an Image:** Section 18.8.2 through **Measure Vertical Features:** Section 18.8.3) to determine the measured depth of the calibration standard with a **+100V Z Center Position**.

4. Record the measured depth. If the depth measured by the extended piezo is correct within two percent, skip to Step 9.

5. Select **Calibrate** > **Z...** to display the **Z Calibration** pane, shown in Figure 18.8f.

6. Click the **Extended offset der** parameter.

7. Perform the following calculation

$$(1 + \text{current offset der}) \bullet \frac{\text{actual depth}}{\text{meas. depth}} - 1$$

For example, if the current offset equals 4% and the measured depth equals 185nm, then:

$$(1 + 0.04) \bullet \frac{\text{actual depth}}{\text{meas. depth}} - 1$$

8. Enter the new percent value from above (example was 12%) for the **Extended offset der** parameter in the **Z Calibration** panel.

9. The procedure for calculating and setting the **Retracted offset der** is exactly the same as for the **Extended offset der**; however, the piezo must be **retracted** to **-100V**. To calculate the **Retracted offset der**:

    a. Use the **TIP DOWN** button in the **Motor Control** panel to retract the piezo.

    b. Retract the **Z Center Position** to **-100V**.

    c. Repeat Step 3 - Step 8 above.

Figure 18.8f Z Calibration Panel

Chapter 19   Maintenance, Troubleshooting and Warranty

This chapter includes the following sections:

- **Maintenance:**

  - **Every three months::**

  - **Cleaning:**

- **Adjustment Screw Maintenance Procedure:**

  - **Quick Start Information:**

  - **Inspection:**

  - **Remove Adjustment Screws:**

  - **Inspect for Physical Damage:**

  - **Clean Guide Bushings:**

  - **Lubricate:**

  - **Reinstall:**

- **Vertical Engagement Scanners:**

- **Calibration:**

- **Fuse Characteristics and Replacement:**

  - **Fuse Replacement Procedure:**

- **Air Tables:**

  - **Air Pressure:**

  - **Air Table Feet:**

- **Troubleshooting:**

19.1   Maintenance

Generally, the MultiMode system requires very little maintenance. A simple schedule of cleaning and calibration check is usually sufficient.

19.1.1 Every three months:

1. Wipe down the major components of the instrument.

2. Check piezo calibration.

19.1.2 Cleaning

Major Components

The MultiMode is essentially an assortment of electronic boxes. You should use the same care with this equipment as with other laboratory instruments. Wipe the exterior of the system with clean paper towels or a cloth dampened with water or alcohol. Dispose of wipes in an appropriately labelled solvent-contaminated waste container.

## 19.2   Adjustment Screw Maintenance Procedure

This section covers maintenance procedures for adjustment screws used on Bruker Small Sample microscopes. These screws are used to support and position the head relative to the scanner, and must be cleaned periodically to ensure smooth operation. Instructions for the removal, cleaning, lubrication and replacement of adjustment screws are included in this section. A schedule of inspection and service is recommended at least **every three months**.

If adjustment screws are inspected and cleaned regularly, they should last the life of the scanner without replacement. Contamination of the screws with grit depends heavily upon the operating environment, types of material(s) being scanned, and operator handling.

19.2.1 Quick Start Information

Your scanner comes with a 1/16 inch hex wrench/key (See Figure 19.2a). This wrench is used to tighten or loosen the setscrews on the side of the scanner which in turn control the tension on the three adjustment screws that run all the way through the scanner. Verify that each adjustment screw turns freely in both directions, but feels snug. (Adjustment screws should turn freely while exhibiting sufficient rigidity to stabilize the head). Adjustment screws that are too loose will allow for drift between the head and the sample which will be evident at small scan sizes. However, if the motor adjustment screw tension is set too tight, the motor may not be able to turn.

Figure 19.2a Setscrew & Adjustment Screw Locations
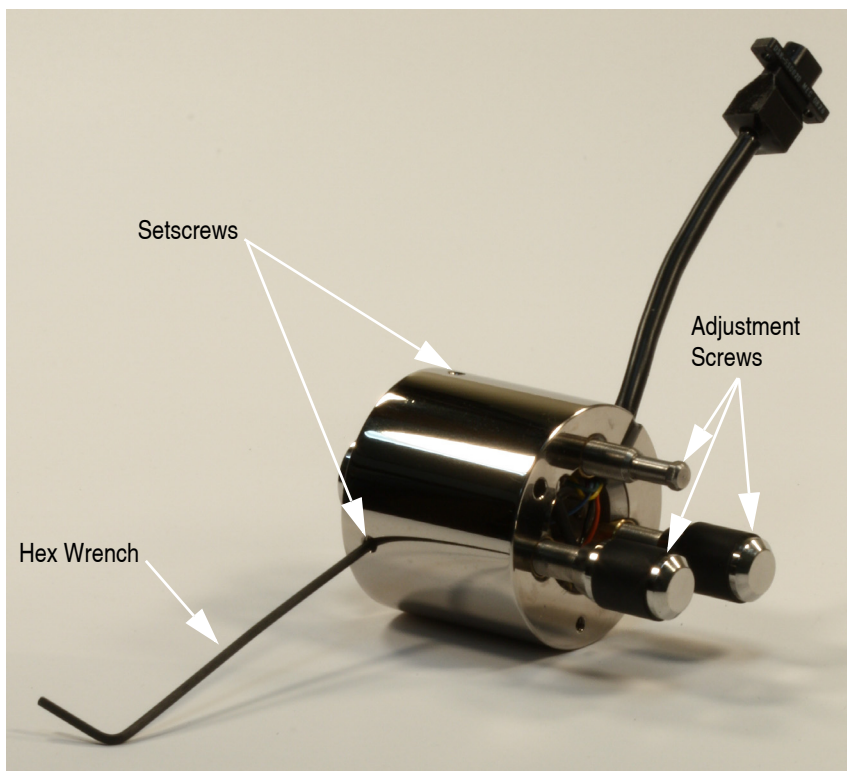
19.2.2 Inspection

