Dimension™ 3100 Manual

Version 4.43C
004-320-000 (standard)
004-320-100 (cleanroom)

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Document Revision History: Dimension 3100 Manual

Revision	Date	Section(s) Affected	Ref. DCR	Approval
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Dimension 3100 Manual

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Chapter 1 System Overview

1.1 Overview

The Dimension 3100 Scanning Probe Microscope (SPM) produces high-resolution, three-dimensional images by scanning a sharp tip over the sample surface. The tip is part of a flexible cantilever mounted on one end of a cylindrical piezoelectric tube mounted near the top of the microscope. Voltages applied to the X and Y electrodes on the piezoelectric tube deflect the tube horizontally to produce a precise raster scan over the sample surface. A voltage applied to the Z electrode on the piezo tube controls the vertical height of the tip. A stepper motor coupled to a lead screw translates a slide with the sample attached. A separate motor drive controls the height of the microscope and tip relative to the sample surface.

This manual details facility requirements, installation requirements and procedures, maintenance requirements and procedures, and applications used with the Dimension 3100 Scanning Probe Microscope. This chapter discusses the following:

- **How to Reach Digital Instruments Veeco:** Section 1.2
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1.2 How to Reach Digital Instruments Veeco

You may obtain technical support via telephone, fax, e-mail, or mail. Your company may have also negotiated a priority service agreement which grants access to the Priority Service Mailbox. When contacting Digital Instruments Veeco, have the NanoScope controller serial number available. The serial number is located on the back of the NanoScope controller. Place your telephone next to the microscope before calling technical support to ensure comfortable access to your system.

Consult Digital Instruments Veeco technical support staff before sending any parts for replacement or repair. All returned parts must have return authorization numbers.

Mailing Address:	Technical Support Department Digital Instruments Veeco 112 Robin Hill Road Santa Barbara, CA 93117		
Telephone:	(800) 873-9750 (805) 967-1400	Fax:	(805) 967-7717
E-mail:	help@di.com bugs@di.com		

If you have a Priority Service Agreement, contact Digital Instruments Veeco at:

24hrs Priority Service Mailbox:	(805) 882-2075	Fax:	(805) 967-7717

For assistance in other than technical support, please contact the appropriate department.

Domestic sales, product information, price quotes:

Richard Puestow (805) 967-2700 ext. 313

International sales, product information, price quotes:

Chris Carnaghi (805) 967-2700 ext. 290
or Contact your local representative.

Pricing, delivery, order information:

Mark Lien (805) 967-2700 ext. 270

Information is also available at the Digital Instruments Veeco web site:

http://www.di.com

1.3 System Overview

There are three typical configurations of the Dimension 3100 Scanning Probe Microscope (See Chapter 3 for detailed information).

- Axiom VT-103-3K with ELCON
- Axiom VT-102
- Axiom IS3K-2

The configurations provide options for acoustic and mechanical vibration isolation, as well as various options for positioning control station components. The features outlined below apply to all configurations.

1.3.1 Dimension 3100 SPM Features

Enhanced Motorized Positioning Stage

- Inspectable Area 120mm x 100mm
- Resolution: 2µm
- Unidirectional Repeatability: 3µm typical, 10µm maximum
- Bidirectional Repeatability: 4µm for x-axis, 6µm for y-axis typical for point to point motion

Integrated Dimension 3100 Controller

The Dimension 3100 controller integrates the illuminator, power supply, and air and vacuum pumps.

Optical Microscope

The optical microscope now includes a computer-controlled illuminator for easier optical focusing and zooming.

Dimension 3100 SPM Features continued...

Video Image Capture Capability

Video image capture capability allows the user to easily incorporate video images into reports and publications.

Computer System

The Dimension 3100 ships with a high quality tower-style Pentium PCI computer system.

1.4 Control Station Overview

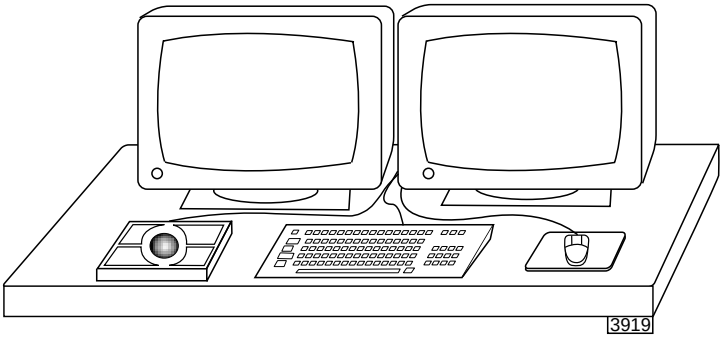
The Dimension 3100 SPM control station consists of four components: input and display devices (keyboard, trackball, monitors), computer, NanoScope controller and Dimension 3100 controller.

1.4.1 Input and Display Devices

Input and display devices include two monitors, a keyboard, mouse, and trackball (See Figure 1.4a). These devices convey information signals to the computer to operate the software and SPM. Depending on the configuration of the system, the input and display devices are located atop the control station table (Axiom VT-103-3) or contained within an enclosure (Axiom IS3K-3).

CAUTION: The monitors contain a hazardous voltage of 120V.

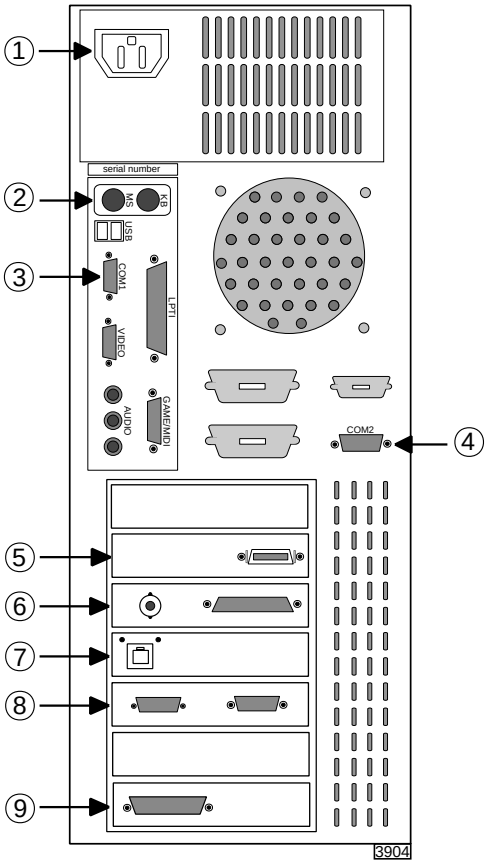
Figure 1.4a Dimension 3100 Input and Display Equipment



1.4.2 Computer

The computer is the main control unit of the Dimension 3100 SPM system; it supports a 100MB Zip® disc drive, CD-ROM drive and 1.44MB floppy disc drive (See Figure 1.4b). 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.

Figure 1.4b Computer (rear view)

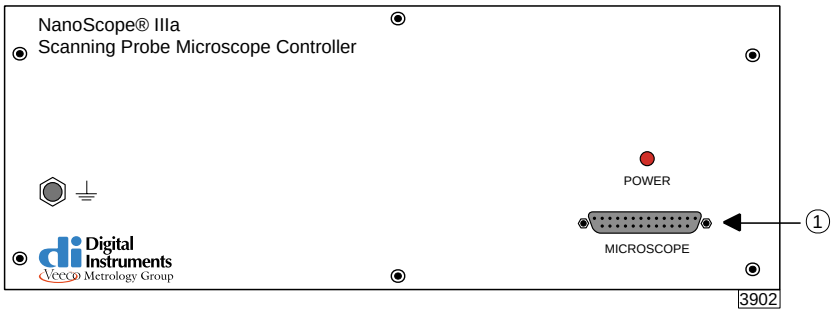


Control Station Overview continued...

1.4.3 NanoScope IIIa Controller

The NanoScope IIIa controller controls the microscope head and scanning. The NanoScope IIIa controller is controlled via a 25-pin D cable connection between it and the computer (See Figure 1.4c).

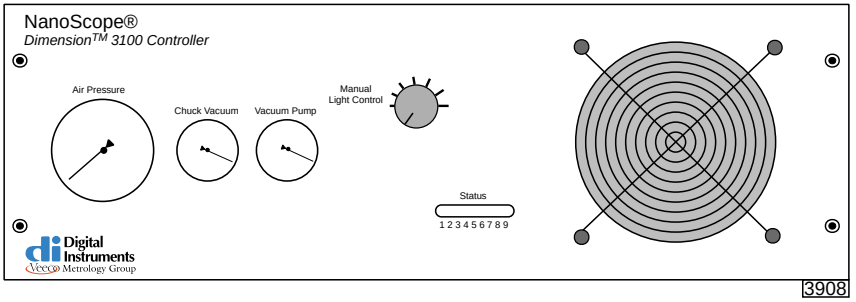
Figure 1.4c NanoScope IIIa Controller (front view)



1.4.4 Dimension 3100 Controller

The Dimension 3100 controller controls the vacuum and air supply and optics illumination. The Dimension 3100 controller is controlled via a serial cable connection between it and the computer (See Figure 1.4d). The Dimension 3100 controller features gauges on the front panel to indicate vacuum and positive pressures. The Dimension 3100 controller channels positive pressure to the underside of the chuck during X-Y movements, allowing the chuck to glide smoothly over the granite.

Figure 1.4d Dimension 3100 Controller (front view)



CAUTION: Do not turn the Dimension 3100 controller on its side. Turning the controller on its side activates an alarm and turns the vacuum pump off.

ATTENTION: Afin d'éviter de sérieux dommages mécaniques, ne tournez pas le boîtier de contrôle sur le côté. Tourner le boîtier sur le côté déclencheune alarme et coupe la pompe à vide.

WARNHINWEIS: Um ernsthafte mechanische Beschädigungen zu vermeiden stellen Sie die Dimension Control Box bitte nicht auf die Seite. Dies würde einen Alarm auslösen und die Vakuumpumpe ausschalten.

Dimension 3100 Controller continued...

CAUTION: The Dimension controller features a special thermostat that sets off an alarm if the controller overheats (> 40° C). The Dimension controller will overheat if the controller ventilation holes are blocked or if the controller is exposed to heat from an outside source.

The Dimension 3100 controller houses the following components:

Power Supply

The power supply is preconfigured at the factory. Verify voltage compatibility before plugging the system into the power source.

Vacuum and Air Pumps

The vacuum hose assembly provides vacuum and positive pressure control.

Illumination System

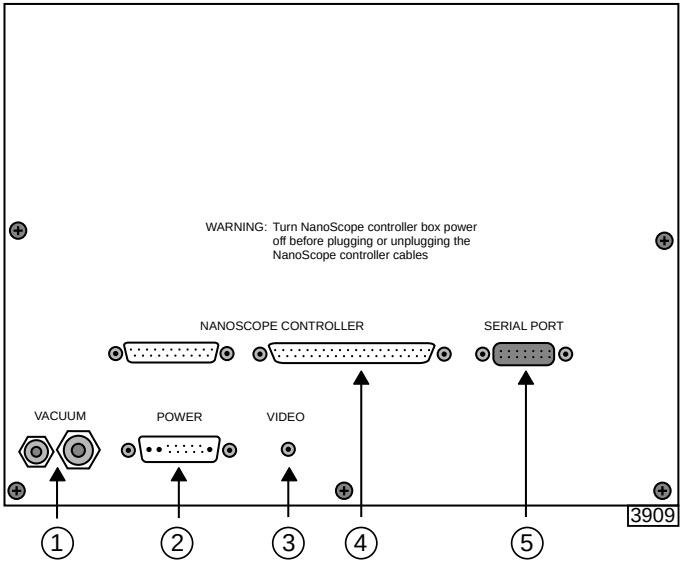
Illumination can be computer or manually controlled. The knob located on the front of the Dimension 3100 controller regulates manual illumination control. For computer control, there is an "illumination" parameter in the **Other Controls** panel to control brightness.

1.5 Dimension 3100 SPM Overview

1.5.1 Dimension 3100 Microscope Electronics Box

The Dimension 3100 Microscope Electronics Box moderates all functions of the microscope, including vacuum and air supply, motor power, optic image signals, microscope control, and stage motor control (See Figure 1.5a).

Figure 1.5a D3100 Microscope Electronics Box (rear view)



The Dimension 3100 Microscope Electronics Box houses the following components:

Main SPM Circuit Board

This smaller circuit board mounts on a larger board and contains switches to route feedback signals from the head. The photodiode signals combine and amplify in various ways to provide the desired information. The main SPM circuit board also contains the circuits to control the microscope in Tapping Mode and non-Contact/MFM modes. The RMS detection circuit for the cantilever vibration signal and cantilever oscillation drive circuit are on this board.

Dimension 3100 Microscope Electronics Box continued...

Larger SPM Mother Board

This larger circuit board provides local computer control of the SPM electronics, microscope motors and vacuum control.

Two Stepper-motor Drive Board

One stepper-motor drive board operates the Z-stage, focus and zoom motors. The second stepper-motor drive board powers the X-Y stage motors.

Vacuum Power Switch

The vacuum power switch toggles with an **ON/OFF** control switch for sample vacuum capabilities.

Note: Dimension 3100 SPMs equipped with Extender™ Electronics Module phase attachments utilize a slightly different electronics architecture than standard models. For more information, contact Digital Instruments Veeco.

1.5.2 Optics and Motors Overview

The optic system assists you in locating the cantilever and tip relative to the sample. The NanoScope software uses this information to engage the tip on the sample surface at the desired location. The system automatically focuses on most samples by adjusting the SPM height, however, the trackball is available for manual focus control.

The objective mounts on a translational tilt stage. The objective can be translated along the optical axis with a servo motor (focus motor) to adjust the focal point approximately 1.5 mm above the cantilever to approximately 3.5 mm below. The range of focus below the cantilever allows you to focus on the sample while leaving up to 3.5 mm clearance between the tip and sample.

1.5.3 Stage System

The improved Dimension 3100 X-Y stage provides substantially better positioning repeatability (3µm unidirectional and 4-6µm bidirectional). The stage is more than twice as fast for moving from one location to another. The improved trackball response also makes it easier to locate features of interest for imaging.

The X-Y stage permits micron-scale positioning of samples beneath the tip with a 120mm (X direction) by 100mm (Y direction) travel range. The stage ships with chucks for fully accessible disks up to 8-inches in diameter.

The Dimension 3100 motorized Z-stage provides accurate, automatic tip engagement and approach. The rigidity of the Z-stage permits low noise and high accuracy imaging. A motorized lead screw drives the Z-stage for coarse approach of the head to the sample. This configuration allows you to image samples of different thicknesses with little difficulty.

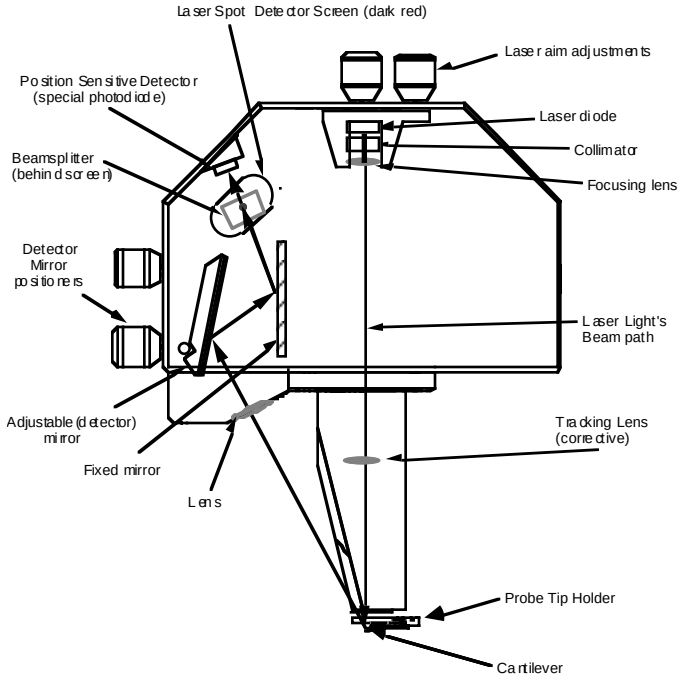
1.5.4 Dimension SPM Head

The Dimension SPM head provides accurate imaging of a stationary sample while scanning the integrated detector probe assembly above the sample. The Dimension SPM head allows optical correction of the laser beam path to track the movement of the probe while scanning under the fixed laser beam assembly. The Dimension SPM head is effective in imaging samples too large or heavy to scan by movement of the sample.

Figure 1.5b depicts the optical path of the laser beam inside a Dimension SPM head. The Dimension SPM head places a corrective, tracking lens within the scanner tube to stabilize the laser beam focal point atop the scanning cantilever. This sharply reduces bowing and attenuation artifacts due to cantilever scanning across the laser beam’s otherwise stationary focal plane. Patents covering the technology developed in the Dimension SPM head are pending.

Dimension SPM Head continued...

Figure 1.5b Dimension SPM Head



The Dimension SPM head scans the tip and generates the cantilever deflection or probe feedback signal for the different imaging modes. A quad photodetector detects the beam emitted by the laser diode (1.0 mW max at 670 nm) as it reflects off the cantilever. The integrated scanner head consists of the following subassemblies:

Preamp Board

The preamp board is located on the inside of the Dimension 3100 SPM microscope head. The preamp board contains a preamplifier circuit for both photodetector signals, a laser diode power supply circuit that regulates the output of the laser, and ±12 volt regulators for the preamp circuit. The voltage regulators include current limiting to protect the circuit. The preamp board is in turn connected to a 21-pin male connector cable plugged into the socket on the front of the stage control electronics box.

Laser Diode Stage

A kinematic tilt stage positions the laser beam on the cantilever. The tilt stage consists of the laser diode, collimator, focusing lens, base plate, and the X and Y laser diode adjustment knobs. The X laser diode adjustment knob moves the beam parallel to the major axis of the cantilever substrate. The Y laser diode adjustment knob moves the beam perpendicular to the major axis of the cantilever substrate. The adjustment knobs are described in more detail in Chapter 7.

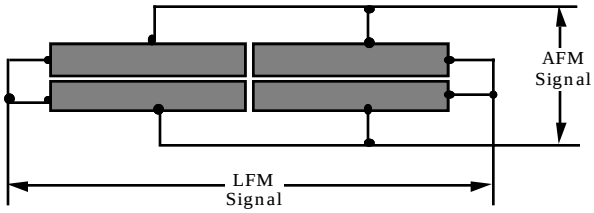
Adjustable Detector Mirror

An adjustable mirror positions the reflected laser spot relative to the four photodetector elements using a kinematic mount and the photodetector mirror adjustment knobs. The photodetector mirror adjustment knobs assist the user in adjusting the position of the mirror to maximize the SUM signal and set the deflection signals.

Photodetector

The four elements of the quad photodetector combine to provide different information depending on the operating mode. In all modes the four elements combine to form the SUM signal. The amplified differential signal between the top two elements and the two bottom elements provides a measure of the deflection of the cantilever. This differential signal is used in Contact AFM mode. The differential signal feeds into an RMS converter (or phase module if attached) for Tapping mode operation. Similarly, the amplified differential signal between the sum of the two left photodiodes and the sum of the two right photodiodes provides a measure of the torsion in the cantilever and (often used in Lateral Force Microscopy). Figure 1.5c illustrates the arrangement of the photodiode elements in the Dimension 3100 SPM head.

Figure 1.5c Quad Photodetector Arrangement



Dimension SPM Head continued...

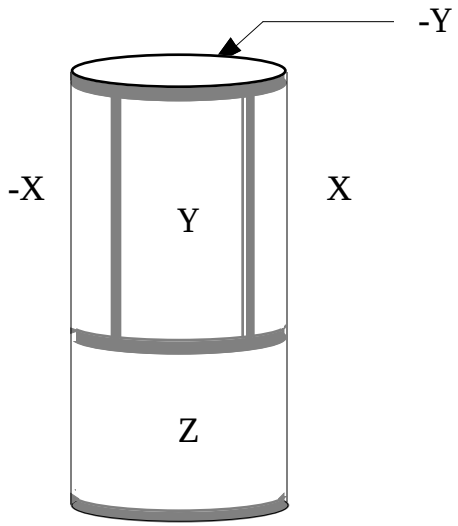
Beamsplitter and Laser Spot Detector Screen

The beamsplitter diverts some of the laser light directed towards the photodetector toward the Laser Spot Detector Screen. This screen provides visual indication of the condition of the reflected spot and its orientation relative to the photodetector.

Scanner Piezo Tube

Figure 1.5d depicts the electrode configuration used on the scanner piezo tube in the Dimension SPM head. The electrodes are oriented as shown when viewing the Dimension 3100 SPM from the front. With the **Scan angle** parameter in the control panel set to **0.00**, the fast-scan direction is parallel to the front or in the direction of the X-axis.

Figure 1.5d Dimension Head Scanner



The end of the scanner allows easy removal of the imaging probe assembly. This permits loading the tip on a separate fixture such that head removal becomes unnecessary.

Scanner Piezo Tube continued...

Table 1.5a: Scanner Piezo Tube Specifications

Travel (approximate scan size)	
x-axis	90 µm
y-axis	90 µm
z-axis	6 µm
Electronic Resolution	16-bit (all axes)
Accuracy	
typical	1%
maximum	2%
Orthogonality	2 degrees
Uncorrected Z bow	
90 µm scan size	50 nm
10 µm scan size	2 nm

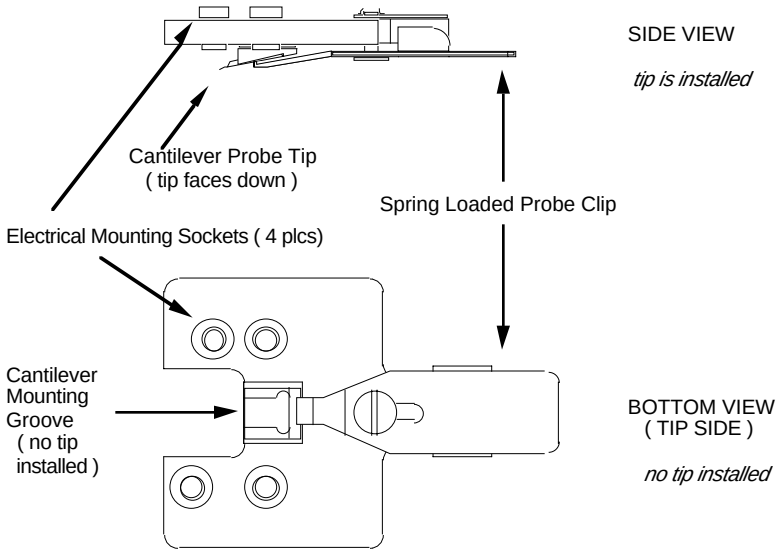
1.5.5 Cantilever Holder

The cantilever holder is a small printed circuit card or plexiglas block that holds the cantilever firmly at the proper angle. The standard cantilever holder is used for operation in air, while a clear glass cantilever holder is used for fluid cell operations. The standard cantilever holder includes the cantilever piezoelectric stack and the electrical contacts to the drive circuits. The fluid cell cantilever holder does not contain a piezoelectric resonator locally. Cantilever holders include gold-plated spring sockets which mate with the gold-plated pins at the end of the scanner. There is also a spring-loaded clip to secure the cantilever probe to the cantilever holder assembly.

You can easily change the cantilever holder on the microscope for different operating modes (See Table 1.5b). The standard cantilever holder shown in Figure 1.5e contains a piezoelectric stack to oscillate the cantilever when operating in Tapping Mode. The same cantilever holder is used for Contact AFM but no voltage is applied to the piezo stack.

Cantilever Holder continued...

Figure 1.5e Standard Cantilever Holder



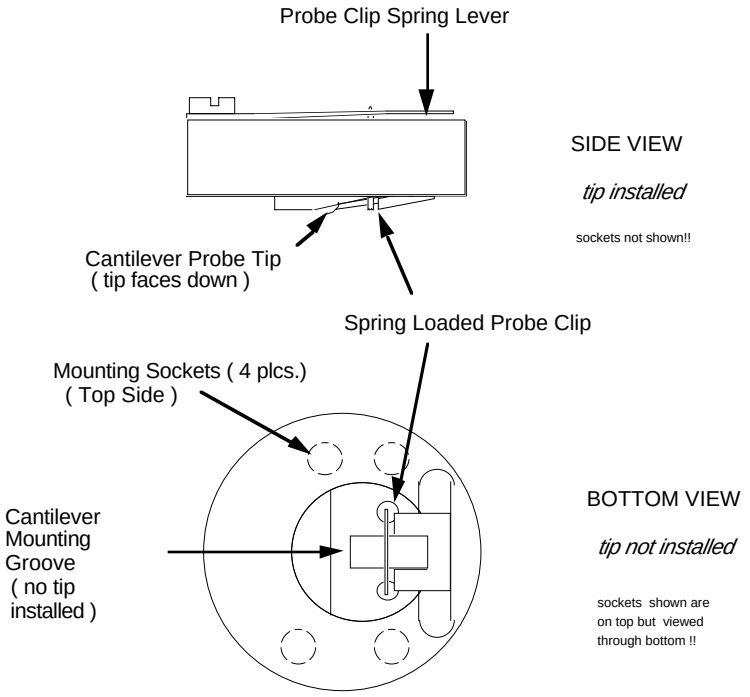
The fluid cell cantilever holder shown in Figure 1.5f provides an optically transparent cover over the back of the cantilever to maintain the optical path of the laser beam constant when the tip is submerged in a fluid medium. If the fluid medium was not covered by the fluid cell, imaging with the tip submerged in fluid would be impossible due to variable surface scattering effects. These effects cause the reflected laser beam to move randomly during imaging, rendering the reflected signal useless for SPM imaging.

Table 1.5b: Cantilever Holder Specifications

Holders	Specifications
Standard	Tapping Mode, Contact AFM, MFM
Optional	STM and Force Modulation
Fluid Cell	Fluid Contact AFM, Fluid Tapping Mode

Cantilever Holder continued...

Figure 1.5f Fluid Cell Cantilever Holder



1.5.6 Video Zoom Microscope

The integrated zoom optical microscope's features include:

- 10x objective (long working distance)
- 2X TV camera tube
- Motorized zoom system
- 410-1845 x magnification range with 13-inch monitor and corresponding field of view of 180—810 µm
- Motorized focus
- Through-the-lens illumination
- Color video camera
- Focus tracking and automated engagement

1.6 Sample Size & Handling

Samples may be up to 8 inches in diameter and 0.5-inch thick using a multipurpose, 8-inch chuck.

Table 1.6a: Sample Specifications

	Specifications
Max Sample Size	Wafers and disk media: 8-inch dia. x 0.5" thick.
Inspectable Area	100mm x 125mm typical Includes interchangeable adapters for centering hard disks and removable wafer locating pins. Vacuum pump included.
Small Samples	Magnetic holder available for mounting small samples on mounting pucks (< 15 mm dia. typ.).
Silicon Wafers	Silicon-dioxide-coated chuck accommodates 2-inch, 100, 125, 150 and 200 mm wafers.
Wet Samples	Optional fluid cell allows immersion of microscope head to max depth of 2 mm.
Chuck Vacuum	The chuck vacuum pneumatics secure samples to the chuck using a separate toggle switch at the front of the stage control electronics box.

1.7 Facilities Specifications

Compliance with the requirements and specifications outlined in Chapter 3 is essential before installation and operation of the Dimension 3100 SPM. Chapter 3 details facility site requirements, safety requirements, and configuration options for the Dimension 3100 SPM.

1.8 Applications

Several applications can be applied using the Dimension 3100 SPM. For specific information regarding these applications, please refer to the appropriate chapters in this manual. The following is a list of common applications used with the Dimension 3100 SPM:

- Contact AFM
- Contact AFM in Fluids
- Tapping Mode
- Tapping Mode in Fluids
- Lateral Force Mode
- Force Imaging
- Interleave Scanning
- Magnetic Force Imaging
- Electric Techniques
- Calibration

1.9 Maintenance and Troubleshooting

Refer to Chapter 18 for detailed information regarding maintenance and troubleshooting issues for the Dimension 3100 SPM.

Chapter 2 Safety

2.1 Overview

This chapter details the safety requirements involved in installation of the Dimension 3100 Scanning Probe Microscope. Specifically, these safety requirements include safety precautions, non-physical conditions, and equipment safety applications. Training and compliance with all safety requirements is essential during installation and operation of the Dimension 3100 SPM.

Specifically, this chapter discusses the following areas of information:

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 tempêratures. 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.

2.3 Safety Precautions

Because the Dimension 3100 SPM features independently 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.
ATTENTION:	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.
ATTENTION:	Il est impératif de suivre les prérogatives imposées tant au niveau gouvernmental 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.

Safety Precautions continued...

CAUTION:	Please contact Digital Instruments Veeco before attempting to move the Dimension 3100 SPM system.
ATTENTION:	Il est impératif de contacter Digital Instruments Veeco avant de déplacer le Dimension 3100 SPM.
VORSICHT:	Bitte kontaktieren Sie Digital Instruments Veeco bevor Sie das Dimension 3100 SPM System transportieren.

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. (Digital Instruments Veeco service personnel, *only*.)
ATTENTION:	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 Digital Instruments Veeco, 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 Digital Instruments Veeco Personal.)

Safety Precautions continued...

WARNING:	Never alter pneumatics or wiring on the Dimension 3100 SPM.
ATTENTION:	Ne jamais toucher les cables et l'installation pneumatique sur le boîtier accoustique du Dimension 3100.
WARNUNG:	Ändern Sie niemals etwas am pneumatischen System oder der Verdrahtung der Schallschutzhaube.

WARNING:	The Dimension 3100 SPM contains a diode laser with an output of less than 1.0mW at 670nm.
ATTENTION:	Le microscope "Dimension 3100 SPM" est équipé d’une diode laser dont la puissance de sortie est inférieure à 1mW à 670nm.
WARNUNG:	Das Dimension 3100 SPM ist mit einem Halbleiterlaser ausgerüstet, dessen Ausgangsleistung kleiner ist als 1.0mW bei 670nm.

WARNING:	Do not use acetone to clean the Dimension 3100 SPM.
ATTENTION:	Ne pas utiliser d’acétone pour nettoyer le Dimension 3100 SPM.
WARNUNG:	Bitte verwenden sie kein Azeton um das Dimension 3100 SPM zu reinigen.

Safety Precautions continued...

WARNING: The Dimension 3100 SPM uses a halogen lamp to illuminate samples. Exposure to non-ionizing radiation from this lamp is well within the current exposure guidelines published by the American Conference of Governmental Industrial Hygienists (ACGIH). Typical IR exposure to the user from the sample illuminator is less than 3 mW/cm^2. UV radiation is not detectable.

ATTENTION: Le microscope Dimension 3100 SPM est équipé d'une lampe halogène pour illuminer les échantillons. L'exposition aux radiations non-ionisantes de cette lampe est trés inférieure aux recommandations publiées par “l' American Conference of Governmental Industrial Hygienists (ACGIH)”. Les radiations IR dues à cette lampe sont typiquement inférieures à 3 mW/cm^2. Les radiations UV ne sont pas détectables.

WARNUNG: Das Dimension 3100 SPM System ist mit einer Halogenlampe ausgestattet, um die Probe zu beleuchten. Die von dieser Lampe ausgehende Strahlenbelastung der nichtionisierenden Strahlung liegt weit unter den publizierten Richtwerten der American Conference of Govermental Industrial Hygienists (ACGIH). Die fuer den Benutzer typische IR Strahlenbelastung der Beleuchtungseinheit ist kleiner als 3 mW/cm^2. UV Strahlung ist nicht nachweisbar.

Safety Precautions continued...

2.3.2 Microscope

To avoid operator injury and equipment damage, observe the following cautions regarding the Dimension 3100 microscope.

CAUTION:	Stage microscopes feature an automated X-Y stage and Z-axis capable of programmed movement. The movements of all axes are slow, but are capable of exerting high forces. A hand caught in the stage of a Dimension 3100 SPM could be injured severely.
ATTENTION:	La platine des microscopes est automatisée dans les directions X, Y et Z, et est programmable. Les mouvements selon ces 3 axes sont lents, but peuvent excercer des forces importantes. La main d'un utilisateur pourrait être sévèrement endommagée si elle se trouvait coincée par cette platine.
VORSICHT:	Mikroskope mit automatisiertem Probentisch können programmierte Bewegungen in X-, Y- und Z-Richtung durchführen. Die Bewegungen in allen drei Richtungen sind langsam, können aber sehr große Kräfte ausüben. Eine Hand, die vom Probentisch des Dimension 3100 SPMs erfaßt wird, kann leicht ernsthaft verletzt werden.

Microscope Safety Precautions continued...

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 Digital Instruments Veeco for repair or replacement.

ATTENTION: 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/ Veeco 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 Digital Instruments Veeco.

CAUTION: 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.

ATTENTION: 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.

VORSICHT: 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.

Microscope Safety Precautions continued...

ATTENTION:	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.
ACHTUNG:	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.

2.3.3 Sample Safeguards

ATTENTION:	**Do not** change samples in the middle of operation. Verify that the stage is clear of tools, objects, and debris at all times. Use alcohol wipes periodically to keep the stage clean of dust.
ATTENTION:	**Ne pas** changer d'échantillon en cours d'utilisation. Vérifier que la platine n'est pas encombrée, par des outils par exemple. Employer des tampons d'alcool régulièrement pour dépoussiérer la platine.
ACHTUNG:	Tauschen Sie **keine** Proben aus, während sich das System im Betrieb befindet. Der Probentisch sollte von Werkzeug, anderen Objekten und Überresten ständig freigehalten werden. Benutzen Sie ein mit Alkohol getränktes Tuch, um den Probentisch regelmäßig von Staub zu reinigen.

Sample Safeguards continued...

ATTENTION:	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.
ACHTUNG:	Alle Sperrvorrichtungen des Systems sind dazu vorgesehen, Personal und Probe zu schützen. Versuchen Sie **nicht**, Sperrvorrichtungen zu umgehen.

2.4 Ergonomics

The Dimension 3100 SPM design promotes compatibility in the integration of user personnel and equipment within a semiconductor manufacturing environment. Specifically, the ergonomics of the Dimension 3100 SPM design prevent personal injury, equipment damage, and minimizes procedural errors.

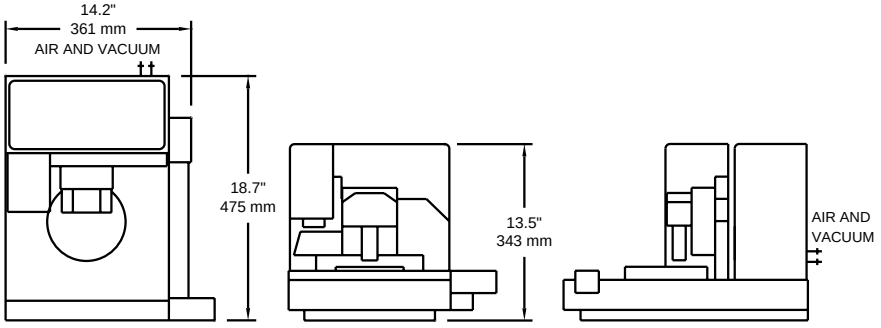
2.5 Non-Physical Conditions

Non-physical conditions that may affect the performance of the Dimension 3100 SPM are vibration and noise. See Chapter 3 for detailed condition specifications.

2.6 Equipment Safety Applications

2.6.1 Dimension 3100 SPM Facility Requirements

Figure 2.6a Dimension 3100 SPM Footprint



2.7 Power-up Sequence (Installation and Service Only)

The following section is required *only* during installation or after servicing and should NOT be used by untrained personnel. For a description of normal power-up procedures, see Section 2.8.

2.7.1 Pre Power-up Checklist

ATTENTION: You must complete the pre power-up checklist before proceeding with facilities connections and the power-up procedure.

ATTENTION: Vous devez effectuer une checklist pour vérifier la mise sous tension avant de mettre en place les connections et commencer la procédure de mise sous tension.

ACHTUNG: Gehen Sie durch die folgende Checkliste („Pre-Power-up Checklist”), bevor Sie Verbindungen zum Netzanschluß und zu den Versorgungsleitungen herstellen und das System einschalten.

Pre-Installation

_____ 1. Verify that there is a minimum installation space of 10' (-305 cm) wide x 7.5' (–230 cm) deep.

Note: Refer to Chapter 3 for facilities requirements specific to the various Dimension 3100 configurations.

_____ 2. Verify that AC power (100V, 120V, 220V-240V single phase) is available to the system.

_____ 3. Verify that clean dry air is available at 60-100 psi to the vibration isolation table.

_____ 4. Verify that vacuum is available at ≥24" Hg to the system (IS3K-2 only).

Pre Power-up Checklist continued...

Module Installation

_____ 1. Uncrate the Dimension 3100 SPM system components.

_____ 2. Verify all facilities requirements outlined in Chapter 3 are met.

_____ 3. Install the Dimension 3100 SPM by completing the following:

- _____ Set the vibration isolation table or microscope platform in place.
- _____ Transition the Dimension 3100 SPM to the final operating location.
- _____ Secure the chuck base and stage.

_____ 4. Install the control station by completing the following:

- _____ Set the table to be used as the control station next to the Dimension 3100 SPM.
- _____ Place the input and display devices on the user console (monitors [2], mouse, keyboard and trackball).
- _____ Place the computer and controllers on the control station.

Pre Power-up Checklist continued...

Connections

ATTENTION: Make sure to power-down all systems at this point to ensure that there is no risk of electrical shock.

ATTENTION: Vérifiez que tous les systèmes ne soient plus sous tension à ce moment, et assurez vous qu'il n'y a pas de risque de choc électrique.

ACHTUNG: Überzeugen Sie sich, daß zu diesem Zeitpunkt alle Geräte ausgeschaltet sind, um die Gefahr eines elektrischen Schocks auszuschließen.

_____ 1. Connect the control station extensions.

- _____ Computer AC power cable to power strip
- _____ Monitor power cables (2) to power strip
- _____ Monitor video cable to computer
- _____ Keyboard to computer
- _____ Mouse to computer
- _____ Trackball to computer

Pre Power-up Checklist continued...

_____ 2. Connect the Dimension 3100 SPM unit extensions.

- _____ Serial cable (12') from computer to Dimension 3100 SPM back panel
- _____ BNC cable from computer to Dimension 3100 SPM back panel
- _____ RJ45 LAN cable from computer to host
- _____ Serial cable (6') from computer to Dimension 3100 controller
- _____ 37-pin D cable from computer to Dimension 3100 controller
- _____ Vacuum hose assembly from Dimension 3100 controller to Dimension 3100 SPM back panel
- _____ 25-pin D cable from computer to NanoScope IIIa controller
- _____ NanoScope IIIa controller AC power cable to power strip
- _____ DC power cable from Dimension 3100 controller to Dimension 3100 SPM back panel
- _____ Fiber optic cable from Dimension 3100 controller to Dimension 3100 SPM objective

Pre Power-up Checklist continued...

Final Installation

ATTENTION: The objective, Dimension head, and vacuum sample chuck should be the final equipment installed due to the sensitive nature of these components.

ATTENTION: L'objectif, la tête du Dimension and la platine porte-échantillon devraient être installés en dernier, à cause de leur fragilité.

ACHTUNG: Das Objektiv, der Mikroskopkopf und der Vakuumprobenhalter sind empfindliche Komponenten des Dimension SPMs, und sollten als letztes installiert werden.

_____ 1. Install the objective.

_____ 2. Install the Dimension head.

_____ 3. Mount the vacuum sample chuck.

Power-up Sequence (Installation and Service Only) continued...

2.7.2 Power-up the Dimension 3100 SPM *(Service and Installation Only)*

Note: Refer to Figure regarding the following power-up instructions.

1. Verify that all system components are plugged into AC power with the correct voltage.
2. Verify that all cables are connected properly.
3. Power-up the computer using the push-button switch located on the front of the computer.
4. Power-up the monitors (2) using the push-button switches located on the front of the monitors.
5. Power-up the NanoScope IIIa controller using the power switch located on the rear of the NanoScope IIIa controller.
6. Power-up the Dimension 3100 controller using the power switch on the rear of the Dimension 3100 controller.

2.7.3 Power-up Checklist *(Service and Installation Only)*

Power-up *(Installation Only)*

_____ 1. Connect the facilities.

_____ Vacuum (VAC): ≥24”Hg (IS3K only)

_____ Clean dry air (CDA): 60-100psi

_____ 2. Verify that all system components are plugged into AC power with the correct voltage.

_____ 3. Verify that all cables are connected properly.

_____ 4. Verify that the computer, controllers, and monitors power-up simultaneously.

2.8 Power-Up Sequence (Normal Usage)

2.8.1 Prepare the System for Power-up *(Normal Usage)*

1. Verify that all system components are plugged into AC power with the correct voltage.
2. Verify that all moving parts are free of obstructions.
3. Verify that all cables are connected properly.
4. Power-up the computer using the push-button switch located on the front of the computer.
5. Power-up the monitors (2) using the push-button switches located on the front of the monitors.
6. Power-up the NanoScope IIIa controller using the power switch located on the rear of the NanoScope IIIa controller.
7. Power-up the Dimension 3100 controller using the power switch on the rear of the Dimension 3100 controller.

2.8.2 Power-up Checklist *(Normal Usage)*

Power-up *(Normal Usage)*

_____ 1. Verify that all system components are plugged into AC power with the correct voltage.

_____ 2. Verify that all moving parts are free of obstructions.

_____ 3. Verify that all cables are connected properly.

_____ 4. Verify that the computer, controllers, and monitors power-up simultaneously.

2.9 Software Power-up

ATTENTION: At this time, all pre power-up and power-up instructions must be completed before continuing.

ATTENTION: A ce moment, toutes les étapes avant la mise en tension et de mise en tension doivent être effectuées avant de continuer.

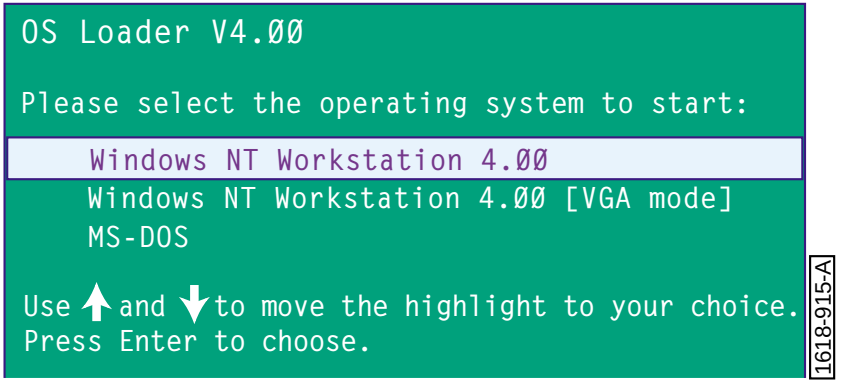
ACHTUNG: An dieser Stelle müssen die „Pre Power-up” und „Power-up” Checklisten komplett sein, bevor Sie fortfahren.

2.9.1 Select Windows NT Workstation 4.00

1. From the control monitor, select the `WINDOWS NT WORKSTATION 4.00` operating mode (See Figure 2.9a).

Note: Do not use the VGA mode. The system should select the correct operating system automatically.

Figure 2.9a Select Operating Mode



Software Power-up continued...

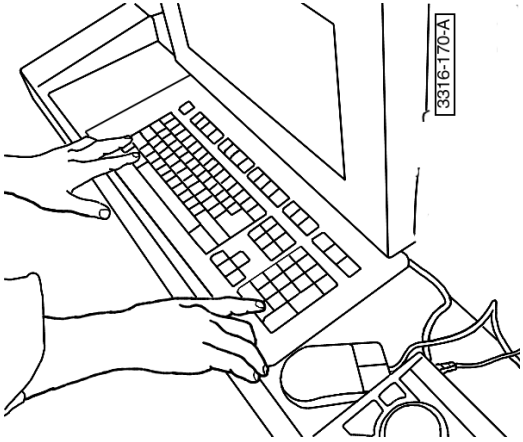
2.9.2 Press CTRL-ALT-DELETE

1. Press **CTRL-ALT-DELETE** to log into Windows NT (See Figure 2.9b).

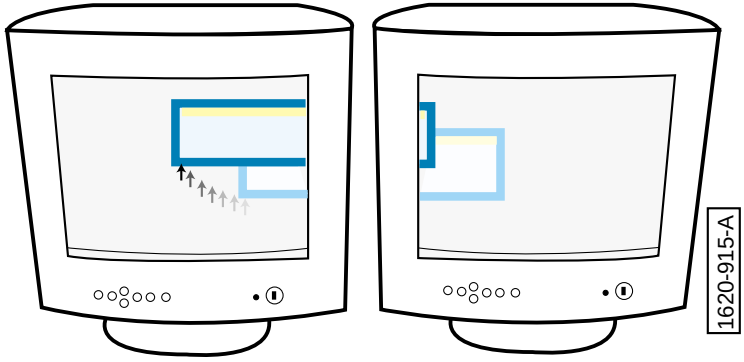
   Note: The screen begins with the panel split across both screens.