Small Sample SPMs utilize fine-pitch (1/4"-80) adjustment screws; in some scanners, two of them are manually turned, and a third (rear) screw is turned by a motor located in the microscope base. The Vertical Engagement "JV" scanner has just one adjustment screw. Figure 19.2b, below, illustrates a screw and adjacent hardware installed on later-model (post-August 1993) microscopes. Earlier models have their brass threaded inserts installed at the top of the scanner body (rather than the bottom) and feature a Teflon bushing. Inspection and service is similar for all types.

Figure 19.2b   Adjustment Screw and Adjacent Hardware Depiction
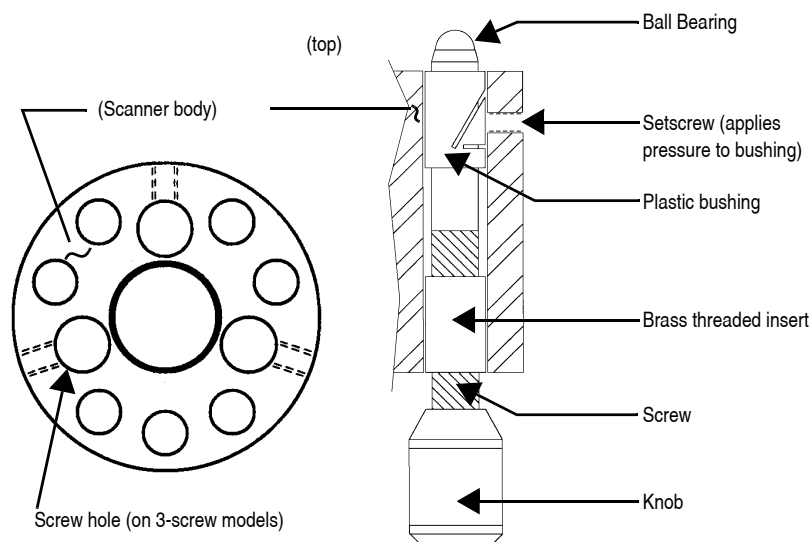
Adjustment screws are threaded into brass inserts, which are affixed to the scanner body with epoxy. Although the screws are not heavily lubricated, a light film of oil is applied to them at the factory to prevent galling. This allows sufficient lubrication for fine adjustment, while minimizing drift (i.e., loosening) between the screws and scanner body due to the slow displacement of lubricant from screw threads.

Problems develop whenever screws become fouled with fine grit: screws may be difficult to turn and/or exhibit any of the following symptoms:

- Eccentric, rotational "limp" (i.e., alternatively easy, then difficult to turn).

- Faint, crunching or grinding noises when rotated.

- Microscope cannot engage sample surface, i.e., motor is unable to rotate rear adjustment screw.

If *any* of these conditions are noted, screws should be backed out and cleaned as described below.

> **Note:**   If screws are frozen (i.e., cannot be rotated), DO NOT attempt to force them! Return the scanner body and screws to Bruker for repair.

The user should inspect screws *at least* every three months, more often if possible. It is a good practice to check screws whenever the scanner body is removed by turning manually and feeling for resistance. This is especially true of the rear, motor-actuated screw, which may be fouled without the user's notice.

19.2.3 Remove Adjustment Screws

To remove adjustment screws, do the following:

1. Remove SPM head and disconnect the scanner body from the Small Sample base by pulling its cable connector straight up. Hold the scanner body firmly in your hand.

2. Gently turn each screw to check for resistance. Turn counterclockwise until backed out of its screw hole. If resistance is experienced in turning the screw, stop, rotate briefly in the opposite direction, then retry. If resistance is experienced on later-model scanners, loosen the setscrew which applies pressure to the plastic bushing (see Figure 19.2b above), then try again.

3. If screws are frozen (i.e., cannot be rotated at all), DO NOT attempt to force them. Return the entire scanner body and screws to Bruker for repair.

19.2.4 Inspect for Physical Damage

Adjustment screws are made of hardened stainless steel; threaded inserts are made of a much softer brass. If a screw becomes fouled with hard grit it may bind against the threaded insert. If the bound adjustment screw is forcefully rotated, the screw will almost always destroy the brass insert: threaded inserts may become cross-threaded, or stripped entirely of threads. If this occurs, they will have to be pressed out and replaced; return to Bruker for repair.

1. Once screws are removed from the scanner body, they may be washed using methanol. Do not use strong solvents such as methyl chloride, MEK, benzene, etc. Use a fine brush or swab to remove grit from between threads and shoulder. Observe caution around rubberized knob surfaces; certain solvents may dissolve them! Be sure to remove all grit from surfaces; air dry.

⚠	**CAUTION:**	Do NOT splash solvent on the scanner tube or wiring at the center of the scanner body—certain components (e.g., wiring insulation) may be dissolved, causing scanner failure!
	ATTENTION:	Ne pas éclabousser le tube en céramique piézo-électrique ou le montage à l'intérieur du tube avec un solvant. Certains composants pourraient être dissous, entraînant une défaillance du tube.
	VORSICHT:	Verwenden Sie keine Lösungsmittel auf dem Scanner-Röhrchen oder den Kabelanschlüssen am Piezo - manche Komponenten (z.B. Isolation der Anschlußdrähte) könnten sich auflösen und eine Fehlfunktion des Scanners nach sich ziehen.

2. Use a swab stick (e.g., Q-tip, Puritan swab, etc.) to <u>carefully</u> clean grit from the threaded brass inserts. Be sure to remove all grit from threads; air dry.

3. Carefully inspect surfaces for signs of wear or damage. If small burrs are visible inside of the threaded brass inserts, they may removed by gently sanding with fine emory cloth, then recleaned using a swab and solvent. If threads are cross-threaded or stripped, the unit will have to returned to Bruker for repair.

19.2.5 Clean Guide Bushings

Plastic guide bushings are installed to stabilize screws and increase rigidity. Tolerances between screws and bushings are very tight, necessitating removal of all grit. Use a swab and methanol as described above to clean the bushings thoroughly.

> **Note:** It is not necessary to remove setscrews from the scanner base to clean the bushings. (Setscrews are used only to apply pressure to the shoulder of each adjustment screw; see Figure 19.2b above.) If the setscrews are removed, they should be retorqued to approximately 4 in/oz., or until the adjustment screw can be turned snugly without binding.

19.2.6 Lubricate

Apply a very thin layer of lubricant to each adjustment screw. Lubricant may consist of high-vacuum grease, optical coupling grease, or equivalent. Screws should exhibit a slight sheen and no more, indicating that they have been finely coated.

> **Note:** Excessive use of oil and grease on screws can cause the head to drift slightly. This is due to a slow displacement of lubricant between the screw and threaded insert. As lubricant is slowly squeezed out between the screw and threaded insert, the screw settles, causing the head to lower itself (and the tip) toward the sample. This is especially apparent during atomic-resolution imaging.

19.2.7 Reinstall

1. Verify that all screws, threaded inserts and plastic bushings are free of grit.

2. Replace adjustment screws in their threaded inserts. Carefully turn them down clockwise until the shoulder of the screw engages the plastic bushing, and the screw's ball bearing appears at the top of the scanner body.

    > **Note:** If screws bind during reinstallation, *stop immediately.* Back the screw out again, reclean as described above, then redo installation. Recheck that the setscrew is properly torqued at the plastic bushing. If screw still cannot be installed without binding, return to Bruker for repair.

3. Verify that each screw turns freely in both directions, but feels snug. (Adjustment screws should turn freely while exhibiting sufficient rigidity to stabilize the head).

19.3   Vertical Engagement Scanners

Figure 19.3a   MultiMode JV Scanner

Bruker offers "E" and "J" scanners which permit vertical engagement without significant lateral movement. The vertical scanners feature the following:

- Completely motorized tip-sample engage.

- Fully vertical engage head does not tilt.

- Easy to locate tip on desired scan area.

- 0.5μm lateral repeatability on subsequent engages.

- Accommodates samples up to 8.0mm thick.

- Integrated fluid seal at top of scanner resists fluid spillage.

- Retaining springs mount permanently to scanner body.

- Stabilizing screw, P/N 130-000-145

19.4   Calibration

A description of the complete scanner calibration is located in Chapter 18. In general the scanner's calibration should be checked every three months. The main change in calibration will be the loss of piezo sensitivity in the first year of use. This is an expected behavior. Loss of 10-15% of the initial scan size is common.

19.5   Fuse Characteristics and Replacement

- **NanoScope V Controller**: 4 fuses. The fuse characteristics are silk-screened on the back of the NanoScope V Controller (see Table 19.5a).

Table 19.5a   Fuse Ratings

Fuse	100V	120V	240V
1	2.5A	2.5A	2.0A
2	800mA	800mA	400mA
3	800mA	800mA	400mA
4	10A	10A	6.3A
ALL FUSES: 250V SLOW BLOW			

19.5.1 Fuse Replacement Procedure

1. Remove all power from the system.

2. Using a screwdriver, press the slot in the fuse holder and rotate the fuse a 1/4 turn counter-clockwise.

3. Gently pull the fuse out.

4. Insert a replacement fuse in the fuse holder.

5. Using a screwdriver, press the slot in the fuse holder and rotate the fuse a 1/4 turn clockwise until the fuse is secure.

6. Reconnect the power.

> **Note:**   If the problem persists, contact Bruker.

19.6 Air Tables

It is very common to see periodic noise in the AFM data due to an under-inflated or improperly balanced air table. It is important to check the air table when you move the instrument.

19.6.1 Air Pressure

The incoming air pressure should be approximately 80PSI. The air table has a built in regulator that is preset for optimum performance.

> **Note:** Incoming pressure higher than 80PSI has not been proven to be harmful. It is just not recommended.

19.6.2 Air Table Feet

The frame of the air table has four legs with feet that support the base plate. The feet should be lowered so that the air table is raised off of the wheels. Periodically check to make sure that the weight of the air table is evenly distributed on all four legs. To do this, simply use the supplied spanner wrench to slightly turn each of the feet in sequence. If the foot you are testing turns easily, there is no weight pressing down on it. Readjust the feet so that each foot is supporting an equal amount of weight.

19.7   Troubleshooting

The MultiMode system is very reliable and generally will not need adjustment once properly calibrated, nor is it likely to develop a problem due to use. In most cases any problems that occur can be linked to some external event.

Some typical examples of such events are:

- Voltage fluctuations.

- Lightning storms that cause power transients can damage circuit boards.

- Fluid spills on electrical components can cause damage.

- Improper installation of the air table when relocating the instrument to a new room can cause noise in the data.

- Improper signal cable installation when relocating the instrument can cause problems.

- After relocating the instrument to a new room, the data may exhibit noise. The room itself may have excessive floor vibration or acoustic noise.