2. Drag the panel to one screen for use (See Figure 2.9b).

Figure 2.9b Log into Windows NT



CTRL-ALT-DELETE



2.9.3 Log On

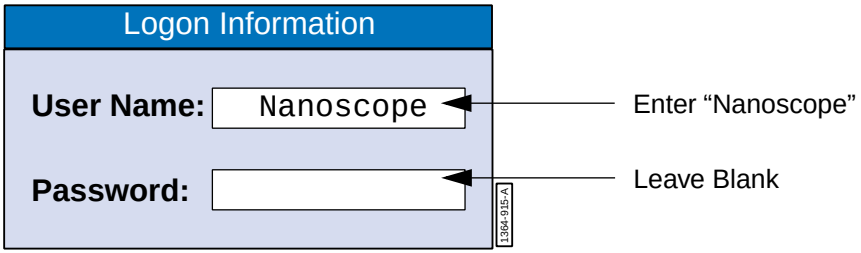
1. In the **Logon Information** window, enter the default settings into the User Name and Password fields (See Figure 2.9c).

   a. **User Name:** NANOSCOPE

   b. **Password:** Leave *blank*

   Note: If you are unable to log on, verify with the process engineer that the password has not been changed. The system is shipped from the factory with a blank password.

Figure 2.9c Logon Window



2.9.4 Start the NanoScope Software

1. Go to the desktop and click on the NanoScope icon or select the **C:\SPM\z.exe** file to start the NanoScope software (See Figure 2.9d).

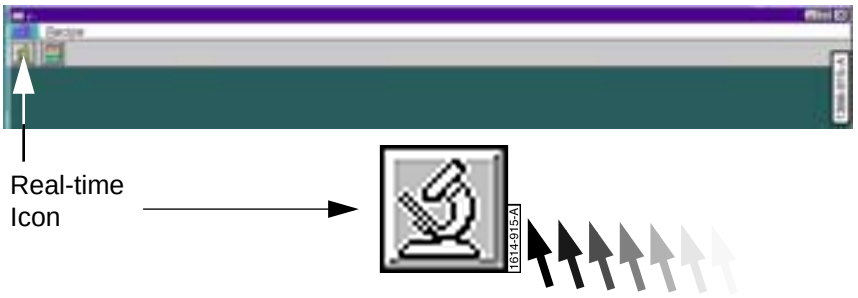
Figure 2.9d Select the NanoScope Icon

Software Power-up continued...

2.9.5 Select Real-Time

1. Click the Real-time icon, or select **Real-time** from the DI menu (See Figure 2.9e). The system automatically initializes for approximately one minute.

Figure 2.9e Select the Real-time Icon



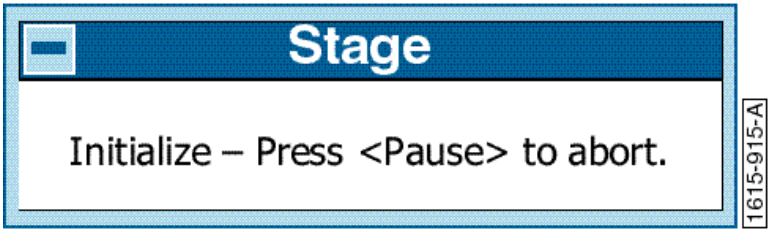
The stage may need to be initialized any time the system or one of its components has been powered-down. This allows the stage controller and computer to locate home positions and reset the coordinate system of the stage. If the focus surface and locate tip icons  are grayed out, the stage needs to be initialized.

2.9.6 Begin Stage Initialization

1. Click on **Real-time**➪**Stage**➪**Initialize**.

Note: Various axes of motion move to home positions (Zoom, Focus, and Z axes) during initialization. The control screen displays a status panel to guide the user through the initialization process (See Figure 2.9f).

Figure 2.9f Status Panel



2.9.7 Software Power-up Checklist

Software Power-up Checklist

_____ 1. Select the **Windows NT workstation 4.00**.

_____ 2. Press **CTRL-ALT-DELETE**.

_____ 3. Log on using your user name and password.

_____ 4. Start the NanoScope software.

_____ 5. Select Real-time.

_____ 6. Begin stage initialization if necessary.

2.10 Hazard Labels

The Dimension 3100 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 Dimension 3100 SPM:

2.10.1 Laser Warning Labels

Laser Explanatory Label

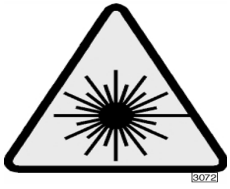
The **Laser Explanatory Label** (See Figure 2.10a) indicates that the area to which the label is affixed is affected by a laser. The Laser Explanatory Label is affixed on the Dimension SPM head.

Figure 2.10a Laser Explanatory Label

Laser Warning Label

The **Laser Warning Label** (See Figure 2.10b) indicates that the area to which the label is affixed is affected by a laser. The Laser Warning Label is affixed on the Dimension SPM head.

Figure 2.10b Laser Warning Label



Noninterlocked Protective Housing Label

The **Noninterlocked Protective Housing Label** (See Figure 2.10c) indicates that the area to which the label is affixed is affected by a laser. The Noninterlocked Protective Housing Label is affixed to the manual access door.

Figure 2.10c Noninterlocked Protective Housing Label

Chapter 3 Facilities Requirements

3.1 Overview

This chapter details facility site requirements, safety requirements, and configuration options for the Dimension 3100 Scanning Probe Microscope. Specifically, this chapter details environmental requirements and equipment facilities drawings. Compliance with the following requirements and specifications is essential before beginning installation.

- **Overview:** Section 3.1
- **Optional Configurations:** Section 3.2
- **Facilities Requirements:** Section 3.3
- **Acoustic/Vibration Isolation Systems:** Section 3.4
- **Facilities Requirements Summary:** Section 3.5
- **Environmental Acoustic/Vibration Specifications:** Section 3.6
- **General Facilities Guidelines:** Section 3.7

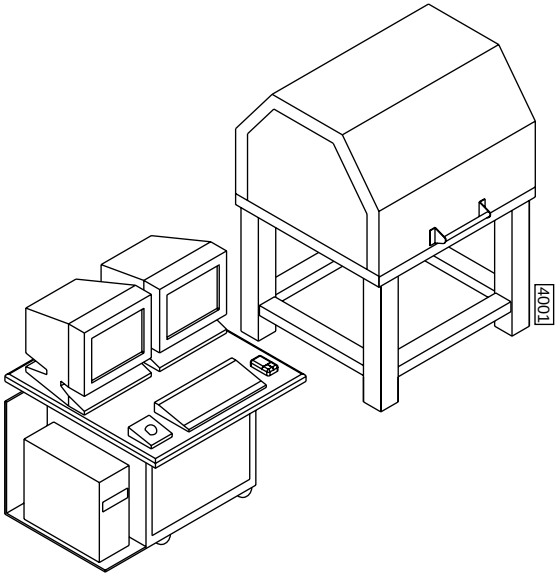
3.2 Optional Configurations

The following are typical configurations for the Dimension 3100 Scanning Probe Microscope, detailing 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.

3.2.1 Axiom VT-103-3K with ELCON

This configuration has two basic elements: the VT-103-3K, which is an integrated air table and acoustic hood for vibration isolation, and the ELCON (electronics console) which is an optional small footprint/compact console (See Figure 3.2a). The VT-103-3K is comprised of an air table, on which the Dimension 3100 microscope rests (not shown) and an acoustic hood which can be raised to access the instrument, and lowered to seal the instrument during operation. The ELCON is a compact control station console used to house the computer and control electronics underneath, and the monitors, mouse and keyboard on top. The VT-103-3K and the ELCON do not require one another and can be used independently.

Figure 3.2a Axiom VT-103-3K with ELCON

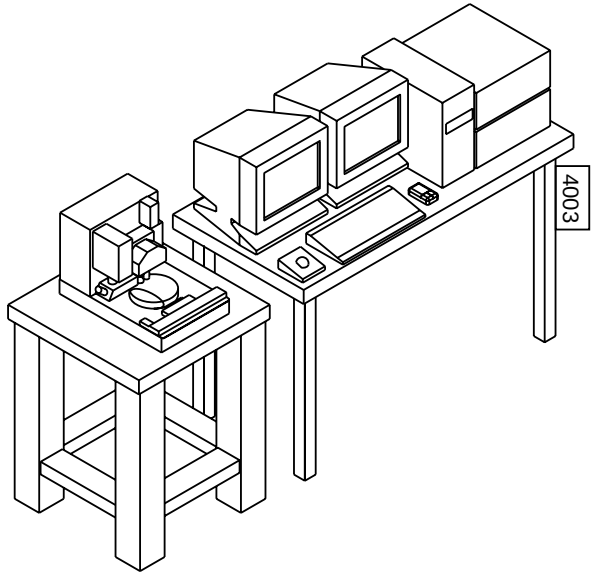


Optional Configurations continued...

3.2.2 Axiom VT-102

This configuration consists of two basic elements: the VT-102 which is an air table for vibration isolation, and a typical "table top" version of the control station (computer, control electronics, and accessories). The VT-102 is a compact air table on which the Dimension 3100 microscope rests and does not include an acoustic hood. Figure 3.2b 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 two electronics boxes stacked atop each other on the far right of the table; the tower computer placed next to them on the table top; and the monitors (2), keyboard, and mouse located on the far left. There are many ways to station the equipment, including placing the electronics boxes on the floor or underneath the monitors, or placing computer on the floor, which may save some space and change the facilities requirements.

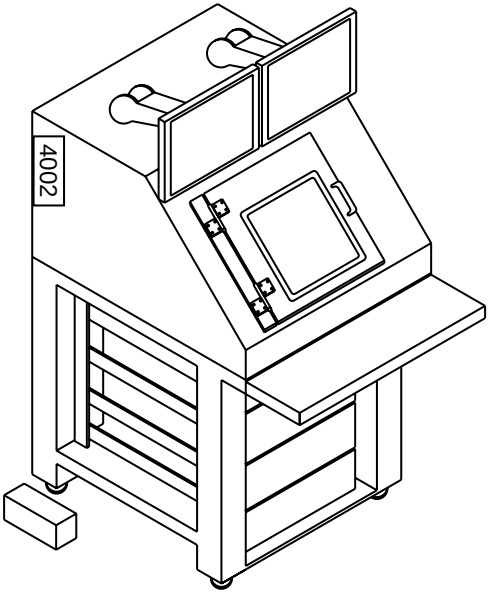
Figure 3.2b Axiom VT-102



3.2.3 Axiom IS3K-2

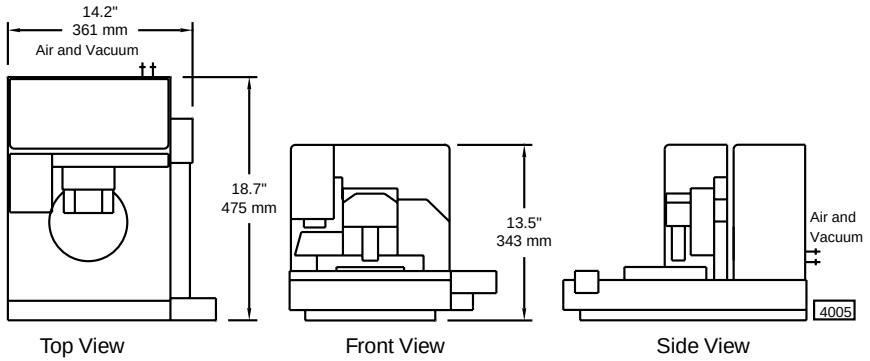
This configuration is comprised of one basic element, the IS3K-2. The IS3K-2 is an ultra compact/small footprint console containing the control station, the microscope, and an integrated vibration isolation and acoustic enclosure (See Figure 3.2c). This setup consists of the following: two control boxes and a rack mount computer mounted underneath the console and between the table legs. The monitors are flat panel displays and mount on the top of the acoustic hood with adjustable arm attachments. The keyboard and mouse (not shown) sit on the small ledge at the front of the instrument. Finally, the Dimension 3100 microscope (not shown) rests on top of the vibration isolation table inside the IS3K-2 acoustic hood. The entire acoustic hood can raise and lower during installation. For general operation, the microscope is accessed via the small door on the front of the acoustic hood. Although the IS3K-2 is extremely compact (ideal for clean rooms), this configuration has limited working space and may require an extra table for sample preparation and cantilever installation.

Figure 3.2c Axiom IS3K-2



3.3 Facilities Requirements

Figure 3.3a Dimension 3100 SPM Facility Requirements



Weight: 150 lbs. for Dimension 3100 SPM assembly only.

Note: Vibration and acoustic isolation is strongly recommended

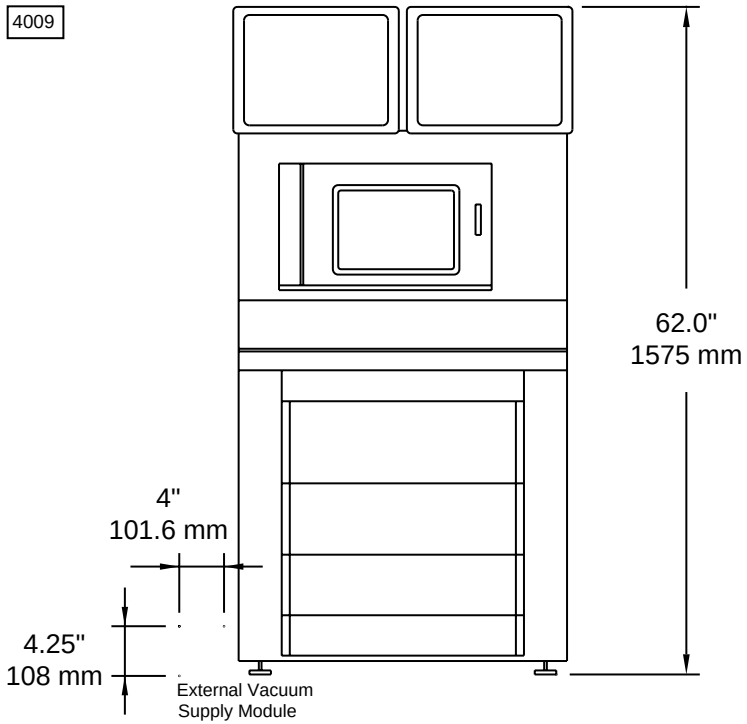
3.4 Acoustic/Vibration Isolation Systems

3.4.1 Axiom IS3K-2 Dimensions, Utilities, and Clearance

The IS3K-2 is painted with cardinal paint 6400 series. The system does not outgas. An all stainless version of the IS3K-2 is available as a special order for an additional cost.

An appropriate seat should be used which brings the user's knees to within 3-4 inches (76-102mm) of the bottom of the keyboard tray.

Figure 3.4a Axiom IS3K-2



Acoustic/Vibration Isolation Systems continued...

Figure 3.4b Axiom IS3K-2

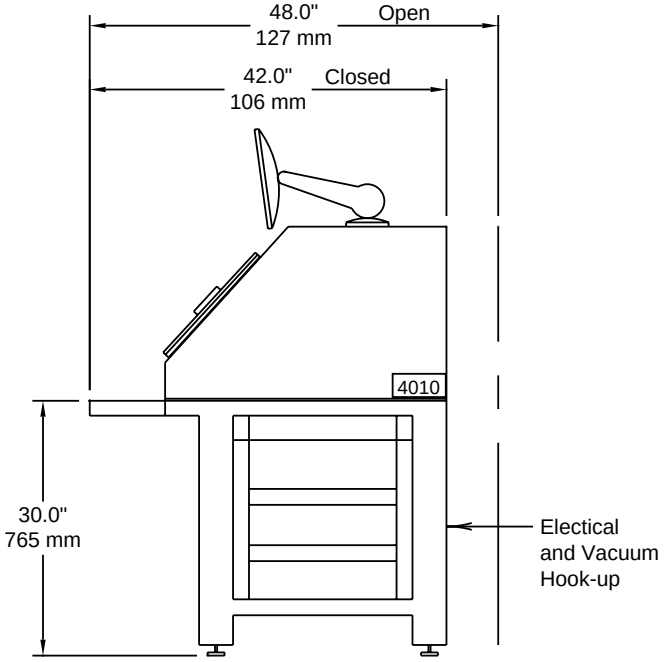


Figure 3.4c Axiom IS3K-2

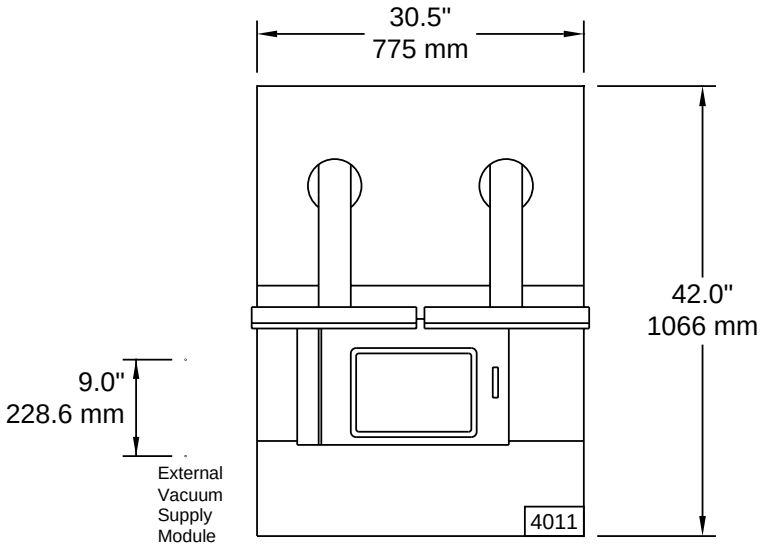


Figure 3.4d IS3K-2 Leveling Feet Location

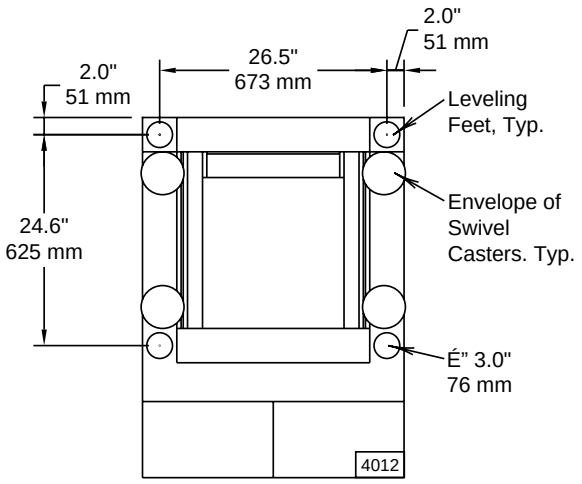
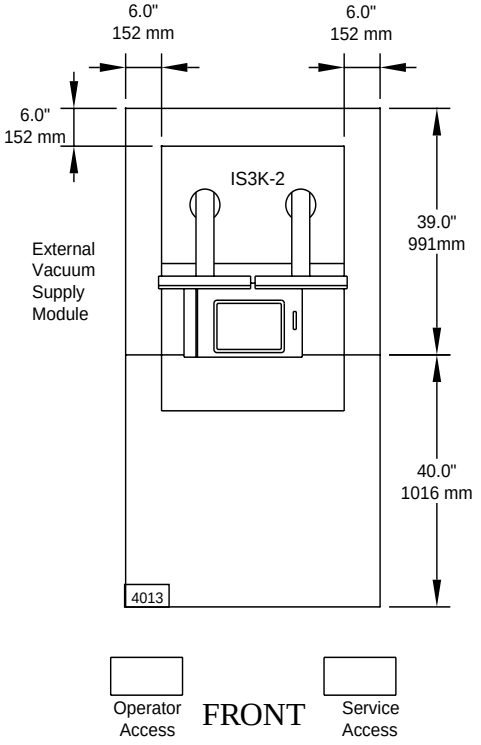


Figure 3.4e IS3K-2 Footprint Requirements



Acoustic/Vibration Isolation Systems continued...

3.4.2 Axiom VT-103-3K Dimensions, Utilities and Clearance

An isolation hood/table is required for acoustic and vibration isolation of the Dimension 3100. The table must be moved to its final location before Digital Instruments Veeco personnel can install and train on the SPM.

Figure 3.4f Axiom VT-103-3K

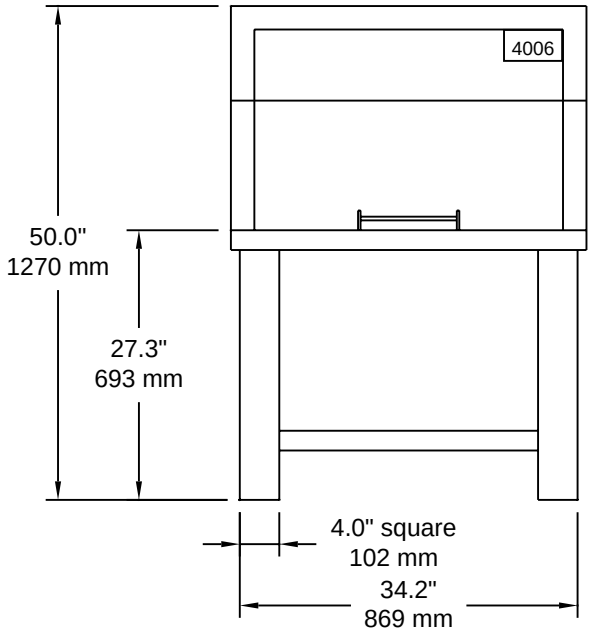


Figure 3.4g Axiom VT-103-3K

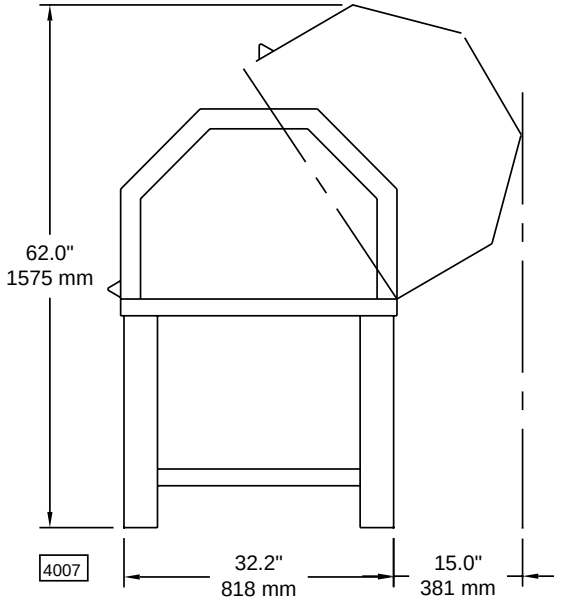
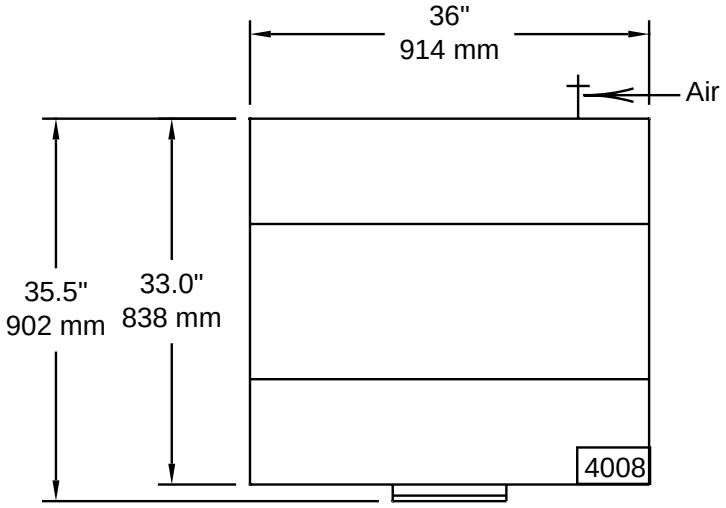


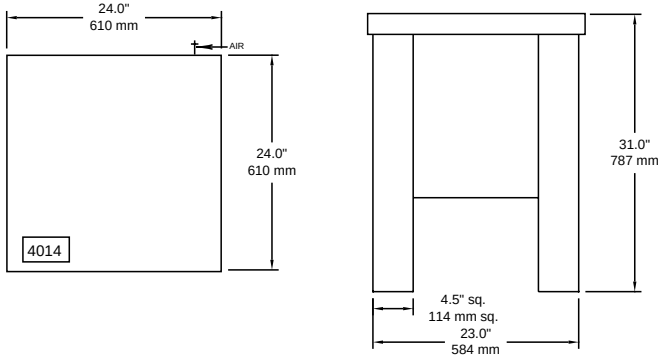
Figure 3.4h Axiom VT-103-3K



3.4.3 Axiom VT-102 Dimensions and Utilities

The vibration isolation table may be supplied in lieu of the VT-103-3K or IS3K-2 for selected applications that do not require acoustic isolation for the desired performance level.The table must be moved to its final location before Digital Instruments Veeco personnel can install and train on the SPM.

Figure 287.3i: Axiom VT-102 Vibration Isolation Table

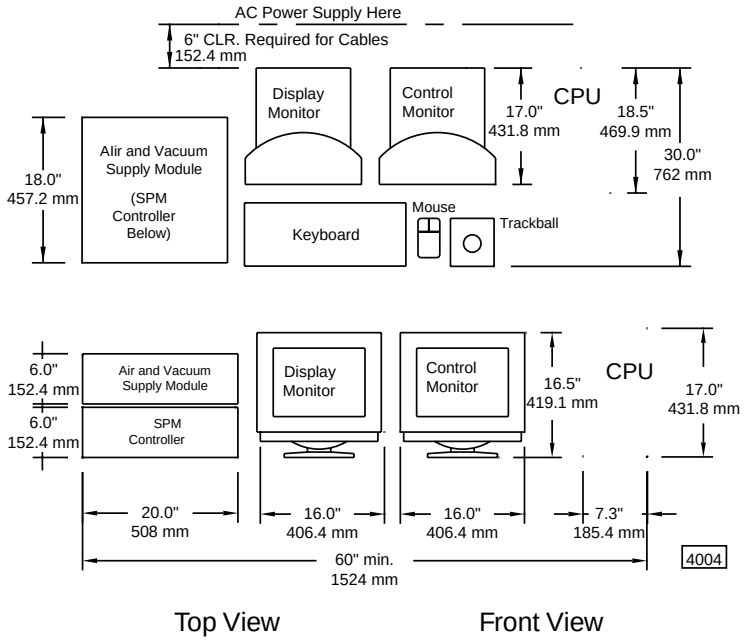


3.4.4 Computer/Controller Facility Requirements

The IS3K-2 allows placement of the computer/controller within the framework of the unit. No additional footprint is required.

Customer must supply computer and controller table or order the optional Elcon console for enclosure.

Figure 3.4j Axiom VT-103-3K and Axiom VT-102



Weight: 170 lbs. nominal total; 140 typically on table top.

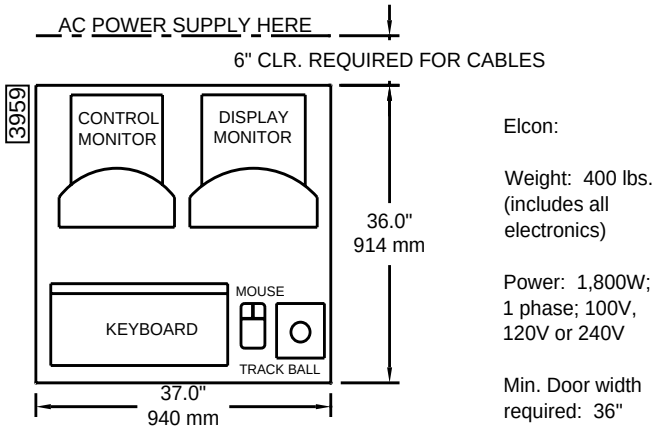
Note: Dimensions shown for computer and controller equipment are approximate and subject to change without notice.

Acoustic/Vibration Isolation Systems continued...

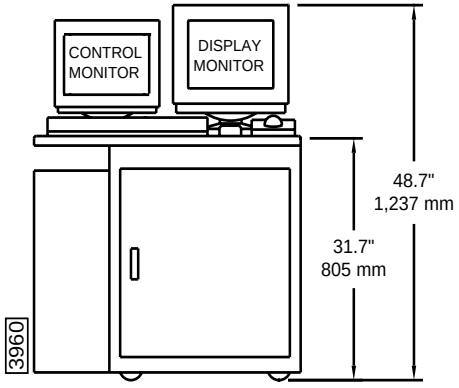
3.4.5 ELCON Console

The ELCON Console is available as an option for the computer/controller enclosure

Figure 3.4k Optional ELCON Console



Top View



Front View

3.5 Facilities Requirements Summary

If the Dimension 3100 system is used in a clean room with raised floors a pedestal must be provided to support leveling feet on the IS3K-2 acoustic hood. House vacuum is optional in all cases. If used, connections should accept a 1/8" or 1/4" male NPT fitting or 170 ID x .250 OD flexible house.

Note: The following summary does not include computer/controller or optional elcon console enclosure requirements.

Table 3.5a: Dimension 3100 Facilities Requirements Summary

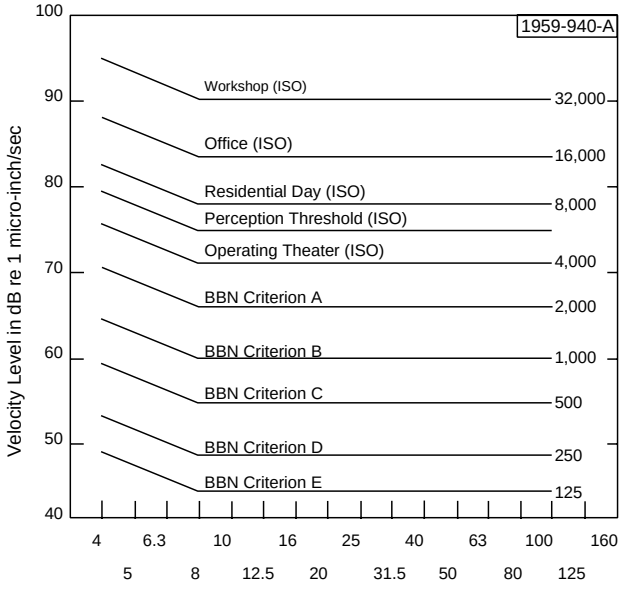
Utility With	Dimension 3100 with VT-103-3K Hood/Table	Dimension 3100 with IS3K-2 Hood/Table	Dimension 3100 with VT-102 Table
Electrical	1,800W; single phase; 100V, 120V or 240V duplex outlet; dedicated circuit	1,800W; single phase;100V, 120V or 240V duplex outlet; dedicated circuit	1,800W; single phase; 100V, 120V or 240V duplex outlet; dedicated circuit
Disconnect or Shutoff	None required	None required	None required
Clean Dry Air for Sample Chuck	Supplied with Dimension 3100 by air and vacuum supply module	Supplied with Dimension 3100 by air and vacuum supply module	Supplied with Dimension 3100 by air and vacuum supply module
Clean Dry Air for Air Table	Requires user supplied air 60-80 PSI, 1 CFM[2]	None required	Requires user supplied air 60-80 PSI, 1 CFM[2]
Vacuum for Sample Holddown	Supplied with Dimension 3100 or house vacuum -25in. Hg[2]	Supplied with Dimension 3100 (ext. vac. supply module) or house vac. -25 in.Hg[2]	Supplied with Dimension 3100 or house vacuum -25in. Hg[2]
Exhaust	None required	None required	None required
Lan Connection	None required but network support card included	None required but network support card is included	None required but network support card is included
Approximate Footprints	36" x 33.0"	30.5" x 42.0"	23.0" x 23.0"
Hood and Table Including SPM	1,100 pounds evenly distributed	1,200 pounds (including computer/controller)	750 pounds evenly distributed
Min. Door Width	36"minimum	32"minimum	26"minimum
Environment/ Noise	See last page	See last page	See last page

3.6 Environmental Acoustic/Vibration Specifications

The following conditions must be met in order to achieve 0.5 angtrom RMS noise 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 3.6a 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

3.7 General Facilities Guidelines

The following list contains general facilities recommendations for the Dimension 3100 system:

- 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 damper the acoustic noise from the roof.
- Temperature is standard laboratory setting +/- 2.5 F˚.
- Humidity is standard laboratory setting +/- 10% RH.
- Power should be in dedicated 115 volt standard duplex outlet (15 amp).
- Clean Dry Air (CDA) is required with a recommended connection of 0.25-inch poly-flow tubing with a range of 0-100 PSI.
- Customer installed house vacuum and vacuum wand is recommended for wafer loading and unloading.
- Customer installed nitrogen is recommended for the blow-off nozzle.

Chapter 4 Installation

4.1 Overview

This chapter details the installation procedure for the Dimension 3100 Scanning Probe Microscope (SPM) system from receiving to full installation. Specifically, the following topics are discussed in this chapter:

- **Overview:** Section 4.1
- **Shipping and Receiving:** Section 4.2
- **Uncrating the System:** Section 4.3
- **Installing the Dimension 3100 System:** Section 4.4
- **Connecting the Dimension 3100 System:** Section 4.5
- **System Power-up:** Section 4.6

CAUTION: Installation should be completed by trained Digital Instruments Veeco personnel *only*. Installation instructions are provided for customer reference *only*.

4.2 Shipping and Receiving

4.2.1 Equipment Requirements

The following equipment is necessary for successful installation of the Dimension 3100 SPM system. Verify the following is on-hand before beginning installation:

Equipment Received

- Dimension 3100 Manual
- Dimension 3100 Controller
- Dimension 3100 Microscope
- Dimension Microscope Head
- Computer
- Hard Drive Back-up on CD
- Mouse, Keyboard and Trackball
- Dimension Accessories Kit
- Cantilever Stand
- Tip Holders (2)
- Wafer Handling Tool Kit
- Stage Calibration Standard
- Vibration Isolation Pad
- Cable Clamp (needed only with vibration isolation table)
- Power Strip
- 8-inch Sample Chuck (option)
- Extender Box (option)

Shipping and Receiving continued...

Cables Received

- Controller-to-Dimension 3100 Cable, 37-pin D
- Dimension 3100 DC Power Cable
- Fiber Optic Cable
- Frame Grabber Video Cable (BNC to BNC)
- Power Cords (2)
- Serial Cable, 9-pin D, 12’
- Serial Cable, 9-pin D, 6’
- Vacuum Hose Assembly

Tools Received

- Allen Wrench (for cable clamp)
- 1/2" Wrench (2)
- Small Flat Head Screwdriver
- Phillips Screwdriver
- Anti-static Wristband

4.3 Uncrating the System

4.3.1 Uncrate the Dimension 3100 SPM System

1. Using scissors, cut and remove the plastic shipping band encircling the Dimension 3100 shipping crate.
2. Lift the cardboard shipping box off of the Dimension 3100 microscope.
3. Unscrew the 2 shipping bolts holding the shipping brackets in place at either side of the Dimension 3100 microscope. Remove the shipping brackets and store with the Dimension 3100 shipping box in the event that the microscope must be shipped for maintenance or repair.
4. Unpack all system components. Each of the items below are shipped in separate boxes:
    - Dimension 3100 Microscope
    - NanoScope IIIa Controller (if applicable)
    - Dimension 3100 Controller
    - Computer and Keyboard
    - Dimension Microscope Head
    - Computer and Microscope Accessories (Dimension accessories box)
    - Chuck Base Hardware (packed with Dimension 3100 microscope)
5. Save and store the shipping box for the Dimension microscope head.

Note: If repair or calibration is necessary, the shipping box is crucial for safe return of the head to the factory. It is also convenient to use for long-term storage.

ATTENTION: Do not remove the plastic wrap from the microscope's X-Y stage. Do not remove the microscope head from its shipping box yet. The SPM microscope head must remain in the box until ready for use.

4.4 Installing the Dimension 3100 System

4.4.1 Install the Dimension 3100 SPM Unit

1. Place the vibration isolation table, or the microscope platform, at the desired location.

2. Place the Dimension 3100 microscope in the operating location on the vibration isolation table or other platform.

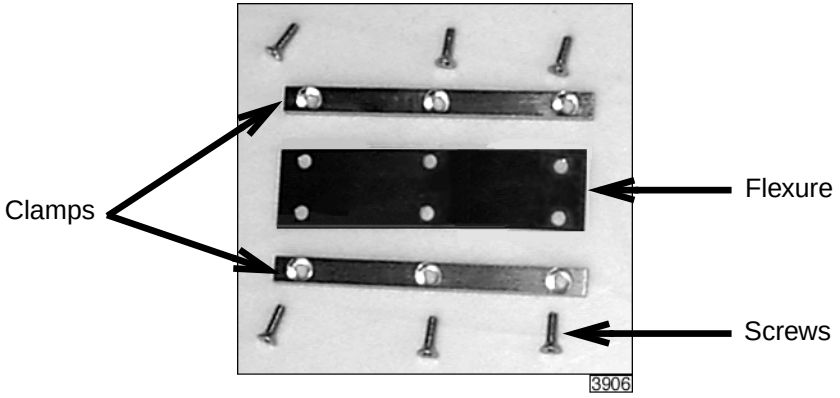
CAUTION: The Dimension 3100 microscope unit exceeds the two-person lift weight limit and should be lifted with a mechanical assist. Use proper lifting technique when removing or replacing the Dimension 3100 microscope. Improper lifting may cause muscle strain or back injury.

3. Carefully remove the plastic wrap from the X-Y stage.

4. Locate the clamps (2), screws (6), and flexure (1) used to secure the chuck base to the granite block (See Figure 4.4a).

Note: An extra flexure and accompanying hardware is included.

Figure 4.4a Hardware for Chuck Securement to Stage



Installing the Dimension 3100 System continued...

5. Carefully slide the chuck base off the X-Y stage.

   Note: Do not stretch or bend the vacuum lines. Do not remove the vacuum lines from the chuck base.

6. Wipe down the granite and underside of the chuck base with isopropyl alcohol. Place the chuck base back onto the granite.

7. Align the two sets of three mounting holes on the long edges of the flexure with the three mounting holes of the chuck base and the three mounting holes on the X-Y stage. Align one clamp over the each set of three mounting holes (See Figure 4.4b).

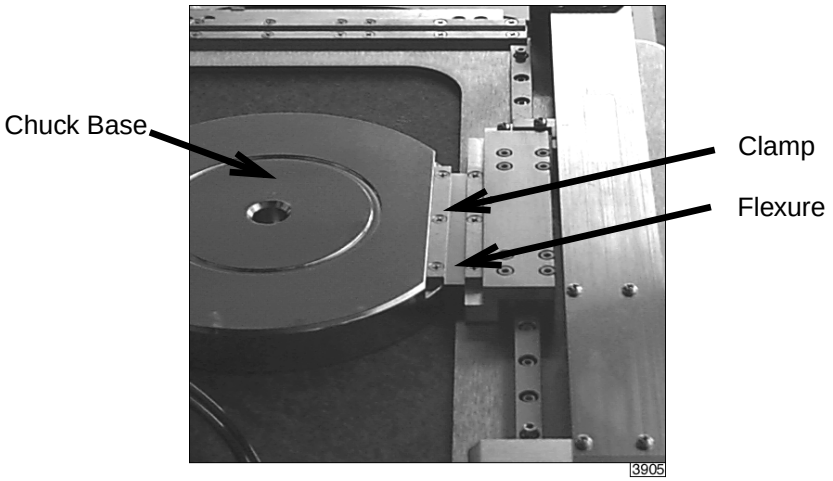
8. Secure the chuck base in place on the X-Y stage by screwing the two clamps down onto the flexure and stage, leaving the six screws slightly loose (See Figure 4.4b).

9. Apply moderate pressure to the chuck base to secure the base to the X-Y stage, and tighten the six screws.

10. Locate the vacuum sample chuck shipped in the Dimension accessories box, and mount it on the chuck base.

11. Wipe down the top of the chuck base and underside of the sample chuck with isopropyl alcohol.

Figure 4.4b Secure the Chuck to the Stage



Installing the Dimension 3100 System continued...

4.4.2 Install the Control Station

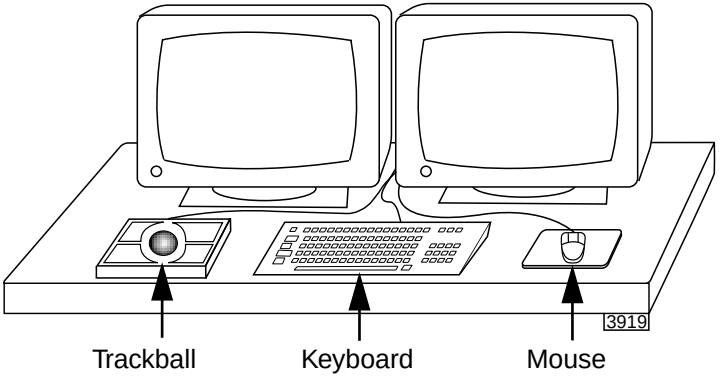
1. Set up a table to be used as the control station next to the Dimension 3100 microscope.

   Note: Keep the control station in close proximity to the Dimension 3100 unit without touching the vibration isolation table.

2. Place the following input and display equipment on the control station (See Figure 4.4c):

   - Monitors (2)
   - Keyboard
   - Mouse and Mouse Pad
   - Trackball

Figure 4.4c Dimension 3100 Input and Display Equipment



3. Place the computer on the side of the input and display devices closest to the Dimension 3100 microscope.

4. Place the NanoScope IIIa controller and Dimension 3100 controller next to the computer.

ATTENTION: Do not set the Dimension 3100 controller on its side**.**

ATTENTION: Do not set the Dimension 3100 controller on the same table as the Dimension 3100 microscope. Vibrations from the vacuum pump affect imaging performance. You may stack the Dimension 3100 controller atop the NanoScope IIIa controller.

4.5 Connecting the Dimension 3100 System

CAUTION: Verify that the machine is powered-down and locked-out before attempting to make any connections.

4.5.1 Connect the Dimension 3100 Control Station Extensions

Connect the Display and Input Devices

1. Connect the monitor power cords (2) to the power strip, but do not power-up.
2. Using the Y-adaptor, connect the monitor video cable to the computer (See Figure 4.5a port #5).
3. Connect the following input devices to the computer (See Figure 4.5a):
    - Keyboard (port #2)
    - Mouse (port #2)
    - Trackball (port #3)

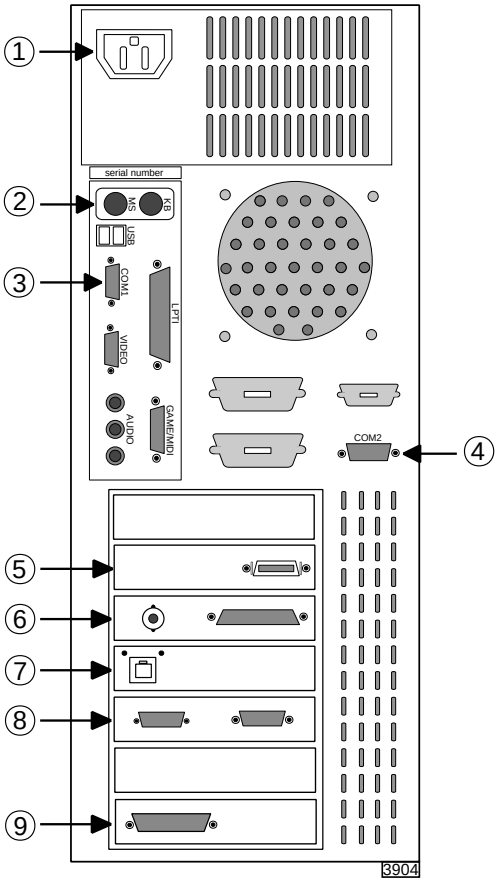
Note: Do not exchange the mouse with the trackball by using your own cable adapters—the trackball's extra buttons are used to manipulate the stage software.

Connect the Computer

ATTENTION: Boards may shift during the course of shipment. Improperly seated boards may cause equipment damage. Remove the computer cover and verify that all the boards are properly seated before powering-up the computer.

Note: Wear an anti-static wristband during this operation to avoid potential damage to the circuit boards.

Figure 4.5a Computer (rear view)



Connect the Computer continued...

Note: The computer ships with the network board disabled to avoid error messages for computers not used on a network.