- Installing a new board other than what is needed to operate the instrument via the computer can cause the software to not load correctly or a hardware conflict within the computer system.

19.7.1 Common Problems

No Laser light from head – Possible Explanations:

- The software is not running.

- The microscope mode is set to STM in software.

- A microscope other than a MultiMode is selected in software.

- The head is not plugged into the MultiMode base.

Fluid spills – Fluid spills will cause numerous problems. The most common problem is that the piezo electric element in the MultiMode scanner can become permanently damaged. Avoid fluid contact with the end of the scanner at all times.

Cables – The wires inside the cable connector on the MultiMode scanner can be broken from the plug. Disconnecting or connecting the scanner by pulling on the cable can cause this.

The scanner plug is very durable and typically does not fail due to use. Never connect or disconnect the MultiMode scanner cable from the base by pulling on the cable. Always connect and disconnect the scanner by holding on to the plug.

19.8   Contact AFM Troubleshooting

Depending on the operating mode being used, the symptoms and subsequent resolution may vary. For this reason, problems listed in this chapter are divided into Contact AFM (see Section 19.8) and TappingMode (see Section 19.9). STM problems are described in Chapter 12 of this manual.

Some of the problems and cures associated with contact AFM are also relevant to TappingMode. If problems occur, it may be helpful to read through the Contact AFM problems and vice-versa.

19.8.1   False engagement

The main cause of false engagement is optical interference on the photodiodes, which causes the vertical deflection (A-B) voltage to slowly move toward the setpoint voltage. Once the vertical deflection voltage reaches the setpoint voltage, the feedback loop assumes the tip has contacted the sample.

The source of the optical interference comes from either: i) the reflective gold coating on the cantilever, allowing some of the laser spot through and onto the sample surface; or, ii) stray laser light from the main beam interacting with the surface directly, causing optical path length related interference. The direct substrate reflection is more of an issue with reflective substrates that have large steps or topography. These effects can cause scattered light to be reflected onto the photodiode assembly along with the reflected beam from the back of the cantilever. The scattered light causes changes in the vertical deflection (A-B) voltage as the AFM head is lowered to the sample during engagement.

IMPORTANT! If a false engagement occurs, it can be detected easily by adjusting the **Setpoint** and observing the **Z Center Position** change. Increasing the **Setpoint** voltage by 1V should cause the **Z Center Position** to change by 1-5V. If the **Z Center Position** changes by a large amount (10-20V), the system is false engaged.

It is a good habit to watch the vertical deflection (A-B) differential voltage while the tip is engaging. The voltage should ideally jump (not drift slowly) from some negative value to the setpoint voltage. If it drifts slowly it means that reflected light is affecting the photodiode voltage. If this is the case, the engagement process can be aborted by clicking the **ABORT** button in the **Engage Status** box shown during engagement. The photodiode position should be adjusted to make the Vertical Deflection voltage more negative than the setpoint voltage before trying the engage sequence again.

Sources of false engagement include:

- Incorrect optical alignment of laser spot on cantilever. This alignment will cause a greater amount of laser light to reflect off of the sample.

- The sample has a region on it that touches the cantilever before the tip does.

- Foreign material stuck on the cantilever beam that is lower than the tip.

- Check the cabling between the computer and the controller, and between the controller and the microscope. Any discontinuity in the microscope signals can cause an immediate engage.

- The **Setpoint** may be set more negative than the vertical deflection (A-B) voltage (this applies only to contact AFM modes). This false engagement is immediate and the computer will not show any motor travel or time delay after one gives the command. To correct for this condition, select **WITHDRAW** and check that the vertical deflection voltage reads a voltage more negative than the setpoint voltage.

- Static charge from the sample.

19.8.2 Head appears engaged but does not track surface features

Occasionally, the head appears to be engaged but surface features do not appear on the display monitor. This usually results from false engagement (see Section 19.8.1 above), poor laser alignment, or something other than the tip coming in contact with the sample surface.

An easy way to check for laser alignment problems is to check the laser beam using a magnifier or slip of paper. Click on the **WITHDRAW** command to remove the cantilever from the surface and recheck alignment with the paper method (see Chapter 7). When satisfied with the alignment, readjust the vertical deflection voltage before re-attempting engagement.

Occasionally, when the optical head is not leveled properly, something other than the cantilever tip (usually the corner of the substrate or the cantilever clip) will contact the surface during engagement. The system may engage, but the image will not contain any features. Testing the SUM signal after withdrawing the tip several times is a good test for this failure mode. Generally, the SUM signal will be far from its original value if something other than the tip contacts the surface. The head should be leveled and the laser realigned if this has occurred.

19.8.3 Head does not engage

The complementary problem to false engagement is not engaging at all. Here are some common causes of this problem:

- The tip may have started too far from the sample. Lower the tip's height above the sample surface, then repeat the engagement procedure.

- The laser may be misaligned and/or the cantilever may be broken. When this occurs, replace the probe, realign the laser, and repeat the engagement procedure.

19.8.4 Head engages immediately

If the microscope engages immediately after the **Engage** icon is selected, the problem may be one the following: 1) The **Setpoint** may be lower than the feedback voltage. Select **WITHDRAW** a few times and verify that the vertical deflection reads a negative voltage of -1.0 to -4.0V. Adjust the **Setpoint** to zero or slightly above and try to engage again. If the microscope still engages immediately, check the cabling between the computer and the NanoScope controller and also between the controller and the microscope. Any discontinuity in the voltage feedback will cause an immediate engage.

19.8.5 Displacement of material

This is generally caused by too much tracking force. Use the **Force Calibration** window after engagement for setting the correct tracking force.