1. To use the computer on a network, select **My Computer\Control\Panel\System\Hardware Profiles\Properties\Network** and click to uncheck the box. Click **OK** twice to exit. Plug in your local network cable and restart the computer.

2. Connect extensions to the **Computer**. See Table 4.5a and Figure 4.5a for list of extensions, connection information and connection location.

Table 4.5a: Computer Connections

Fig Ref	Cable	Part Number	Box	Function
1	AC Power Cable	466-000-004	Power Strip	Computer power
2	Mouse	n/a	Computer	Input from mouse
2	Keyboard	n/a	Computer	Input from keyboard
3	Trackball	860-000-022	Computer	Input from trackball
4	Serial Cable (12’)	464-000-010	D3100 Back Panel	Motor control for stage
5	Monitor Cable	n/a	Monitors	Y adaptor to video signal out
6	BNC Cable	462-000-002	D3100 Back Panel	Optical image signal from framegrabber
7	Cable, RJ45 LAN	n/a	Host	Network connection
8	Serial Cable (6’)	464-000-024	Dimension Controller	Serial communication w/ Dimension controller
9	Cable, 25-pin D	464-000-012	NanoScope Controller	Computer to controller electronic interface

Connect the NanoScope IIIa Controller

1. Connect extensions to the **NanoScope IIIa Controller**. See Table 4.5b, Figure 4.5b, and Figure 4.5c for list of extensions, connection information and connection location.

Table 4.5b: NanoScope IIIA Controller Connections

Fig Ref	Cable	Part Number	Box	Function
1	Cable, 37-pin D	464-000-002	D3100 Back Panel	Microscope control
2	AC Power Cable	466-000-004	Power Strip	NanoScope IIIa controller power
3	Cable, 25-pin D	464-000-012	Computer	Computer to controller electronic interface

Figure 4.5b NanoScope Controller (front view)

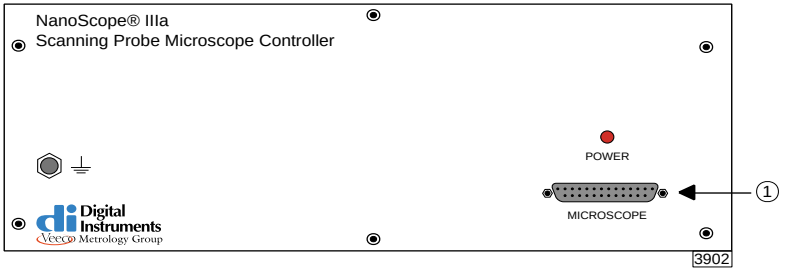
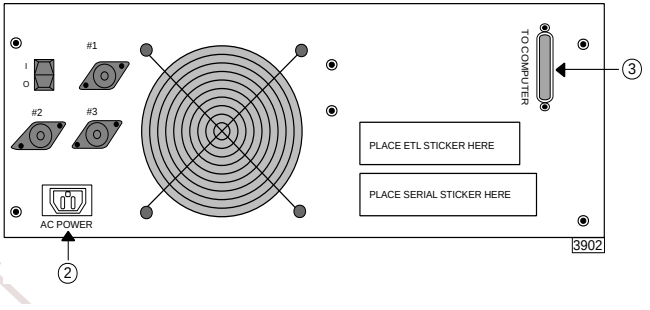


Figure 4.5c NanoScope Controller (rear view)



Connect the NanoScope IIIa Controller continued...

Note: Be sure to tighten the cable connectors' locking screws at both ends to prevent accidental removal of the cable while the NanoScope IIIa Controller operates.

ATTENTION: Do not remove or install the cable while the NanoScope IIIa Controller is powered-up or in operation.

Connect the Dimension 3100 Controller

1. Connect extensions to the **Dimension 3100 Controller**. See Table 4.5c and Figure 4.5d for list of extensions, connection information and connection location.

Note: The Dimension 3100 cables are bundled through the cable clamp provided if the system includes a vibration isolation table. (See section titled Route the VT103 Air Table Cabling at the end of the chapter).

Table 4.5c: Dimension 3100 Controller Connections

Fig Ref	Cable	Part Number	Box	Function
1	AC Power	466-000-004	Power Strip	Dimension 3100 controller Power
2	Vacuum Hose Assembly	860-000-012	D3100 Back Panel	Vacuum and air supply
3	Serial Cable (6’)	464-000-024	Computer	Serial communication w/ computer
4	DC Power Cable	820-000-006	D3100 Back Panel	Motor power
5	Fiber Optic Cable	443-000-001	D3100 Microscope Optics	Optics illumination

Figure 4.5d Dimension 3100 Controller (rear view)

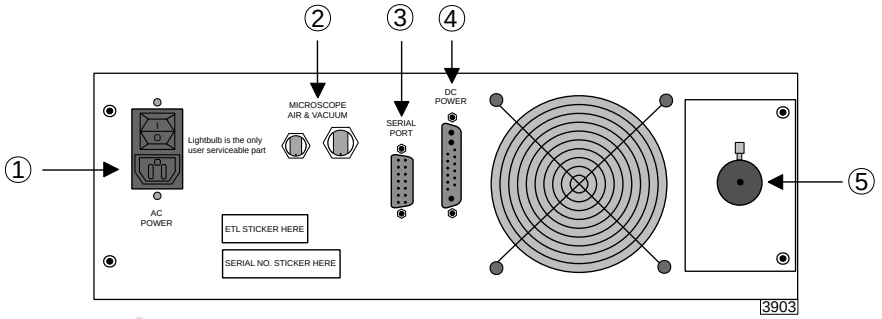
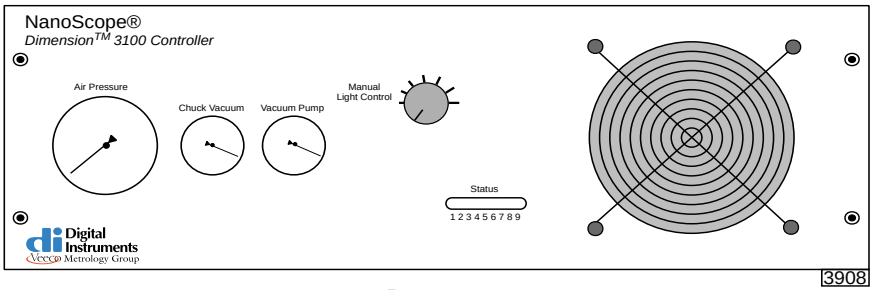


Figure 4.5e Dimension 3100 Controller (front view)



2. To set illumination, toggle the manual control switch on the front of the Dimension 3100 controller to **ON** for manual control or **OFF** for computer control (See Figure 4.5e).

   Note: Each illumination system may vary in intensity. Adjust to preferred levels once the system is operational—for computer adjustment there is an "illumination" parameter on the **Other Controls** panel.

3. Lock the fiber optic cable in place after establishing connection to the Dimension 3100 controller and to the Dimension 3100 microscope optics. Tighten the respective locking screws until they are snug.

4. Remove the temporary cover from the vacuum/air ports.

4.5.2 Connect the Dimension 3100 Microscope Extensions

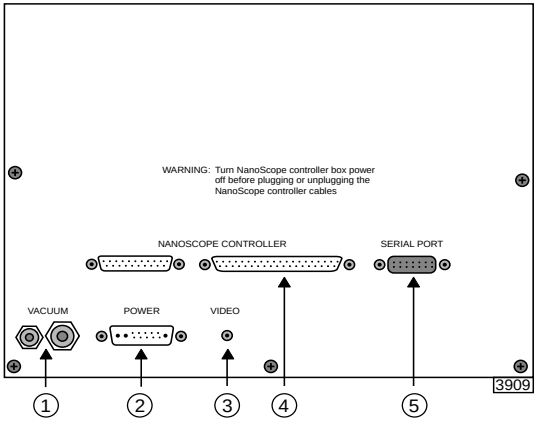
Connect the Dimension 3100 Microscope Electronics Box

1. Connect extensions to the **Dimension 3100 Microscope Electronics Box**. See Table 4.5d and Figure 4.5f for list of extensions, connection information and connection location.

Table 4.5d: Dimension 3100 Microscope Electronics Box Connections

Fig Ref	Cable	Part Number	Box	Function
1	Vacuum Hose Assembly	860-000-012	Dimension Controller	Vacuum and air supply
2	DC Power Cable	820-000-002	Dimension Controller	Motor power
3	BNC Cable	462-000-002	Computer	Optical image signal from framegrabber
4	Cable, 37-pin D	464-000-002	NanoScope Controller	Microscope control
5	Serial Cable (12’)	464-000-010	Computer	Motor control for stage

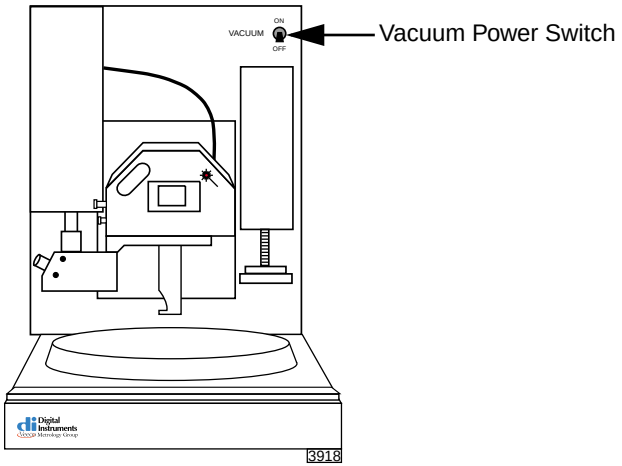
Figure 4.5f D3100 Microscope Electronics Box (rear view)



Connect the Dimension 3100 Microscope Electronics Box continued...

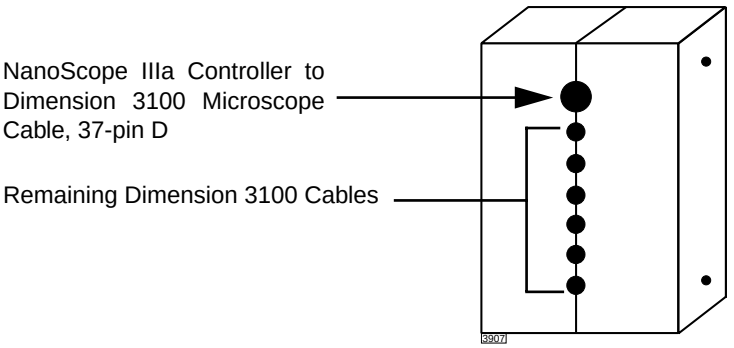
2. Toggle the vacuum power switch, located on the front of the microscope unit, to **OFF** (See Figure 4.5g).

Figure 4.5g Vacuum Power Switch



Route the VT103 Air Table Cabling

Figure 4.5h Cable Clamp



1. Route all cables connected to the Dimension 3100 microscope through the slot located in the far right region of the VT103 vibration isolation table.

2. Position the Dimension 3100 microscope in the desired location.

Route the VT103 Air Table Cabling continued...

3. Mark the cables with a marker to reference each cable's position in the cable clamp (See Figure 4.5h).

   Note: When secured in place, the cable clamp is located underneath the vibration isolation table, near the aforementioned slot.

4. Disconnect all cables from the Dimension 3100 microscope, and remove the cables from the slot.

5. Route cables through the cable clamp and secure the bolts using the supplied allen wrench. Verify that the cables are not pinched in the cable clamp.

6. Re-route cables through the slot and re-connect cables to the Dimension 3100 microscope.

7. Secure the cable clamp by tightening the four bolts underneath the vibration isolation table.

4.6 System Power-up

ATTENTION: The following section is required only during installation or after servicing and should NOT be used by untrained personnel.

Prepare the System for Power-up

1. Verify that the power cord is plugged into a power receptacle with the correct voltage.

2. Verify that all cables are connected properly, especially the NanoScope III SPM controller to the Dimension 3100 microscope electronics box.

CAUTION: Cables should not be removed or installed while power is applied to the system. These restrictions do not apply to the small black cable that connects the microscope head to the stage control electronics.

Power-up Sequence

1. Power-up the computer using the push-button switch located on the front of the computer.

2. Power-up the monitors (2) using the push-button switches located on the front of the monitors.

3. Power-up the NanoScope IIIa controller using the power switch located on the rear of the NanoScope IIIa controller.

4. Power-up the Dimension 3100 controller, using the power switch on the rear of the Dimension 3100 controller.

Chapter 5 Stage System

5.1 Overview

The Dimension 3100 Scanning Probe Microscope (SPM) features a large sample stage capable of positioning large samples such as silicon wafers and computer hard drive media, as well as small samples. The X-Y stage consists of a pair of stacked, perpendicular slides and uses an open loop (unencoded) architecture with stepper motors to drive the stage to user-specified coordinates.

This chapter details procedures for mounting samples and dedicated stage menu software commands. Specifically, this chapter discusses the following:

- **Overview:** Section 5.1
- **Mounting of Samples:** Section 5.2
- **Stage Menu Commands:** Section 5.3

5.2 Mounting of Samples

There are two methods widely used for mounting samples: vacuum chucks and magnetic pucks. Regardless of the method used, verify that samples are mounted flat and parallel to the stage. This is especially important for larger samples inspected over more than one site. Grossly tilted samples may require raising the head higher whenever the sample is indexed, increasing cycle time and the risk of probe-sample collision. Similarly, large numbers of identical samples should be mounted the same way whenever possible to allow use of the same settings between samples.

5.2.1 Vacuum Chucks

Stages are equipped with vacuum chucks which are often employed for securing samples.

CAUTION: Operators should be cautious when handling larger samples with the vacuum chuck—an 8-inch wafer held using 5 psi vacuum sustains a loading of 250 lbs.

ATTENTION: If debris is trapped between the stage and wafer, the wafer may become scratched or broken. Keep the stage clean at all times using isopropyl alcohol. When cleaning dust from the stage area, use a vacuum cleaner with a soft brush. DO NOT clean stages using compressed air.

5.2.2 Magnetic Pucks

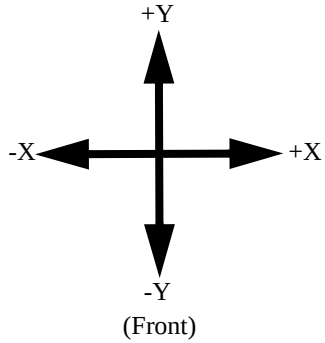
Samples may also be secured to the stage using magnetic pucks. This system allows for easy mounting and removal of small samples. For detailed mounting instructions, see Chapter 7.

Mounting of Samples continued...

5.2.3 Axis Orientation—Motorized X-Y Stages

When viewing the Dimension 3100 SPM from the front, stage movements are defined over two axes of motion: X (left-right), and Y (front-back). For X-axis movements, lesser (decreasing) coordinates are located to the left. For Y-axis movements, positive (increasing) coordinates are located toward the rear of the machine, decreasing values are located forward (See Figure 5.2a).

Figure 5.2a Stage X-Y Axis Orientation



5.3 Stage Menu Commands

Important stage menu commands are discussed in detail in the following sections:

- **Load New Sample:** Section 5.3.1
- **Locate Tip:** Section 5.3.2
- **Align Laser:** Section 5.3.3
- **Focus Surface:** Section 5.3.4
- **Move To (X,Y):** Section 5.3.5
- **Set Reference:** Section 5.3.6
- **Stage Menu Commands continued...:** Section
- **Initialize:** Section 5.3.8
- **SPM Parameters:** Section 5.3.9

5.3.1 Load New Sample

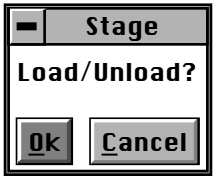
ATTENTION: Do not move the stage while the microscope is scanning.

Stage software is designed to function between vertical movements of the head. Functions such as **Load New Sample** first lift the head's probe from the sample surface, then index the stage to a preprogrammed location (usually front-center) where samples may be rapidly reloaded.

To load or unload a sample from the stage using the **Load New Sample** function, complete the following:

1. Select **Load New Sample** from the **Stage** pop-down menu. The screen displays a dialog box (See Figure 5.3a).

Figure 5.3a Stage Load/Unload Prompt



Load New Sample continued...

2. To load a new sample, click **OK**. The head raises to the Load/ Unload height, and the stage indexes to the front-center position.

   Note: Verify that the tip is still usable. If the tip has been used for a lengthy period, or if damage is suspected, change the tip now per instructions provided in Chapter 7 of this manual.

3. To release the vacuum chuck's hold on a sample, use the pneumatic toggle switch located on the upper-right corner of the electronics box. When the vacuum is released, remove the sample.

4. Verify that the stage and vacuum chuck are clean of debris. If debris is present, clean the stage using a lint-free wipe and isopropyl alcohol.

5. Place the new sample on the stage.

6. Verify that the sample is centered and seated flat against the chuck's contact points.

7. Activate the vacuum chuck by toggling the vacuum chuck switch. Use the **Stage/Move To (x,y)** command to move the stage back.

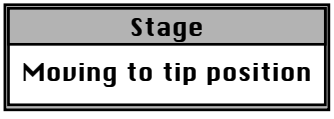
5.3.2 Locate Tip

When installing new tips, execute the locate tip function is executed before focusing on the sample surface. This function enables the operator to find the probe tip using the optical system. To locate the tip, complete the following:

1. Verify that a tip is installed in the probe tip holder, then select the **Stage / Locate Tip** option or use the tool bar icon.

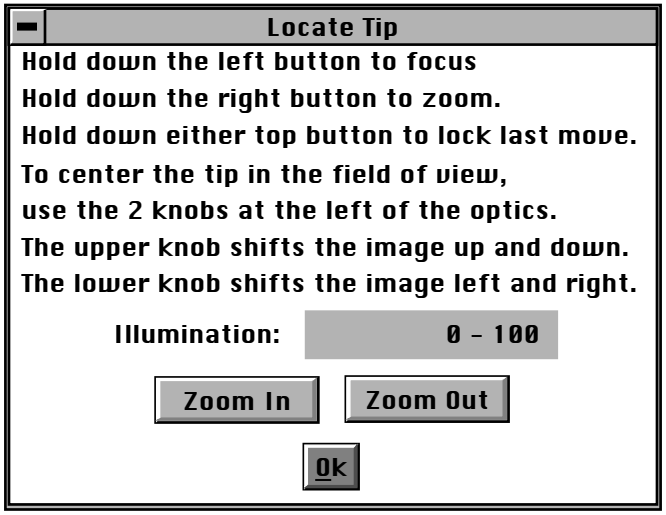
   Note: The screen displays a caution indicating that the microscope objective is in motion (See Figure 5.3b).

Figure 5.3b Moving to Tip Position Caution



Note: As the objective moves positions, the tip should begin to come into focus. When the focus position is attained, the screen displays trackball instructions for achieving a focus (See Figure 5.3c).

Figure 5.3c Locate Tip Prompt



2. Bring the tip into focus by holding down the left trackball button while rolling the trackball.

   Note: The tip may be off from center in the field of view, particularly if installing a new type of tip. Zooming out may aid in locating the tip.

3. Rotate the small, metal knobs located on the front of the on-axis viewing assembly to center the tip in the field of view as indicated in the screen prompt.

4. Verify that a sufficiently large field of view is utilized to locate the tip. If locating the tip proves difficult, turn the metal knobs clockwise until they stop, then turn back counter-clockwise approximately 1.5 turns.

5. When the tip is in focus and centered in the field of view, click **OK** in the **Locate Tip** prompt.

Stage Menu Commands continued...

5.3.3 Align Laser

This option offers a menu-driven method for aligning lasers onto cantilevers, and is especially useful for beginners. This alignment procedure requires approximately 10 minutes.

1. Verify that the tip is withdrawn from the surface of the sample and raised as high as possible. Select **Stage / Align Laser**. The screen displays a prompt to continue (See Figure 5.3d).

Figure 5.3d Align Laser Prompt

2. To continue, click **OK**. You are prompted to attach a mirror cap to the bottom of the head's tube (See Figure 5.3e).

Figure 5.3e Mirror Cap Prompt

3. Attach the mirror cap, then click **OK**. Additional detailed instructions are provided from on the screen.

Note: The mirror cap ships as part of the Dimension Accessories Kit.

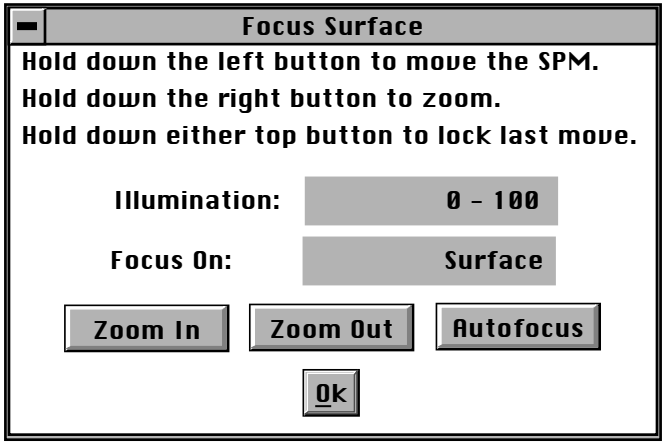
5.3.4 Focus Surface

This function focuses the sample surface. The operator may choose to manually focus on the surface using the trackball or allow the optics to focus automatically by choosing the **Autofocus** feature. When the surface is already partially in focus (or close to it), use the **Autofocus** feature.

To use the **Focus Surface** feature complete the following:

1. Select **Focus Surface** from the **Stage** pop-down menu. The screen displays trackball instructions for achieving a focus (See Figure 5.3f).

Figure 5.3f Focus Surface Prompt



2. Use the left button on the trackball to focus on the surface (which moves the SPM or Z stage up and down).

3. Use the right button on the trackball to zoom out completely when trying to focus on the surface.

4. If the sample is not already under the microscope, use the trackball (with neither the left nor right buttons pressed) to move the X-Y stage until the sample is in position.

Focus Surface continued...

5. If the surface is partially in focus, use the **Autofocus** option to complete the focusing process.

6. To focus on the sample "surface" (normal operation) or the "tip reflection" (for extremely clean samples), change the **Focus On** parameter accordingly.

   Note: For reflective or semi-reflective samples, the tip reflection is easier to bring into focus than the surface, especially if the sample is very flat or clean.

CAUTION: When moving the SPM stage up and down, it is possible to crash the tip into the surface. To prevent a crash while focusing on the surface, watch the optical image **and** tip-to-sample proximity. The sample should be in focus when the tip is 1nm (1000mm) above the surface.

7. For samples which are difficult to bring into focus, move to an edge of the sample, which is easy to find in the optical image, and bring the top of the edge into focus.

8. Move the sample back to the desired X-Y position.

9. Verify that the surface remains in focus.

5.3.5 Move To (X,Y)

This option enables the operator to quickly index the stage to a defined X-Y coordinate. If the origin has not been preset using the **Stage / Set Reference** panel, the stage automatically defaults to the last origin that was previously used. (All subsequent X-Y moves are done from the origin.)

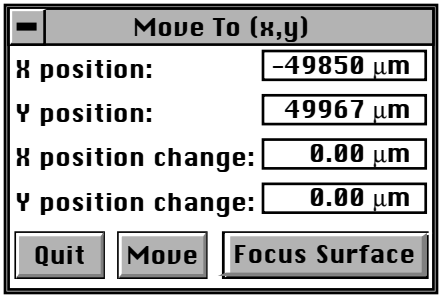
Move To (X,Y) continued...

To move the X-Y stage to a specified X-Y coordinate, complete the following:

1. Verify that the stage origin (position 0, 0) is either:
   - At the default, or,
   - Reset to a new position using the **Set Reference** panel under the **Stage** pop-down menu.
2. Select the **Move To (x,y)** panel under the **Stage** pop-down menu. The screen presents a panel with four fields (See Figure 5.3g).

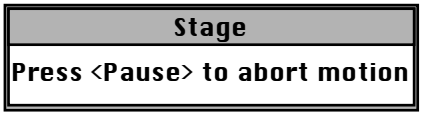
   Note: The first two fields define absolute moves relative to the currently set origin (0,0). The third and fourth fields define relative moves from the *current position.*

Figure 5.3g Move To Prompt



3. Enter the X and Y coordinates of the desired move to position, then click **MOVE**.
4. The stage moves to the new position; meanwhile, the screen reminds the operator to monitor the **F12** button. Should it become necessary to halt movement, click on the **PAUSE** button (See Figure 5.3h).

Figure 5.3h Abort Motion Prompt



Move To (X,Y) continued...

CAUTION: Always verify that the tip is off the surface before attempting stage movements. If manual stage movements are attempted during engagement (by turning the leadscrew knobs on the stage's X-Y slide assemblies) the tip and/or sample may be damaged.

ATTENTION: Il est impératif de toujours s'assurer que la pointe ne touche pas la surface avant de bouger la platine porte-échantillon. Si la platine porte échantillons est déplacée manuellement (en tournant les vis des moteurs de la platine) la pointe et/ou l'échantillon pourraient être endommagés.

WARNHINWEIS: Überprüfen Sie immer zunächst, daß die Meßspitze nicht mehr auf der Oberfläche ist, ehe Sie den XY-Verschiebetisch bewegen. Wenn der XY-Verschiebetisch von Hand bewegt wird (indem die Drehknöpfe an den Gewindestangen des XY-Verschiebetisches gedreht werden), während sich das Mikroskop im Engage-Zustand befindet, können Meßspitze und/oder Probe beschädigt werden.

5. To exit the **Move To (x,y)** dialog box, click the **CLOSE** button (upper-left corner of dialog box), or press the **F12** key.

5.3.6 Set Reference

The **Set Reference** panel is used to set the origin point on the sample surface to be used for all subsequent **Move To** and **Programmed Move** operations. It is important to verify the reference point (0,0) with programmed move sequences, since all moves are relative to the current origin. For example, a program designed to move the stage to the four corners of a square sample when the origin (0, 0) is set to the upper-left corner will *not* probe the same positions if the origin is changed to the lower-right corner. It will avoid confusion to select a standard position as the usual origin reference (e.g., lower-left corner), then reuse the same reference position with all samples.

On samples having a grid-like aspect (e.g., integrated circuits), the reference may be initially defined from a line (two points) rather than a single point. The reference line is defined parallel to some feature on the sample surface (e.g., an electronic trace on an integrated circuit). You may complete this by defining two points: an origin and a second point. Defining the second point compensates for sample rotation. To set a reference on the sample, complete the following:

1. Select the **Focus Surface** function under the **Stage** pop-down menu to focus on the sample and move the sample to the desired origin.

2. Select the **Set Reference** option under the **Stage** pop-down menu. The screen offers five options to the operator (See Figure 5.3i).

Figure 5.3i Set Reference Prompt

3. Click **ORIGIN** in the **Set Reference** panel; this is the first of two points to be used for defining a reference line on the coordinate grid.

4. Quit the **Set Reference** menu, and use the **Focus Surface** option and trackball to either move the stage right or upward as necessary.

Set Reference continued...

Move Stage Right

a. Move the stage to the right to a second point on the linear feature. (This second point, along with the point of origin, defines the X-axis.)

b. Quit the **Focus Surface** option, and return to the **Set Reference** option under the **Stage** pop-down menu.

c. Click on the **X Axis** option. This establishes the (Y = 0) reference line.

Move Stage Upward

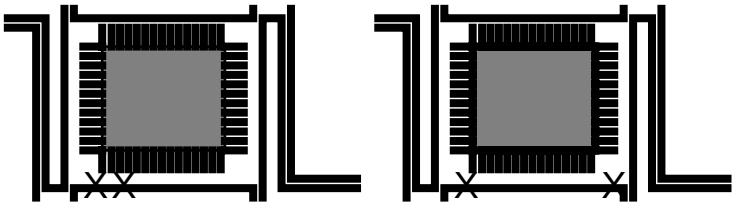
a. Move the stage upward to a second point on the linear feature. (This second point, along with the point of origin, defines the Y-axis.)

b. Quit the **Focus Surface** option, and return to the **Set Reference** option under the **Stage** pop-down menu.

c. Select the **Y Axis** option. This establishes the (X = 0) reference line.

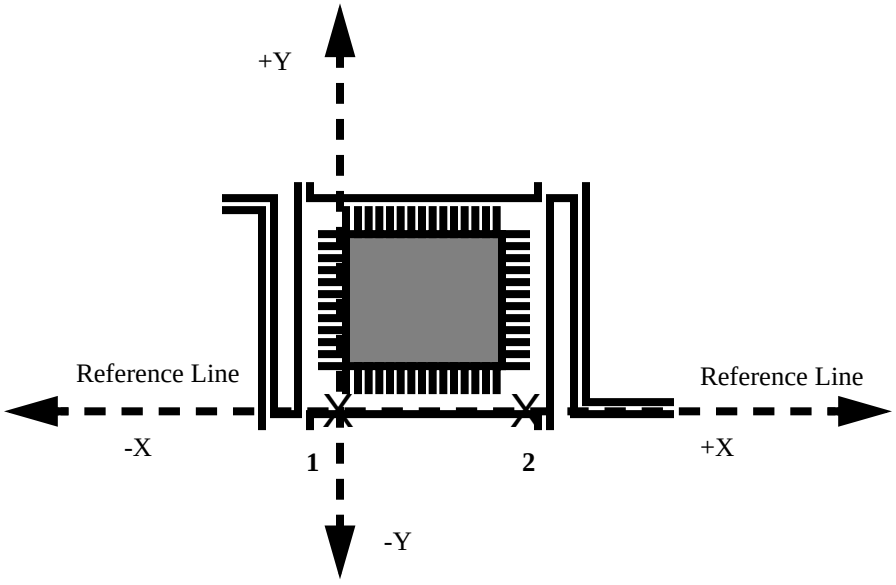
Note: Accuracy improves if the two points defining a reference line are located at some distance to each other. For maximum angular accuracy, the two points should be located at opposite sides of the sample (See Figure 5.3j). The left image depicts two closely located points to define the X-axis. The right image depicts a more accurate reference line defined by two, widely spaced points.

Figure 5.3j Defining the X-Axis



Set Reference continued...

Figure 5.3k Resultant Reference Line

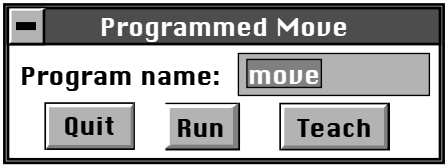


5.3.7 Programmed Move

The **Programmed Move** function allows the stage to be automatically positioned using a series of memorized positions. These positions are programmed into the controller's computer, then executed automatically in sequence. This function is particularly useful for statistical quality assurance runs on large numbers of identical samples, and as a basic inspection aid.

To use this option, select the **Programmed Move** function under the **Stage** pop-down menu. The screen displays a panel of options (See Figure 5.3l).

Figure 5.3l Programmed Move Prompt



The name of the move program currently loaded is displayed in the **Program name** panel (in the above example, the program is called "move"). Moves are first programmed using the **Teach** function. Later they may be executed using the **Run** function.

Teaching a Programmed Move

To program (teach) a series of moves, complete the following:

1. Draw a simple map of the sample, along with each programmed position, before programmed moves are entered. Note the origin point at the time of programming.

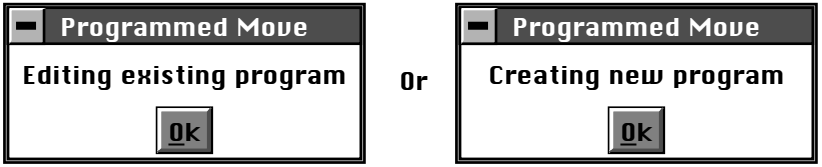
   Note: If reducing cycle time is important, position order should be optimized to reduce stage travel

2. Click on the **Program name** option in the **Programmed Move** panel (See Figure 5.3l) and enter the name to be used.

   Note: Program names must be eight characters or less and follow DOS protocol.

3. From the **Programmed Move** panel, click on **TEACH**. The screen prompts with (See Figure 5.3m):

Figure 5.3m Editing or Creating New Program Prompts



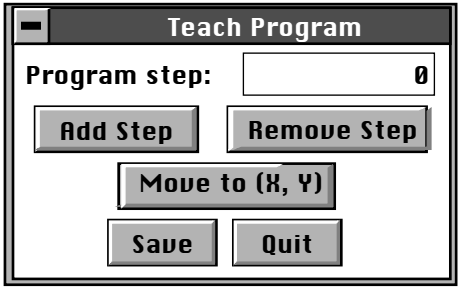
If the program name already exists on the computer.

If the program name is new (not previously used).

4. Click **OK** to proceed with programming moves. A new panel displays, featuring the basic teaching commands (See Figure 5.3n).

5. To add a first step, click **ADD STEP** to invoke the **Teach Mode** panel (See Figure 5.3n).

Figure 5.3n Teach Program Prompt



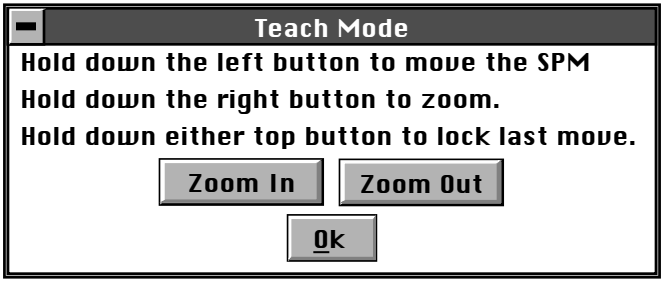
6. Use the controls to position the stage and sample to the desired location(s) to be programmed into the computer.

Note: These controls are identical to the **Focus Surface** controls explained in Chapter 5 of this manual.

Programmed Move continued...

7. Click on **OK** in the **Teach Mode** panel when the stage has0 moved to a desired position (See Figure 5.3o).

Figure 5.3o Teach Mode Prompt



8. To add another position, repeat steps 4-6 until all desired positions have been programmed up to a maximum of 100 steps.

   Note: Each time the **Teach Program** panel is reentered, the **Program step** increments by another count and automatically assigns a program step number.

9. When all stage positions are entered into the program, click **SAVE** in the **Teach Program** panel, then click **QUIT**. Verify the program is entered and saved under the correct program name.

   Note: Save steps before exiting the **Teach Program** panel; otherwise, programmed steps will be lost.

10. Enter additional programs in the same manner, using a separate program name for each.

Other Information Regarding the Teach Program Option

To remove a step from the programmed sequence:

1. Go to the **Stage** pop-down menu to the **Programmed Move** panel, and select **Teach Program**.

2. Select **Program step** from the **Teach Program** panel.

Other Information Regarding the Teach Program Option continued...

3. Enter the step number to be removed or drag the mouse to index to the step number. The stage simultaneously moves to the new step position.

4. Select the **Remove Step** option.

   Note: When individual program steps are removed, all subsequent steps are "moved up" by one count.

5. Click **SAVE** to save the edited version of program.

To insert a step into an existing program sequence:

1. Go to the **Stage** pop-down menu to the **Programmed Move** panel, and select **Teach Program**.

2. Select **Program step** in the **Teach Program** panel.

3. Enter *one less* than the step number to be added to the sequence, or drag the mouse to index to the desired step number.

   Note: For example, to add a new program step #7, while leaving all preexisting steps intact, enter "6" in the **Program step** panel. The stage simultaneously moves to the preexisting step position #6.

4. Select the **Add Step** option. The **Teach Mode** panel appears.

5. Use the trackball to move the stage to the new position to be added.

6. Click **QUIT** to exit the **Teach Mode** panel. Verify the new position is now added.

   Note: When new program steps are added, all preexisting steps beyond the new entry are "moved up" by one count.

7. Click **SAVE** to save the edited version of program.

Programmed Move continued...

Origin Points

Programmed Move positions are memorized relative to the current origin at the time of programming. If the origin has been shifted from its original position since the time of programming, it is necessary to reestablish the original origin point to locate the same positions on the sample. When generating maps of programmed moves, always indicate the origin point.

Running a Programmed Move

Once a program sequence has been taught to the computer, you may run the program from the **Programmed Move** panel. The stage moves to each position in the same order taught, relative to the current origin point. At each program position, the sample is scanned for 1.5 frames, captured, and then indexed to the next position. The system stores captured data from each position is stored on the hard disk; there should be sufficient memory to record 100 frames at 256 samples each.

To run a series of programmed moves, complete the following:

1. Select **Programmed Move** from the **Stage** pop-down menu.

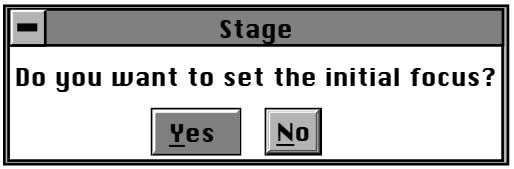
2. Select the **Program name** field, and enter the name of the program to be run.

3. Select the **Run** option. If you are loading and running the program from the beginning, the screen prompts the operator whether to refocus the screen.

    a. Clicking **YES** transfers you to the **Focus Surface** panel; you may then make focusing adjustments to better view the surface.

    b. Clicking **NO** initiates the programmed move from step #1.

Figure 5.3p Initial Focus Prompt



Note: If the program was previously run without finishing (aborted), the screen requests whether to begin the program sequence at the aborted step.

4. If you wish to run the entire program from its beginning (step #1); click **NO**. Otherwise, click **YES** (See Figure 5.3p).

5. The screen displays the current program step in progress (See Figure 5.3q). To initiate the program sequence from another starting point, use the **Teach Program** panel to remove any unwanted step(s), and run the program again from the new “step 1” position.

Figure 5.3q Programmed Move Prompt

5.3.8 Initialize

The **Initialize** option allows the system software to locate the top (positive) limit switches on the SPM axis and optics axis stages. If the NanoScope computer determines that it is unsure of the stage position, it will not allow any functions under the **Stage** menu (except **Initialize**) to be selected.

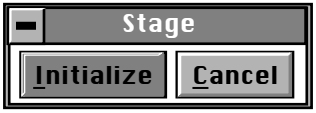
To initialize the stage, complete the following:

1. Before beginning initialization, verify that the stage is clear of loose items and debris.

Stage Menu Commands continued...

2. Select **Initialize** from under the **Stage** pop-down menu. The dialog box offers two options (See Figure 5.3r).

Figure 5.3r Stage Initialize/Cancel Prompt

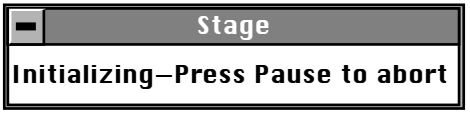


3. To begin initialization, click **INITIALIZE**. As the stage begins a series of motorized movements, the screen indicates operating status (See Figure 5.3s).

Figure 5.3s SPM Move to Top of Travel Prompt

Note: The initialization sequence may be aborted at any time by clicking **PAUSE** (See Figure 5.3t).

Figure 5.3t Stage Initializing Prompt

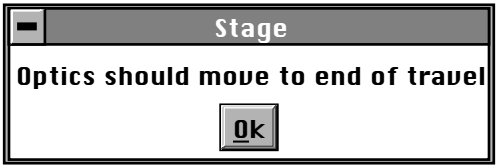


Initialize continued...

4. The microscope's Z-axis moves to the top of its travel. When this is achieved, click **OK** to continue.

5. The microscope focuses its camera optics to the extent of its travel. When this is achieved, click **OK** to continue (See Figure 5.3u).

Figure 5.3u Optics Move to End of Travel Prompt

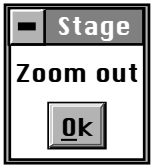


6. Next, the camera zoom optics assembly zooms. When the camera zooms in to the limit of its travel, click **OK** to continue (See Figure 5.3v).

Figure 5.3v Stage Zoom Prompt

7. Finally, the camera's optics zooms out. When the camera zooms out to the limit of its travel, click **OK** to complete the stage initialization process (See Figure 5.3w). The dialog box removes itself from the screen.

Figure 5.3w Stage Zoom Out Prompt



5.3.9 SPM Parameters

The SPM parameters menu lists the most important Z-axis parameter values for loading/unloading the stage and engaging samples. These values are explained in more detail in Chapter 7.

Chapter 6 Cantilever Preparation

6.1 Overview

The Dimension 3100 Scanning Probe Microscope comes furnished 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:

- **Overview:** Section 6.1
- **Silicon Cantilever Substrates:** Section 6.2
- **Silicon Nitride Cantilever Substrates:** Section 6.3

6.2 Silicon Cantilever Substrates

ATTENTION: The cantilevers are stored tip-side-up and that the silicon is very brittle. Contacting the cantilever during this operation will break it off of the substrate.

6.2.1 Wafer Tool Kit

A wafer tool kit for working with silicon cantilever substrates is included with the Dimension 3100 SPM system. The kit contains the following:

- Wafer tweezers
- Flat, substrate tweezers
- Regular tweezers
- Curved, sharp-pointed tweezers
- Flat, L-shaped tweezers

6.2.2 Cantilever Preparation

The silicon cantilever substrates used in TappingMode™ are removed from the wafer with the following procedure.

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 6.2a).

Cantilever Preparation continued...

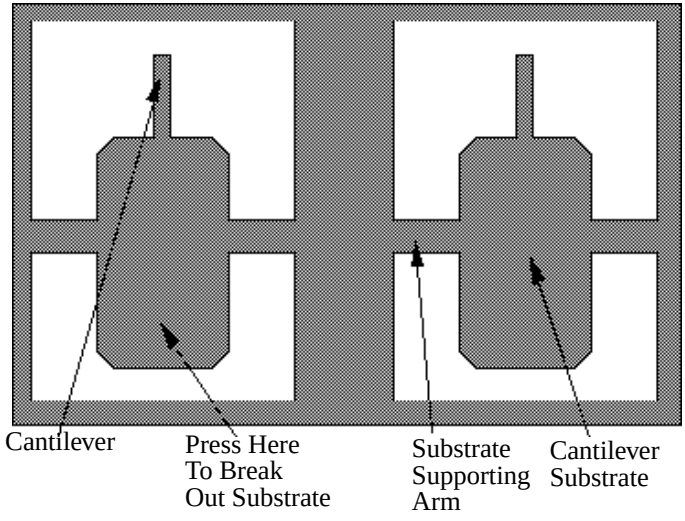
Note: The supporting arms connecting the substrate to the bulk of the wafer shatter when pressure is applied. It may be convenient to break several substrates from the wafer at one time. Extras may be safely stored in a specially prepared petri dish.

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 6.2a Silicon Cantilever Substrates in Wafer



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.

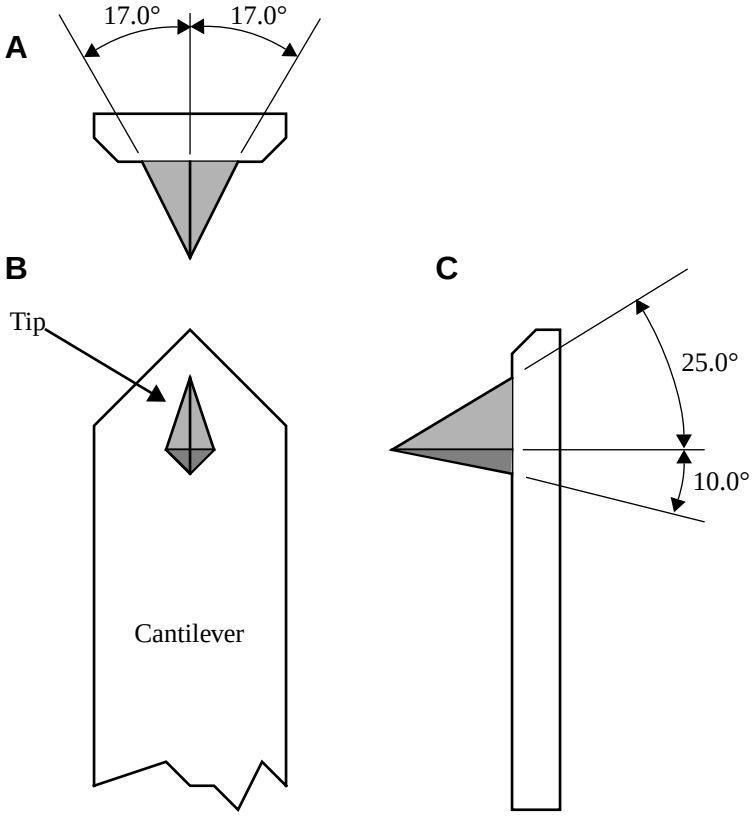
Note: It may be helpful to tip the substrate to one side to help grasp it in the tweezers.

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

6.2.3 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 6.2b).

Figure 6.2b Theoretical Tip Shape of Silicon Probes

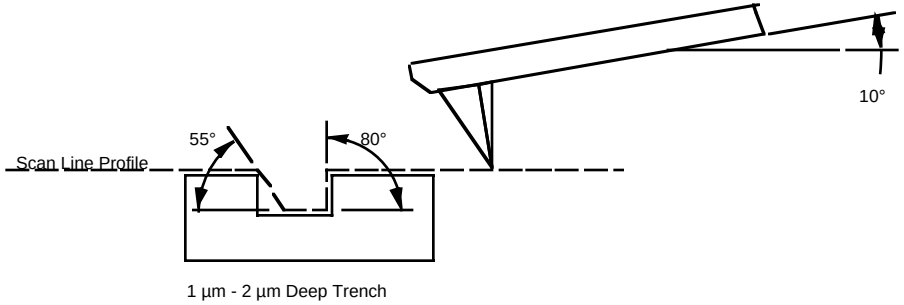


Tip Shape of Etched Silicon Probes continued...

The present process creates a tip which is symmetric from side-to-side with a 17± 2 ° half cone angle (See A of Figure 6.2b) and asymmetric from front-to-back, along the length of the lever (See C of Figure 6.2b).

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 6.2c Silicon Probe Tip Profile Artifact (Front-to-Back)



Note: Any wall angle on the left wall that is > 55 deg. will be shown as 55 deg. in the image.

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

Scan direction = 0 degrees

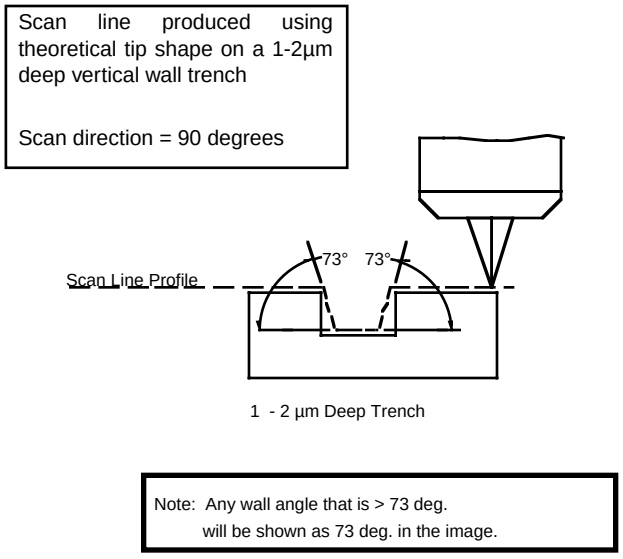
Tip Shape of Etched Silicon Probes continued...

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 6.2c). Using the back edge, step angles approaching 90 degrees can be measured routinely, depending on the step height.

ATTENTION: 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 6.2d Silicon Probe Tip Step Profile Artifact (Side-to-Side)



Tip Shape of Etched Silicon Probes continued...

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 6.2e).

Figure 6.2e Common Silicon Probe Profile (Resultant Scan Artifact)

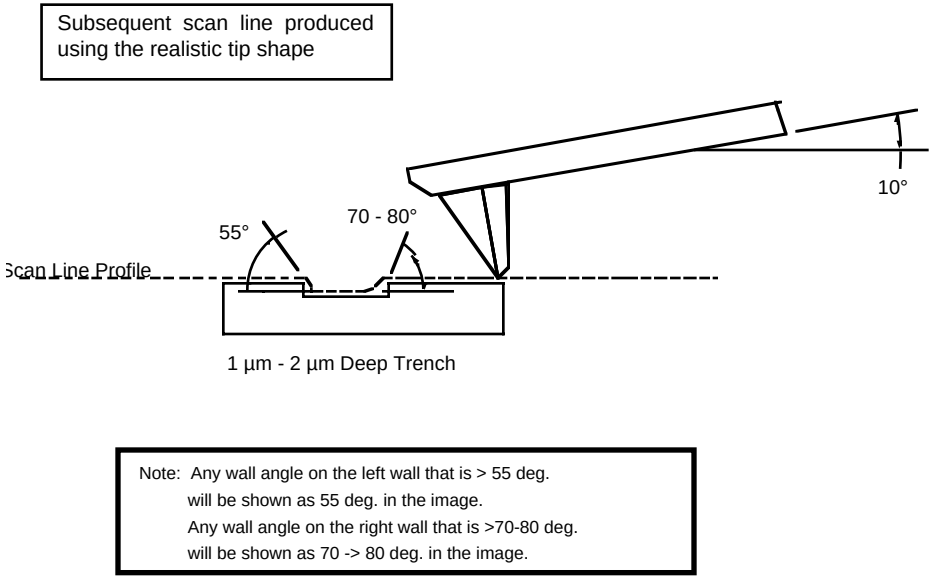


Figure 6.2e depicts the resultant effect of the angled back ridge on the step angle measurement for a deeper trench depth. This is tip and topography dependent.

In addition to microscopic scale shape characteristics (See Figure 6.2b-Figure 6.2e), 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 5–10 nm (on a very good tip).

6.3 Silicon Nitride Cantilever Substrates

When using the Dimension 3100 microscope for the first time, begin with the provided strip of cantilevers and skip to Step 5 of the following instructions. Step 5 provides instructions on how to separate substrates from strips.

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

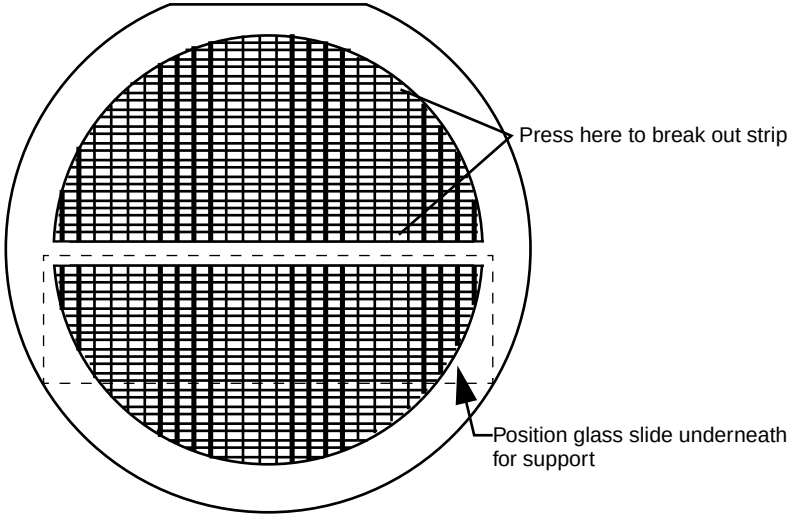
2. Inspect the wafer with an optical microscope.

   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, tungsten carbide scribe and a pin-vise.

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

4. Applying downward pressure with the tweezers until the strip breaks free from the silicon ring (See Figure 6.3a).

Figure 6.3a Silicon Nitride Cantilevers in a Wafer



Silicon Nitride Cantilever Substrates continued...

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.

ATTENTION: 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.

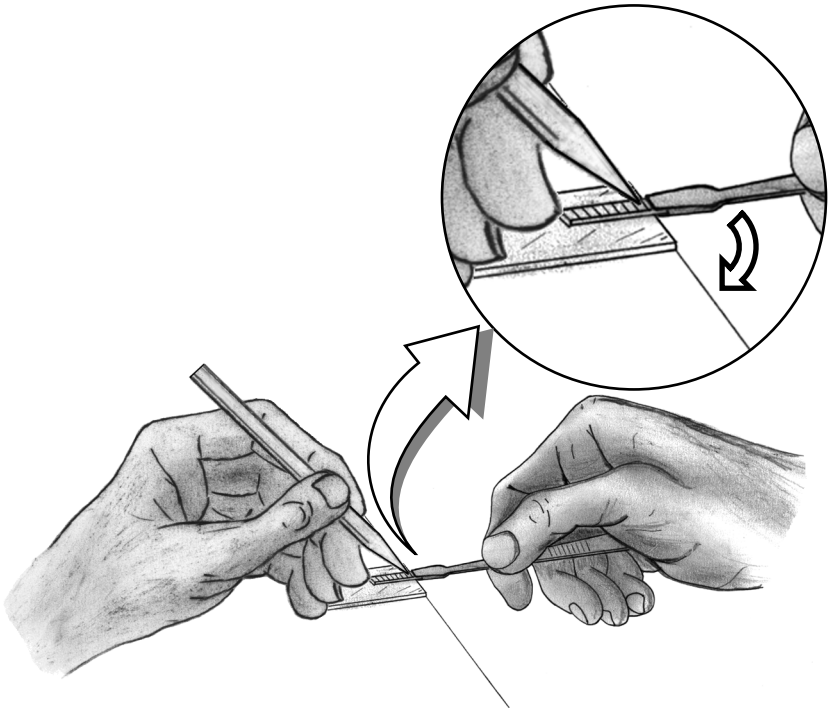
6. Handling the strip by its ends, place it on a glass slide (taped down to the edge of a table) with the substrate or spacer piece to be removed hanging over the edge (See Figure 6.3b).

   Note: 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 6.3b Substrate Break-off



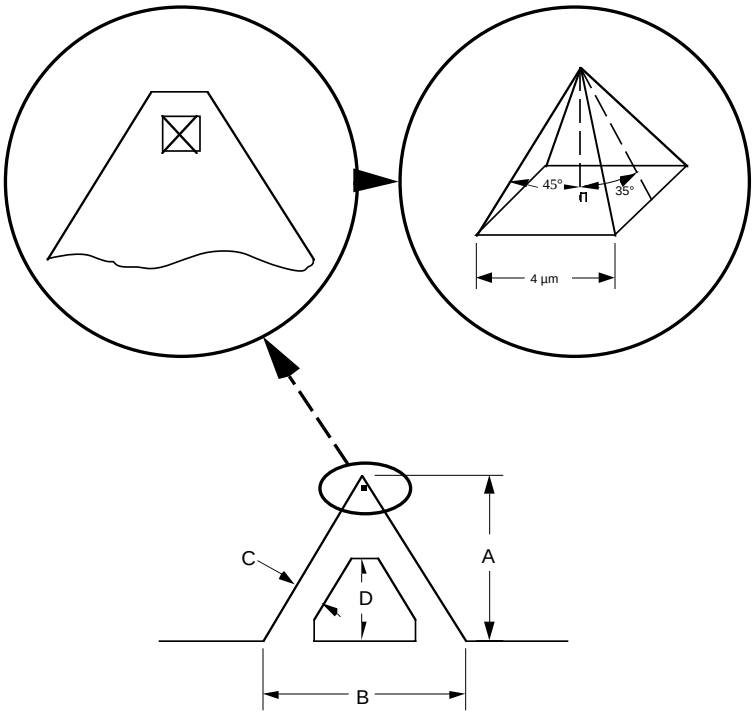
Note: Extra substrates are easily stored in a covered petri dish. The shipped substrates are secured with X0-grade, GEL-PAK™ adhesive strips. The strips are used to permit easy removal of the substrates. If GEL-PAK adhesive strips cannot be found, a simple substitute is the adhesive area from a Post-it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

Each substrate has two cantilevers on each end of the substrate. Both 100 and 200-micron 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 200-micron cantilever with the wider legs. For atomic scale images, the 100-micron triangular cantilever with the wider legs yields good results.

6.3.1 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 6.3c along with dimensions and approximate values for spring constants and resonant frequencies.

Figure 6.3c Silicon Nitride Cantilevers

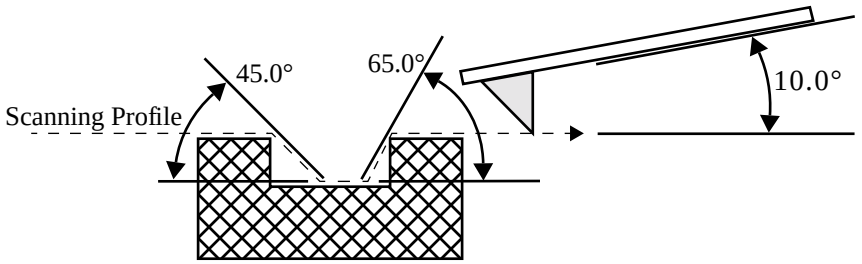


Lever Type	A	B	C	D	Spring constant (N/m)	Measured Fr (kHz)
100 µm Wide	115	122	21	60	0.58**	40
200 µm Wide	193	205	36	113	0.12**	12.3-22.1 †
100 µm Narrow	115	122	15	69	0.38**	-
200 µm Narrow	193	205	20	150	0.06**	-

Tip Shape of Silicon Nitride Probes continued...

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 6.3d) 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 6.3d 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, & Sample Preparation

7.1 Overview

This chapter includes information regarding the Dimension 3100 Scanning Probe Microscope (SPM) setup and operation procedures for Contact Mode and Tapping Mode. Specifically, this chapter details removal and installation of the microscope head, mounting the cantilever, changing the tip, loading and positioning samples, focusing the optics, and general information regarding engaging and withdrawing the tip.

Chapter 10 discusses SPM operation in Tapping Mode in more detail, while Chapter 8 reviews SPM operation in Contact Mode AFM. This chapter contains the following:

- **Overview:** Section 7.1
- **System Information:** Section 7.2
- **Basic AFM Operation:** Section 7.3
- **Advanced AFM Operation:** Section 7.4

7.2 System Information

7.2.1 Mouse versus Trackball

The mouse exclusively operates the NanoScope software with the exception of functions related to direct control of the stage. These commands are only initiated with the trackball. Operator-initiated movement of motors via the Zoom, Focus, Move SPM (Z-stage) and Move XY (in motorized version) commands are controlled by the trackball and its buttons. Motor operation is permitted only when the appropriate menu enables movement.

7.2.2 Motor Interlock

SPM stage movement is software interlocked against motion if a valid sum (laser signal) is not present. The sum signal must be between 0.5-9.5 volts, otherwise all commands which try to initiate stage motion will not move the SPM X, Y, or Z stages. However, an invalid sum/laser signal still permits upward Z-stage travel. This interlock reduces the possibility of inadvertently crashing the tip and scanner crystal into the sample or stage, minimizing tip and scanner damage. If during the engage sequence the sum signal changes to a value outside the interlocked limits, the downward motion stops, preventing damage to scanners had the Z-stage continued downward.

7.2.3 Laser Requirements

CAUTION: When the Dimension head is plugged into the microscope control electronics, laser light emits.

CAUTION: The Dimension head features an internal tilt switch to shut power off to the laser whenever it is inverted, however, heed all precautions below.

This instrument uses a semiconductor laser emitting a maximum 1.0mW beam at 670 nm. The light is emitted down the center of the scanner tube and during normal operation reflects back into the system’s optics from the back surface of the cantilever probe.

Laser Requirements continued...

WARNING: During and prior to set up of the laser, it is important to avoid looking directly at the laser beam or at the laser spot. Never plug the laser head should never be plugged into the microscope control electronics unless the head is installed in the Z-stage mount. Take care when inserting highly reflective samples on the chuck. Avoid looking at all reflected laser light. Use care to avoid staring into beams that may be reflected from sample surfaces.

ATTENTION: Avant de faire fonctionner le laser, et durant tout le temps pendant lequel il fonctionne, il est impératif de ne pas regarder directement le faisceau du laser ou l'image qu'il réfléchit. La sonde laser ne doit jamais être branchés sur l'électronique de contrôle du microscope, tant que la téte de mesure n'est pas installée dans son support. 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. Der Laser sollte niemals an die Mikroskopelektronik angeschlossen werden, wenn er nicht in der Halterung der Z-Verschiebeeinheit installiert ist. 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.

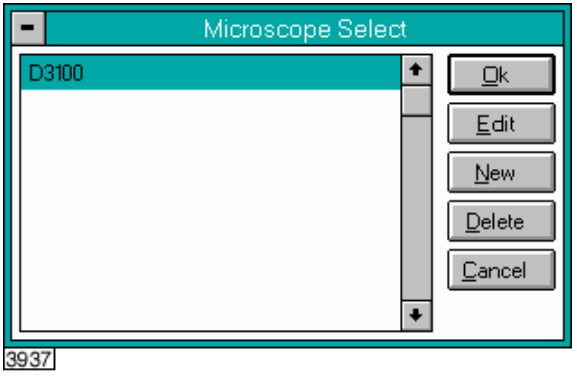
7.3 Basic AFM Operation

7.3.1 Select the Microscope Head

1. Set the microscope head configuration by selecting the **Microscope Select** panel from the **Di** pop-down menu (See Figure 7.3a).

2. Click **OK** to close the **Microscope / Select** dialog box. When enabled, the selected buttons are black.

Figure 7.3a Microscope Select Prompt



7.3.2 Select Mode of Operation

1. Select **Microscope/Profile**.

2. Select the desired mode of operation and click **OK**.

Note: Contact Mode AFM and TappingMode are the primary modes of operation used with the Dimension 3100 SPM.

7.3.3 Prepare the Cantilever Holder

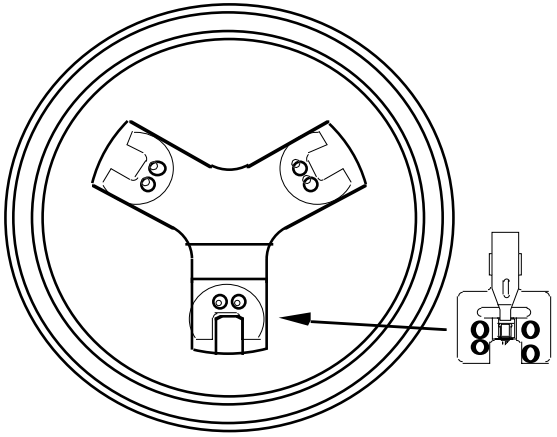
Before loading the substrate onto the cantilever holder, place the cantilever holder on the cantilever holder stand (See Figure 7.3b). The cantilever holder stand is a 2.5-inch diameter black anodized aluminum cylinder that ships with the Dimension 3100 SPM for easy installation of the substrate onto the cantilever holder.

Basic AFM Operation continued...

The cantilever holder stand has three stations: 1) standard AFM, 2) fluid imaging AFM and 3) STM. A small block in the center of the gold connector identifies the standard AFM load station. The center block helps to support the cantilever holder you place the holder on the cantilever holder stand.

To prepare the cantilever holder, with the AFM cantilever holder's large spring clip face up, mate the standard AFM cantilever holder sockets to the pins of the AFM cantilever holder stand (See Figure 7.3b).

Figure 7.3b Cantilever Holder Stand (top view)



Note: You may install the AFM cantilever holder in only one orientation because the pins are asymmetrical.

7.3.4 Load the Cantilever Holder

Contact Mode AFM

Silicon nitride substrates are used for SPM operation in Contact Mode AFM. Silicon nitride substrates consist of a cantilever integrated with a sharp tip on the end. For Contact Mode AFM imaging, the cantilever must be soft enough to deflect very small forces and have a high enough resonant frequency to avoid vibrational instabilities.

Note: Install silicon nitride substrates face-up so the tip points away from the AFM cantilever holder. This ensures that the cantilever and tip face toward the sample once the cantilever holder is mounted on the head.

To install a silicon nitride substrate on the AFM cantilever holder, complete the following:

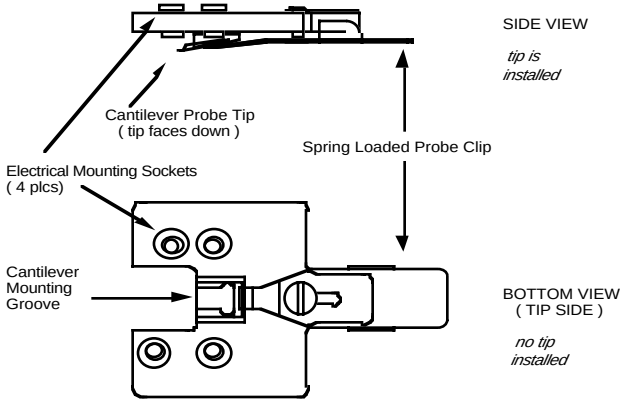
1. Using sharp tweezers, grasp the substrate firmly on the sides and lift to free the substrate from the wafer or gel-pack.

   Note: Refer to Chapter 6 for detailed information on how to break out each substrate from the wafer.

CAUTION: Do not turn the substrate upside-down. Loading a substrate on a cantilever holder upside-down can permanently damage the cantilever and tip.

2. Press down and slide back the spring clip of the standard AFM cantilever holder (See Figure 7.3c).

Figure 7.3c Standard AFM Cantilever Holder



3. Verify that the substrate is face-up (gold-plated side down) with the nitride film side attached to the cantilever oriented up away from the cantilever holder.

Load the Cantilever Holder continued...

4. Place the substrate on the AFM cantilever holder groove.

5. Carefully maneuver the substrate until the substrate is flush against one side and laying flat in the cantilever holder groove.

6. Press the spring-loaded probe clip down, gently push forward over the substrate, and release the spring clip to hold the substrate in place in the cantilever holder groove.

7. Verify the substrate remains flush against one side and lies flat in the cantilever holder groove. This keeps the tip oriented in the correct direction.

> **Note:** Although this step is not required, it will improve repeatability of tip location between runs which makes aligning the laser onto the cantilever quicker and easier.

TappingMode

The procedure for installing TappingMode, single crystal silicon substrates is the same for installing silicon nitride substrates in Contact Mode AFM.

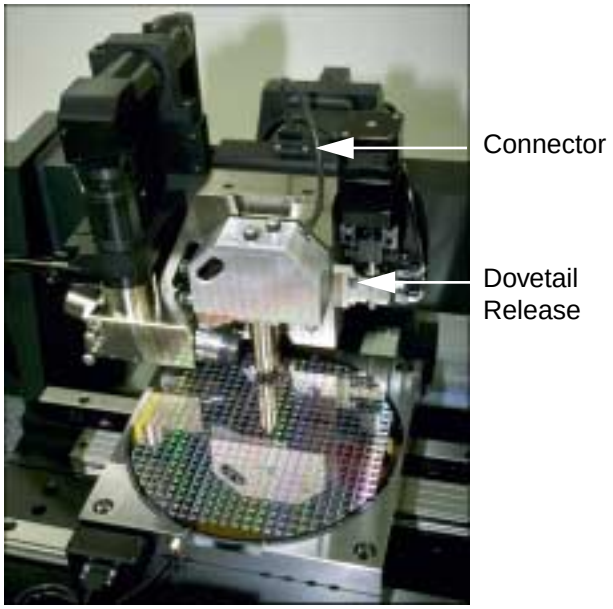
> **Note:** Single crystal silicon substrates are installed face-up so the tip points away from the AFM cantilever holder. This ensures that the cantilever and tip face toward the sample once the cantilever holder is mounted on the head.

7.3.5 Remove the Dimension SPM Head

1. Tighten the screw located on the right side of the Dimension SPM head dovetail to release the Dimension SPM head (See Figure 7.3d).

Load the Cantilever Holder continued...

Figure 7.3d SPM Head Dovetail and Signal Connector



Note: Dovetail engagement actuates with a spring. Failing to engage the spring-loaded dovetail causes a large increase in image noise due to reduced rigidity of the mechanical support of the SPM head.

2. Carefully slide the Dimension SPM head up and out of the dovetail groove.

7.3.6 Install the Cantilever Holder

1. Mate the cantilever holder sockets to the Dimension SPM head pins to install the loaded AFM cantilever holder.

2. Verify the tip points down and away from the head. The tip-end of the cantilever end must point in the direction of the optics assembly.

3. Verify the AFM cantilever holder mounts flat against each pin on the end of the head.

Basic AFM Operation continued...

7.3.7 Replace the Dimension SPM Head

1. Carefully slide the Dimension SPM head down into the dovetail groove of the Z-stage SPM.

2. Loosen the screw located on the right side of the Dimension SPM head dovetail to lock the head in place (See Figure 7.3d).

3. Verify that sufficient clearance exists between the sample and tip to prevent the tip and head scanner from crashing into the stage or sample upon engaging.

4. If it appears the Dimension SPM head may crash once fully inserted, remove the Dimension SPM head and execute the **Motor / Withdraw** command several times.

7.3.8 Connect the Dimension Head

Insert the Dimension SPM head black 21-pin connector plug into the socket just behind the Z-stage located on the stage control electronics box (See Figure 7.3d).

7.3.9 Align Laser

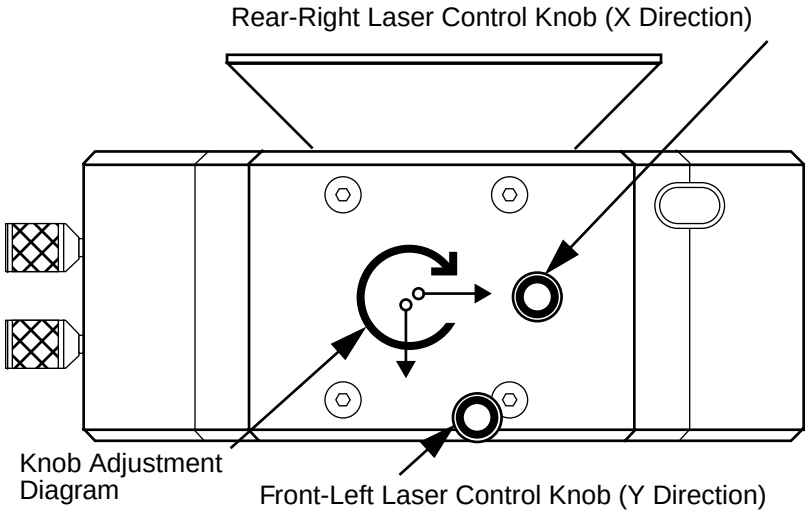
ATTENTION: Turn down the illuminator intensity before proceeding with laser alignment.

For both Tapping Mode and Contact Mode AFM, the user aligns the laser by moving the laser beam relative to the cantilever while observing the laser spot on the granite surface (a piece of white paper also works well) below the Dimension 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.

Figure 7.3e displays the laser control knobs located on the top of the Dimension head. There is a diagram printed on top of the Dimension head illustrating which direction the laser moves when turning the laser control knobs counter-clockwise.

Align Laser continued...

Figure 7.3e Dimension Head Laser Control Knobs



The X direction runs along the major axis of the substrate (parallel to the length of the cantilever). The right-rear laser control knob, atop the SPM head, controls the laser beam movement along the X direction. The front-left laser knob, atop the SPM 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 between Tapping Mode and Contact Mode AFM. The sections below detail the procedures for aligning the laser on the cantilever and tips for each mode respectively.

Etched Silicon Tips (Tapping Mode)

1. Execute the **Stage / Load New Sample** command to position the sample stage to the front.

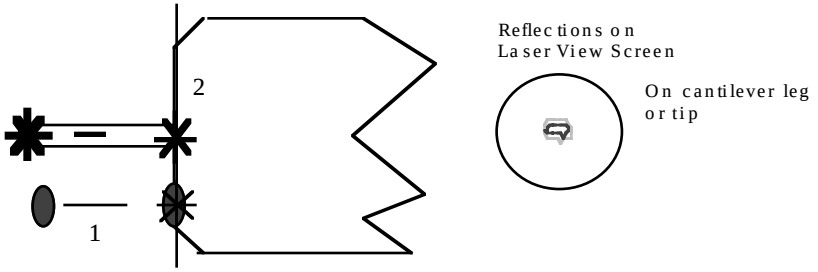
   Note: This positions the stage out of the optical objective's line of sight to provide a dark background and improve image quality during laser alignment.

Align Laser continued...

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

3. Turn the rear-right laser control knob clockwise to move the laser in the X positive direction until the laser spot disappears from the surface below. 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 7.3f).

Figure 7.3f Etched Silicon Tip Laser Alignment



4. Turn the front-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. The laser is now positioned over the cantilever (See Point 2 in Figure 7.3f).

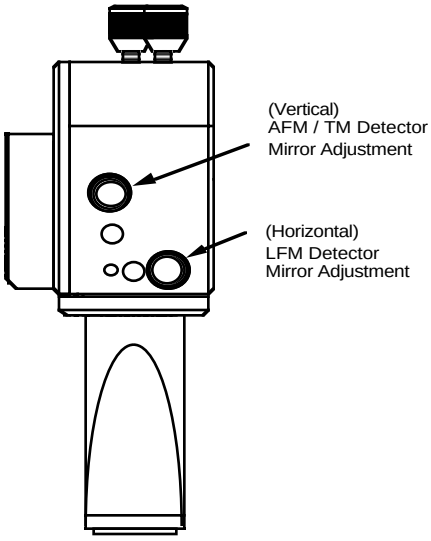
5. Verify that the laser is deflecting off the cantilever by moving the laser on, over, and off the cantilever by turning the front-left laser control knob less than 1/8 of a turn.

6. Turn the rear-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 rear-right laser control knob clockwise until the spot disappears from the surface below (See Point 3 in Figure 7.3f).

8. Verify that a laser spot appears in the Dimension head filter screen. If there is not laser spot, adjust the photodetector mirror using the photodetector adjustment knobs located on the left side of the SPM head (See Figure 7.3g).

Figure 7.3g Photodetector Adjustment Knobs



Note: You may now restore the illuminator lamp to increased intensity for improved video image quality.

Silicon Nitride Tips (Contact Mode AFM)

1. Execute the **Stage / Load New Sample** command to position the sample stage to the front.

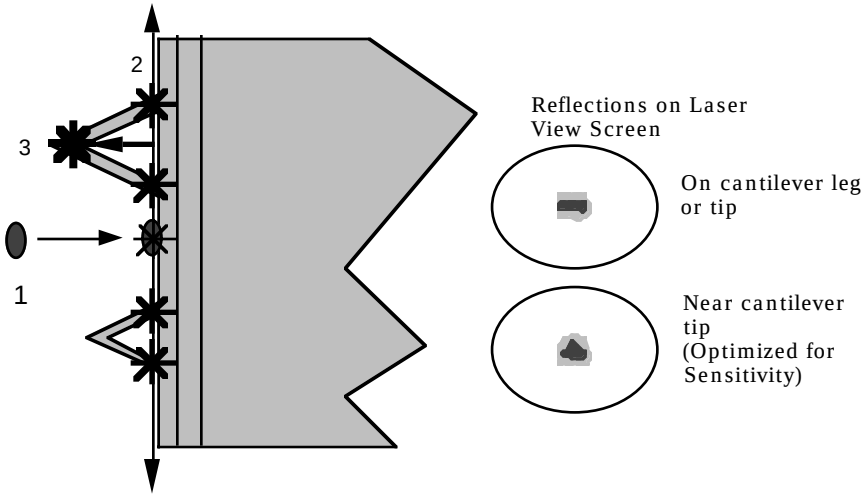
   Note: This positions the stage out of the optical objective's line of sight to provide a dark background and improve image quality during laser alignment.

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

Silicon Nitride Tips (Contact Mode AFM) continued...

3. Turn the rear-right laser control knob clockwise to move the laser in the X positive direction until the laser spot disappears from the surface below. Turn the right-rear laser control knob counter-clockwise until the laser spot just reappears and stop turning the knob. The laser is now positioned at the edge of the substrate (See Point 1 in Figure 7.3h).

Figure 7.3h Silicon Nitride Laser Alignment



4. Once the laser is positioned at the end of the substrate, use the front-left laser control knob to move in the Y direction (parallel to the edge of the substrate) until the laser crosses a maximum of four legs on the two V-shaped cantilevers (See Point 2 in Figure 7.3h).

> **Note:** This occurs when moving in one direction (either up or down from end to end). You should detect four distinct occurrences of the laser spot disappearing and reappearing on the surface below.

5. If the laser is positioned between a pair of legs of one cantilever (laser spot on surface below) turn the rear-right laser control knob counter-clockwise to move the laser left in the X direction until the laser spot disappears on the surface below (See Point 3 in Figure 7.3h).

Silicon Nitride Tips (Contact Mode AFM) continued...

6. Verify the laser is located on the portion of the cantilever connecting the two lever arms near the tip location (See Figure 7.3h).

7. Turn the rear-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 (See Figure 7.3h).

8. Move the laser onto the tip-end of the cantilever by reversing the direction of the rear-right laser control knob clockwise until the spot disappears from the surface below.

9. Verify that a laser spot appears in the Dimension head filter screen. If there is not laser spot, adjust the photodetector mirror using the photodetector adjustment knobs located on the left side of the SPM head.

7.3.10 Adjust Photodetector

1. Verify that the SPM head is fitted with a tip.

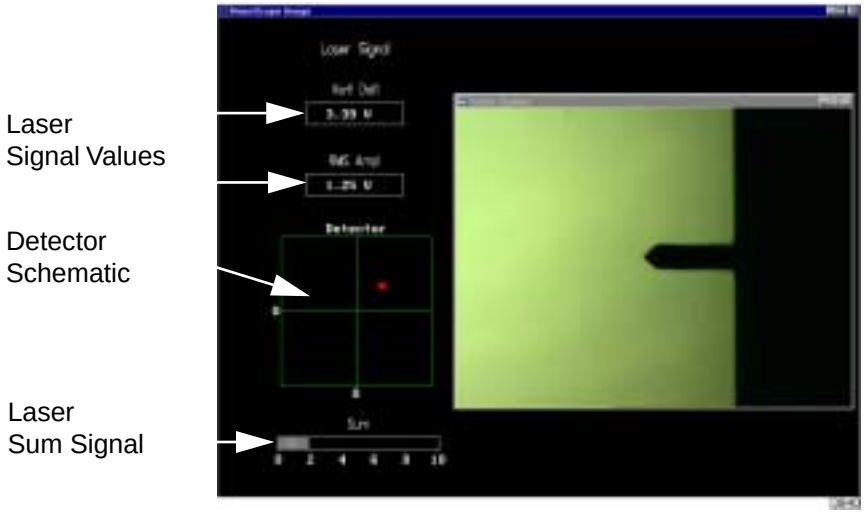
2. Verify that the laser beam is positioned on the back of the cantilever, with a spot visible on the Dimension head filter screen.

3. Verify there is an appropriate sum signal displayed on the image monitor (See Figure 7.3i).

    - **Contact Mode AFM: 4-6 volts**
    - **Tapping Mode: 2 volts**

Basic AFM Operation continued...

Figure 7.3i Vision System Window



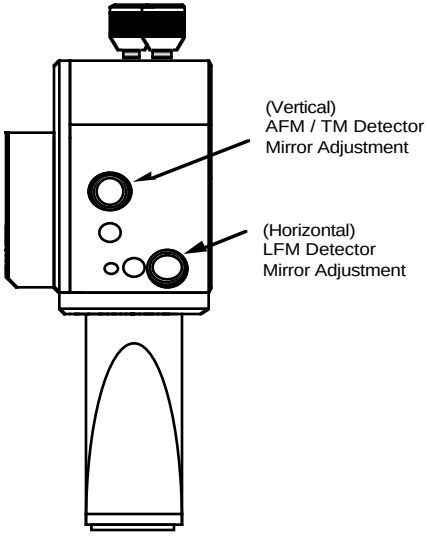
4. Center the laser detector signal using the photodetector adjustment knobs located on the left side of the Dimension head.

Note: The image monitor displays the laser signal values and a schematic of the detector quadrants labeled **Detector**. The position of the laser is denoted by a red dot on the detector schematic (See Figure 7.3i).

Note: The **RMS Ampl** is an AC signal and does not have any real magnitude until the cantilever tune has been completed (Tapping Mode only).

Note: The detector graphic on the computer screen has the laser side-to-side (long axis) in the dark screen on the SPM head.

Figure 7.3j Photodetector Mirror Adjustment Knobs



5. For **Contact Mode AFM** only, offset the vertical deflection value to **-2 volts** using the photodetector adjustment knobs.

7.3.11 Locate Tip

For initial start-up where the Z-stage and optics have been almost fully retracted, use the **Focus Surface** command to move the tip closer to the surface. It is easier to focus on the tip with more light reflecting from the surface into the optics.

To locate the tip, complete the following steps:

1. Initiate the **Locate Tip** command in the **Stage** menu by clicking on **Locate Tip**.

   Note: This command locates the tip position (Z height) using optical focal distance measurements. When this step completes, the computer records the tip position in memory.

2. Zoom out as far as possible. This helps you locate the cantilever.

Basic AFM Operation continued...

3. Once you locate the tip, use the optic adjustment knobs (lower-left corner of the zoom optics assembly) to align the optical microscope lens so the tip is centered in the video display window.

4. Focus on the cantilever using the trackball.

5. Quit the **Locate Tip** sequence by pressing **ENTER** on the keyboard or clicking **OK** at the bottom of the **Locate Tip** dialog box.

7.3.12 Load the Sample

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

Small Sample Preparation

Place the calibration sample or other small sample on one of the 1.5nm diameter metal disks used for sample mounting. The Dimension 3100 SPM ships with several steel sample disks that you may attach to a magnetic sample holder located in the Dimension Accessories Kit. Also provided with the instrument are red and white colored double-sided adhesive patches, or “sticky tabs,” designed to hold the sample chip to the disk.

1. Peel off a “sticky tab” from the provided sheet, and place it on the steel small sample puck (See Figure 7.3k).

2. Peel off the red-and-white paper to leave a patch of the two-sided adhesive on the steel sample disk holding the sample chip to the disk.

3. Using tweezers, place the small sample to be imaged firmly on the adhesive (See Figure 7.3l).

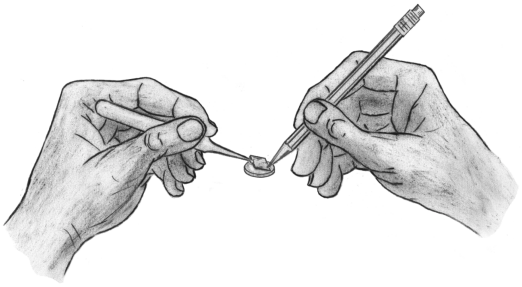
> **Note:** Alternatively, you may glue a small sample down to the sample puck using cyanoacrylate glue (super glue).

4. Place the small sample disk on the magnetic small sample holder.

Note: The small sample holder provided with the Dimension 3100 is not optimal for imaging soft, magnetic materials due to the magnetic hold-down properties. If magnetic imaging is intended, use double-sided tape to hold your sample onto the chuck directly. Or, if your sample is flat enough, use the vacuum chuck.

Figure 7.3k Securing Double-sided Tape to the Sample Disk

Figure 7.3l Securing the Sample



5. Secure the sample atop the stage.

Large Sample Preparation

You may place large, flat samples directly on the vacuum chuck which are held down using vacuum. To engage the vacuum, use the toggle switch labelled **Vacuum** on the front of the black SPM electronics box.

Basic AFM Operation continued...

7.3.13 Focus Surface

1. Select the **Focus Surface** option under the **Stage** pop-down menu, or click on the **Focus Surface** icon.
2. Focus on the sample surface by rolling the trackball up or down while pressing the bottom-left button. This adjustment raises or lowers the vertical engage stage on which the SPM and optics are mounted.
3. To move long distances hold both left trackball buttons down simultaneously and roll the trackball with high speed to lock the peak speed of motion. Release these two buttons to stop the motors.

CAUTION: Be careful when making this adjustment to ensure that the tip does not hit the sample surface.

7.3.14 Cantilever Tune (TappingMode only)

1. Select **Cantilever Tune** from the **View** pop-down menu, or click on the **CANTILEVER TUNE** icon.
2. For **Auto Tune Controls**, verify the following:
   - **Start Frequency** is set to **100 kHz**
   - **End Frequency** is set to **500kHz**.
   - **Target Amplitude** is set to **2 volts**.
3. Click on **AUTO TUNE**.
4. When the procedure ends, click on **QUIT** to exit the function.

7.3.15 Set Initial Scan Parameters

1. In the **Scan Controls** panel, set the following:
   - Initial **Scan Size** is set to **1um**.
   - **X** and **Y Offsets** are set to **0**.
   - **Scan Angle** is set to **0**.

2. In **Tapping Mode**, under the **Feedback Controls** panel, set the following:
    - **Integral Gain** is set to **0.5**.
    - **Proportional Gain** is set to **0.7**.
    - **Scan Rate** is set to **2Hz**.
3. In **Contact Mode AFM**, under the **Feedback Controls** panel, set the following:
    - **Setpoint** is set to **0 volts**.
    - **Integral Gain** is set to **2.0**.
    - **Proportional Gain** is set to **3.0**.
    - **Scan Rate** is set to **2Hz**.

7.3.16 Engage

1. Select **Engage** under the **Motor** pop-down menu or click on the **ENGAGE** icon.

7.3.17 Establish Tip Clearance

Raise the Z stage to a safe height to ensure tip clearance when the microscope head is installed by executing one of the following options:

a. Execute multiple **Withdraw (Alt-W)** commands.

b. Use **Stage/Load New Sample**.

c. Use the **Stage/Focus Surface** command. Use the trackball and its buttons to raise the head by holding down the left buttons while rolling the trackball.

> **Note:** It is impossible to move the Dimension head if the laser sum signal is too low. For example, if the laser was not properly set up during a previous imaging session.

7.4 Advanced AFM Operation

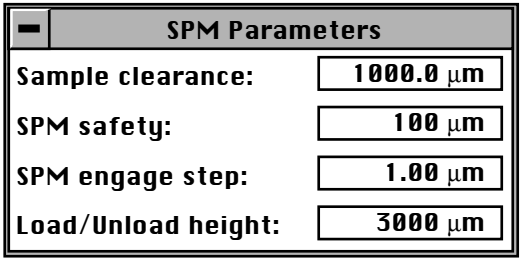
7.4.1 Stage Parameters

The system ships with the following default stage parameters (See Figure 7.4a). Once you have become familiar with using the system, you may want to adjust the stage parameters to speed up the engage sequence.

1. To access the stage parameters panel, select **SPM Parameters** from the **Stage** pop-down menu.

2. For initial set-up, use the following values. When done, click **OK** to close the **SPM Parameters** window.

Figure 7.4a Default SPM Stage Parameters



Definitions of **Stage / SPM Parameters** fields are as follows:

Sample clearance

Defines the height of the probe tip over the sample prior to **Engage**. You may change the sample clearance parameter once you are experienced with the system. The default parameter is 1000µm. To speed up the engage sequence and reduce the cantilever's effect on the optical image resolution when withdrawn, reduce this number to 500-600 µm.

SPM safety

Defines the height of the probe tip over the sample where the fast approach changes over to the slow approach. The default parameter is 100µm. You may reduce to 50-75µm once you are experienced with the system. Reducing this number speeds up the approach sequence.

Stage Parameters continued...

SPM engage step

Defines the step size of the Z-stage during engage. The default parameter is 1µm. Increasing this increases the speed of the approach.

CAUTION: Do not change this parameter by more than 0.5-1 µm because the step size must be some modest fraction of the total Z range of the scanner (i.e. 6-7 µm).

Load/Unload height

This parameter defines how high the tip moves above the previously defined, or found, sample clearance height. You may set the sample clearance height using the **Stage** command under the **Load/Unload Sample** panel. The stage command changes the cantilever or the sample. The Z stage raises the head the amount specified previously in the **Load/Unload height** parameter. The sample stage moves as far forward in the Y direction (towards the user) as possible.

Chapter 8 Contact AFM

8.1 Overview

This chapter covers procedures for operating the Dimension 3100 Scanning Probe Microscope (SPM) in Contact Mode AFM. It is assumed that the operator has previously prepared a Contact Mode tip and aligned the laser per instructions provided in Chapter 7 of this manual. Specific information regarding tip preparation is also provided in Chapter 6.

- **Overview:** Section 8.1
- **Basic Contact Mode AFM Operation:** Section 8.2
- **Advanced Atomic Force Operation:** Section 8.3
- **Optimization of Scanning Parameters:** Section 8.4
- **Force Calibration Mode:** Section 8.5

8.2 Basic Contact Mode AFM Operation

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

8.2.1 Select the Microscope Head

1. Set the microscope head configuration by selecting the **Microscope / Select** panel from the **Di** pop-down menu.
2. Click **OK** to close the **Microscope / Select** dialog box. When enabled, the selected buttons are black.

8.2.2 Select Mode of Operation

1. Select **Microscope/Profile**.
2. Select **Contact Mode** as the mode of operation.

8.2.3 Head, Cantilever and Sample Preparation

1. Install a silicon nitride tip onto an AFM cantilever holder (See Chapter 7).
2. Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head.

8.2.4 Align Laser

1. Align the laser using the laser control knobs.
2. Verify the laser beam is positioned on the back of the cantilever, with a spot visible in the Dimension head filter screen and a sum signal of **4-6 volts**.

Basic Contact Mode AFM Operation continued...

8.2.5 Adjust Photodetector

1. Adjust the photodetector so that the red dot moves toward the center of the Dimension head filter screen using the two photodetector adjustment knobs located on the side of the Dimension head.
2. Verify that the red dot is centered and elliptical in shape in the Dimension head filter screen.
3. Set the **Vertical Deflection** to **-2 volts**.