The AFM can be tuned to operate in the attractive region of the force curve. This will take advantage of the fluid layer which captures and holds the tip to the sample. This effectively reduces the tracking force. Be aware of the fact that this means the tip is pulling away from the sample. This operating condition might cause the tip to pop off of the sample surface.

19.8.6 Lines in the image

Lines can be caused by:

*   AFM tip picking up contamination. The tip will effectively become longer and this will cause the feedback loop to raise the tip to keep the same tracking force. The contamination can come off of the tip which will cause another level shift in the image. This problem will show up as large bands in the captured image.

*   Friction. Some samples have a stronger frictional interaction with the tip than others. The cantilever will bend and straighten due to the tip sticking and slipping as it is dragged across the surface of the sample. The result is a line-by-line level shift in the captured image. The trace and retrace scan directions can actually be inverted from each other if the friction is high enough. A good practice is to use the scope window in dual trace display. The trace and retrace directions should be close to each other. Trace and retrace can invert if there is friction present between the tip and the sample surface.

19.8.7 Problems with silicon nitride cantilevers

Sometimes nitride cantilevers can be a problem due to lateral warping or torquing of the nitride beams. This causes the reflected light to spill off to the side as the tip begins to engage the sample. Warping or torquing of the cantilever is associated with some older cantilever wafers. If engagement does not work the first time, try changing the actual lever used, i.e., go to the next lever on the same chip. Usually, the wide 200μm-long lever has the most lateral warping. If this is the case, try the wide, short cantilever next to it. Generally, the narrow-legged silicon nitride

cantilevers (both of which are on the same side of the chip) will have difficulties with the laser optics due to laser beam spillage over the side of the cantilever. This effect is more pronounced for samples which are highly reflective.

19.8.8 Image vertical dimensions are not correct

- When invoking the **Highpass filter**, height information will not be accurate. The Highpass filter removes the DC component of scan information. This will invalidate the height information in the image.

- The **Z Sensitivity** parameter must be calculated (as described in Section 18.8) or height data will be inaccurate.

- When acquiring deflection data for height measurements, gains must be set low to reduce movement of the Z piezo.

19.8.9 *Z Center Position* goes out of range

The **Z Center Position** voltage is a measure of the average voltage to the Z electrode. The image will disappear when the Z center position reaches either the fully extended or the fully retracted ends of the Z center indicator. Possible causes include:

- Having the **Z Limit** too low.

- Tilted sample. The sample should be as level as possible, particularly for larger scans, because tilt in the sample can cause the Z scan to run out of range.

- Mechanical drift will cause the sample-to-tip distance to change slowly bringing them apart completely, or too close together. If this is the case, the Z center will show either +220 (extended) or -220 (retracted), respectively.

- Drift in the optical path

Differentiating between optical path or mechanical drift is the first step in eliminating this problem. The force curve is a very useful tool for this purpose. Go to the force curve immediately after engaging. The force curve has two main regions: the sloping regions when the sample is in contact with the tip, and the flat region when the tip is free. Intersection of these two regions is what is important. Watch the force curve and determine whether the curve drifts vertically or horizontally; vertical drift is indicative of optical path drift, while a horizontal drift is due to a mechanical change in tip-to-sample separation.

Use the following as first steps in correcting drift:

- Verify that the sample, tip and stage are all stabilized. There should be no free movement between any of these components

- Recheck that the cantilever is firmly seated in its place and that there are no dirt particles wedged beneath it. Check that everything is tight and secure. Once this is checked, there may still be some drift. Using the **Tip Up** and **Tip Down** command can bring the sample back into range. (This may have to be done due to any number of factors).

- Check for thermal stability. The SPM should not be located directly in the path of heating or air conditioning ducts; also, avoid locating the SPM near large windows which trap solar heat. Thermally caused drift due to thermal expansion of SPM components is the most common cause of mechanical drift. The MultiMode tends to heat up and drift when used with the acoustic isolation hood because it traps heat produced by electronics in the base and head.

19.8.10 Poor image quality

If the tip is engaged with the vertical deflection reading a stable, near-setpoint value and the Z center position is not overly sensitive to small changes in **Setpoint**, then laser alignment is probably good and the tip is scanning the sample. If image quality is poor with distorted shapes and low contrast, try adjusting the gains first, then optimize the scan direction to take advantage of the best tip shape to improve image quality. Vary the **Scan Angle** parameter in the **Scan** panel and see if this cleans up the image. There is some variation in the shape of tip from substrate-to-substrate and the tip shape can affect the engagement process. (For example, a blunt tip may tend to engage falsely.) Therefore, changing the angle at which the tip scans some surfaces may have a significant effect.

Other scan parameters may also have a significant effect on the image quality. Varying the **Scan Rate**, **X** and **Y Offsets**, filtering, feedback gains and ranges all may improve the image (or make it worse). Once engaged, adjust parameters in the **Scan** panel to improve the image quality. In general, the **Scan Rate** should be lower for large scans and for samples having tall features. The **Scan Rate** can be increased on flat samples.

The offset and zoom commands should be used to locate good clean regions on the sample. Filtering can improve atomic-scale images, but it is usually better to go without filtering on larger scans. Highpass filtering will distort the height information in all but atomic scans. For height data, the **Integral** and **Proportional Gains** should be high but not high enough to cause oscillations. For deflection data, the feedback gains should all be low (close to zero). Please review the descriptions of all the parameters in the *NanoScope Software Manual*, and experiment with parameters on a known sample.

Selecting a large **Scan Size** and a high scan rate for a few scans can "sweep" an area clear. Decreasing the **Scan Size** to image within the "swept" area can improve the quality of atomic images.