8.2.6 Locate Tip

1. Select **Stage/Locate Tip** or click on the **Locate Tip** icon.
2. Center the tip end of the cantilever under the cross hairs using the two optics adjustment knobs located left of the optical microscope objective.
3. Use the trackball with the bottom left button depressed to focus on the tip end of the cantilever.

8.2.7 Focus Surface

1. Select **Stage/Focus Surface** or click on the **Focus Surface** icon.
2. Focus on the sample surface using the trackball with the bottom-left button depressed.

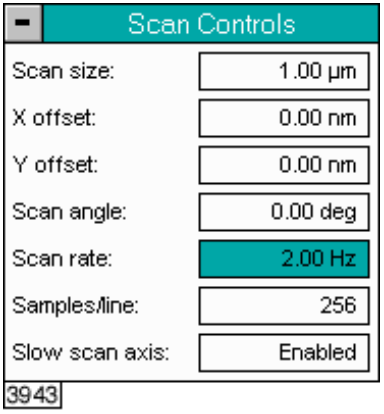
8.2.8 Set Initial Scan Parameters

Scan Controls Panel

In the **Scan Controls** panel, set the following initial scan parameters (See Figure 8.2a).

1. Set the **Scan Rate** to **2 Hz**.
2. Set the **Scan Size** to **1µm**.
3. Set the **Scan Angle** to **0**.
4. Set **X** and **Y Offsets** to **0**.

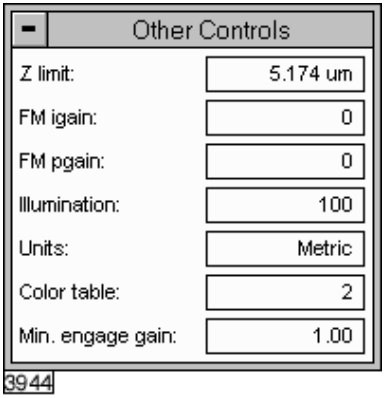
Figure 8.2a Suggested Scan Controls Settings



Other Controls Panel

Keep the **Z limit** at its maximum value.

Figure 8.2b Suggested Other Controls Settings

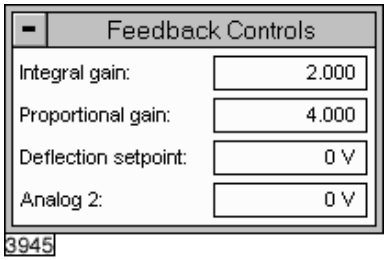


Feedback Controls Panel

1. Set the **Integral gain** to **2.0** and the **Proportional gain** to **4.0** (See Figure).
2. Set the **Deflection Setpoint** to **0 volts**.

Set Initial Scan Parameters continued...

Figure 8.2c Suggested Feedback Controls Settings

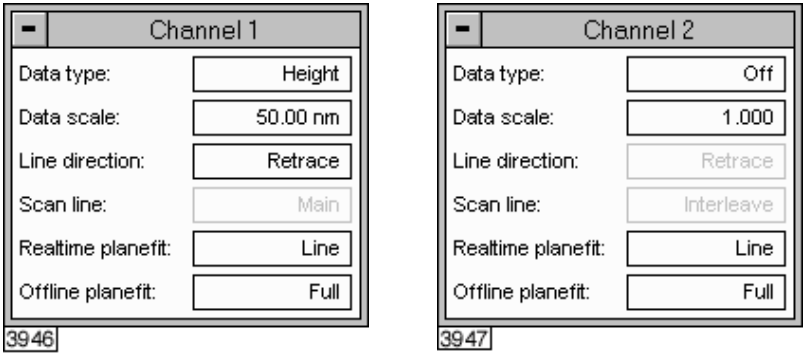


Channel Panels 1 and 2

1. In the **Channel 1** panel, set **Data type** to **Height** (See Figure 8.2d).
2. Set **Z range** to a reasonable value for the sample.

   Note: For example, for a 200 µm step height calibration sample, a reasonable Z range setting is 300 µm initially.
3. Set **Line direction** to either **Trace** or **Retrace**.
4. On the **Channel 2 panel,** verify **Data type** is set to **Off** to disable the panel.

Figure 8.2d Suggested Channel Controls Settings



8.2.9 Engage

1. Select **Motor**/**Engage**. A pre-engage check begins, followed by Z-stage motor motion.
2. To move to another area of the sample, execute a **Withdraw** command to avoid damaging the tip and scanner.
3. Move the stage using the trackball to the next area of interest on the sample.
4. Select **Motor**/**Engage.**

> **Note:** After the tip engages, adjust the control panel values to provide the desired scan parameters.

8.3 Advanced Atomic Force Operation

Although a great deal can be accomplished with basic knowledge of AFM operation, there is far more to operating the AFM.

Cantilever selection is critical and becomes more important as tip and cantilever technology continues to develop. A clear understanding of the parameters in the Real-time control panel allows the user to tune the microscope to accommodate a wide variety of samples. This section provides more detailed information on the operation of the Dimension microscope in Contact Mode AFM.

8.3.1 Cantilever Selection

Two basic cantilever styles are available for Contact Mode AFM. 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 smaller end radius than the silicon nitride cantilevers (Model ESP).

Note: There are a wide variety of tips available for Contact Mode AFM. Check the tip buying guide on the Digital Instruments Veeco website (www.di.com) for more information.

Silicon Nitride Cantilevers

Silicon nitride cantilevers for the Dimension 3100 SPM are available in two process variations: standard and sharpened. Sharpened silicon nitride cantilevers (Model DNPS) are almost identical in appearance to the standard silicon nitride cantilevers, but have a slightly sharper end at the very tip. Sharpened silicon nitride cantilevers are available by mail order through Digital Instruments Veeco.

Note: Although the Dimension 3100 SPM system does not require the “stand-alone” silicon nitride tips used in some older, interferometric microscope heads, you may still use them.

Silicon Nitride Cantilevers continued...

Each silicon nitride cantilever substrate includes four cantilever tips with different size and spring-constants. Two of the cantilevers on each substrate measure 115µm from the substrate to the apex of the triangular cantilever (referred to as 100µm cantilevers) while the remaining two cantilevers measure 193µm from the substrate to the apex of the triangular cantilever (referred to as 200µm cantilevers). Both cantilever lengths are available with wide legs and narrow legs; however, thickness of both cantilevers are equal. The calculated spring-constant for each of the cantilever configurations is listed below in Table 8.3a. These values are approximations and significant variability occurs. The tabulated values are used to approximate contact force unless more accurate values are measured by the user.

Table 8.3a: Cantilever Spring Constants

Cantilever Type	k (N/m) Narrow Legs	k (N/m) Wide Legs
100 micron (triangular)	.38	.58
200 micron (triangular)	.06	.12

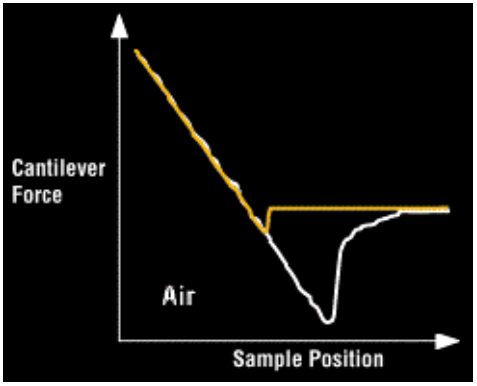
The 100µm wide-legged cantilever are used on most samples. If the image degrades rapidly because the tip damages the sample surface, switch to a cantilever with a lower spring-constant. Cantilevers with smaller spring-constants are used on softer samples which are destroyed by imaging with high-contact forces.

Etched Silicon Tips

Etched silicon tips have a higher aspect ratio and smaller end radius than the silicon nitride cantilevers. Because they are sharper, etched silicon tips provide better resolution and have less applied capillary forces. Most samples in air are covered by a thin layer of water and other condensed contaminants. These contaminants often form a capillary bridge between the tip and sample, generating large adhesive forces, also referred to as capillary forces (See Figure 8.3a). For more force curve information, see Chapter 13.

Cantilever Selection continued...

Figure 8.3a Force Curve



CAUTION: Large offsets are not recommended between engage and disengage (2 volts) with etched silicon cantilevers in Contact Mode AFM (450µm long only) because breakage is likely.

ATTENTION: Il est recommandé de ne pas utiliser de forts décentrements pendant l'engagement et le désengagement des céramiques piézo-électriques (2 V) avec des pointes en silicium (longueur: 450 µm) sous peine de destruction de celles-ci.

WARNHINWEIS: Wir empfehlen, im Kontakt-Modus mit den geätzten Siliziumspitzen (nur mit den 450 µm langen) keine großen Offsets zwischen engage und disengage-Zustand (2 Volt) zu haben, da diese sonst leicht brechen könnten.

Note: The smaller end radius of etched silicon tips creates greater force between the tip and sample. Therefore, etched silicon tips are not suggested for soft samples.

8.4 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 Real-time control panels in the NanoScope III *Command Reference Manual* before making bold changes. The following section analyzes the effects of the most important parameters.

8.4.1 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** 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. To collect accurate topographical data, set the **Data type** parameter to **Height**. Topographical **Deflection** data is only reasonable on very smooth, flat samples. Topograhpical **Deflection** data is used infrequently and only for high resolution work.

Collect **Deflection** 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** data is not automatically calibrated in units of distance. You must measure the sensitivity using the procedures discussed in Chapter 13.

Data Type continued...

Deflection 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-signal) data alone does not yield quantitative height information.

8.4.2 Gain Settings

The **Integral**, **Proportional**, and **LookAhead** gains in the **Feedback Controls** 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** 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 **2-3 volts** 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 Real-time image. Piezo oscillations are more easily observed in **View/Scope Mode**.
4. For **Deflection** 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.
5. Set the **LookAhead gain** to **0.7** initially for samples with step-like features oriented perpendicular to the fast scan direction. Otherwise, it should be left at 0.00.

Note: The **LookAhead gain** includes information from the previous scan line to determine the current gain setting. **LookAhead gain** controls are not included on some software releases. For more information, contact Digital Instruments Veeco.

8.4.3 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.

8.4.4 Setpoint

The **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 **Setpoint**; therefore, the cantilever deflection remains nearly constant.

Adjust the **Setpoint** to increase or decrease the cantilever deflection and, therefore, the contact force of the tip on the sample. The **Force Calibration** command in the **Real-time / View** 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 13, minimizes the contact force of the tip on the sample. This is especially important on soft materials such as biological samples.

8.4.5 Lowpass Filter

The **Lowpass filter** invokes a digital, one-pole, lowpass filter to remove high-frequency noise from Real-time data. The filter operates on the collected digital data regardless of the scan direction. Settings for this item range from **Off** through **9**. **Off** implies no lowpass filtering of the data, while settings of **1** through **9**, successively, lower the cut-off frequency of the filter applied to the data stream. The standard setting is **Off**.

8.4.6 Highpass Filter

The **Highpass filter** parameter invokes a digital, two-pole, highpass filter to remove low frequency effects such as ripples caused by torsional forces on the cantilever when the scan reverses direction. As with the **Lowpass filter**, the **Highpass filter** also operates on the digital data stream regardless of scan direction. This parameter can be **Off** or set from **1** through **9**. Settings of 1 through 9, successively, lower the cut-off frequency of the filter applied to the data stream. Note that in removing low frequency information from the image, the **Highpass filter** distorts the height information in the image. As a result, this filter must be **Off** when accurate height information is desired. The **Highpass filter** is typically used only for atomic images.

8.5 Force Calibration Mode

The **Force Calibration** command in the **View / Force Mode / Calibration** menu allows you to check the interaction between the cantilever and the sample surface. For detailed information regarding **Force Calibration**, see Chapter 13.

Chapter 9 Contact AFM in Fluids

9.1 Overview

The imaging of samples under fluid is an ever-increasing realm for SPM technology. Imaging samples under fluid minimizes surface forces on delicate samples, allows observation of biological specimens in their natural, fluid environments, and allows real-time observations of samples undergoing electrochemical reactions (ECAFM). In order to conduct ECAFM observations with electrical potentials, an external potentiostat unit is necessary.

This chapter details basic operation of the Dimension 3100 SPM in fluid. Methods of preparing the sample for fluid operation are also included. Specifically, this chapter addresses the following:

- **Overview:** Section 9.1
- **Basic Principles of Contact AFM in Fluids:** Section 9.2
- **Fluid Operation Hardware:** Section 9.3
- **Sample Mounting:** Section 9.4
- **Fluid Operation Procedure:** Section 9.5

9.2 Basic Principles of Contact AFM in Fluids

Attractive forces due to surface tension effects are eliminated when imaging samples under fluid. This enables the sample surface to be imaged with minimal cantilever tip force—a decided advantage when imaging biological specimens and delicate materials. The procedure for observing samples under fluid is the same as that for Contact 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 assumes familiarity with standard operation of the Dimension 3100 AFM in air. If you are not familiar with air operation of the AFM, please follow the procedures outlined in Chapter 8 before attempting to operate the AFM under fluid. Images of submerged samples may also be obtained in Tapping Mode (See Chapter 11).

9.3 Fluid Operation Hardware

9.3.1 Fluid Tip Holder

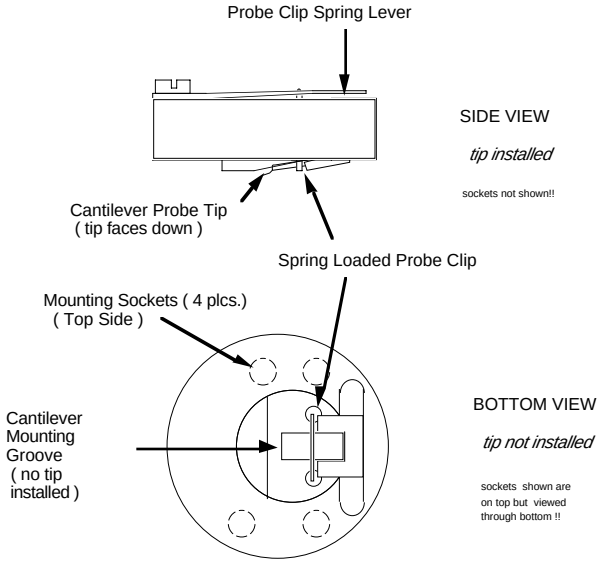
The Dimension 3100 can be shipped with an optional tip holder that permits operation under fluid (See Figure 9.3a). The fluid tip holder interchanges with the standard tip holder quickly and easily.

The fluid tip holder consists of a small glass assembly with a wire clip for holding an AFM cantilever substrate. The glass surfaces provide a flat, beveled interface so that the AFM laser beam may pass into the fluid without being distorted by an unstable fluid surface. The four sockets located on the top of the fluid tip holder are used to attach the tip holder to the four pins at the end of the scanner tube.

Use the fluid tip holder in an open environment where the holder is dipped into a user-supplied fluid container (or into a drop of fluid on larger samples). Methods for mounting samples for fluid operation are illustrated in Figure 9.4a in Section 9.4.

Fluid Operation Hardware continued...

Figure 9.3a Fluid Tip Holder



9.3.2 Tip Suggestions

Soft cantilevers, particularly oxide sharpened tips, produce the best results for biological applications. These tips are typically 100 microns long, narrow-legged with oxide sharpened silicon-nitride tips. Models DNP-S or DNP-STT are good examples. Users should experiment to find which cantilevers work best for their sample.

Note: For additional information on selecting a cantilever, please refer to the Digital Instruments Veeco website (www.di.com).

Removing Organic Contamination

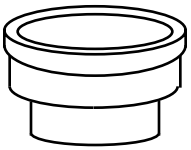
Contaminants on the cantilever tip may limit the AFM's resolution. Use ultraviolet light to remove contaminants as follows:

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 unit for two minutes at full intensity.

9.3.3 Rubber Protective Skirt

A rubber protective skirt (See Figure 9.3b) is also provided with the fluid tip holder. This protective skirt is used to protect the Dimension scanner from getting liquid on it. It is recommended that the protective skirt be used with all wet samples. Because the skirt does not seal against the tip holder, tips should not be immersed in fluids deeper than 2 mm. Skirts wear over time and may need to be replaced.

Figure 9.3b Rubber Protective Skirt



9.4 Sample Mounting

9.4.1 General Notes on Sample Binding

Immobilize samples for AFM imaging on a rigid support. Macroscopic samples (biomaterials, crystals, polymer membranes, etc.) can be attached directly to a stainless steel sample disk with an adhesive. Biological samples like cells, proteins, DNA, etc. are usually bound to a flat substrate such as mica or glass. Many sample preparations have been developed and AFM applications articles are an excellent source of information or sample binding.

Note: For a list of articles describing biological applications of AFM, including sample preparation techniques, contact Digital Instruments Veeco.

A special sample container for fluid operation is provided for wet samples.

CAUTION: Do not attempt to operate the standard air tip holder in a fluid environment. The standard tip holder has exposed electrical signal lines that could short circuit if exposed to a conducting fluid.

ATTENTION: En milieu liquide, ne pas utiliser le support de pointe standard prévu pour une utilisation à l'air. Le support de pointe standard présente des contacts électriques qui peuvent être endommagés (court-circuit) suite à une exposition à un liquide conducteur d'électricité.

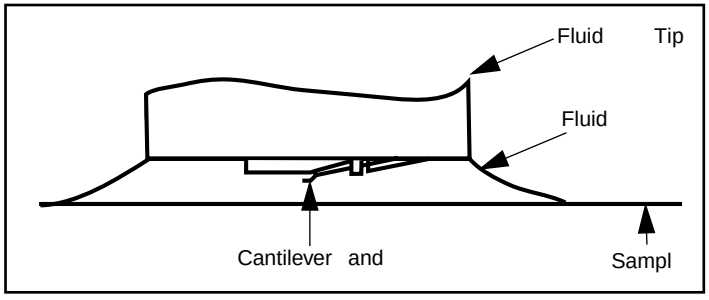
WARNHINWEIS: Versuchen Sie nicht, den Standard-Luft-Cantileverhalter in Flüssigkeiten zu betreiben. Am Standard-Cantileverhalter liegen elektrische Leitungen frei, die in leitfähigen Flüssigkeiten kurzgeschlossen werden könnten.

9.4.2 Larger Samples

For larger samples, it may be desirable to image a small region under a drop of fluid (See Figure 9.4a).

1. Put the drop of fluid onto the sample.
2. Lower the AFM fluid tip holder into the drop.

Figure 9.4a Imaging a Sample Covered by a Drop of Fluid



9.4.3 Smaller Samples

Alternatively, it may be desirable to image smaller samples that are totally immersed in a liquid bath.

Sample Mounting continued...

1. Place the sample in the plastic petri dish supplied with the unit.

   Note: If you wish to construct a custom sample holder, please feel free to contact Digital Instruments Veeco for more information.

2. Secure the sample to the bottom of the petri dish lid or other sample holder with epoxy or other non water-soluble adhesive.

   Note: For non-critical applications, Devcon 2-Ton Epoxy works well. For applications where contamination control is more critical, use a more inert, solvent-free epoxy like Master Bond EP21LV, EP21AR, or a hot melt adhesive.

3. Follow the manufacturer's directions for mixing and curing to obtain the best resistance to leaching and chemical attack. For lower resolution applications where it is desirable to image a sample quickly without waiting for an adhesive to cure, you can use double-sided tape as long as fluid never touches the tape.

   Note: Do not use cyanoacrylate glue (like super glue) for mounting samples in fluid.

CAUTION: When imaging fluid samples, use extraordinary precautions against spillage. Fluids must NOT be spilled on or around the sample stage, electronics boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise, damage may result. In the case of a spill, immediately clean and dry all affected surfaces carefully.

ATTENTION: Lors d'un travail en milieu liquide, prendre toute précaution pour éviter des fuites. Les liquides ne doivent pas se répandre sur la platine porte échantillons, le boîtier électronique ou toute autre partie du microscope contenant de l'électronique. Eviter toute fuite de liquide corrosif sur les surfaces exposées. Le non respect de cette recommandation peut entraîner des dommages. En cas de fuite, nettoyer et sécher immédiatement les surfaces touchées.

WARNHINWEIS: Falls Sie Proben in Flüssigkeiten abbilden, lassen Sie äußerste Vorsicht walten, damit keine Flüssigkeit verspritzt wird. Flüssigkeiten dürfen nicht auf die oder nahe der Probenhalterung, der Elektronikbox oder anderen Komponenten, die elektronische Bauteile enthalten, verspritzt werden. Vermeiden Sie bitte, korrosive Flüssigkeiten auf freiliegende Oberflächen zu verspritzen; andernfalls wären Beschädigungen die Folge! Falls Sie Flüssigkeit verspritzt haben, säubern und trocknen Sie alle betroffenen Flächen sorgfältig.

9.5 Fluid Operation Procedure

9.5.1 Load the Cantilever Substrate

The cantilever substrate is held in a small pocket on the bottom side of the tip holder by a gold-plated stainless steel wire. The cantilever installation fixture has three docking stations where different tip holders may be mounted. Use the docking station that has a small wire between two of the sockets. The steel wire is held against the cantilever substrate by a leaf spring mounted on the top of the tip holder.

1. To mount a cantilever substrate in the holder, turn the holder over so that the leaf spring and four sockets are facing down and plug the holder onto the appropriate docking station of the cantilever installation fixture.
2. Grip the tip holder by the edges and gently push down on the holder.

   Note: When pushing down on the holder, the steel wire in the docking station will press on the leaf spring to raise the gold-plated wire that will hold the cantilever substrate into its pocket.
3. With the gold wire raised, use tweezers to slide a cantilever substrate under the wire and into the pocket.
4. Check that the cantilever substrate is squarely set against one side of the pocket and flush against the back.

CAUTION: Avoid scratching the tip holder's glass surface with the tweezers or the cantilever substrate, especially in the area under the cantilever itself.

5. Gently lift the tip holder off the docking station.

   Note: When removing the tip holder from the docking station, the leaf spring should pull the gold wire tight against the cantilever substrate.
6. Check that the cantilever substrate is held firmly by the wire.

Fluid Operation Procedure continued...

9.5.2 Install the Cantilever Holder

CAUTION: When cleaning the cantilever holder, take care to avoid scratching the glass surfaces in the center of the cantilever holder. Be careful to prevent dripping any liquid onto the Dimension 3100 system, especially onto the SPM head.

1. Check that there is sufficient clearance between the bottom of the SPM head and the sample.

   Note: The cantilever tip position extends roughly 1 mm further towards the sample with the fluid cantilever holder over the standard air cantilever holder.

2. If the height of the SPM head needs adjustment, use one of the following methods: **Focus Surface**, **Motor / Step Motor**, or **Withdraw**.

3. Gently unplug any cantilever holders attached to the base of the scanner.

   Note: If necessary, lift the SPM head out of the dovetail to allow for easier access to the base of the scanner.

4. Pull the cantilever holder straight off to prevent bending any of the pins on the cantilever holder or scanner cap.

5. Fit the fluid cantilever holder onto the four pins of the scanner cap.

   Note: The four sockets on the cantilever holder will only align with the pins on the scanner cap when the cantilever tip points to the left of the microscope.

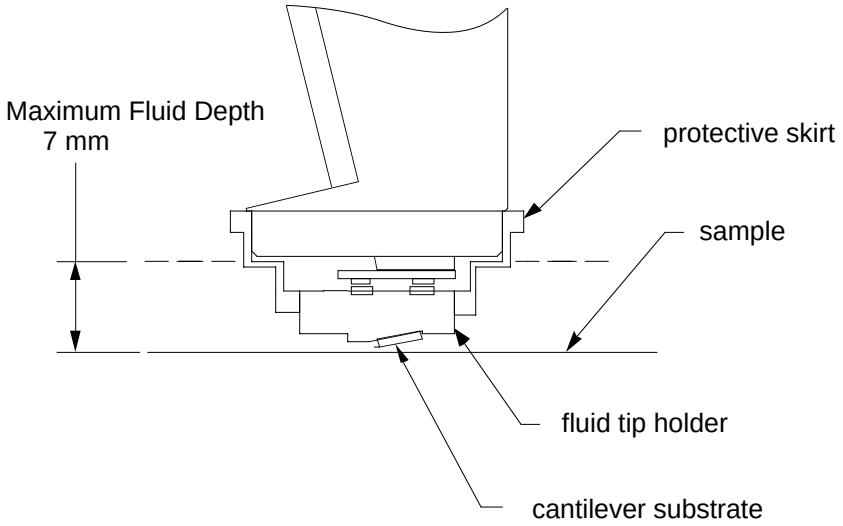
6. Set the head back into the dovetail and lock into place by releasing the knurled head clamp screw, located at the upper-right of the Z-stage, until the thread is just loosened.

7. Turn the head clamp screw an additional 1-1.5 turns to allow the spring loaded clamp to engage the SPM head's dovetail, locking the head to the Z-stage.

9.5.3 Install the Protective Skirt

The protective skirt is a rubber seal used to protect the SPM scanner tube from liquids. Install it by sliding it over the fluid tip holder and onto the shoulder on the SPM tube. Make sure that the seal is tight on both the tip holder and the SPM tube. See Figure 9.5a below for an illustration of the SPM head and the fluid tip holder with the protective skirt installed.

Figure 9.5a Tip Holder Installed With Protective Skirt



9.5.4 Align Laser

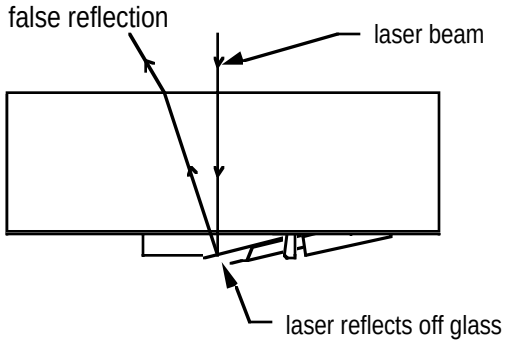
Use the technique for aligning the laser onto the cantilever discussed in Chapter 7 of this manual.

9.5.5 “False” Reflections

The cantilever substrate rests on a smooth, angled, glass surface. The laser reflects off the angled glass surface resulting in a visible laser reflection on the laser viewing window on the microscope head, even when the laser is not aimed at the cantilever (See Figure 9.5b). This reflection from the glass surface does not affect the operation of the AFM, but it can be a source of confusion when aligning the laser on the cantilever. Ignore this fainter reflection and look instead for the much brighter reflection off the cantilever.

Fluid Operation Procedure continued...

Figure 9.5b False Reflections from Fluid Tip Holder



Once the fluid tip holder is plugged into the bottom of the AFM scanner, note the faint laser reflection visible on the glass in the tip holder (in the laser viewing window).

Note: The SUM signal on the display monitor will typically show less than 1 V when the laser is not yet aligned on the cantilever. The SUM signal should rise well above 1 V when the laser is truly reflecting off the cantilever.

9.5.6 Lower Tip Holder into Fluid

1. Lower the head, allowing the tip holder to enter the liquid. The fluid should reach up to form a meniscus around the fluid tip holder (See Figure 9.4a).

Note: The laser sum signal must be greater than 0.5 V to use the **Z motor**. It may be necessary to use the detector mirror adjustment screws to roughly center the reflected laser beam on the detector before using either the **Focus Surface** or **Stepper Motor** commands.

9.5.7 Readjust Laser Alignment

Lowering the tip holder into fluid causes the laser spot to move towards the fixed end of the cantilever by roughly 25 microns. To compensate for this shift, proceed with the following:

1. Turn the right-rear laser aiming screw slightly counter-clockwise to move the laser spot back to the free end of the cantilever.
2. Check that a bright reflected laser spot is still present on the laser viewing window.

Occasionally, air bubbles will become trapped near the cantilever and these may interfere with the laser beam's optical path. If this occurs, it may be impossible to get a good reflection from the cantilever after putting the tip holder into fluid. In this case, do the following:

3. Remove the SPM head from the dovetail.
4. Lightly rinse the tip holder with liquid.
5. Replace the SPM head.

9.5.8 Adjust Detector Offsets and Setpoint

1. Turn the detector mirror adjustment screws to center the laser spot on the laser detector as described in Chapter 7.
2. Set the vertical deflection signal to roughly -1 V and the **Setpoint** to 0 V which are typical starting parameters.

Note: The difference between the vertical deflection before engaging and the setpoint is related to the amount of force that the cantilever probe tip applies to the sample.

ATTENTION: Verify that there is not too large a difference between the setpoint and the vertical deflection signal before engaging. Samples are typically softer in liquid than in air. It may be desirable to reduce the setpoint once engaged to obtain the minimum tracking force.

Fluid Operation Procedure continued...

9.5.9 Locate Tip

1. Using the mouse, select **Locate Tip** from under the **Stage** pop-down menu or click on the Locate Tip icon.
2. Center the tip on the cantilever under the cross hairs using the two adjustment screws located to the left of the optical objective on the microscope.
3. Focus on the tip end of the cantilever using the trackball while holding the bottom left button.

Note: See Chapter 7 for more detailed instructions for locating the tip.

9.5.10 Focus Surface

When focusing on the sample surface in **air** before adding fluid to the sample area, the following procedure is necessary:

a. Align Laser

b. Locate Tip

c. Focus Surface

d. Add Fluid

e. Readjust Laser Alignment

f. Adjust Laser Alignment in Photodetector

Fluid changes the optical pathlength as the angle of refraction changes, therefore, you must offset the focus position in **Focus Surface** by 300µm to a point below the surface when focusing in fluid.

Note: For example, if the surface is in focus with the Z motor positioned at -5000µm, you must move the Z motor position down until it is at -5300µm.

Fluid Operation Procedure continued...

When focusing on the sample surface directly in **fluid**, the following procedure is necessary:

a. Align Laser (in air)

b. Locate Tip (in fluid)

c. Focus Surface (hit engage several times)

OR

Focus Surface (beyond surface by another 300um)

d. Realign Laser

e. Realign Laser in Photodetector

Focus Surface continued...

Focus Surface Procedure

1. Use the **Stage / Focus Surface** command to bring the sample into focus on the video camera monitor, as described in Chapter 7.
2. Move the X-Y sample stage to bring an area of interest under the AFM cantilever tip.

CAUTION: Use extreme caution moving the X-Y stage if the sample holder has a lip that extends above the sample surface (i.e. petri dish). The SPM head can be destroyed if the sample stage is moved such that the tip holder crashes sideways into the lip of your sample holder. Also, check that the position of the lip of the sample holder will not interfere with the movement of the Z-stage.

ATTENTION: Il est recommandé de déplacer avec une extréme vigilance la platine quand le support d'échantillon présente un rebord qui se trouve à une hauteur supérieure à mesurer (comme une boîte de Petri). La tête de mesure peut être endommagée si le support de pointe vient heurter le rebord du support d'échantillon lors d'un déplacement de la platine. De plus, il est impératif de vérifier que le rebord du support d'échantillon ne viendra pas perturber le mouvement vertical du moteur de la tête de mesure.

WARNHINWEIS: Seien Sie extrem vorsichtig mit XY-Bewegungen des Probentisches, wenn der Rand Ihrer Probenbefestigung über die Probenfläche hinausragt (wie zum Beispiel bei einer Petrischale). Der SPM-Kopf kann zerstört werden, falls der Probentisch derart bewegt wird, daß der Spitzenhalter seitlich gegen den Rand der Probenbefestigung schlägt. Versichern Sie sich außerdem, daß der Rand Ihrer Probenbefestigung bei Bewegung der Z-Verschiebeeinheit nicht im Weg ist.

9.5.11 Check Scan Parameters

Check that various scan parameters such as **Scan rate**, **Scan size**, and **Integral gain** are reasonable. Suggested parameter values are listed in Chapter 9.

Note: It may be desirable to select a large **X** and **Y offset** before engaging if the sample is easily damaged by the tip.

9.5.12 Engage

1. Click on **ENGAGE** under the **Motor** pop-down menu.

9.5.13 Adjust Scan Parameters

Once engaged, the scan parameters should be adjusted to obtain the best image. This procedure will be similar to operation in air with the following exception:

Samples are often softer in fluids

Adjusting the applied force becomes much more critical because samples are often softer in fluids. To adjust the applied force and avoid sample damage, proceed with the following:

1. Adjust the **Setpoint** to as low a value as possible using the cursor keys until the cantilever pulls off the surface (and the Z-center voltage jumps to -220 V).
2. Increase the **Setpoint** slightly until the cantilever begins to touch the surface again and an image appears. Or, use the **Force Calibration** command to select the Setpoint and estimate the contact force, as described in Chapter 17.

Fluid Operation Procedure continued...

Note: The cantilever will typically adhere to the sample surface much less in fluid; therefore, it is often possible to image at much smaller contact forces in liquid than in air.

9.5.14 Clean Fluid Cell and Protective Skirt

To reduce contamination problems and to obtain high quality images, clean the fluid cell and protective skirt as follows:

1. When sample imaging is complete, carefully remove the protective skirt and fluid tip holder.
2. Place the fluid cell and protective skirt in warm, soapy water and place a few drops of liquid dish soap on them.
3. Gently rub the fluid cell and protective skirt with a cotton swab or finger.

CAUTION: Avoid scratching the glass surface with abrasive material.

4. Using distilled water, rinse the fluid cell and protective skirt completely.
5. Using 0.2 mm-filtered, compressed air or dry nitrogen, blow dry the fluid cell until all moisture evaporates to prevent the buildup of salts or other contaminants on the parts.

Chapter 10 Tapping Mode AFM

10.1 Overview

This chapter details procedures for operating the Dimension 3100 SPM in Tapping Mode in air. For information regarding Tapping Mode in fluids, see Chapter 11. For information regarding loading a Tapping Mode tip and aligning the SPM laser see Chapter 7 of this manual. Additional information regarding cantilever preparation is provided in Chapter 6.

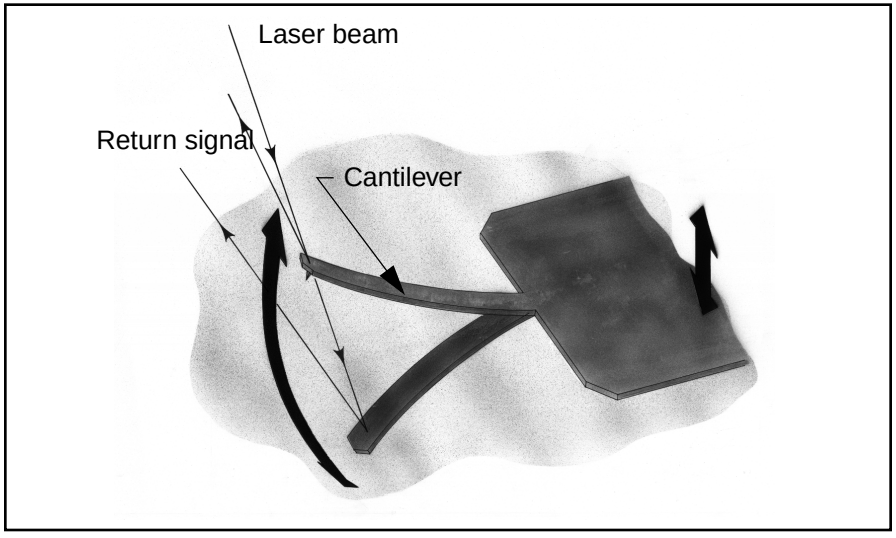
This chapter addresses the following:

- **Overview:** Section 10.1
- **Principles of Tapping Mode:** Section 10.2
- **Basic Tapping Mode AFM Operation:** Section 10.3
- **Withdraw the Tip:** Section 10.4
- **Advanced Tapping Mode AFM Operation:** Section 10.5
- **Troubleshooting:** Section 10.6

10.2 Principles of Tapping Mode

Figure 10.2a 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.

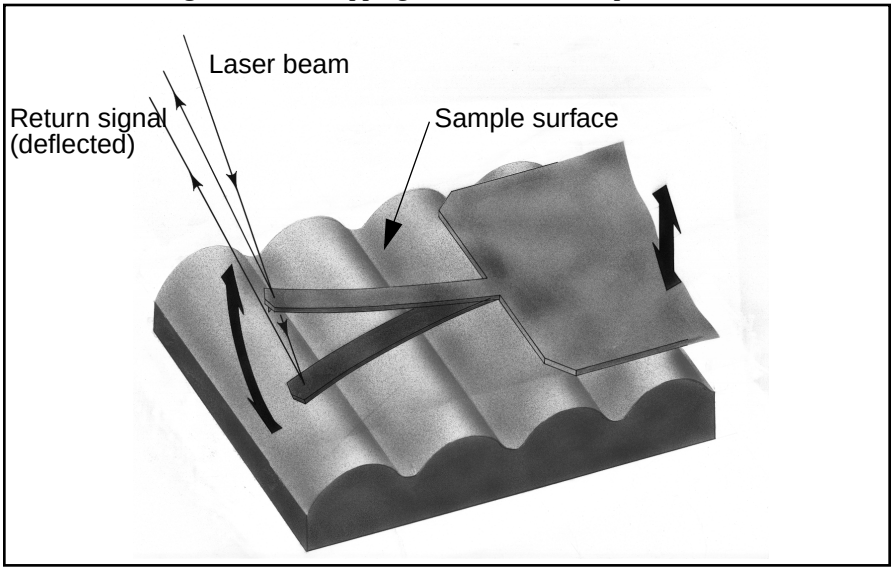
Figure 10.2a Tapping Cantilever in Free Air



Principles of Tapping Mode continued...

Figure 10.2b represents the same cantilever at the sample surface. Although the piezo stack continues to excite the cantilever substrate with the same energy, the tip deflects 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 10.2b Tapping Cantilever on Sample Surface



Note: Deflection of the cantilever and return signal are exaggerated in the figure for illustrative purposes.

10.3 Basic Tapping Mode AFM Operation

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

10.3.1 Select the Microscope Head

1. Set the microscope head configuration by selecting the **Microscope / Select** panel from the **Di** pop-down menu.

2. Click **OK** to close the **Microscope / Select** dialog box. When enabled, the selected buttons are black.

10.3.2 Select Mode of Operation

1. Select **Microscope/Profile**.

2. Select **Tapping Mode** as the mode of operation.

10.3.3 Head, Cantilever and Sample Preparation

1. Install an etched single crystal silicon tip onto an AFM cantilever holder (See Chapter 7).

2. Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head.

10.3.4 Align Laser

1. Align the laser using the laser control knobs.

2. Verify the laser beam is positioned on the back of the cantilever, with a spot visible in the Dimension head filter screen and a sum signal of **2 volts**.

Basic Tapping Mode AFM Operation continued...

10.3.5 Adjust Photodetector

1. Adjust the photodetector so that the red dot moves toward the center of the Dimension head filter screen using the two photodetector adjustment knobs located on the side of the Dimension head.

2. Verify that the red dot is centered and elliptical in shape in the Dimension head filter screen.

3. Set the **Vertical Deflection** to **0 volts**.

10.3.6 Locate Tip

1. Select **Stage/Locate Tip** or click on the **Locate Tip** icon.

2. Center the tip end of the cantilever under the crosshairs using the two optics adjustment knobs located left of the optical microscope objective.

3. Use the trackball with the bottom left button depressed to focus on the tip end of the cantilever.

10.3.7 Focus Surface

1. Select **Stage/Focus Surface** or click on the **Focus Surface** icon.

2. Focus on the sample surface using the trackball with the bottom left button depressed.

10.3.8 Cantilever Tune

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

1. Select **View / Cantilever Tune**, or click on the **Cantilever Tune** icon. The initial **Cantilever Tune** panel appears with the **Frequency Sweep** (a plot of cantilever response as a function of applied oscillation frequency) on the display monitor.

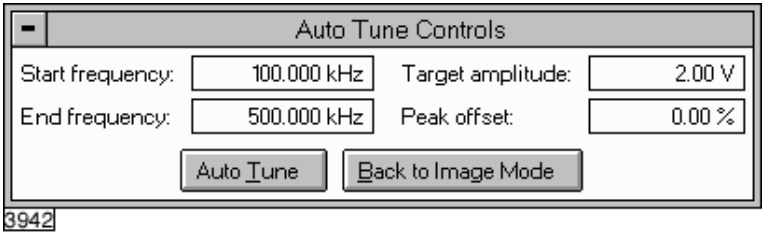
2. Choose either the manual or automatic tuning method (See Automatic Tuning and Manual Cantilever Tuning).

Automatic Tuning

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

1. Click on **AUTO TUNE**. The computer and controller begin automatic tuning (See Figure 10.3a).

Figure 10.3a Auto Tune Control Panel



Cantilever Tune continued...

Manual Cantilever Tuning

With Force Modulation or Fluid Tapping applications, it may be useful to tune the cantilever manually.

> **Note:** More than one type of cantilever exists. Cantilevers can have different dimensions and different resonance frequencies. 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 **Cantilever Tune** panel, set the **Drive frequency** parameter to a value near the center of the resonance 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. Set the **Drive amplitude** to **200 mV**.

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

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

4. Zero the **Setpoint**.

5. Set the **Amplitude limit** to **2.5V**.

6. Center the peak on the frequency sweep plot shown on the display monitor 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.

7. Increase the **Setpoint** until the peak appears.

8. 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 Tapping Mode 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 10.3d).

Figure 10.3b Cantilever Tune Control Panels for Main Controls

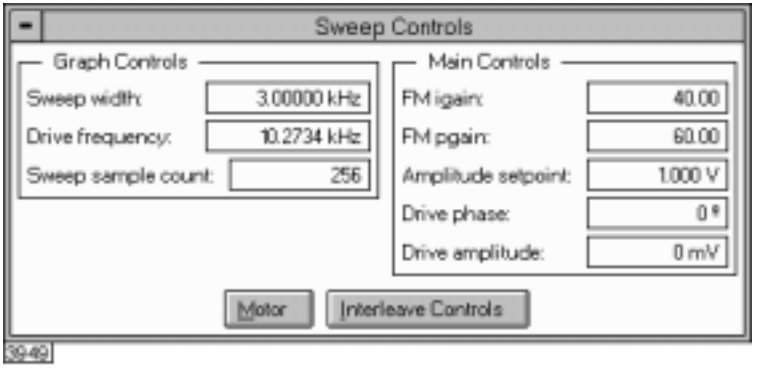
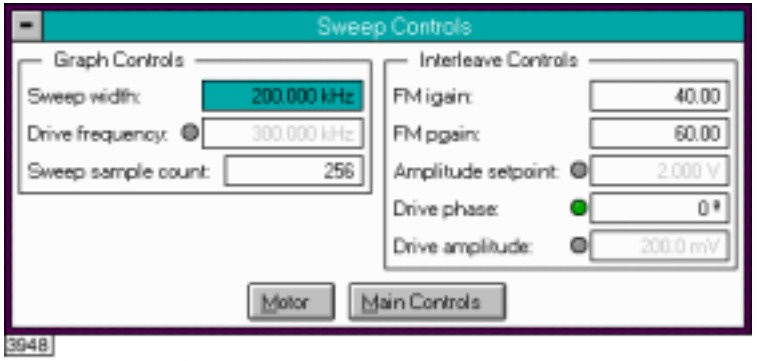


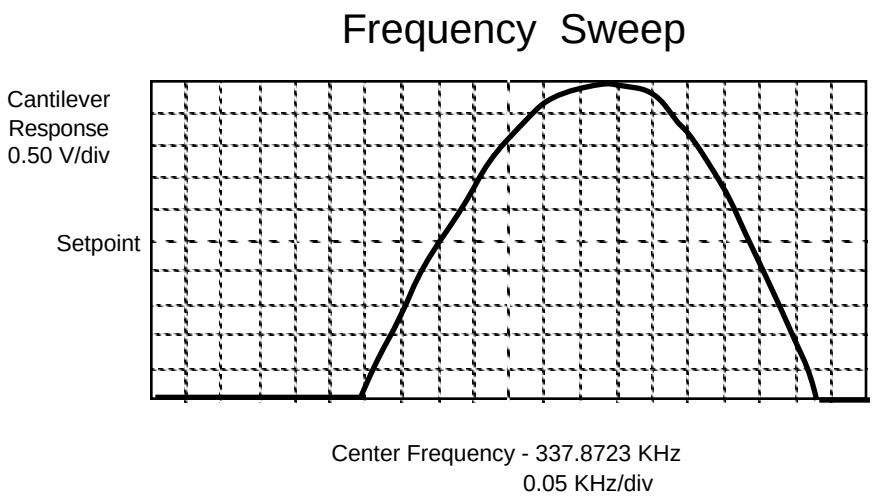
Figure 10.3c Cantilever Tune Control Panels for Interleave Controls



Manual Cantilever Tuning continued...

9. 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 10.3d Cantilever Tune Frequency Sweep



10. Click **OK**. The parameters set in the **Cantilever Tune** control panel appear in the **Real-time** control panel.

11. Click on **CANCEL** to exit the **Cantilever Tune** command and leave the parameters unchanged.

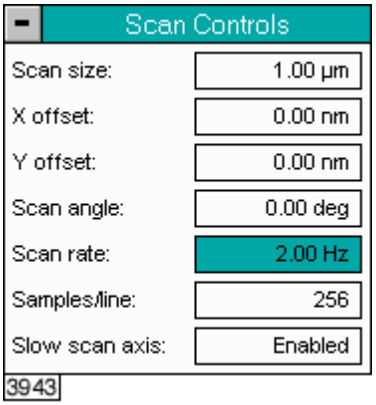
10.3.9 Set Initial Scan Parameters

Scan Controls Panel

In the **Scan Controls** panel, set the following initial scan parameters (See Figure 10.3e).

1. Set the **Scan Rate** to **2 Hz**.
2. Set the **Scan Size** to **1µm**.
3. Set the **Scan Angle** to **0**.
4. Set **X** and **Y Offsets** to **0**.

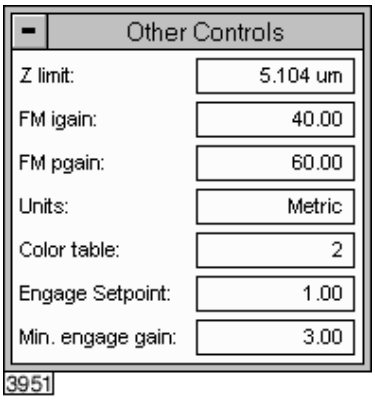
Figure 10.3e Suggested Scan Controls Settings



Basic Tapping Mode AFM Operation continued...

Other Controls Panel

Figure 10.3f Suggested Other Controls Settings

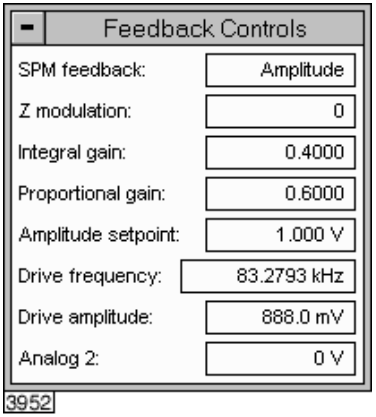


Feedback Controls Panel

1. Set the **Integral gain** to **0.5** and the **Proportional gain** to **0.7** (See Figure 10.3g).

2. Set the **Look Ahead gain** to zero.

Figure 10.3g Suggested Feedback Controls Settings



10.3.10Engage

1. Select **Motor**/**Engage**. A pre-engage check begins, followed by Z-stage motor motion.

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

3. Move the stage using the trackball to the next area of interest on the sample.

4. Select **Motor**/**Engage.**

> **Note:** After the tip engages, adjust the control panel values to provide the desired scan parameters. Refer to Section 10.5.4 for scan parameter optimization.

10.4 Withdraw the Tip

1. Select **Withdraw** from the **Motor** menu. The SPM stops scanning, and ascends to the sample clearance height defined in the **SPM Parameters** menu.

2. Select the **Stage / Load New Sample** option to replace or move the the sample.

3. Use the **Focus Surface** command to move the SPM up if you desire more clearance between the tip and sample.

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

10.5 Advanced Tapping Mode AFM Operation

This section discusses the more subtle aspects involved in operating the Dimension 3100 in Tapping Mode.

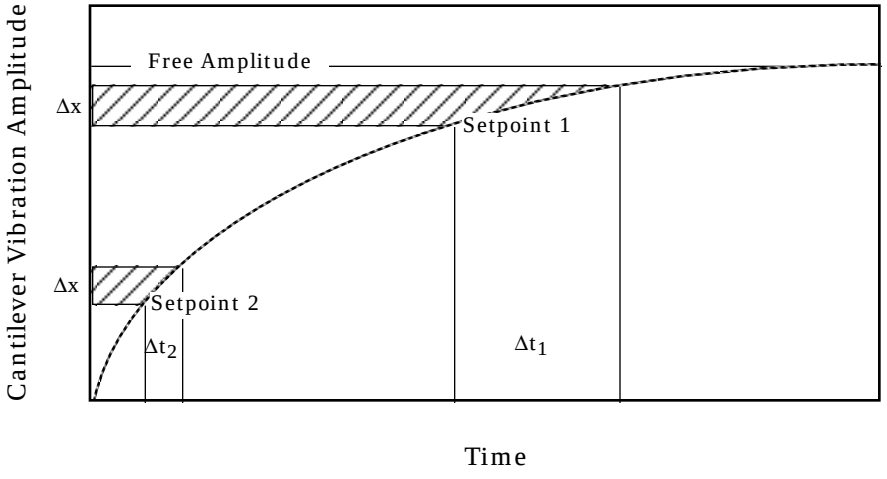
10.5.1 Resonating Techniques

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 **Force Calibration Mode in Contact AFM** and **Force Calibration Mode in Tapping Mode** (See Chapter 13).

10.5.2 Cantilever Oscillation

The response of the cantilever to inputs plays an important role in the operation of the Dimension 3100 SPM while in Tapping Mode. 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 10.5a 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 10.5a Cantilever Response Curve



Cantilever Oscillation continued...

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. Unfortunately, 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 **Setpoint amplitude**. Soft samples that are relatively flat should run with higher **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, **Scope Mode** is very useful when setting scan parameters. As the tip descends, features are evaluated by comparing the **Trace** and **Retrace** in **Scope Mode**. Figure 10.5b 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 10.5b Scope Trace with High Scan Rate

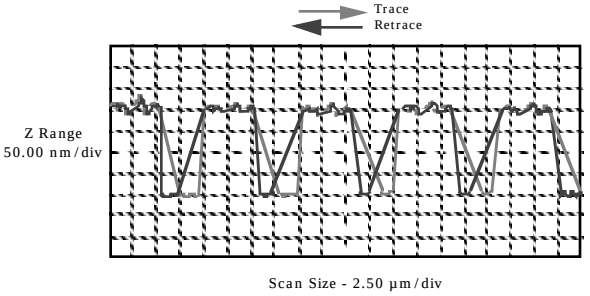
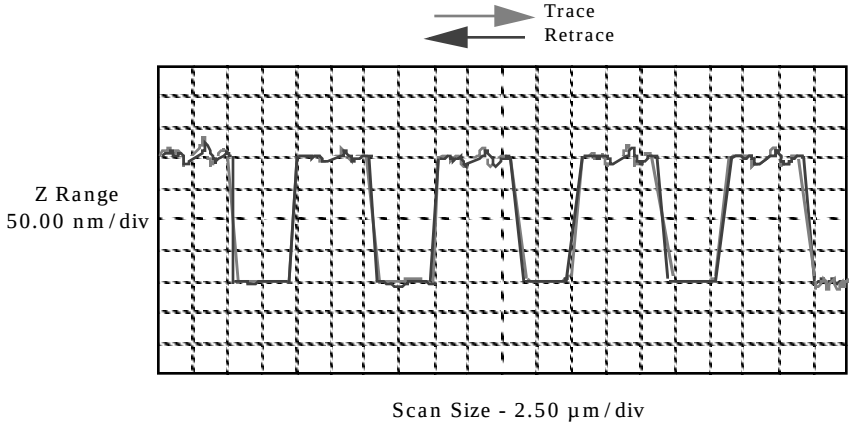


Figure 10.5c depicts the same sample with a slight increase in the **Integral gain** and a twofold decrease in the **Scan rate**. The tip now tracks the surface when it descends into the pit as well as when it exits. The **Trace** and **Retrace** lines now coincide closely.

Figure 10.5c Scope Trace with Correct Scan Rate



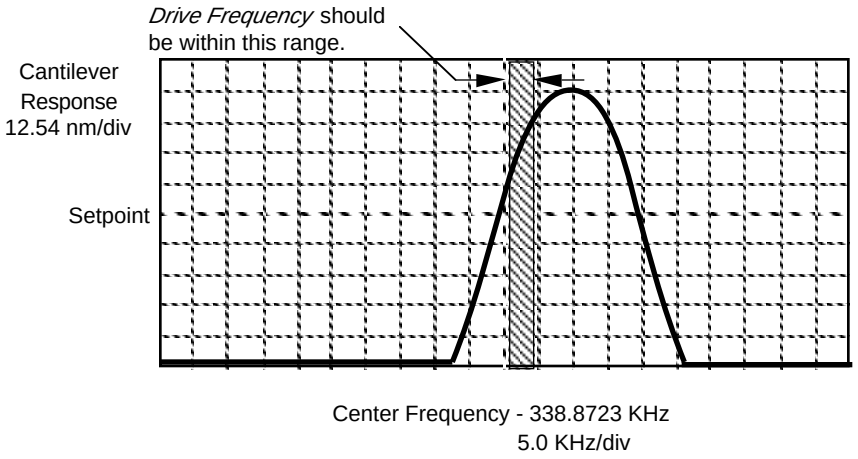
10.5.3 Decreasing the Cantilever Drive Frequency

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

Decreasing the Cantilever Drive Frequency continued...

The microscope produces better data in Tapping Mode when the **Drive frequency** is set lower than the resonance 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 10.5d 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 10.5d Example of Cantilever Tune Frequency Sweep



10.5.4 Optimization of Scanning Parameters

The user is encouraged to review Section 8.4 which discusses parameter optimization for Contact AFM.

10.5.5 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.

Data Type continued...

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 Tapping Mode 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. With the dual screen mode it is possible to capture both **Height** and **Amplitude data** simultaneously.

10.5.6 Gain Settings

The **Integral** and **Proportional gains** on the **Feedback Controls** 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 **1—2)**, 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**.

10.5.7 Scan Size, Scan Rate, and Setpoint

The **Scan size**, **Scan rate**, and **Setpoint** values effect 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.

Scan Size, Scan Rate, and Setpoint continued...

The **Setpoint** parameter defines the desired voltage for the feedback loop. The **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 **Setpoint**; therefore, the oscillation amplitude remains nearly constant.

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

10.6 Troubleshooting

10.6.1 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.

10.6.2 Engaging the Sample

If the engage aborts because the tip is still too far away from the surface, return to **Stage/Focus Surface** and use the trackball to relocate the surface. After successful re-engaging, a well formed image appears on the display monitor.

Chapter 11 TappingMode AFM in Fluids

11.1 Overview

Tapping Mode also functions in fluid. This technique is available on the Dimension 3100 Scanning Probe Microscope using a fluid cell tip holder in conjunction with the NanoScope IIIa controller, or with a modified NanoScope III controller with a “Fluid Tapping Mode” switch on the front panel. For information regarding required modifications, contact Digital Instruments’ technical support department.

11.2 Principles of Tapping Mode in Fluids

Operation of Tapping Mode in fluid provides many of the same advantages of Tapping Mode in air, with the additional ability to image samples under native liquid conditions. In fluid Tapping Mode, the tip oscillates so that it only intermittently contacts the sample surface. This reduces or eliminates frictional forces that often damage soft samples in Contact AFM. The following sections provide general instructions for Tapping Mode imaging in fluid, as well as provide sample instructions for imaging RNA polymerase—a biological sample.

Before attempting Tapping Mode in fluids, you should become familiar with standard Tapping Mode operation in air (See Chapter 10) and Contact AFM in fluid (See Chapter 9).

11.2.1 Acknowledgments

Digital Instruments Veeco wishes to express its appreciation to the following individuals for their assistance in preparing the following sections: Monika Fritz, Manfred Radmacher, Magdalena Benzanilla, Helen G. Hansma, Jan H. Ho, Craig B. Prater.

11.3 Precautions

11.3.1 Spillage Precautions

Throughout all procedures outlined in the following sections, you will work with fluids on and around the Dimension 3100 SPM. When handling fluids, keep a quantity of filter paper and/or paper towels nearby for wicking away any spilled fluid. The Dimension head is designed to be immersed in no more than 3 mm of fluid when used with a protective skirt.

CAUTION: Users occasionally experience problems with moisture wicking up through fluid cell seals. Users should avoid prolonged cell immersion in fluids. Always remove the cell from the fluid, detach it from the scanner, and dry thoroughly prior to storage. Any moisture present on the end of the scanner must be dried **immediately** to prevent shorting the piezo.

ATTENTION: A la suite de problemes occasionnels, certains utilisateurs ont experimentes des fuites du joint de la cellule liquide. Il est donc recommande d'eviter les immersions prolongees dans un liquide. Une fois l'experience termine, veuillez enlever la cellule de la solution, la retirer de la sonde de balayage piezo et la ranger apres sechage. Afin d'eviter un endommagement du piezo, l'extremite de la sonde de balayage piezo doit etre nettoyer et secher des que la moindre trace d'humidite et/ou de moisissure apparaissent.

WARNHINWEIS: Da es zu Problemen mit Flüssigkeit kommen kann, die durch die Silikonabdichtung an der Meßzelle bis hinauf zum Scanner wandert, beachten Sie bitte das Folgende:Es wird empfohlen, ein längeres Verweilen des Meßkopfes (fluid-cell) in Flüssigkeit zu vermeiden. Nach Abschluß der Messungen entfernen Sie bitte immer die Meßzelle aus der Flüssigkeit, ziehen sie vom Scanner ab und trocknen sie vorsichtig und gründlich bevor sie gelagert wird. Jegliche Flüssigkeit am Ende des Scanners muß SOFORT entfernt werden, um einem Kurzschluß des Piezos vorzubeugen.

Precautions continued...

CAUTION: When imaging fluid samples, use extraordinary precautions against spillage. Fluids must **not** be spilled on or around the sample stage, electronics boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise, damage may result. In the case of a spill, immediately clean and dry all affected surfaces carefully.

ATTENTION: Lors d'un travail en milieu liquide, prendre toute précaution pour éviter des fuites. Les liquides ne doivent pas se répandre sur la platine porte échantillons, le boîtier électronique ou toute autre partie du microscope contenant de l'électronique. Eviter toute fuite de liquide corrosif sur les surfaces exposées. Le non respect de cette recommandation peut entraîner des dommages. En cas de fuite, nettoyer et sécher immédiatement les surfaces touchées.

WARNHINWEIS: Falls Sie Proben in Flüssigkeiten abbilden, lassen Sie äußerste Vorsicht walten, damit keine Flüssigkeit verspritzt wird. Flüssigkeiten dürfen nicht auf die oder nahe der Probenhalterung, der Elektronikbox oder anderen Komponenten, die elektronische Bauteile enthalten, verspritzt werden. Vermeiden Sie bitte, korrosive Flüssigkeiten auf freiliegende Oberflächen zu verspritzen; andernfalls wären Beschädigungen die Folge! Falls Sie Flüssigkeit verspritzt haben, säubern und trocknen Sie alle betroffenen Flächen sorgfältig.

11.4 Basic Tapping Mode AFM in Fluids Operation

The following is a general outline of basic operational procedures involved in Tapping Mode AFM in fluids. For information on Tapping Mode AFM in air, see Chapter 10. For more detailed instructions on head, cantilever, and sample preparation, refer to Chapter 7 of this manual.

11.4.1 Select the Microscope Head

1. Set the microscope head configuration by selecting the **Microscope / Select** panel from the **Di** pop-down menu.

2. Click **OK** to close the **Microscope / Select** dialog box. When enabled, the selected buttons are black.

11.4.2 Select Mode of Operation

1. Select **Microscope/Profile**.

2. Select **Tapping Mode** as the mode of operation.

11.4.3 Head, Cantilever and Sample Preparation

1. Install an etched single crystal silicon tip onto an AFM cantilever holder (See Chapter 7).

2. Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head.

11.4.4 Align Laser

1. Align the laser using the laser diode control knobs.

2. Verify the laser beam is positioned on the back of the cantilever, with a spot visible in the laser viewing window.

11.4.5 Adjust Photodetector

1. Adjust the photodetector so that the red dot moves toward the center of the laser alignment window using the two photodetector adjustment knobs located on the side of the Dimension head.

2. Set the **vertical deflection** to **0**.

3. Verify that the red dot is centered and elliptical in shape in the laser alignment window.

11.4.6 Locate Tip

1. Select **Stage/Locate Tip** or click on the **Locate Tip** icon.

2. Center the tip end of the cantilever under the crosshairs using the two optics adjustment knobs located left of the optical microscope objective.

3. Use the trackball with the bottom left button depressed to focus on the tip end of the cantilever.

11.4.7 Focus Surface

1. Select **Stage/Focus Surface** or click on the **Focus Surface** icon.

2. Focus on the sample surface using the trackball with the bottom left button depressed.

11.4.8 Cantilever Tune

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

1. Select **View / Cantilever Tune**, or click on the **Cantilever Tune** icon. The initial **Cantilever Tune** panel appears with the **Frequency Sweep** (a plot of cantilever response as a function of applied oscillation frequency) on the display monitor.

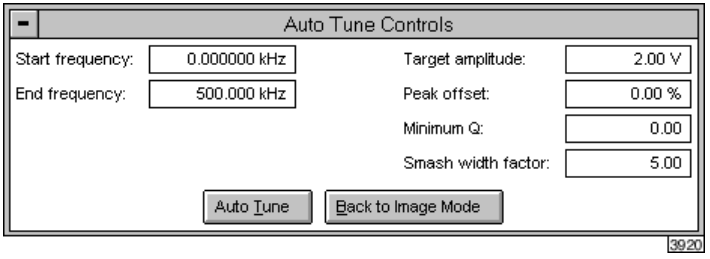
2. Choose either the manual or automatic tuning method (See Automatic Tuning and Manual Cantilever Tuning).

Automatic Tuning

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

1. Click on **AUTO TUNE**. The computer and controller commence with automatic tuning (See Figure 11.4a).

Figure 11.4a Cantilever Tune Control Panel



Cantilever Tune continued...

Manual Cantilever Tuning

With Force Modulation or Fluid Tapping applications, it may be useful to tune the cantilever manually.

> **Note:** More than one type of cantilever exists. Cantilevers can have different dimensions and different resonance frequencies. 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 **Cantilever Tune** panel, set the **Drive frequency** parameter to a value near the center of the resonance 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. Set the **Drive amplitude** to **200 mV**.

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

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

4. Zero the **Setpoint**.

5. Set the **Amplitude limit** to **2.5V**.

6. Center the peak on the frequency sweep plot shown on the display monitor 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.

Manual Cantilever Tuning continued...

7. Increase the **Setpoint** until the peak appears.

8. 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 Tapping Mode 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 11.4d).

Figure 11.4b Cantilever Tune Control Panels for Main Controls

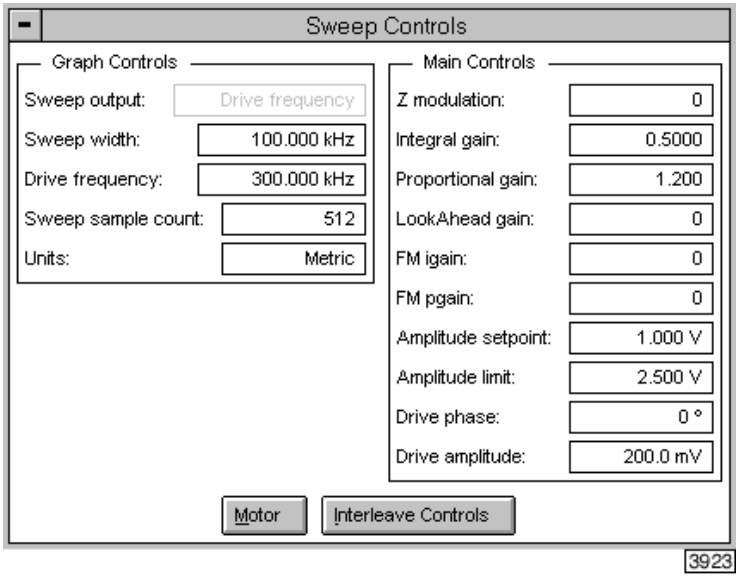
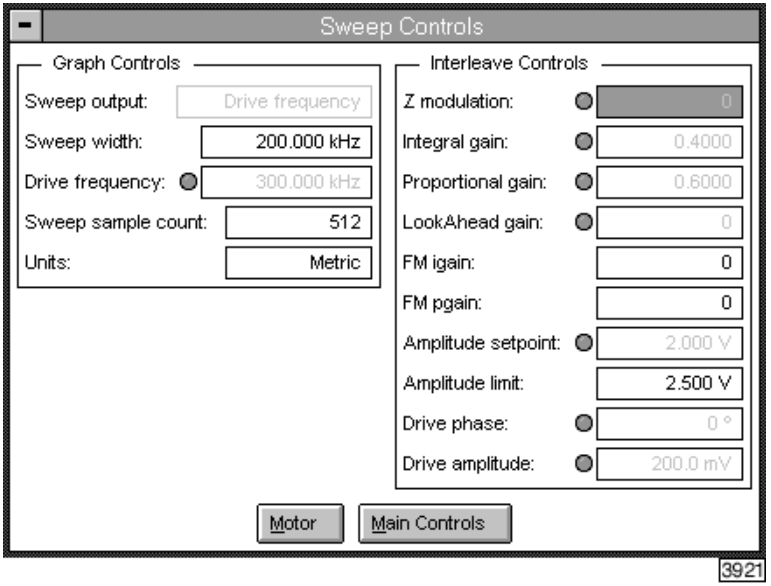
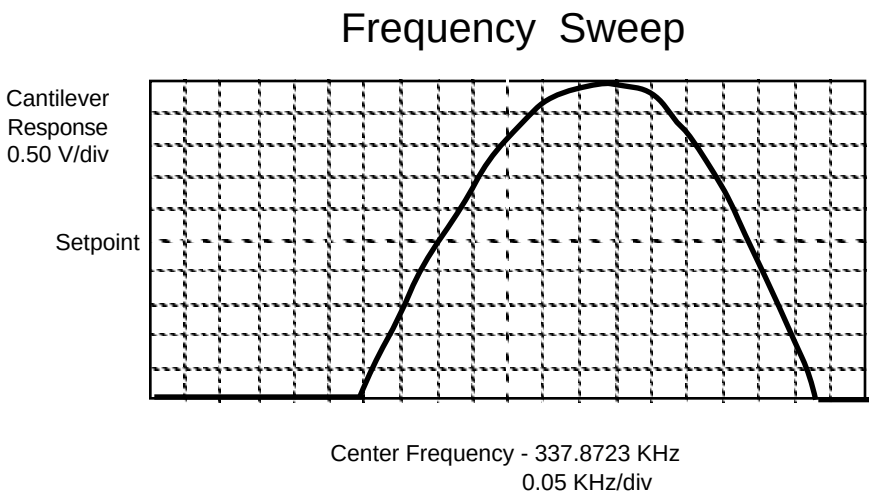


Figure 11.4c Cantilever Tune Control Panels for Interleave Controls



9. 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 11.4d Cantilever Tune Frequency Sweep



Manual Cantilever Tuning continued...

10. Click **OK**. The parameters set in the **Cantilever Tune** control panel appear in the **Real-time** control panel.

11. Click on **CANCEL** to exit the **Cantilever Tune** command and leave the parameters unchanged.

11.4.9 Set Initial Scan Parameters

Scan Controls Panel

In the **Scan Controls** panel, set the following initial scan parameters (See Figure 11.4e)

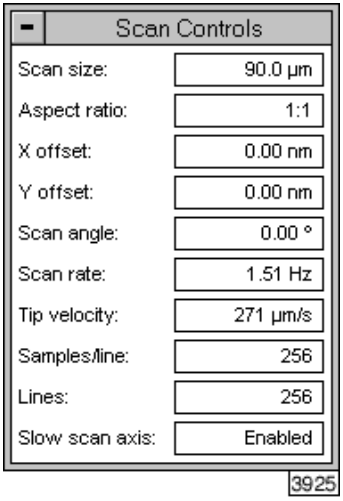
1. Set the **Scan Rate** to **2 Hz**.

2. Set the **Scan Size** to **1µm**.

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

4. Set **X** and **Y Offsets** to **O**.

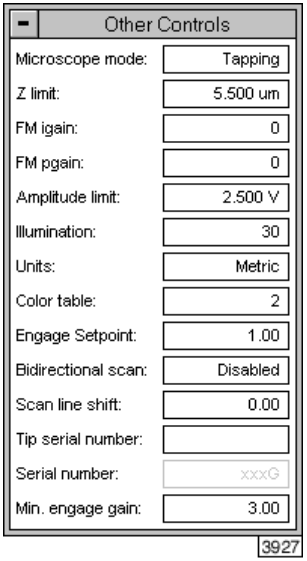
Figure 11.4e Suggested Scan Controls Settings during Tapping Mode



Other Controls Panel

1. Set the **Amplitude Setpoint** to **2.5 volts** on the **Other Controls** panel before imaging (See Figure 11.4f).

Figure 11.4f Suggested Other Controls Settings during Tapping Mode



Feedback Controls Panel

1. Set the **Integral gain** to **0.5** and the **Proportional gain** to **0.7** (See Figure 11.4g).

2. Set the **Look Ahead gain** to zero.

Figure 11.4g Suggested Feedback Controls Settings

Basic Tapping Mode AFM Operation continued...

11.4.10Engage

1. Select **Motor**/**Engage**. A pre-engage check begins, followed by Z-stage motor motion.

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

3. Move the stage using the trackball.

Note: After the tip is engaged, adjust the control panel values to provide the desired scan parameters.

11.4.11Clean the Fluid Cantilever Holder

Before beginning Tapping Mode in fluid, make certain that the fluid cell is completely clean. This prevents contamination of new samples and reduce the entrapment of air bubbles. Generally, a simple bath in a solution of liquid detergent (e.g. dishwashing soap) and distilled water has been found to succeed well. The interior of the cell should be gently scrubbed with an antiseptic swab, then dried using compressed and filtered air (0.2 µm filter). Take care not to scratch the glass surfaces of the cantilever holder.

11.4.12Select a Tip and Mount into the Tip Holder

Selection of a tip will generally be restricted to one of two choices: 1) oxide-sharpened silicon nitride tips; or, 2) electron beam deposition (EBD) tips. In most instances, oxide-sharpened silicon nitride tips will suffice. Since EBD tips are expensive, try oxide sharpened tips first.

1. Install a cantilever into the recess of the fluid cantilever holder.

2. Verify that the cantilever chip (substrate) is flush against the bottom of the recess.

3. Flush the fluid cell and cantilever with distilled water to remove any minuscule chips produced due to handling of the cantilever.

11.4.13Attach Protective Skirt

1. Attach the protective skirt to the fluid cell by gently sliding it over the periphery of the cell.

11.4.14Plug Fluid Cell in Dimension Head

1. Plug the fluid cell into the end of the (unplugged) Dimension head.
2. Slide the skirt up until secure around the edge of the scanner tube.

CAUTION: The protective skirt does not serve as a seal to block fluid from the Dimension head. When the edge of the skirt is immersed in fluid, fluid may wick up inside the skirt and short the Dimension head.

11.4.15Locate the Tip

1. Secure the head in its mount by turning the release screw clockwise.
2. Locate the tip with the optical microscope objective. Detailed instructions are provided in Chapter 7 of this manual.
3. After the optical microscope aligns with the tip, raise the motorized Z-axis by using the **Motor / Step Motor / Tip Up** option on the Real-Time menu, or use the **Withdraw** command.

   Note: Maximize the **SPM step size** parameter at 200 µm.

4. Verify that the head rises far enough above the stage to avoid interference with the sample.

Basic Tapping Mode AFM Operation continued...

11.4.16Prepare the Sample for Imaging

Set the sample on the microscope's stage. For Tapping Mode in fluid, use a petri dish, or if the sample is hydrophobic, image the surface within a drop of fluid.

Note: Immersed samples may tend to dry out during imaging due to evaporation; therefore, keep a quantity of fluid nearby to periodically replenish lost fluid.

11.4.17Remount the Dimension Head

With the sample prepared and positioned on the stage, *slowly* remount the Dimension head in its dovetailed, vertical mount.

CAUTION: If the fluid level is too high, the end of the scanner tube may be dunked too far into fluid. Do not allow this to happen. Permanent damage to the scanner tube may occur.

ATTENTION: Si le niveau du liquide est trop important, l'extrémité des céramiques piézo-électriques peut tremper dans le liquide. Ceci ne doit jamais arriver. Ce cas de figure peut entraîner des dommages définitifs sur les céramiques piézo-électriques.

WARNHINWEIS: Falls der Flüssigkeitsspiegel zu hoch ist, könnte das Ende des Scannerröhrchens in die Flüssigkeit eintauchen-dies darf auf keinen Fall passieren. Es könnte sonst eine dauerhafte Schädigung des Scannerröhrchens eintreten.

Remount the Dimension Head continued...

Ideally, the Dimension head should come to rest in its mount at a level where the tip is just above the level of the fluid. If the head rests too low in its mount, use either of the following methods to raise the mount:

- Raise the mount further by using the **Motor / Step Motor / Tip Up** command.
- Remove the head from its mount and lower the sample into the fluid level. Remount the head.

> **Note:** Because the tip is lowered through a fluid layer, the laser beam will refract slightly requiring minor adjustments. Refer to Chapter 9 of this manual for more information regarding aiming the laser through a fluid layer.

Remount the Dimension Head continued...

At this point, the fluid cell should be prepared for Tapping Mode imaging: the cantilever is aligned with the laser, the sample is prepared and the tip is immersed in fluid and positioned one millimeter above the sample surface. All that remains is to tune the cantilever.

11.4.18Verify that the Microscope is Dry

Verify that all Dimension 3100 surfaces are free of spilled fluid. Wick away moisture and droplets with filter paper.

11.4.19Switch to Tapping Mode

Transfer to the **Other Controls** panel and set the **AFM mode** parameter to **Tapping**.

11.4.20Set Initial Scan Parameters

For initial imaging, try the following settings:

- Drive frequency = 18 kHz.
- Drive amplitude = 1.0 volts
- Scan speed = 2 Hz
- Setpoint = (set automatically during engagement)
- Integral gain = 4.0 volts
- Proportional gain = 1.0 volt
- Z range = 50 nm
- Channel 1 Data type = Height
- Channel 2 Data type = Amplitude

The settings listed above have given good results on RNA polymerase, but the user should try to optimize them for other samples or imaging conditions.

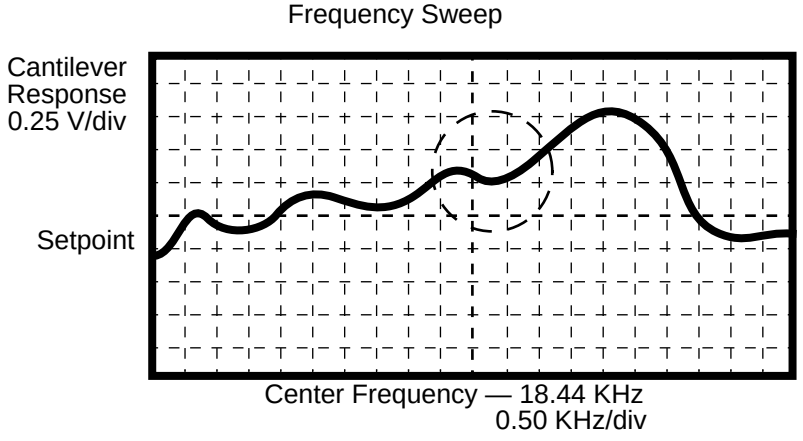
11.4.21Cantilever Tune

Cantilever Tune is the counterpart to the step used in standard (air) Tapping Mode to find the resonant frequency of the cantilever. In liquid, however, the cantilever resonance is largely damped. Instead, this step is used to find an oscillating frequency specific to the fluid and cantilever holder where the cantilever can be driven into oscillation.

Enter the **View / Cantilever Tune** menu 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—19 kHz and 8—12 kHz; higher frequencies have also been used. Start with a **Sweep width** of 10 kHz. Users of Tapping Mode in air will notice that their 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.

A typical **Cantilever Tune** screen is shown in Figure 11.4h. It is necessary to select a frequency where there is some cantilever response, (i.e., near a peak), but experience suggests that it is best to avoid the tops and sides of extremely sharp peaks. The best frequencies appear to be on the side of a peak or in a shallow valley between peaks. The circled region in Figure 11.4h shows a typical operating frequency that produces good fluid tapping images.

Figure 11.4h Typical Cantilever Tune Curve for Silicon Nitride Tip in Fluid



Basic Tapping Mode AFM Operation continued...

11.4.22Adjust the Drive amplitude

Adjust the **Drive amplitude** until obtaining an RMS Amplitude of 0.3-0.6 volts. This value has produced good results for protein samples like RNA polymerase and lysozyme. In general, larger RMS amplitudes (approximately 2 volts) work better for taller samples such as cells.

11.4.23Re-center the Photodiode

Readjust the photodiode until the cantilever deflection is roughly zero. The cantilever deflection signal can drift when the cantilever is in fluid, so it is best to readjust prior to engaging.

11.4.24Engage

Use the **Engage** command to bring the tip into tapping range. The NanoScope software will automatically select a setpoint and will stop engagement when tapping is detected.

11.4.25Adjust the Setpoint

Usually the best images are obtained at setpoints 5—10 percent less than the cantilever RMS amplitude before engaging. The setpoint may be optimized in one of two ways:

Use the Force Calibration Command

The Force Calibration command plots the cantilever amplitude versus the scanner position. A typical Force Calibration is shown in Figure 11.4h. The curve should show a mostly flat region where the cantilever has not yet reached the surface and a sloped region where the amplitude is being reduced by the tapping interaction. Set up the Force Calibration as described for Tapping Mode in air (Experienced users may use the Force Step command instead). To protect the tip and sample, take care that the cantilever amplitude is never reduced to zero. Adjust the setpoint until the green “setpoint line” on the graph is just barely below the flat region of the Force Calibration curve. This is the setpoint that applies the lowest force to the sample.

Adjust the Setpoint continued...

Note: The slope of the Force Calibration curve shows the sensitivity of the fluid Tapping Mode measurement. In general higher sensitivities will give better image quality. If the sensitivity is very poor, consider changing to a different drive frequency or check the mounting of the sample and fluid cell.

Optimize the Image Quality

The setpoint can also be adjusted by simply monitoring the image quality. Select a Scan size of 500 nm. Increase the Setpoint in small increments until the cantilever pulls off the surface and the Z center voltage goes to -220 V. Then reduce the Setpoint in small increments until an image appears. Continue reducing the Setpoint until the image is optimized. Usually the best images are obtained at setpoints just below where an image appears.

11.4.26Optimize Other Scan Parameters

The NanoScope III will attempt to keep the cantilever amplitude constant during the scan. Optimize the **Integral gain** and **Proportional gain** so that the **Height** image shows the sharpest contrast and that there are minimal variations in the Amplitude image (the error signal). It may be helpful to optimize the scan speed to get the sharpest image.

11.5 Troubleshooting

11.5.1 Cantilever Tune Plot Looks Bad

Become familiar with the characteristics of the Cantilever Tune plot when you successfully obtain good images. You may use the Cantilever Tune plot as a diagnostic tool. If the plot looks substantially different from previous successful experiments, there may be a problem with the fluid cell or the cantilever may be loose in its holder.

11.5.2 Laser Sum Signal Absent or Weak

Remove all air bubbles from the cantilever. Bubbles may attach themselves to the cantilever, causing the laser beam to diffract. Remove bubbles by gently squirting the tip and sample with a stream of fluid, taking care not to squirt or splash fluid into spaces above the protective skirt.

11.5.3 Poor Image Quality

Some types of samples may adhere to the cantilever and tip (e.g., certain proteins). If you suspect tip contamination, you must protect the tip against contamination using either of the following methods:

- If the tip adheres to a sample surface through diffusion (e.g., diffusion of protein onto mica), first diffuse the sample substance into the substrate, then flush away stray substance using a straight fluid media. Lower the tip into a fluid containing little or no stray substances that may adhere to the tip.
- If the sample is short-lived and must be imaged quickly, mask the tip against contamination by bringing the tip into gentle contact with an uncontaminated substrate surface. Set the Dimension 3100 in Contact AFM mode by switching the **AFM mode** parameter on the Other Controls panel to **Contact**. Engage the substrate surface using a zero scan size. While keeping the tip gently in contact with the substrate surface, add the sample substance to be imaged to the petri dish and allow the sample substance to settle onto the substrate. Once diffusion is reached, quickly lift the tip from the substrate surface. Switch to **Tapping Mode** and image the sample.

11.5.4 Unable to Locate Particulate Samples

Some particulate samples (i.e., proteins) may prove difficult to find directly beneath a cantilever if the cantilever remains 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 are located; they are stuck to the cantilever. If you suspect this problem, simply shift the imaging site to a location outboard of the tip and cantilever. You should find more individual samples there.

Chapter 12 Lateral Force Mode

12.1 Overview

The Dimension 3100 Scanning Probe Microscope (SPM) is capable of measuring frictional forces on sample surfaces using a special feature known as Lateral Force Microscopy (LFM). With LFM, the cantilever scans laterally (perpendicular to their lengths). The cantilever torques more while transiting high-friction sites while low-friction sites tend to torque the cantilever less. The relative measure of lateral forces encountered along the surface yields a map of high- and low-friction sites.

After obtaining a good topographical image in AFM mode, it is relatively easy to switch to LFM mode to view and acquire lateral force data. It is important to obtain a good image in AFM mode before switching to LFM mode. The NanoScope system continues to run the feedback based on the AFM signal and feedback gains while generating and displaying LFM data.

This chapter details the following topics:

12.2 Basic LFM Operation

The following is a general outline of basic operational procedures involved in Lateral Force Mode (LFM). For more detailed instructions, refer to Chapter 7 of this manual. To run the microscope in LFM, set up the system as you would for Contact Mode AFM with the following exceptions: assign the **Channel 1** image to **Data Type: Height**, set the **Channel 2** image to **Data Type: Friction**, and set the **Scan angle** to **90 degrees**.

12.2.1 Select the Microscope Head

1. Set the microscope head configuration by selecting the **Microscope / Select** panel from the **Di** pop-down menu.

2. Click **OK** to close the **Microscope / Select** dialog box. When enabled, the selected buttons are black.

12.2.2 Select Mode of Operation

1. Select **Microscope/Profile**.

2. Select **Contact Mode** as the mode of operation.

12.2.3 Head, Cantilever, and Sample Preparation

1. Install a tip onto an AFM cantilever holder (See Chapter 7).

2. Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head.

12.2.4 Align Laser

1. Align the laser using the laser diode control knobs.

2. Verify the laser beam is positioned on the back of the cantilever, with a spot visible in the Dimension head filter screen and a sum signal between 4-6 volts.

Basic LFM Operation continued...

12.2.5 Adjust Photodetector

1. Adjust the photodetector so that the red dot moves toward the center of the Dimension head filter screen using the two photodetector adjustment knobs located on the side of the Dimension head.

2. Verify that the red dot is centered and elliptical in shape in the Dimension head filter screen.

3. Set the **Vertical Deflection** to **-2.0 Volts**.

12.2.6 Locate Tip

1. Select **Stage/Locate Tip** or click on the **Locate Tip** icon.

2. Center the tip end of the cantilever under the crosshairs using the two optics adjustment knobs located left of the optical microscope objective.

3. Use the trackball with the bottom left button depressed to focus on the tip end of the cantilever.

12.2.7 Focus Surface

1. Select **Stage/Focus Surface** or click on the **Focus Surface** icon.

2. Focus on the sample surface using the trackball with the bottom-left button depressed.

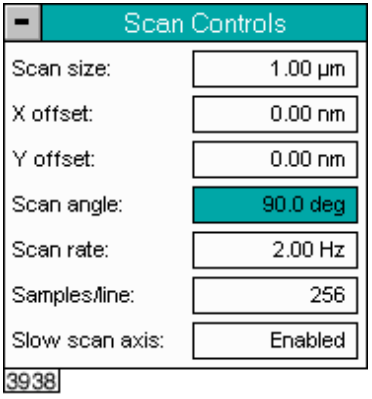
12.2.8 Set Initial Scan Parameters

Scan Controls Panel

In the **Scan Controls** panel, set the following initial scan parameters (See Figure 12.2a).

1. Set the **Scan Rate** to **2 Hz**.
2. Set the **Scan Size** to **1µm**.
3. Set the **Scan Angle** to **90 degrees**.
4. Set **X** and **Y Offsets** to **0**.

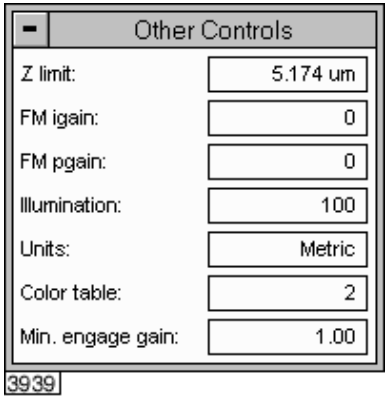
Figure 12.2a Suggested Scan Controls Settings



Set Initial Scan Parameters continued...

Other Controls Panel

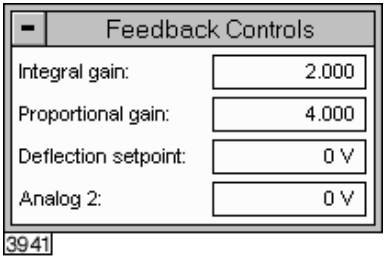
Figure 12.2b Suggested Other Controls Settings



Feedback Controls Panel

1. Set the **Integral gain** to **2.0** and the **Proportional gain** to **4.0** (See Figure 12.2c).

Figure 12.2c Suggested Feedback Controls Settings



Channel Panels 1 and 2

1. In the **Channel 1** panel, set **Data type** to **Height** (See Figure 12.2d).

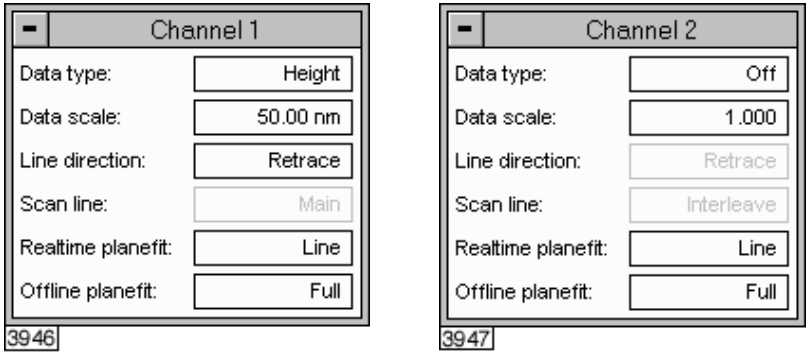
2. Set **Z range** to a reasonable value for the sample.

   Note: For example, for a 200 µm step height calibration sample, a reasonable Z range setting is 300 µm initially.

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

4. On the **Channel 2 panel,** verify **Data type** is set to **Friction** to disable the panel.

Figure 12.2d Suggested Channel Controls Settings



Basic LFM Operation continued...

12.2.9 Engage

1. Select **Motor**/**Engage**. A pre-engage check begins, followed by Z-stage motor motion.

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

3. Move the stage using the trackball to the next area of interest on the sample.

4. Select **Motor**/**Engage.**

Note: After the tip engages, adjust the control panel values to provide the desired scan parameters.

12.3 Advanced LFM Operation

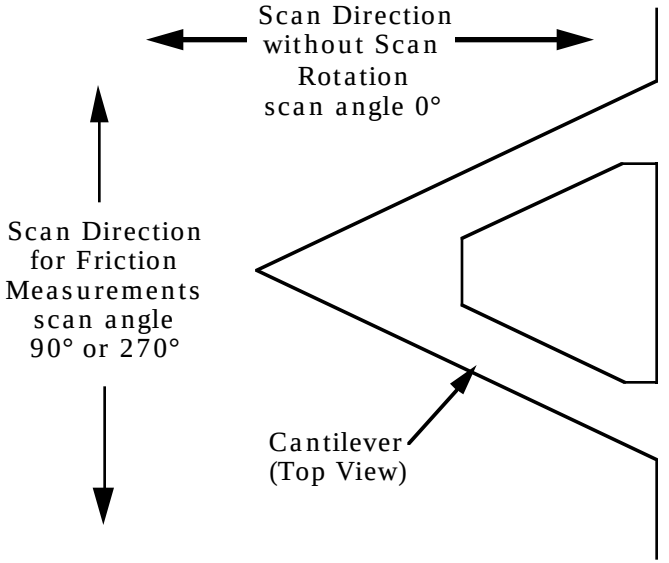
The following sections provide more detailed information concerning the operation of LFM in friction mode.