Finally, adjust the force exerted on the sample. Engagement requires a positive deflection of the cantilever, but the microscope will operate at much lower forces and lowering the force sometimes improves the image quality. Decrease the **Setpoint** in small increments to lower the force. Once engaged and the **Scan Size** is significantly increased, sometimes more force is necessary to keep the tip scanning on the sample surface. Increase the **Setpoint** in small increments to increase the force.

19.8.11 Force Calibration command does not seem to work

Most often, the **Force Cal.** command will not provide a meaningful display, when first invoked. **Z Scan Start** and **Scan Size** as well as **Setpoint** affect the position and shape of the curve, and will probably require adjustment to achieve a useful display. The negatively sloped sensitivity line should, however, always be straight for a proper force curve.

19.8.12 Image features appear washed out

The **Data Scale** in the active **Channel** panel might be too large. Decrease the scale for more contrast between features.

19.8.13 Image is only black and white

The **Data Scale** in the active **Channel** panel is too small. Increase the scale for less contrast between features.

19.8.14 Image goes white

If, when using an "A" scanner, the image goes white after a couple of scans and the Z center voltage is still within range, check the **Realtime Plane fit** parameter on the active **Channel** panel. When using an "A" scanner, it should be set to **Line** or **Offset** instead of **None**. In general, this will be a problem anytime there is a significant amount of drift in the Z center voltage, but drift is especially prevalent with "A" scanners.

19.9   TappingMode AFM Troubleshooting

Depending on the operating mode being used, the symptoms and subsequent resolution may vary. For this reason, problems listed in this section are divided into Contact Mode (see Section 19.8) and TappingMode (see Section 19.9). STM problems are described in Chapter 12 of this manual.

Some of the problems and cures associated with contact AFM are also relevant to TappingMode. If problems occur, it may be helpful to read through the Contact AFM problems and vice-versa. The most common problem while operating in TappingMode is due to unusual resonance peaks in the **Cantilever Tune** command. Double peaks in the resonance curve can be caused by improper seating of the cantilever or a fracture in the cantilever. In general, bad cantilevers produce strange resonance curves, so the first thing to do when an unusual resonance curve occurs is to clean the groove on the cantilever mount and reseat the cantilever. If this does not improve the resonance curve, install a new cantilever.

19.9.1   Streaks on the trailing edge of surface features

Streaks are an indication of the tip not tracking the surface due to either: 1) insufficient tapping force; 2) an excessively fast scan rate; or, 3) gain values set too low. Streaking may also result from any combination of these factors (see Figure 19.9a).

Try the following procedures to eliminate this condition:

- Reduce the setpoint voltage. This increases the amount of tapping force on the surface. This is probably the thing that will be most effective. Be careful when doing this on soft samples. The sample surface can still be disturbed even though the forces are very small.

- Reduce the scan rate. The scan rate needs to be slower in TappingMode than in contact AFM. Typically it should be around 1-3Hz.

- Increase the **Integral** and **Proportional Gains**. This will speed up the response time of the Z piezo transducer.

Figure 19.9a Images With/Without Streaks

With Streaks

Without Streaks
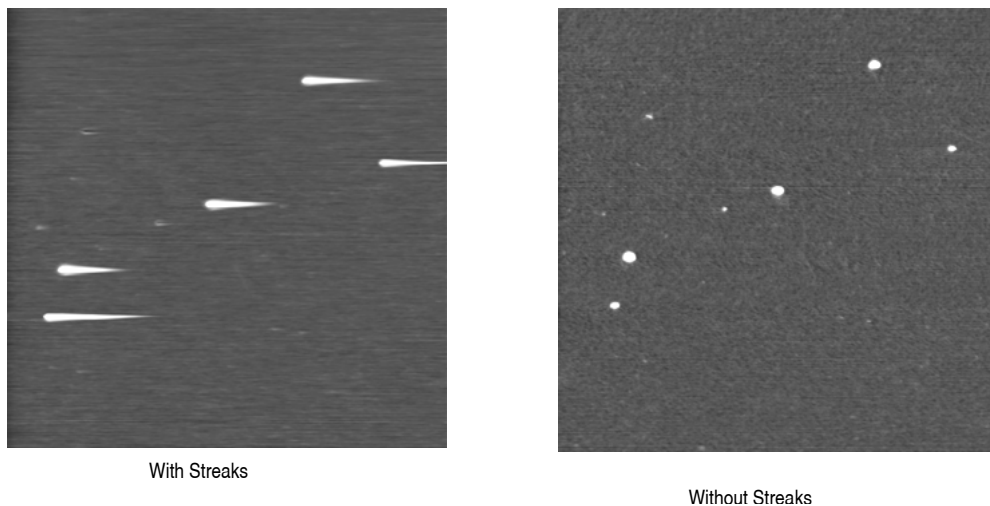
19.9.2 Lines across the image

Lines oriented in the fast scan direction can be caused by the tip sticking to the surface. This condition may be remedied by increasing the RMS voltage. Working with a larger RMS has the effect of giving the tip more energy to pull off of the surface. To correct this condition, try the following approach:

1. Use arrow keys (p) to increment the **Setpoint** voltage positively. Do this while monitoring the **Z Center Position** voltage on the display monitor. Increase the **Setpoint** voltage until the **Z Center Position** voltage jumps to the fully retracted position.

2. Note the current **Setpoint** voltage value. This value is just slightly greater than the RMS voltage currently used.

3. Increase the **Setpoint** voltage another 2V.

4. Use arrow keys to increase the **Drive Amplitude** (press 2-3 times). This will increase the RMS voltage output from the microscope. (Increasing the RMS voltage means the cantilever is oscillated harder, making it less subject to capture by a sticky surface.)