12.3.1 Optimal Setup for Frictional Measurements

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 12.3a. The **Scan angle** parameter in the **Scan Controls** panel must be set to **90°** or **270°** to produce this scan direction.

Figure 12.3a Scan Angle Selection



Scan Direction continued...

The Contact Mode setpoint voltage slightly adjusts 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 is necessary to change 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.

Tip Selection

The analog-to-digital converter on the auxiliary input channel which is used for LFM data has a maximum input range of ±10 volts. This, and the anticipated interaction between tip and sample, define the selection of the cantilever to be used for the measurement.

The 200-micron 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 ± 10.00 volts with the 200-µm wide-legged cantilever, one of the stiffer 100-µm cantilevers should be used. If the signal level is too small, the narrow-legged 200-µm cantilever provides a larger signal.

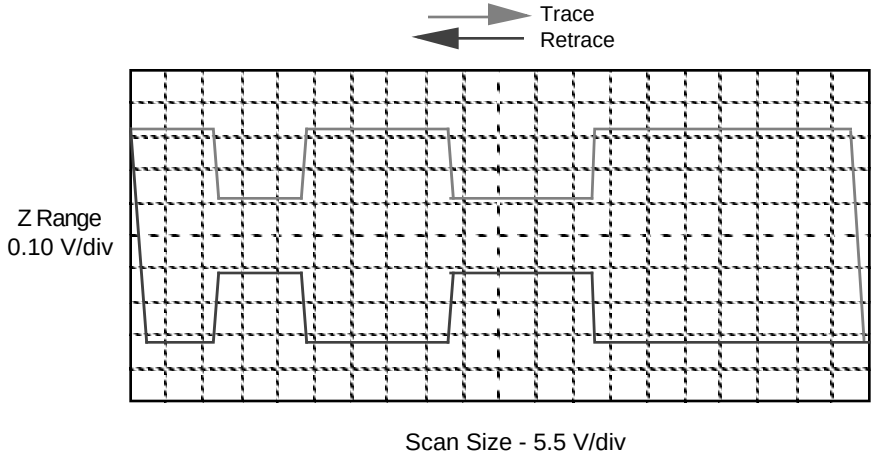
12.3.2 Identification of Friction

The lateral force on the tip is caused by both friction and the tip “running into” or “tripping on” the edges of features. There are a few things the user can do to verify that data obtained in **Data Type: Friction** is a result of friction between the tip and the sample. The topographical information should be compared to the **Friction** data in both scan directions. The **Scope Mode** can also be very useful when analyzing frictional data.

Scope Mode

The LFM user should be familiar with the **Scope Mode** of viewing the data. When using **Scope Mode**, verify that the **Rounding** parameter in the **Scanner Parameters** window (selected with the **Microscope/ Calibrate / Scanner** command) is set to zero. In the dual-trace mode, the data from the trace and the retrace of the sample under the tip is shown in an oscilloscope format. The vertical axis represents the differential signal from the left and right photodiodes, while the horizontal axis represents the tip position along the fast scan direction. There are several general features to observe in the scope signal (See Figure 12.3b).

Figure 12.3b Example of a Scope Trace with Low-Friction Areas



With the scan direction rotated, the separation between the trace and retrace lines in **Scope Mode** provides a general indication of the amount of friction between the sample and the tip. For example, if there is no friction between the sample and the tip, the trace and retrace will coincide vertically. As the frictional forces between the tip and the sample increase, the cantilever twists to a larger degree and the vertical separation between the trace and the retrace increases.

Note: There is virtually always some friction between the tip and the sample.

Scope Mode continued...

The trace and retrace signals move closer together when regions of low friction are encountered. Figure 12.3b shows a theorized example of the scope trace for a sample with two areas of relatively low friction. Note how the distance between the traces decreases over low-friction areas as the torsional deflection of the cantilever decreases for both trace directions. When trace and retrace both deflect in the same direction, this is often caused by non-frictional effects (e.g., optical interference). In general, samples with areas of varying friction will include step-like features in the **Scope Mode** view.

Compare to Topography

Comparing frictional data to topographical data for the same scan area provides another means of deducing the origin of the frictional features. Transient frictional features often occur at the edges of topographical features. The tip “trips” over the feature, producing the transient frictional feature.

Reverse Scan Direction

Reversing the scan direction in **Image Mode** while viewing the friction channel is also useful in verifying the origin of the data. Note that changing the **Line direction** parameter in the Real-time control panel changes the direction of the scan during which data is collected. If the data results from friction between tip and sample, the relative signal strengths invert as the scan direction is reversed. For most color tables, the image produced from the trace in Figure 12.3b (with the **Line direction** parameter set to **Trace**) would be darker in low-friction areas than would be in high-friction areas. Conversely, for the same color table, the image produced from the retrace (with the **Line** direction parameter set to **Retrace**) would be lighter in low-friction areas.

12.3.3 Identification of Forces Other Than Friction

There are a few phenomena that will produce false features in friction data on the auxiliary data channel.

Tripping

Tripping occurs when the probe encounters a step on the sample surface. The side of the probe strikes the feature, causing the cantilever to twist. In **Image Mode** on the auxiliary channel, tripping appears as highlights coinciding with topographical features. In the **Scope Mode** on the auxiliary channel, tripping produces transients similar to those shown in Figure 12.3c. Regions of high-friction can also produce transients similar to those shown in Figure 12.3c, so the user must be careful to analyze the data carefully.

Optical interference

Optical interference between the laser light reflected off the cantilever and secondary sources (e.g., laser light spilling over the sides of the cantilever, light transmitted through the cantilever, or light reflected off the sample and back through the cantilever) produce evenly spaced lines in the image. The spacing of the lines ranges between 1.5 and 2.0 microns.

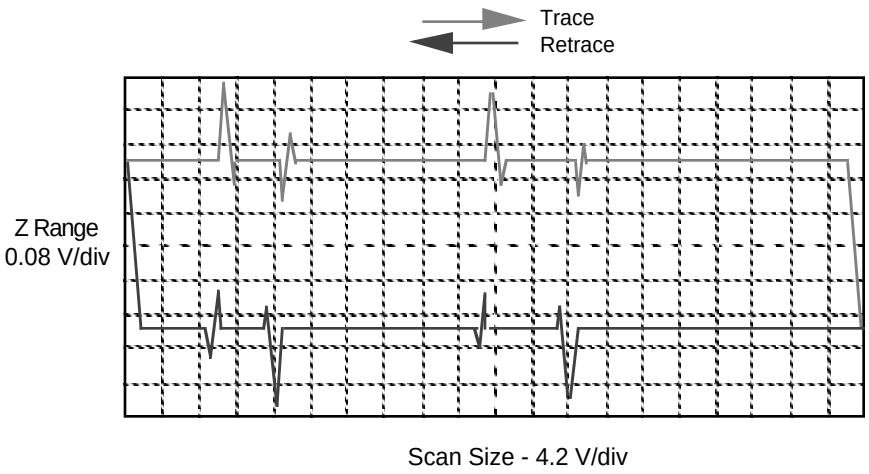
Identification of Forces Other Than Friction continued...

Rounding

Use the **Calibrate** command under the **Microscope** pop-down menu in **Real-time** to set the **Rounding** parameter to zero before looking at frictional data. In **Scope Mode,** the Rounding function shifts the trace and retrace lines horizontally which makes it difficult to directly compare the two lines.

Figure 12.3c Tripping Transients in Scope Mode

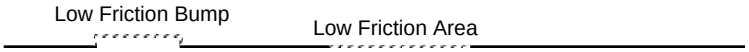
Example of Sample Surface with Uniform Friction



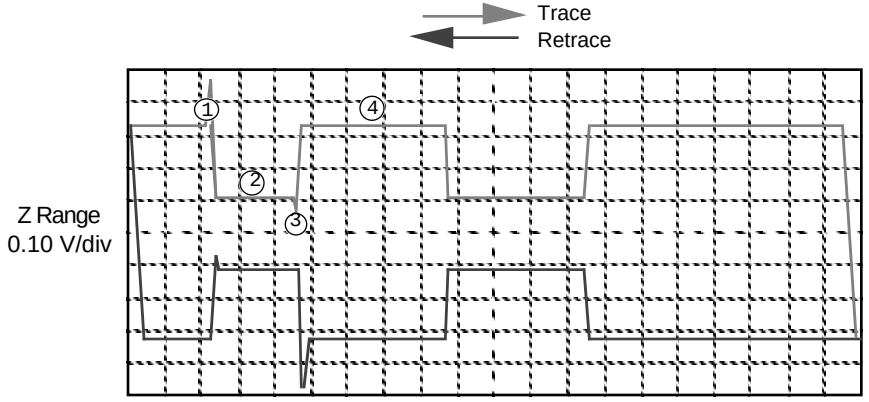
12.3.4 Example of Frictional Data

Rarely will samples produce traces like the ones shown in Figure 12.3b and Figure 12.3c. Typically, combinations of the features discussed above will be present in each trace and retrace. For example, Figure 12.3d depicts the data Scope Mode might generate for a sample with two low-friction areas where one is a flat bump (e.g., low-friction foreign material sticking up on a high-friction surface) and the other a low-friction area even with the sample surface. In Figure 12.3d note that as the tip strikes the edge of the bump (point 1), the lateral force increases momentarily and then decreases as the tip scans across the low-friction bump (point 2). At the far edge of the bump, the lateral force may decrease somewhat as the tip comes off the bump (point 3), and then increase as the tip scans across the high-friction surface (point 4). At the second low-friction area, the spikes due to topography are not present. On the retrace, notice that the sign of the frictional forces will change, but the shape will remain the same. The lateral forces due to topography shift to the other side of the bump and also change sign.

Figure 12.3d Scope Mode View of Example Surface



Example of Sample Surface with Two Low Friction Areas



Chapter 13 Force Imaging

13.1 Overview

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:

13.2 Force Plots–An Analogy

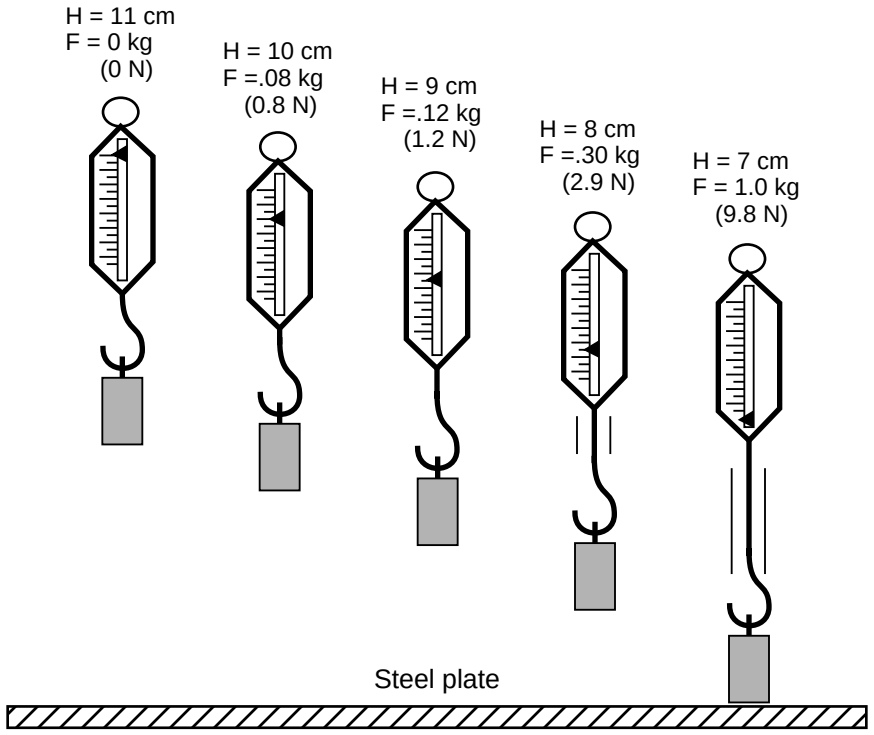
A force plot is an observation of tip-sample interactions which yields information regarding the sample and tip. By way of analogy, suppose a materials researcher must determine how powerful two different types of magnets are. One magnet is made of iron, the other is a stronger, so-called “rare earth” 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, 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 13.2a.

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).

Force Plots–An Analogy continued...

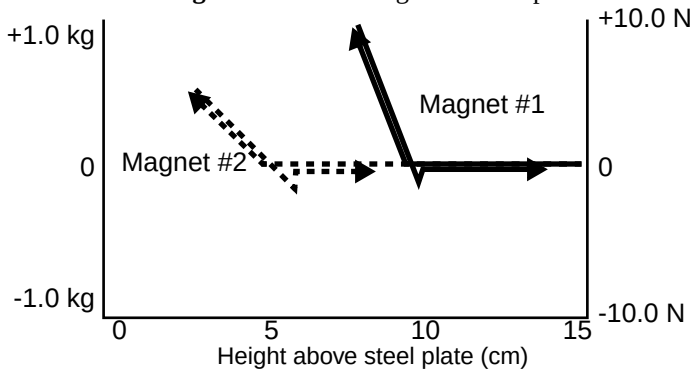
Figure 13.2a Comparative Index of Pulling Forces



The pulling force is measured at 1 cm height intervals while the scale and magnet lower and lift in a controlled cycle. Figure 13.2b 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 power. First, magnet #1 is stronger, attaching to the steel plate with 1.0 kg of pulling force at 7 cm, and magnet #2 is weaker, attaching at 2 cm with only 0.6 kg. Secondly, the slope of the plots reveal information about the range and density of each magnet's field. Magnet #1 exhibits quick attachment and pull-off from the plate with a steep curve, magnet #2 exhibits more sluggish attachment and pull-off with a less-sloped curve, due perhaps to a weaker magnetic field.

Force Plots–An Analogy continued...

Figure 13.2b Pulling Forces Graph

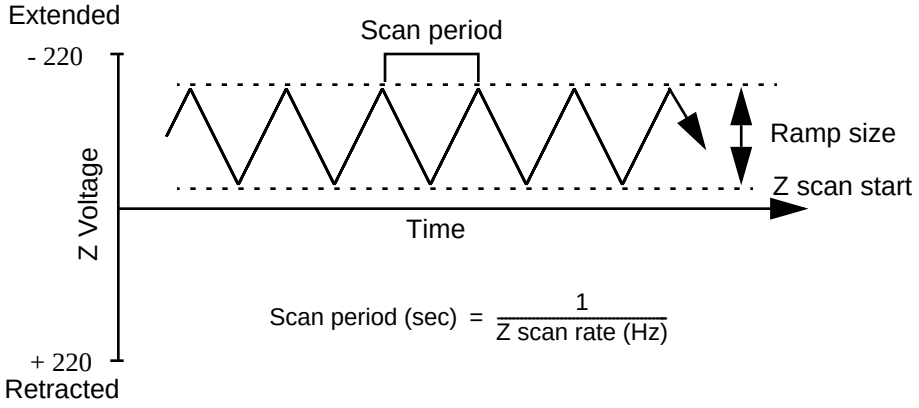


This oversimplified model depicts activity between SPM tips and various materials. In reality, SPM force plots reveal far more. For example, by combining force curves at regularly spaced intervals across the sample, you may generate a force map of the sample's electric properties, elastic modulus, and chemical bonding strengths.

13.3 Force Calibration

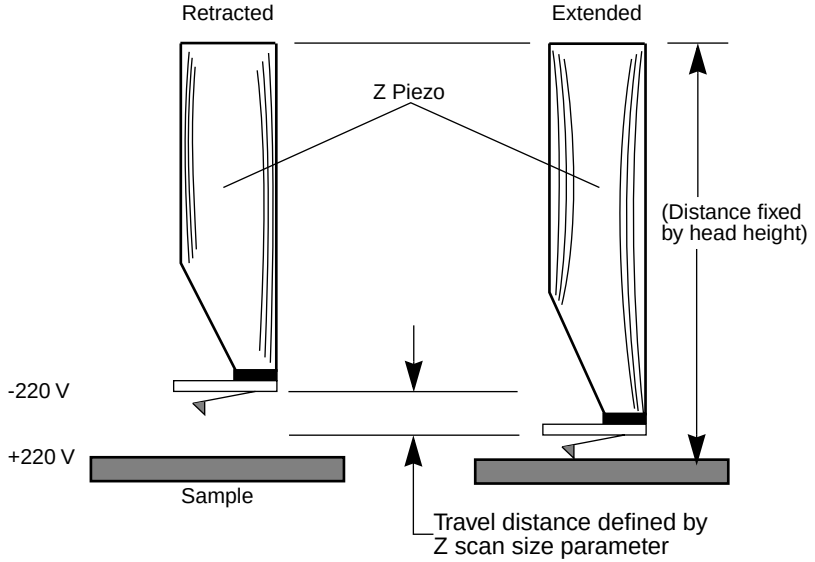
The **Force Calibration** command in the **View / Force Mode / Calibration** menu allows you to quickly check the interaction between the cantilever and the sample surface. In **Force Calibration** mode, the X and Y voltages applied to the piezo tube are held at zero and a triangular waveform similar to the one depicted in Figure 13.3a is applied to the Z electrodes of the piezo tube.

Figure 13.3a Force Calibration Z Waveform



As a result of the applied voltage, the cantilever tip moves up and down relative to the stationary sample as shown in Figure 13.3b. The **Z scan start** parameter 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 **+220** volts, with the **Ramp size** set to **440** volts.

Figure 13.3b Piezo Travel in Force Calibration Mode



As the piezo moves the tip 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 on the display monitor. The control panel detailing parameters controlling the microscope in **Force Calibration** mode displays on the control monitor.

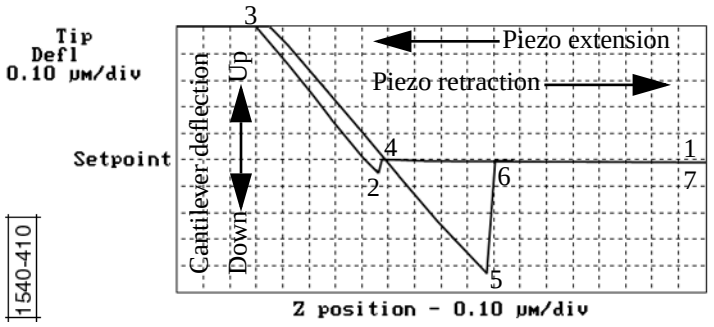
Force Calibration mode is frequently used to adjust and calculate contact forces between the cantilever and the sample. Other uses of **Force Calibration** mode include characterization of the forces on the cantilever tip, diagnosing AFM performance, and determination of the sensitivity of the cantilever deflection voltage in terms of voltage applied to the piezo. If used correctly, **Force Calibration** mode provides a variety of useful information.

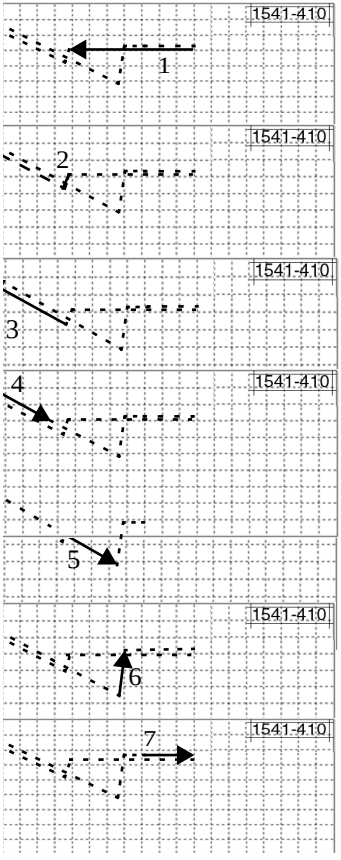
13.3.1 Example Force Plot

A Contact Mode AFM force plot using a silicon nitride tip is the most simple SPM force plot. Because of the pliant property (and lower spring constant) of silicon nitride tips, they are sensitive to attractive and repulsive forces. A force plot in Contact Mode AFM is shown in Figure 13.3c.

Example Force Plot continued...

Figure 13.3c 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.

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.

Example Force Plot continued...

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 right-to-left in yellow on the NanoScope display monitor. By retracting the Z-axis piezo crystal, the tip ascends away from the sample and the tip-sample distance increases. The ascent plots from left-to-right in white on the NanoScope display monitor.

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.

The graph detailed in Figure 13.3a reveals the following types of information:

Adjusting the Force Between Tip and Sample

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.

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.

Example Force Plot continued...

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**.

13.4 Force Calibration Control Panels

The control panel window (See Figure 13.4a) manipulates the microscope in **Force Calibration** mode and displays on the control monitor. The parameters control the 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. With the **Motor Control / Tip Up** and **Tip Down** buttons (See Figure 13.4b), you can use the stepper-motor to adjust the position of the cantilever tip relative to the sample. The **Capture** button stores the force curve for **Off-line** viewing. Some parameters influence the **Real-time** operation of the microscope; most of the menu parameters affect only **Force Calibration** mode.

Figure 13.4a Force Calibration Control Panel (Contact Mode AFM)

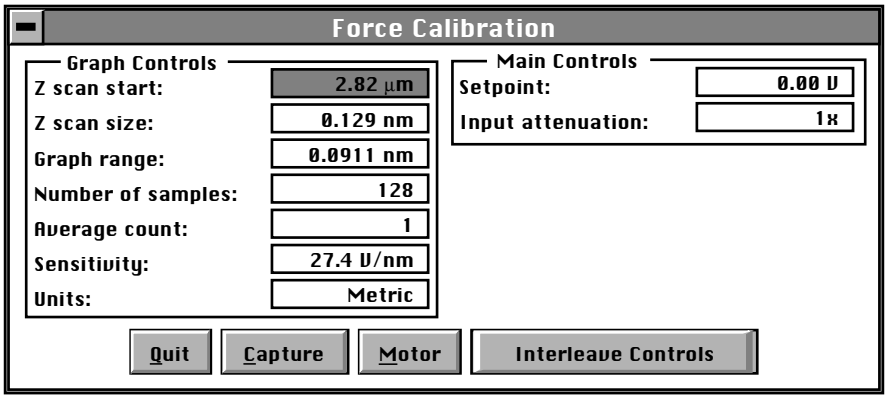
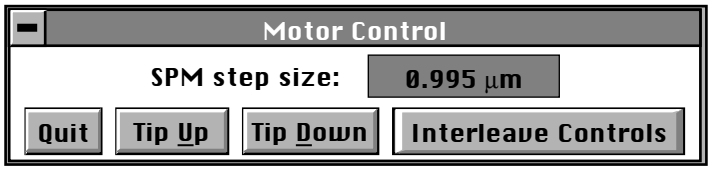


Figure 13.4b Motor Control Panel



The **Force Calibration** menu allows you to precisely control tip interaction with the sample surface. This is useful during Contact Mode AFM procedures as it directly influences image quality and the degree to which forces from the tip influence the sample. Items in the **Force Calibration** control panel are discussed individually below.

Force Calibration Control Panels continued...

13.4.1 Main Controls (Ramp Controls)

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 13.3a displaces up and down in relation to the value of **Z scan start**. Increasing the value of the **Z scan start** parameter moves the sample closer to the cantilever 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 **Force Calibration** mode. Decreasing the value of this parameter shifts the force curve on the display to the left, while increasing the parameter shifts the curve to the right.

Ramp Size

As shown in Figure 13.3a, 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 compresses the curve on the display.

Data Scale

This parameter defines the vertical scale of the cantilever deflection signal versus Z voltage graph shown in **Force Calibration** mode. Increasing this parameter expands the display range about the centerline causing more of the force curve to fall on the graph. This parameter should generally be set to full range (2.5 volts).

Number of Samples

This parameter defines the number of data points captured during each extend and retract operation of the Z piezo during **Force Calibration**. This parameter does not affect the number of samples used in **Image Mode**.

Average Count

This parameter defines the number of **Force Calibration** scans taken to average the display of the **Force Calibration** graph. Set to **1** unless the user needs to reduce noise. Otherwise, set between **1** and **1024**.

13.4.2 Main Controls (Display)

Units

This item selects the units, either metric lengths or volts, that define the parameters and the horizontal axis of the graph. Changing this item in **Force Calibration** mode also changes it in the **Real-time** imaging mode.

X Rotate

X Rotate allows you to move the tip in the X direction during indentation. 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. When indenting in the Z direction, the **X Rotate** parameter allows you to add movement to the scanner in the X direction. **X Rotate** moves the scanner in the opposite direction in which the cantilever points. Without **X Rotate** control, the tip pitches forward during indentation. Normally, it is set to about **22.0 degrees**. This parameter typically ranges between **15** and **25 degrees.**

13.4.3 Channel 1, 2, 3 Panels

Data Scale

This parameter vertically scales the cantilever deflection signal versus Z voltage. 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.

Channel 1, 2, 3 Panels continued...

Deflection Sensitivity

This item relates the cantilever deflection signal to the Z travel of the piezo. It equals the slope of the deflection versus Z voltage line when the tip is in contact with the sample as shown in Figure 13.3c. The NanoScope system automatically calculates and enters the value from the graph once you use the mouse to fit a line to the graph. You must properly calculate and enter the deflection sensitivity value before reliable deflection data in nanometers display in **Real-time** mode. For a proper force curve, the line has a negative slope with typical values of 10-50 mV/nm; however, by convention, values appear as positive in the menu.

Amplitude Sensitivity (Tapping Mode)

Amplitude Sensitivity relates the vibrational amplitude of the cantilever to the Z travel of the piezo. To calculate the **Amplitude Sensitivity**, measure the slope of the **RMS amplitude** versus the **Z voltage** when the tip is in contact with the sample. The NanoScope system automatically calculates and enters the value from the graph after you use the mouse to fit a line to the graph. You must accurately calibrate the **Amplitude Sensitivity** before amplitude data obtained in LiftMode is accurate.

13.4.4 Feedback Controls

Deflection Setpoint (Contact Mode AFM)

By changing the deflection setpoint, you can adjust the cantilever deflection voltage maintained by the feedback loop in **Real-time** mode. In **Force Calibration** mode, this parameter defines the centerline of the vertical, “Cantilever Deflection Voltage” axis of the force calibration curve shown on the display monitor. Changing the setpoint shifts the force calibration curve on the graph, because the centerline value is always equal to the setpoint. For example, if the setpoint is set to -3.0 volts, the cantilever deflection axis of the graph centers around -3.0 volts. Raising the setpoint to -2.0 volts shifts the force calibration curve down by one volt so the graph centers at -2.0 volts. Changing the value of this parameter in **Force Calibration** mode also changes the Real-time setpoint in the **Real-time** control panel.

Amplitude Setpoint (Tapping Mode AFM)

The RMS value of the cantilever deflection voltage maintained by the feedback loop in **Real-time** mode can be adjusted by changing **Setpoint**. In the TappingMode force plot mode, setpoint defines the centerline of the vertical, “Tip Amplitude” axis of the amplitude calibration plot shown on the display monitor. Changing the **Setpoint** shifts the amplitude curve on the graph, because the centerline value is always equal to the Setpoint. For example, if the setpoint is at 3.0 volts, the RMS amplitude axis of the graph will be centered around 3.0 volts; raising the setpoint to 4.0 volts will shift the amplitude curve down by one volt, so the graph will be centered at 4.0 volts. Changing the value of this parameter in the **Amplitude Calibration** mode also changes the setpoint parameter in the **Real-time** control panel.

Deflection/Amplitude Limit

This parameter changes the gain applied to the input signal. You can reduce the input signal by a factor of eight to see more of the **Force Calibration** plot signal amplitude. This parameter also changes in the **Real-time** menu and affects the resolution of the system; therefore, change the value to **2.5 V** before returning to imaging.

Drive Frequency (Tapping Mode AFM)

This parameter defines the frequency at which the cantilever oscillates. 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 **Real-time** control panel.

Feedback Controls continued...

Drive Amplitude (Tapping Mode AFM)

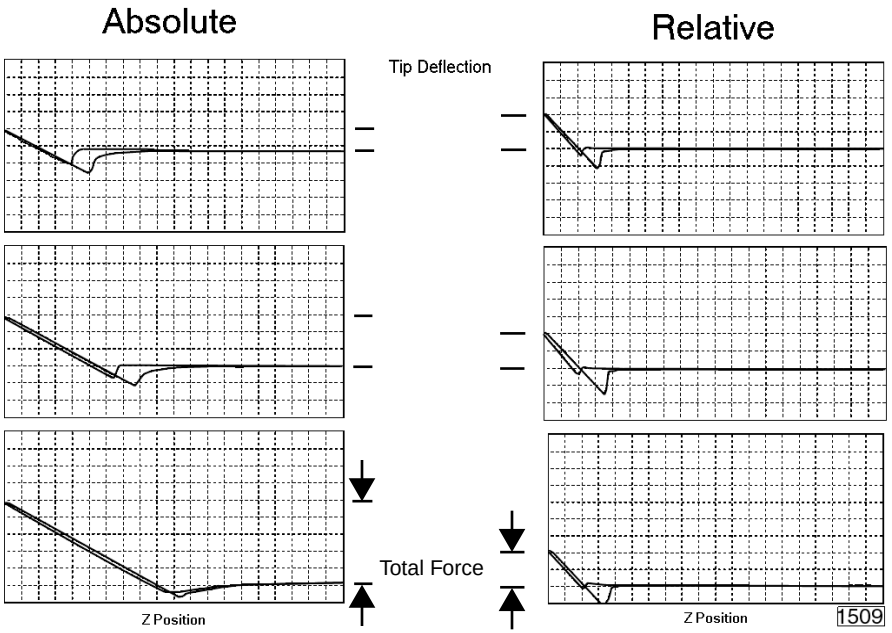
This parameter defines the amplitude of the voltage applied to the piezo system which drives the cantilever vibration. Changing the value of this parameter in **Force Plot** mode also changes the **Drive amplitude** parameter in the **Real-time** control panel. Start with a small value and increase incrementally to avoid fracturing the cantilever by using too high of a drive amplitude. If the amplitude calibration plot consists of a flat line all the way across, changing the value of this parameter shifts the level of the curve. If it does not, the tip is fully extended into the surface. Withdraw the tip before proceeding.

13.4.5 Scan Mode

Triggering

The **Scan Mode** panel allows you to use various triggers when obtaining **Force Plot** and **Force Volume** plots. The idea of a trigger 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 13.4a Absolute and Relative Triggers



The plots in Figure 13.4a show the effect of drift on each of the two trigger types. The plot series shown on the right side of Figure 13.4a utilizes a **Relative** trigger and maintains force at a constant level defined by the **Trig threshold** parameter. As illustrated, the total force is maintained, even as the system drifts.

The plot series on the left side of Figure 13.4a 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.

13.4.6 Menu Bar Commands

Capture

The **Capture** button stores the force plot for **Off-line** viewing.

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. **Probe** menu commands are helpful when performing force plots in Tapping Mode and are described in more detail in the *Command Reference Manual*.

Stepper Motor

Within the **Force Calibration** menu, a separate motor control window (See Figure 13.4b) allows you to move the tip at will. Use this control during the **Force Calibration** procedure to adjust the tip height above the sample surface with the Z stage stepper motor.

- **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.

- **Enable Motion Keys**

  This permits rapid upward movement of the SPM using the **Shift** keys, or down using the **CTRL** or **ALT** keys. To move a small vertical distance toward the surface, set the **SPM step size** value to the desired distance increment and click on **Tip Down**.

Stepper Motor continued...

CAUTION:	Do not use the **Enable Motion Keys** control to move the tip down if the tip is already located on the surface of the sample; damage will result.
ATTENTION:	Si la pointe est engagée sur la surface de l’échantillon, n’UTILISEZ pas la commande "Engage Motion Keys" pour faire descendre la pointe. Le non respect de cette procédure peut entraîner des dommages.
WARNHINWEIS:	Benutzen Sie NIEMALS die „Enable Motion Keys“ Funktion um die Meßspitze nach unten zu bewegen, falls sich die Meßspitze schon auf der Oberfläche befindet; andernfalls sind Beschädigungen die Folge.

13.5 Force Calibration (Contact Mode AFM)

13.5.1 Obtaining a Good Force Curve

Figure 13.5a Typical Force Calibration Curve

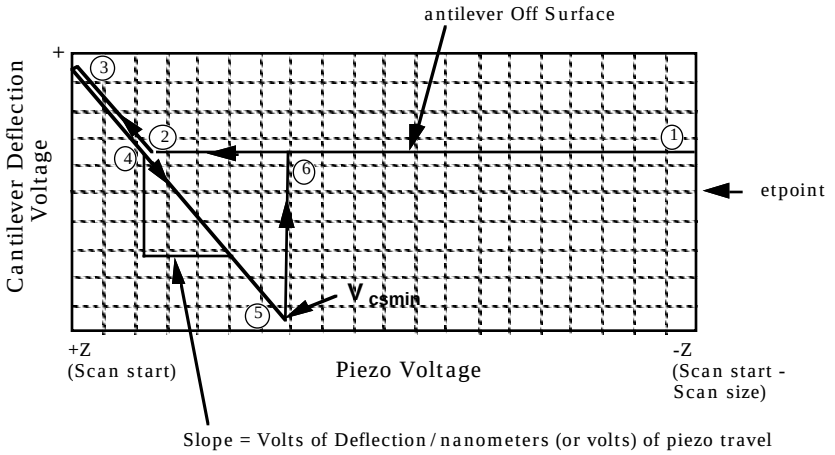
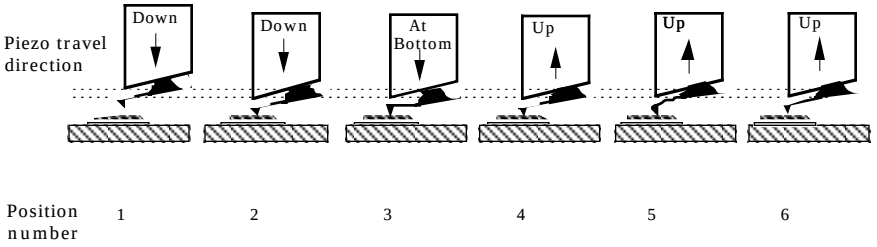


Figure 13.5b Piezo Positions for Typical Force Curve



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 13.5b. However, the force curve rarely looks "typical" right after invoking the **Force Calibration** menu. This section discusses general approach adjustments to improve force curves obtained after engaging the microscope.

Obtaining a Good Force Curve continued...

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. Maximize the **Data Scale** parameter by setting it to **2.5 volts**.

2. Adjust the **Ramp size** parameter.

3. For the Dimension 90-micron, G scanner, set the **Ramp size** to **100 volts** or the equivalent in nanometers (generally it is easier to work in the **Force Calibration** mode with the **Units** parameter set to **Volts**). This setting moves the tip far enough away from the sample to overcome the attractive forces and pull off of the surface.

4. If the **Ramp size** is not set high enough, increase the **Z scan start** value. The maximum **Ramp size** and the **Z scan start**, in volts, are related by the following equation:

   $$\textbf{(Z scan size}_{\textbf{MAX.}}\textbf{) = 220 volts + (Z scan start)}$$

5. As the **Z scan start** increases, the traces on the force curve move to the right. As **Ramp size** increases, the slope of the traces should increase.

6. 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 horizontal centerline of the graph is always equal to the **Setpoint** 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.

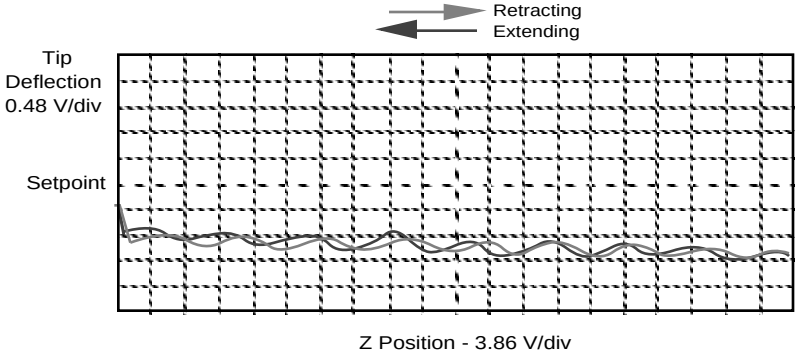
Obtaining a Good Force Curve continued...

If the force curve does not show the tip pull-off with the **Setpoint** adjusted to the minimum value of **-10 volts**, increase the **Ramp size**. If the **Ramp size** is set to **440 volts** with the **Setpoint** at -10 volts and the pull-off cannot be seen, see Section 13.5.2 that follows. If that does not help, call Digital Instruments Veeco.

Examples for 90-µm G Scanners

Figure 13.5c illustrates an initial force curve for a 90-micron scanner before adjustment. Figure 6.13 represents an adjusted, desirable force curve for the 90-micron scanners. (Note that the **Graph range** is set to the maximum voltage for all of the following examples.)

Figure 13.5c Unadjusted Force Curve for 90 µm Scanner



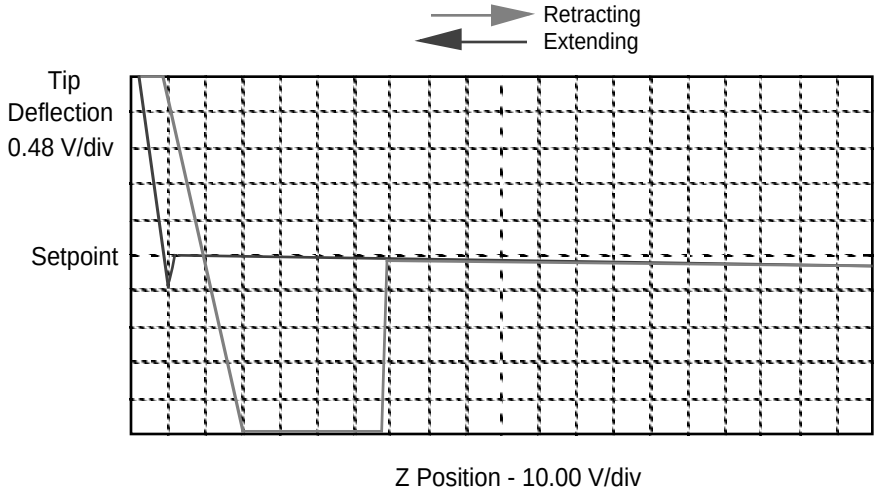
For the 90-micron scanner, try the following steps to get from the initial force curve, showing flat lines across the bottom of the graph, to a force curve similar to the one shown in Figure 13.5a.

1. Increase or decrease the **Setpoint** until you observe the curve (not railed on the top or bottom of the graph).

2. Verify the **Ramp size** is at **50-100V**.

3. Increase **Z scan start**.

Figure 13.5d Adjusted Force Curve for 90 µm Scanner



13.5.2 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 **Force Calibration** mode are included.

False Engagement

Figure 13.5e 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.

Although you can use the **Tip Down** button to move the tip down to the surface, 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 has the effect of pushing the cantilever farther up before the Setpoint is reached.

Helpful Suggestions continued...

Figure 13.5e False Engagement (G Scanner)

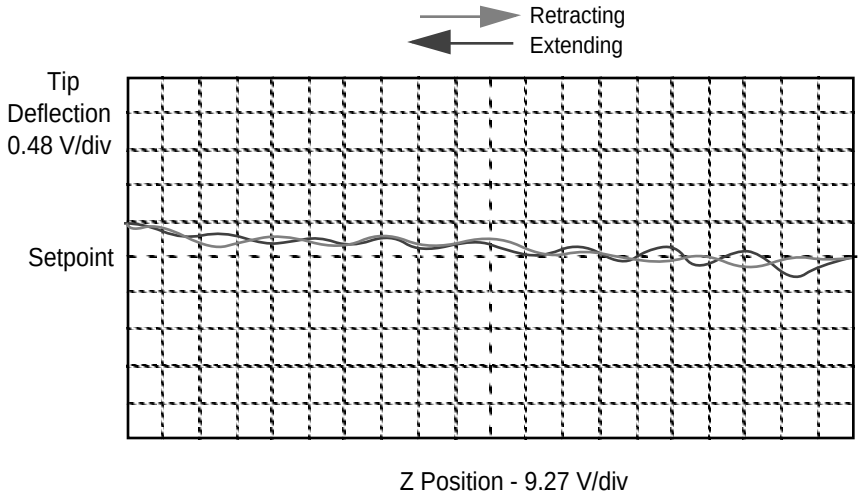
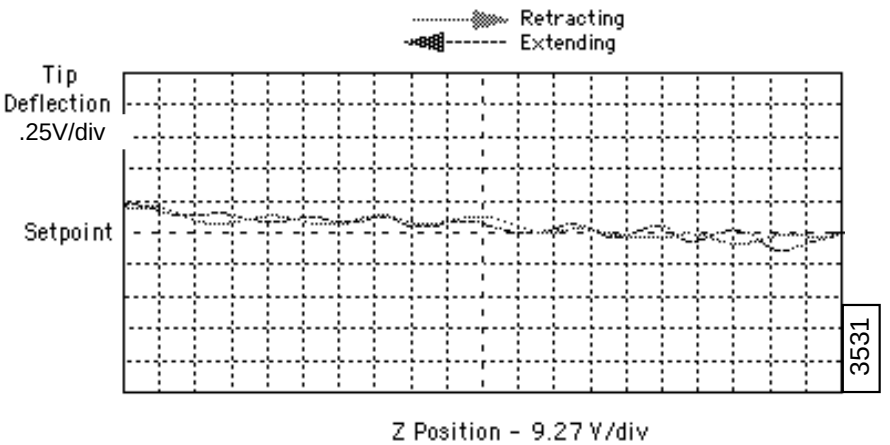


Figure 13.5f False Engagement (J Scanner)



Motor Control

Motor Control / Tip Up and **Tip Down** buttons provide coarse adjustment of **Z center** voltage. With these buttons the SPM head moves vertically. The Z stage stepper motor causes the Z piezo to adjust according to the 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, rotate the leadscrew several turns by clicking on the **Tip Up** or **Tip Down** buttons before obtaining movement.

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. Moving the photodiode down by rotating the photodiode adjustment knobs counter-clockwise shifts the curve down, just as decreasing the **Setpoint** parameter shifts the curve down. Conversely, rotating the photodiode adjustment knobs clockwise moves the curve up by moving the photodiode up.

Force Calibration (Contact Mode AFM) continued...

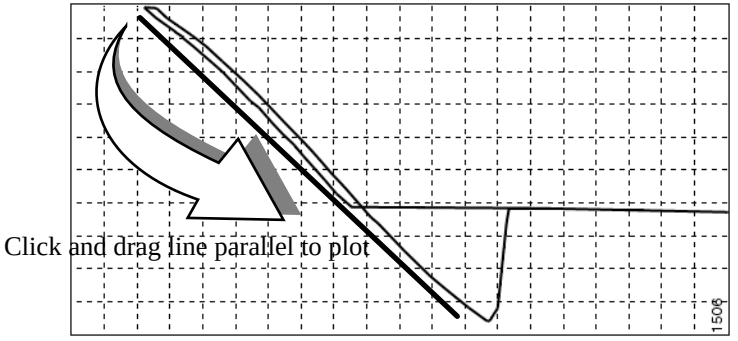
13.5.3 Advanced Techniques

Sensitivity Determination

Sensitivity represents the cantilever deflection signal versus voltage applied to the piezo and is normally set from the **Force Calibration** mode. You must calibrate the sensitivity before you can generate accurate deflection data. Sensitivity is equal to the slope of the force curve while the cantilever is in contact with the sample surface. Complete the following steps to calculate the sensitivity:

1. Obtain a good force curve on the display monitor.
2. Position the cursor on one end of the contact portion of the curve.
3. Click on the left mouse button to fix the line segment.
4. Drag the mouse to position the “rubber band line” parallel to the contact portion of the force curve Figure 13.5g.

Figure 13.5g Set the Sensitivity Parameter



5. Clicking on the mouse a second time tells the system to calculate the slope of the line segment and enter the calculated value as the **Sensitivity** in the menu.
6. Clicking on the right mouse button removes the line segment from the screen.

Sensitivity Determination continued...

Sensitivity can be expressed in terms of the photodiode voltage versus the distance traveled by the piezo, or the voltage applied to the piezo, depending on the setting of the **Units** parameter. If you calibrate **Sensitivity** on a material much stiffer than the cantilever, it measures the value of the AFM optical lever sensitivity (i.e., how many volts of deflection signal are produced by a given deflection of the cantilever tip). The sensitivity changes for different cantilever lengths and styles (shorter cantilevers give higher sensitivities). Sensitivity also changes with the position of the laser on the cantilever and the quality of the laser beam reflection from the cantilever.

ATTENTION: Calibrate the **Sensitivity** parameter on a hard substrate before using the force curve's vertical scale for quantitative measurements. Use a relatively hard sample for the sensitivity measurement.