5. Use arrow keys to reduce the **Setpoint** voltage until the **Z Center Position** voltage begins to move away from the retracted position. Continue to reduce the **Setpoint** voltage until the topographic image "pops" into clear view.

19.9.3 Rings around features on the surface

This effect might also be described as the image looking as though it is half submerged beneath water (see Figure 19.9b). This is caused by operating with a drive frequency too close to cantilever resonance.

Use the arrow keys to increment the drive frequency a little lower. Do this while watching the Realtime scan. Be aware that the RMS voltage might also reduce. Repeat the steps in the "lines across the image" troubleshooting description to adjust the RMS voltage.

Figure 19.9b   Rings During High Frequency Operation

With Rings

Without Rings
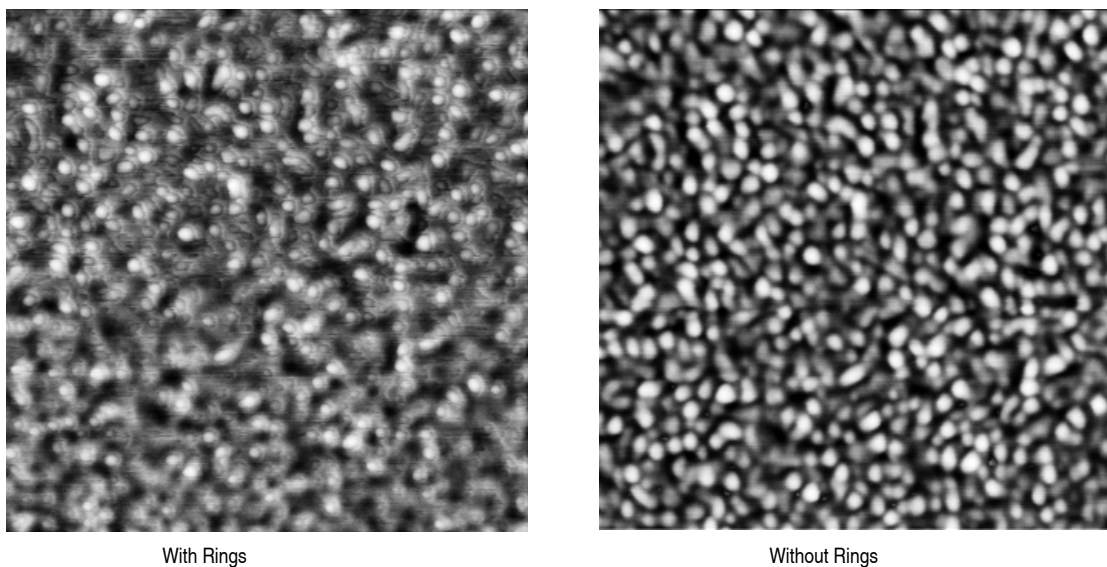
19.9.4 Multiple or repeating patterns

The tip is probably chipped (see Figure 19.9c and Figure 19.9d). This is usually caused by using too much tapping force on the surface, or because the tip encountered a feature too high to successfully traverse. If this occurs, change the tip.

> **Note:** Operating with a smaller difference between the RMS voltage and the **Setpoint** voltage means that less tapping force is being used. This may damage the tip because the tip may not be following the surface, allowing it to crash into vertical features.

Figure 19.9c Dull or Dirty Tip
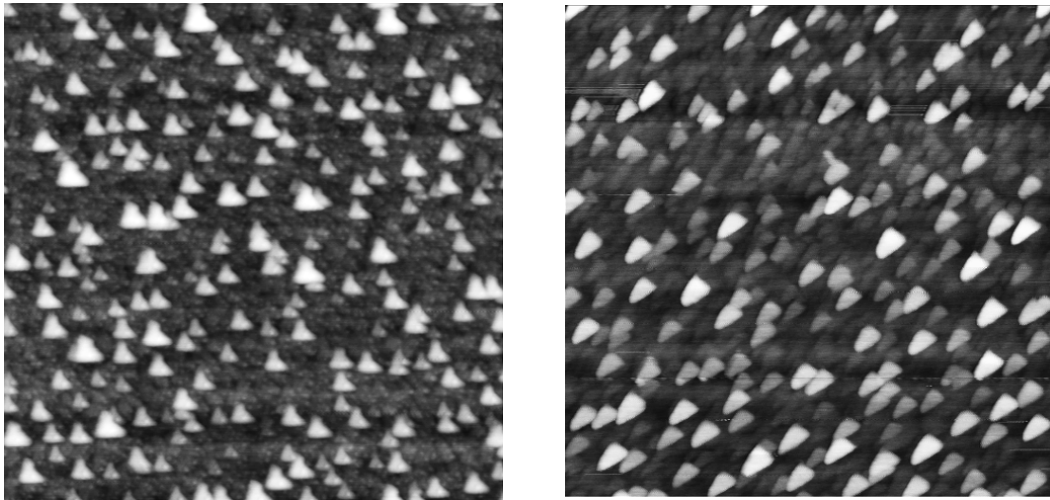
Figure 19.9d Double or Multiple Tips
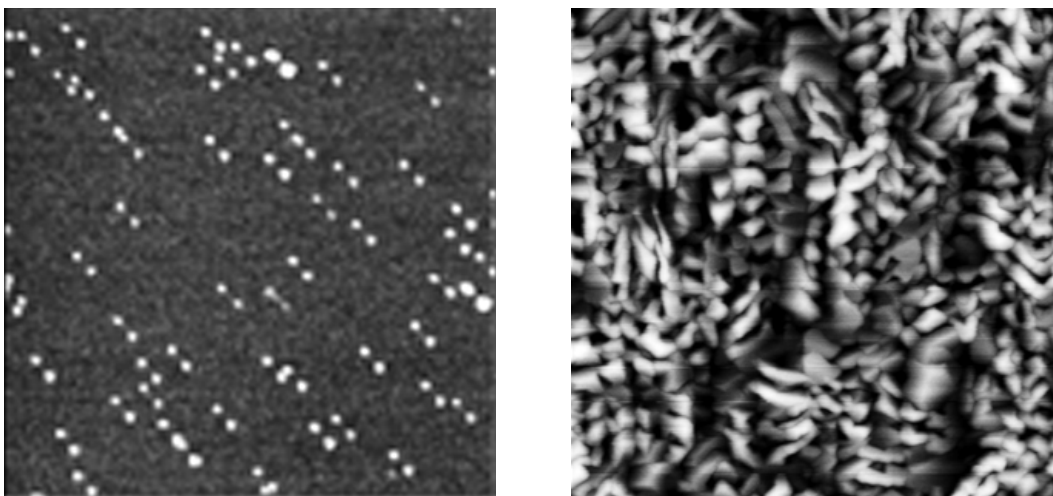
19.9.5 Image goes white or black

If the image goes white or black after a few scans and the **Z Center Position** voltage is still within range, check the Offline **Planefit** sub-command. Offline **Planefit** is normally set to **Full**. (See full description of the Offline **Planefit** command in the *Command Reference Manual*.

19.10 Fluid Imaging Troubleshooting

19.10.1 Image drifts

Occasionally, there may be some image drift due to the O-ring (used to contain fluid) sliding across the sample surface. To reduce movement of the O-ring, the fluid cell needs to be set up such that there is minimal lateral movement of the SPM head with respect to the sample once the O-ring is installed. This is best accomplished by keeping the head level and positioning the tip as closely as possible to the surface before installing the O-ring so that the lateral movement of the tip during engagement is minimized. Also, moving the positioning screws at the bottom of the optical head should also be minimized once the O-ring is installed. Further suggestions for solving this problem consist of the following:

- Use the fluid cell without the O-ring as described in Chapter 11 of this manual. This technique works best on hydrophobic surfaces. If you have a surface that does not allow a liquid drop meniscus to form or stabilize (e.g., mica), a water repellent marker may be used to outline a boundary for holding the drop. This type of marker, the "PAP PEN," can be purchased from the The Binding Site, 5889 Oberlin Drive #101, San Diego, CA 92121; 1-800-633-4484; Fax: 619-453-9189.

  Note: For applications where fluid flow is necessary, this option should not be used. Be certain to check for leaks and wipe up any spilled fluid as soon as possible to avoid damage to the scanner.

- The vertical engage scanners allow the tip to approach the sample without the lateral movement caused by the stepper motor. Thus, lateral stress on the O-ring is absent. Vertical engage scanners are available in the 125μm ("J"), and 10μm ("E") sizes. Contact Bruker for more information.

- Lightly coating the area of the O-ring which contacts the sample surface with white petrolatum or vacuum grease will allow the O-ring to slide across the surface, minimizing lateral stress. This also forms a better fluid-tight seal between the O-ring and sample. Be aware that some solvents (e.g., non-polar organic solvents) may dissolve some lubricants into the fluid.

- When positioning the O-ring on the surface, adjust the positioning knob at the base of the head to move it slightly forward. This will counter some of the lateral stress on the O-ring due to the head shifting rearward during engagement.

19.11 Warranty

All new catalog-listed standard equipment sold and/or manufactured under Bruker labels is warranted by Bruker to be free of defects in material and workmanship if properly operated and maintained. This one-year warranty covers the cost of necessary parts and labor (including, where applicable as determined by Bruker, field service labor and field service engineer transportation) during the warranty period.

Warranty period takes effect upon date of shipment. Except as detailed below, these warranties extend to parts which are components of standard catalog items and manufactured by persons other than Bruker. The manufacturer warranty covers purchased equipment incorporated into any item supplied by Bruker.

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Equipment made or modified to purchaser specifications on special order shall carry the above warranties with respect to material and workmanship, but shall be specifically excluded from any other warranties, express or implied, including those related to performance specifications. Special components shall only carry the original manufacturer warranties.

Warranty Claims

Bruker's obligation under these warranties is limited to repairing or replacing at Bruker's option defective non-expendable parts. Bruker's obligation shall not extend to defects that do not impair service. No claim will be allowed for any defect unless Bruker has received notice of the defect within thirty days following its discovery by purchaser.

Claims for Shipment Damage

No claim will be allowed for equipment damaged in shipment sold under standard terms of F.O.B. Factory. Within thirty days of purchaser's receipt of equipment, Bruker must receive notice of any defect which purchaser could have discovered by prompt inspection of equipment. In any event, Bruker shall have the option of inspection at purchaser's premises or at Bruker's plant before allowing or rejecting the claim.

Warranty Eligibility

To be eligible for the above warranties, purchaser must perform preventative maintenance in accordance with the schedule set forth in the manual provided. Bruker assumes no liability under the above warranties for equipment or system failures resulting from improper operation, improper preventative maintenance, abuse or modifications of the equipment or system from the original configuration.

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Service

Field service is available nationwide. Service and installations are performed by factory trained Bruker service personnel. Contact the Bruker sales/service office for prompt service.

Bruker Corporation
112 Robin Hill Road
Santa Barbara CA 93117
Attn.: Service Center

Phone: (805) 967-2700
Fax: (805) 967-7717

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