Force Minimization

Force Calibration 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 nominal deflection of the cantilever and, therefore, the nominal 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. The system cannot engage to a negative deflection operating point, because the engagement process requires a setpoint which is greater than the voltage at zero deflection. However, you can change the operating point after engaging the cantilever.

In **Force Calibration**, you can adjust 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}. The contact force is at its minimum when V_{CSmin} is on the centerline of the deflection-signal axis.

Force Minimization continued...

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 Controls** panel changes the setting of the **Setpoint** parameter in its **Real-time** counterpart when you exit the **Force Calibration** routine. 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 top/bottom differential signal on the display 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.

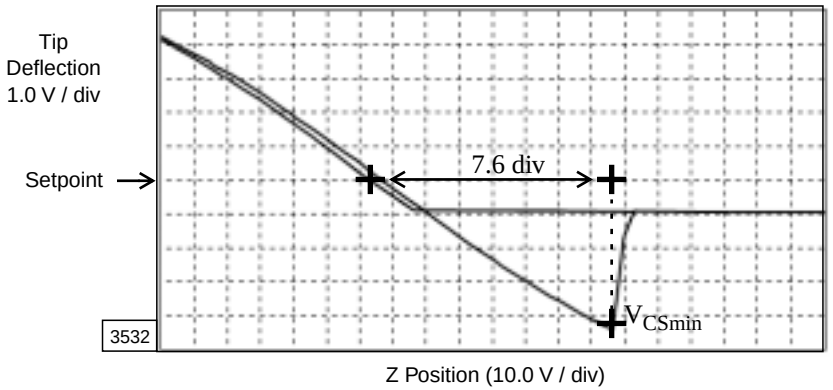
Contact Force

The force curve illustrates 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, you can use the force curve to calculate the nominal 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:

$$F = k(\Delta Z)$$

where ΔZ is the Z distance from the control point to V_{CSmin} in nanometers. An example of how to compute the contact force from the **Force Calibration** graph is shown in Figure 13.5h.

Figure 13.5h Computing Contact Force



Recalling that contact force F is equal to $k(\Delta Z)$, you can calculate the contact force from the sample plot above. For example, assume that the spring constant of the cantilever is $k = 0.6\ N/m$ and $Z\ \text{piezo sensitivity} = 12\ nm/V$. You may measure the plot above at the points where the retract portion of the curve intersects the setpoint to tip pull-off (rebound). Multiply the distance times the Z piezo sensitivity to obtain ΔZ. In this example:

$$\Delta Z = 7.6\ \text{div} \times 10.0\ V/\text{div} \times 12\ nm/V = 912\ nm$$

Therefore, the contact force calculates:

$$F = 0.6\ N/m \times 912\ nm$$
$$= 547.2\ nN$$

When you set the **Data type** to **Height** with the feedback gains set high, the tip tracks the sample surface with nearly constant deflection on the cantilever. When the cantilever deflection is constant, the force is constant and the force calculation determines the force between the tip and the surface over the entire scan area.

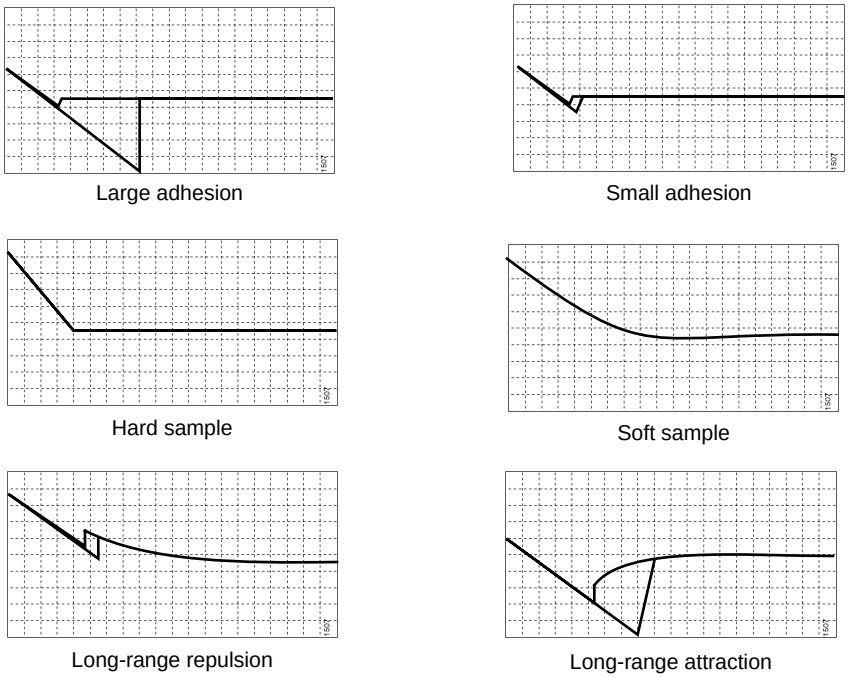
Contact Force continued...

Force calculations are not as straightforward on images captured with the **Data type** set to **Deflection**. When collecting deflection data, the feedback gains are set low so the sample stays at a constant position relative to the cantilever holder. In this case, the cantilever deflection (and therefore the force applied to the sample) varies as the tip encounters features on the surface. You can calculate the nominal force applied to the surface from the force equation. You can calculate the force applied at other points on the sample relative to the nominal force by using deflection data and the spring-constant of the cantilever. The **Sensitivity** parameter must be accurate—that is, previously determined and entered—before you can generate accurate deflection data.

Interpreting Force Curves

An examination of force curves is useful for determining adhesion and hardness characteristics of samples. The examples in Figure 13.5i represent some of the general variations in force curves.

Figure 13.5i Force Curve Examples



13.6 Force Calibration (Tapping Mode)

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 Calibration allows the imaging of forces between the tip and surface, including chemical bonds, electrostatic forces, surface tension and magnetic forces. In Tapping Mode, you can observe forces by measuring changes in tip RMS amplitude, phase, or deflection. The user may represent force plots in one of two forms: **Force Calibration** 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.

13.6.1 Force Plots

When performing **Force Calibration** in Tapping Mode, 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. However, in Tapping Mode, the force plot is a graph of the piezo's extension versus oscillating tip amplitude, phase or deflection.

Uses of **Force Calibration** in Tapping Mode include characterizing forces on the cantilever tip, diagnosing SPM performance, and calibrating the RMS amplitude, deflection or phase of the cantilever as a function of tip-sample distance. 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 228-13.

Force Plots continued...

Figure 13.6a Piezo Extension Versus RMS Amplitude and Deflection

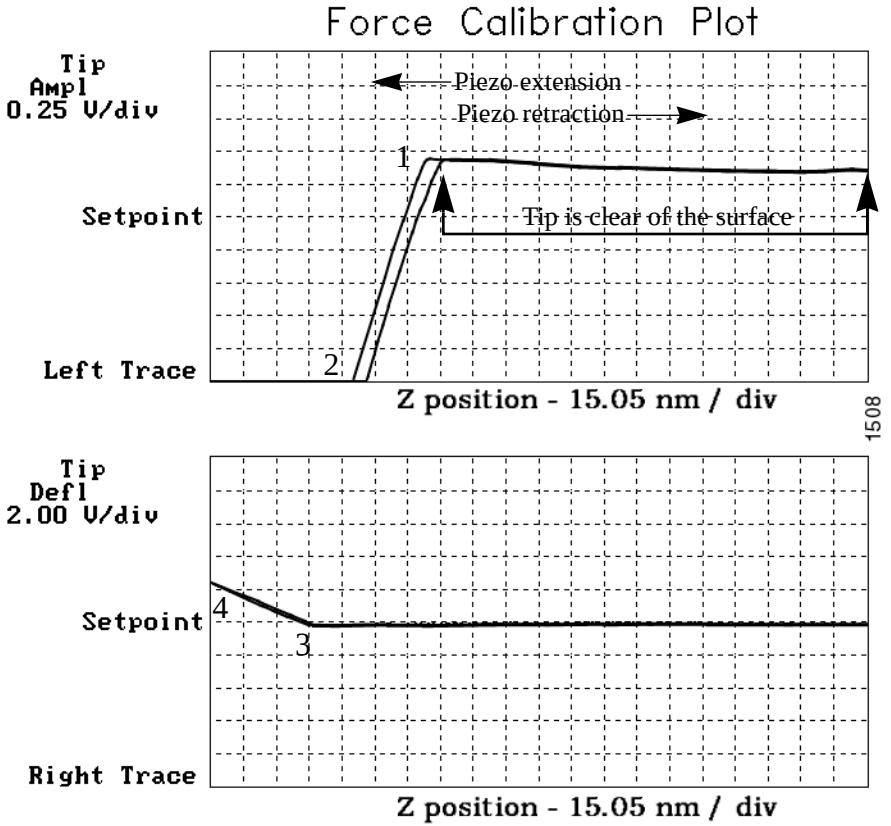


Figure 13.6a illustrates a two-channel Tapping Mode force plot. The vertical axes of the graphs represent the RMS amplitude (top) and deflection signal (bottom) of the cantilever. The position of the Z piezo plots along the horizontal axis. **Channel 1** (top) demonstrates how the cantilever RMS amplitude decreases as the tip moves closer to the sample. The plot represents the RMS amplitude for one complete extension-retraction cycle of the piezo. The **scan rate** parameter in the **Main Controls** 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 recedes, dropping off as the tip moves closer to the sample surface. Between points 1 and 2, voltage to the Z piezo increases, bringing the tip about 30 nm closer to the surface. Over the same interval, the cantilever amplitude diminishes about 1.75 volts due to dampening effects.

Force Plots continued...

Dividing the change in amplitude by the change in Z piezo position gives the responsiveness of the tip-sample interaction, displayed as a **Sensitivity** value in the **Main Controls** menu. You can determine this value by using the mouse to draw a line parallel to the plot's slope in the region between points 1 and 2 where the tip amplitude dampens. Tip dampening occurs as a result of mechanical-acoustic coupling between the tip and sample. As the tip descends closer and closer to the sample, oscillation eventually ceases and the amplitude drops to zero.

For Tapping Mode in air, each nanometer decrease in the cantilever position decreases the oscillation amplitude of the cantilever by two nanometers. Once the tip encounters the sample surface, the oscillation amplitude of the cantilever decreases as the probe lowers. When the piezo turns around and begins to retract the tip, the oscillation amplitude of the cantilever increases until the tip is free of the surface, leveling off at the free-air amplitude.

Channel 2 (bottom) in Figure 13.6a plots average cantilever deflection versus piezo extension. The deflection signal is low-pass filtered to eliminate the high-frequency Tapping Mode 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 moves so close to the sample that all oscillation ceases. Pressing the tip still further causes the average deflection to increase, applying a constant force to the sample.

At point 3 in Figure 13.6a, the cantilever begins to deflect. The region between points 3 and 4 may be hazardous to the tip, because the tip is pressed tightly against the sample surface. Most single crystal silicon Tapping Mode tips fail in this region, depending upon the hardness of the sample.

Obtaining a Force Plot (Tapping Mode) continued...

13.6.2 Obtaining a Force Plot (Tapping Mode)

CAUTION: Use **Force Calibration** 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 Tapping Mode, set up scan parameters so that the reduction of amplitude is minimal (approximately 25 percent of the free air amplitude). If the amplitude reduces to zero, the tip and sample may sustain damage.

To generate a Tapping Mode force plot of a silicon calibration reference, complete the following steps:

1. Verify the cantilever holder is loaded with a Tapping Mode tip.
2. Mount the calibration reference on the SPM stage.
3. Set the **AFM mode** parameter to **Tapping** and obtain a TappingMode image.

   Note: You are now in **Image mode**.

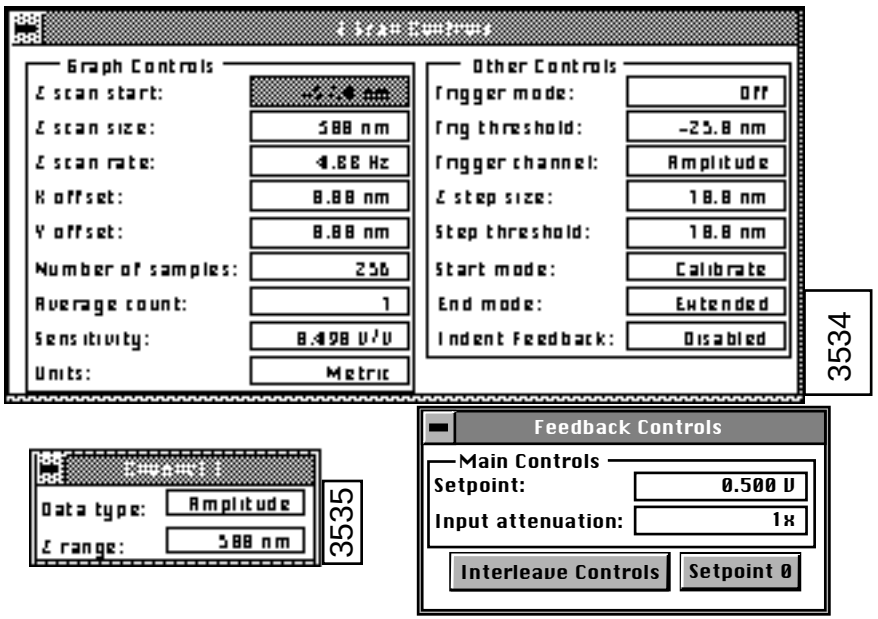
4. To switch to **Force Mode**, click on the **Real-time / View / Force Mode / Calibration** option. At least three panels should be visible:
   - **Z Scan Controls**
   - **Feedback Controls**
   - **Channel Panel**

Note: Collectively, these panels control tip-sample interactions. If any panels do not appear, pull down the **Panels** menu to select them. The top menu bar offers a number of tip approach options detailed in the *Command Reference Manual*. These buttons are not generally used for Tapping Mode and may be ignored.

5. Set the **Z Scan Controls**, **Other Controls** and **Channel** panel parameters to the settings shown in Figure 13.6b.

Note: The **Sensitivity** value shown in Figure 13.6b may differ from yours.

Figure 13.6b Tapping Mode Force Plot Parameter Settings



Obtaining a Force Plot (Tapping Mode) continued...

6. Set the **Data type** parameter to **Amplitude** under the **Channel 1** panel.

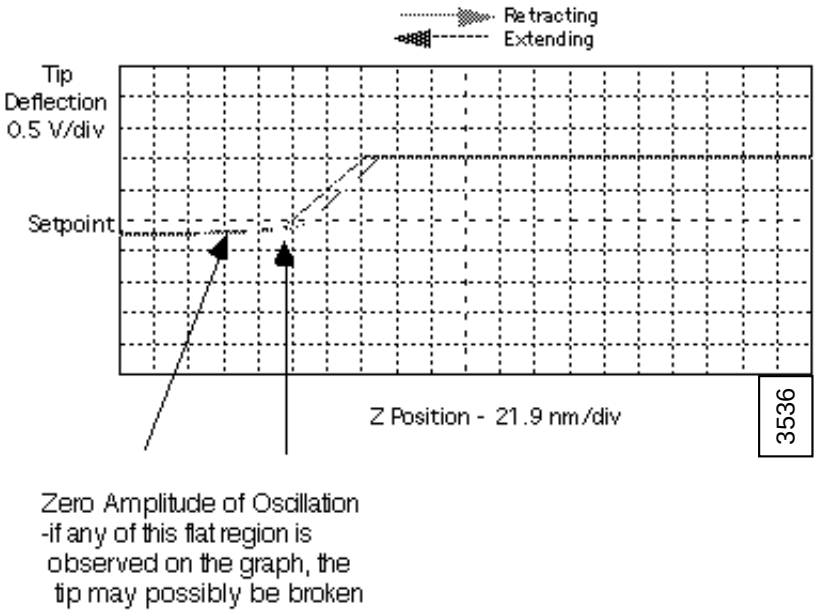
7. Adjust the **Z scan start** parameter to obtain a satisfactory force plot using the left-right arrow keys.

13.6.3 High Contact Force

Figure 13.6c shows a curve produced when the tip pushes too far into the sample. This is a very dangerous condition because the cantilevers used in Tapping Mode are stiff and brittle. Tips break when driven into the sample surface until oscillation amplitude changes to zero.

The flat portion on the left side of the amplitude curve inFigure 13.6c 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 Tapping Mode cantilevers. The cantilever cracks or the tip shatters if deflection continues.

Figure 13.6c Amplitude Force Plot with High Contact Force



You can avoid this situation by using triggers (See Section 13.4.5) or by reducing the value of **Z scan start** until there is no flat portion on the left 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.

13.6.4 Plotting Phase Versus Frequency in Tapping Mode

This is available only for SPMs with an Extender™ Electronics Module. Just as imaging magnetic-electric forces in phase gives better resolution than amplitude, plotting force in terms of phase versus frequency allows improved observation of long-range attractive and repulsive forces between the tip and sample. Precise interpretation of force plots using phase versus frequency remains under investigation. Contact Digital Instruments Veeco for more information.

Tip Selection

You may use virtually any Tapping Mode tip to obtain Tapping Mode force plots; however, the ultimate choice depends upon the delicacy of the sample and the magnitude of the forces to be gauged. Longer tips have lower spring constants (i.e., they are more pliant) and therefore offer greater sensitivity for most samples. Shorter tips afford better control when gauging strong attractive forces and are less prone to entrapment by surface tension forces. Experiment to determine the tip that best meets your needs.

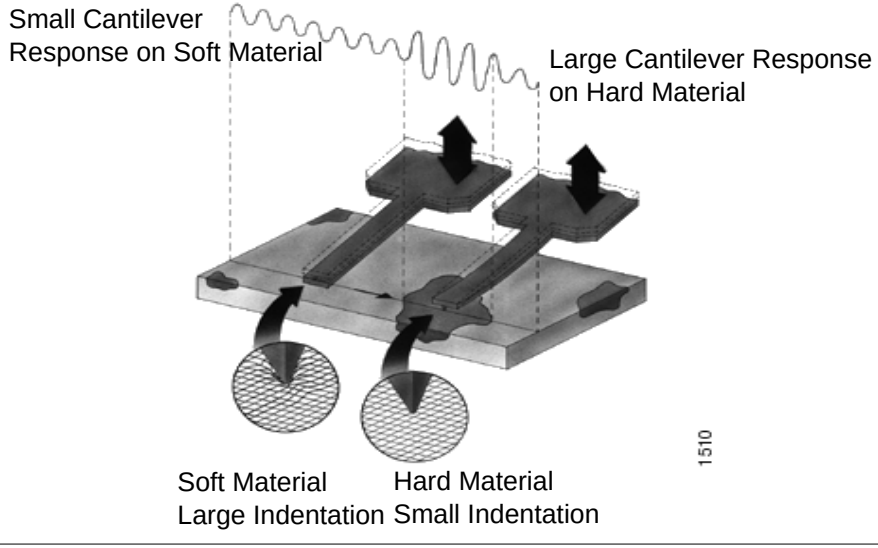
13.7 Force Modulation

13.7.1 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 8). It is useful, but not essential, to have experience operating in Tapping Mode (See Chapter 10).

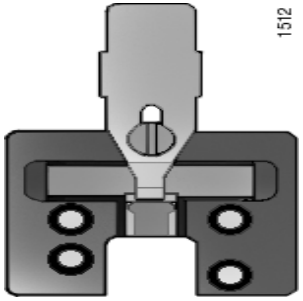
Force modulation measures local sample elasticity by oscillating a cantilever such that the tip indents slightly into a sample. The tip indents soft materials more easily than harder materials. The amount of cantilever deflection is inversely related to the amount of indentation. For example, a soft 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 13.7a. The cantilever scanning the surface from left-to-right generates contrast. When the cantilever encounters a hard site (dark area) embedded in a softer medium, the harder material absorbs less of the cantilever's energy causing an increase in the cantilever's response and signal amplitude.

Figure 13.7a Contrast Generation in Force Modulation Mode



Force modulation requires the use of a special optional cantilever holder, shown in Figure 13.7a. 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 cantilever holder; however, it does not have a piezo stack used for Tapping Mode operation.

Figure 13.7a Force Modulation Cantilever Holder



13.7.2 Selecting a Force Modulation Tip

The key consideration when selecting a force modulation cantilever is its spring constant. Ideally, the cantilever must have a spring constant which compliments 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 (elasticity) 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 Digital Instruments Veeco recommends using 225 µm long force modulation (Model # FESP) silicon cantilevers. For more delicate, biological samples, use 450 µm long silicon cantilevers or silicon nitride cantilevers. For extremely hard materials, use stiffer (“harder”) tips like the 225 µm single crystal silicon Tapping Mode tips.

Selecting a Force Modulation Tip continued...

Digital Instruments Veeco offers cantilevers with a wide range of spring constants (See Table 13.7a). Choosing a tip depends upon how hard the sample is. For samples of known hardness, try using a stiffer cantilever then working toward softer cantilevers. For samples of unknown hardness, start with a force modulation cantilever (Model FESP) and determine whether the tip is sufficiently stiff then adjust accordingly.

Table 13.7a: Force Modulation Tips

Cantilever	Model No.	Cantilever Length	Spring Constant
Standard Silicon Nitride	NP, DNP	100—200 µm	0.01—0.6 N/m
Oxide-sharpened Silicon Nitride	NP-S, DNP-S	100—200 µm	0.01—0.6 N/m
Contact AFM Etched Silicon	ESP	450 µm	0.02—0.1 N/m
Force Modulation Etched Silicon	FESP	225 µm	1—5 N/m
TappingMode Etched Silicon	LTESP	225 µm	20—70 N/m
TappingMode Etched Silicon	TESP	125 µm	20—100 N/m

SOFTER

HARDER

13.7.3 Operating Principle

Force modulation mode is very similar to Contact Mode AFM. The NanoScope system scans the cantilever over the sample surface while trying to keep the cantilever deflection constant. The deflection setpoint determines the average deflection during operation. In addition, the cantilever oscillates up and down by a piezoelectric bimorph in the tipholder 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, indicating 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 creates a small 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 creates a large amplitude which displays as a dark area on the image.

13.7.4 Force Modulation Procedure

This section gives instructions for operating in **Force Modulation** mode.

1. Choose the **Force Modulation** profile under **Microscope/Profile**.

2. Verify that the **Microscope** mode parameter in the **Other Controls** panel is set to **Tapping** and the **SPM Feedback** in the **Feedback Controls** panel is set to **TM Deflect**.

3. Load the special force modulation cantilever holder with a cantilever. The procedure for loading a cantilever is exactly the same as for operation with the standard air cantilever holder.

   Note: Refer to the section on Tip Selection earlier in this chapter to help you choose an appropriate cantilever.

4. Install the cantilever holder on the Dimension SPM head.

5. Align the laser on the cantilever.

   Note: Methods for aligning the laser are discussed in Chapter 7.

6. Adjust the **Setpoint**. Begin with a **Setpoint** of **0 V**. Recall that higher setpoints cause the cantilever to push harder on the surface during scanning.

7. Turn the photodetector adjustment knobs to center the laser spot on the laser detector. Adjust the **Vertical Deflection** to approximately **-0.5 V** for FESP cantilevers.

   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**.

8. Select **View / Sweep / Cantilever Tune** or click on the Cantilever Tune icon.

Force Modulation Procedure continued...

Cantilever Oscillation

The cantilever oscillates 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.

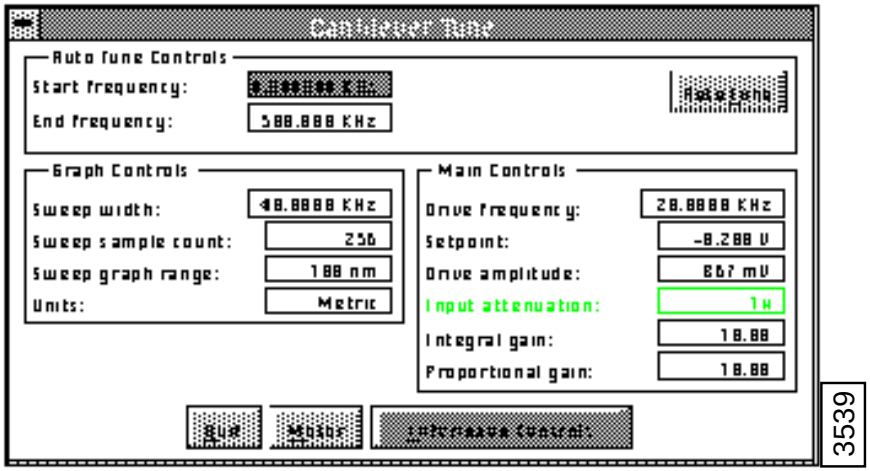
1. Find the bimorph resonance manually using the **Sweep Controls** panel.

   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.

2. The display monitor plots the **Frequency Sweep**, showing cantilever oscillation amplitude versus frequency. To set the parameters controlling the **Frequency Sweep** plot, use the parameters in the **Sweep Controls** panel and the commands on the top menu bar of the display monitor.

Force Modulation Procedure continued...

Figure 13.7a Auto Tune Controls Panel



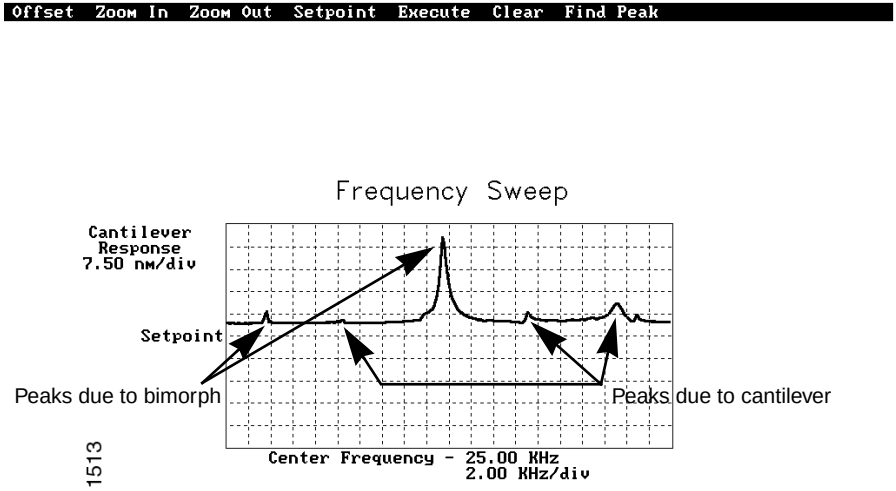
3. Set the **Drive frequency** to **15 kHz** and the **Sweep width** to **30 kHz**.

4. Set the **Drive amplitude** at **5 V** to start. You can readjust this value later.

> **Note:** A series of peaks display on the **Frequency Sweep** plot. A typical **Frequency Sweep** plot is shown in Figure 13.7b.

Force Modulation Procedure continued...

Figure 13.7b Typical Frequency Sweep Plot



Note: The large drive amplitude is necessary because peaks are smaller than normally seen during Tapping Mode operation. This is due to the cantilever 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 **8-20 kHz**.

6. Choose a peak, then click on the **Zoom In** command (top menu bar of display monitor) with the left mouse button. Two vertical lines appear on the **Frequency Sweep** plot.

7. Use the mouse to move the vertical lines until the selected peak is centered between the lines.

8. Increase or decrease the zoom range by clicking the left mouse button.

Force Modulation Procedure continued...

9. When the peak centers and the zoom width is adjusted, click the right mouse button twice to automatically adjust the **Drive Frequency** and **Sweep width,** and zoom in on the selected peak.

10. Use the **Offset** command on the display monitor to center the peak in the graph. Click on the **Offset** command with the left mouse button; a vertical, green line appears on the plot.

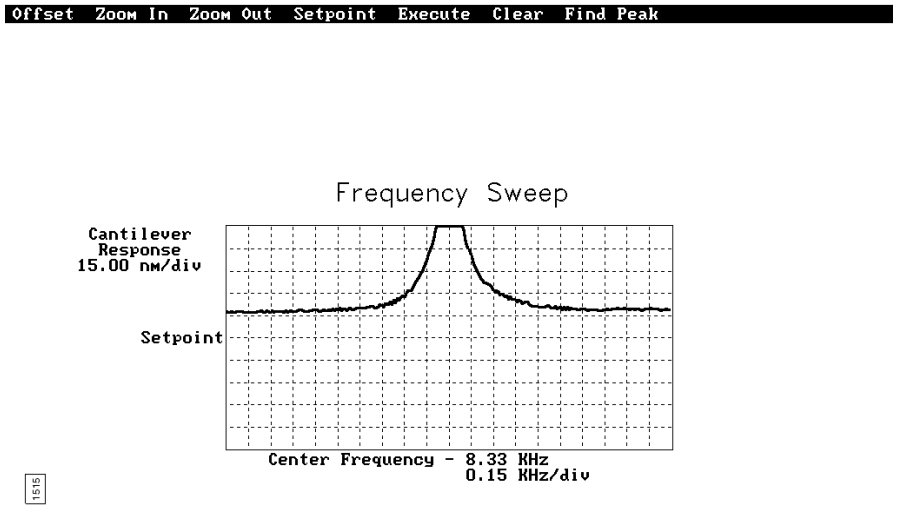
11. Move the line until centered on the desired peak (usually the frequency with the highest amplitude), then click the left button to lock it.

12. Click on **Execute**. The computer automatically changes the **Drive Frequency** so that the peak centers. You may need to click on this command more than once to properly center the peak.

    Note: Once you choose the desired frequency, the **Frequency Sweep** plot should be recentered similarly to the plot shown in Figure 228-23.

Force Modulation Procedure continued...

Figure 13.7c Correctly Tuned Force Modulation Frequency



Note: You may also change the **Drive Frequency** by clicking on the **Drive Frequency** parameter on the control monitor's **Feedback Controls** panel, and entering a new value.

ATTENTION: 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.

11. Select **Stage/Locate Tip** or click on the **Locate Tip** icon.

12. Select **Stage/Focus Surface** or click on the **Focus Surface** icon.

13. Check the scan parameters **Scan speed**, **Scan size** and **Integral gain** to verify they are reasonable for Contact Mode AFM imaging. Also, it may be desirable to readjust the detector mirror adjustment screws if the **Vertical Difference** signal has drifted from the value originally set.

Force Modulation Procedure continued...

14. For the **Channel 1** image, select **Data Type: Height**. Select **Data Type: Amplitude** for the **Channel 2** image. The multi-image capability views both the topography data and the force modulation (elasticity) data at the same time.

    Note: The **Data Type** chosen for the **Channel 1** image displays when using the **Browse** command. If desired, the above choice can be reversed so that the surface topography (**Height**) is chosen for **Channel 2**.

15. Reduce the **Drive Amplitude** to **0** before engaging.

16. Initiate the **Engage** command.

17. Select the **Motor / Engage** command.

    Note: The motor moves the SPM head down towards the sample and stops once the cantilever deflects to the chosen **Setpoint**. Under some conditions the system may “false engage” before the cantilever actually reaches the surface. Increase the **Setpoint** and engage again.

18. 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 **10-20** 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 **-220 V**.

19. Record the Setpoint value where the cantilever pulls off the surface (the "pull-off value").

Force Modulation Procedure continued...

20. Increase the **Setpoint** until the cantilever touches the surface, and an image appears.

    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.

21. 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. As the **Drive amplitude** increases, contrast remains unchanged 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

a. Start with a small **Drive amplitude** value of **100 mV**.

b. Increase the **Drive amplitude** with the right arrow keys or type in new values. The contrast of the **Amplitude** (force modulation) image increases.

c. 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**.

22. Readjust gains (if necessary).

23. 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.

> **Note:** If the gains are set too high, parallel diagonal lines display on the **Height** image. If an oscillation is seen, the feedback system is attempting to cancel the cantilever oscillation by moving the Z-axis piezo in the opposite direction. If there is evidence of an oscillation in the **Height** data, reduce the gains until the oscillation stops. You may view oscillations more easily in **Scope Mode**.

Force Modulation Procedure continued...

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 Section 13.7.5.

13.7.5 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 (1-3 Hz) 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 **Scan 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.

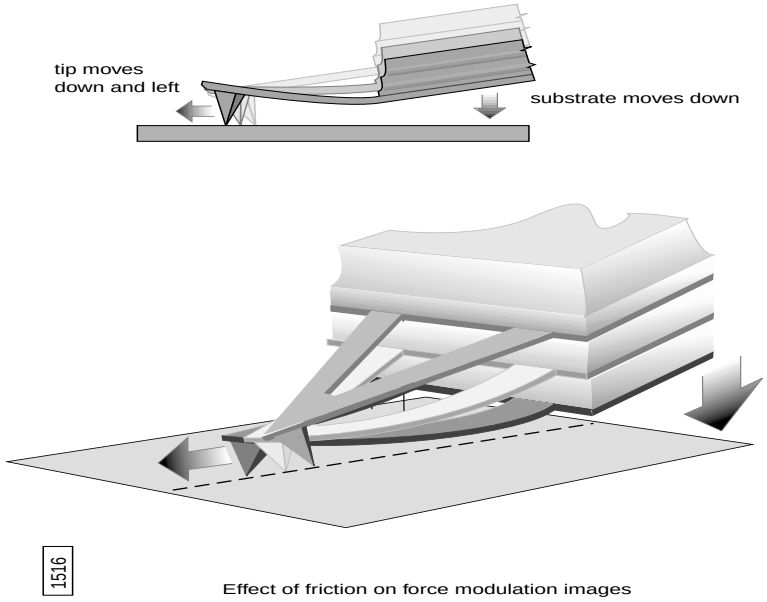
Notes About Artifacts continued...

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 13.7d). 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**.

Notes About Artifacts continued...

Figure 13.7d 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 13.8.4).

13.8 Force Modulation with ‘Negative LiftMode’

A new form of force modulation imaging utilizing Tapping Mode 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 (< 500 nm vertical features); however, Digital Instruments Veeco encourages experimenting with this technique on rougher surfaces as well. A general procedure for Force Modulation with "negative LiftMode" is described in Section 13.8.4.

> **Note:** Force modulation contrast is very sensitive to the spring constant of the tip, which varies according to the length and thickness of the cantilever. You may perform Tapping Mode easily with either force modulation or Tapping Mode cantilevers, but you may have difficulty with Contact Mode AFM silicon and silicon nitride cantilevers. (See Table 13.7a).

13.8.1 Find Resonance of the Bimorph

The force modulation cantilever holder features a bimorph, which excites the probe and cantilever mount.

1. Verify the probe is withdrawn from the sample surface.
2. Switch modes under **Microscope/Profile** to **Tapping Mode**.
3. Set the **Drive amplitude** on the **Interleave Controls** panel to **200-400 mV**.
4. Select the **Real-time / View / Sweep / Cantilever Tune** dialog box.

   > **Note:** For version 4.10 or later software, click on the **Manual** button within this box.

5. Click on the **Interleave Controls** button to access tuning controls for interleaved scans.

Find Resonance of the Bimorph continued...

6. Set an initial **Sweep width** of **10 KHz** and a **Drive frequency** of **10 kHz**. Determine the **Drive frequency**.

7. Verify that the **Drive frequency** parameter in the **Interleave Controls** panels is set to the bimorph's resonant frequency.

8. Click on the enable button next to the **Drive frequency** parameter; the enable button appears green.

9. Quit the **Cantilever Tune** dialog box.

13.8.2 Set Interleave Controls

1. Transfer to the **Real-time / Interleave Controls** panel.

2. Set **Lift start height** and **Lift scan height** to **0.00 nm**.

3. For now, set the **Interleave mode** parameter to **Disabled**.

13.8.3 Obtain a Tapping Mode Image

While negative LiftMode force modulation data is imaged using **Channel 2**, height data is obtained using Tapping Mode on **Channel 1**. You must obtain a satisfactory Tapping Mode image to generate good data.

1. Verify that the **Interleave mode** parameter on the **Interleave Controls** panel is **Disabled**.

2. Verify that the **AFM mode** parameter in the **Other Controls** panel is set to **Tapping**.

3. Select **Real Time / View / Sweep / Cantilever Tune**. On version 4.XX software, you may tune the cantilever using **Auto Tune**. Enter a main controls **Start frequency** value of **5 KHz** and **End frequency** value of **400 KHz**.

4. Set **Target Amplitude** to **1-2V**, and click on the **AUTO TUNE** button.

5. Exit the **Cantilever Tune** dialog box.

Obtain A Tapping Mode Image continued...

6. Verify that all **Scan controls** and **Feedback Controls** parameters are set for obtaining a Tapping Mode image.

7. Set the **Channel 1** panel **Data type** parameter to **Height**.

8. Set the **Line direction** to **Retrace** and enter an appropriate **Data Scale** value for your sample.

9. Engage the tip on the surface and obtain a good Tapping Mode image.

13.8.4 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 Controls** 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 Controls** panel from zero until you reach the surface. In most cases, you should not go below **-60.0 nm**.

> **Note:** This is sometimes best performed while in **Real-time / View / Scope Mode**.When the interleaved scan is enabled, the tip's Tapping Mode 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.

Obtain a Negative LiftMode Force Modulation Image continued...

4. Adjust the interleaved **Drive amplitude, Interleave Controls** 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**. Adjust gains slightly lower than when performing normal Tapping Mode imagery. Verify by setting a **Lift scan height** of **100 nm**, 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 **0.0 nm** and approach the surface.

13.9 Force Volume

Force volume imaging with the atomic force microscope (AFM), available with NanoScope III software versions 4.22 and higher, 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, contact Digital Instruments Veeco for a copy of *Support Note 240A, Force Volume*.

Chapter 14 *Interleave Scanning*

14.1 Overview

Interleave Scanning is an advanced feature of the Nanoscope III software which allows simultaneous acquisition of two data types. To achieve this, Interleave Scanning 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 produces an image concurrently with the main scan.

This chapter provides general information regarding the Interleave procedure and commands, with emphasis on **Lift Mode**. Specifically, this chapter details the following topics:

14.2 Interleave Scanning

Typical applications of Interleave Scanning include Magnetic Force Microscopy (MFM) and Electric Force Microscopy (EFM) measurements. During the interleave scan in **Lift Mode**™, feedback is disabled and the tip lifts to a user-selected height above the surface to perform far field measurements such as MFM and EFM. Topography data recorded during the main pass keeps the tip a constant distance from the surface during the interleave trace and retrace. Magnetic and electric forces on the tip result in cantilever deflection or resonance shifts. A magnetic force or electric force image is produced by recording these shifts. **Lift Mode** was developed to isolate purely MFM and EFM data from topographic data. Chapter 15 provides detailed instructions for obtaining MFM images of a standard magnetic sample. Chapter 16 details instructions for obtaining EFM images.

The Interleave commands include a set of **Interleave Controls** which allow you to set several scan controls (**Drive amplitude**, **Setpoint**, and various **Gains**) independently of those in the main scan controls.

14.3 Basic Interleave Scanning Operation

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

14.3.1 Select the Microscope Head

1. Set the microscope head configuration by selecting the **Microscope / Select** panel from the **Di** pop-down menu.
2. Click **OK** to close the **Microscope / Select** dialog box. When enabled, the selected buttons are black.

14.3.2 Select Mode of Operation

1. Select **Microscope/Profile**.
2. Select **Tapping Mode** as the mode of operation.

14.3.3 Head, Cantilever and Sample Preparation

1. Install an etched single crystal silicon tip onto an AFM cantilever holder (See Chapter 7).
2. Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head.

14.3.4 Align Laser

1. Align the laser using the laser control knobs.
2. Verify the laser beam is positioned on the back of the cantilever, with a spot visible in the Dimension head filter screen and a sum signal of **2 volts**.

14.3.5 Adjust Photodetector

1. Adjust the photodetector so that the red dot moves toward the center of the Dimension head filter screen using the two photodetector adjustment knobs located on the side of the Dimension head.
2. Verify that the red dot is centered and elliptical in shape in the Dimension head filter screen.
3. Set the **Vertical Deflection** to **0 volts**.

14.3.6 Locate Tip

1. Select **Stage/Locate Tip** or click on the **Locate Tip** icon.
2. Center the tip end of the cantilever under the crosshairs using the two optics adjustment knobs located left of the optical microscope objective.
3. Use the trackball with the bottom left button depressed to focus on the tip end of the cantilever.

14.3.7 Focus Surface

1. Select **Stage/Focus Surface** or click on the **Focus Surface** icon.
2. Focus on the sample surface using the trackball with the bottom left button depressed.

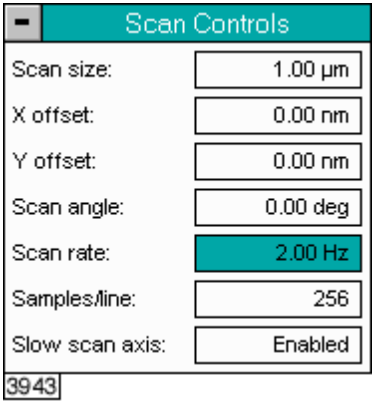
14.3.8 Set Initial Scan Parameters

Scan Controls Panel

In the **Scan Controls** panel, set the following initial scan parameters (See Figure 14.3a).

1. Set the **Scan Rate** to **2 Hz**.
2. Set the **Scan Size** to **1µm**.
3. Set the **Scan Angle** to **0**.
4. Set **X** and **Y Offsets** to **0**.

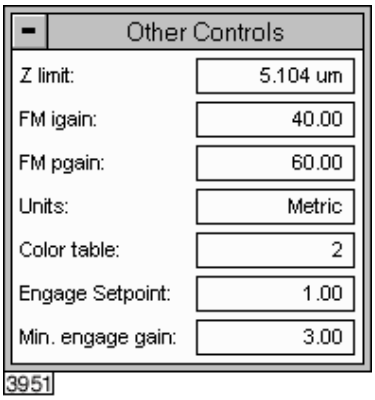
Figure 14.3a Suggested Scan Controls Settings



Set Initial Scan Parameters continued...

Other Controls Panel

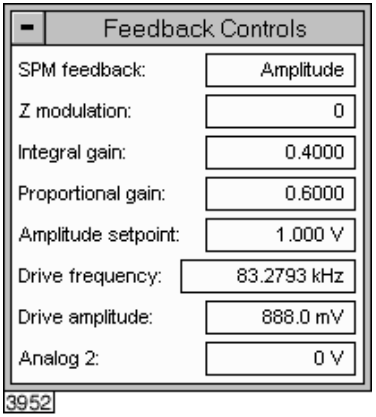
Figure 14.3b Suggested Other Controls Settings



Feedback Controls Panel

1. Set the **Integral gain** to **0.5** and the **Proportional gain** to **0.7** (See Figure 14.3c).
2. Set the **Look Ahead gain** to zero.

Figure 14.3c Suggested Feedback Controls Settings



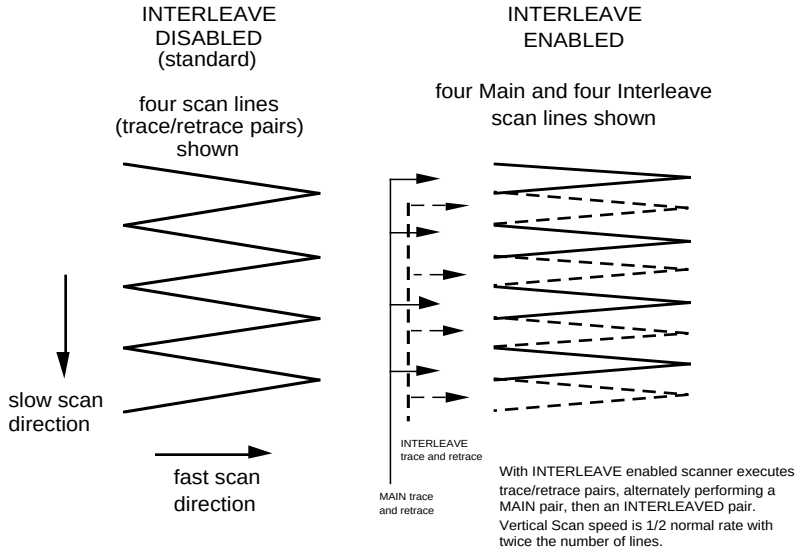
14.3.9 Engage

1. Select **Motor**/**Engage**. A pre-engage check begins, followed by Z-stage motor motion.
2. To move to another area of the sample, execute a **Withdraw** command to avoid damaging the tip and scanner.
3. Move the stage using the trackball to the next area of interest on the sample.
4. Select **Motor**/**Engage.**

> **Note:** After the tip engages, adjust the control panel values to provide the desired scan parameters.

In **Interleave mode,** the scan pattern of the tip relative to the imaged area changes. 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 in Figure 14.3d. This is the standard scan pattern of the NanoScope III.

Figure 14.3d Figure 12.1. X-Y Scan Pattern



Engage continued...

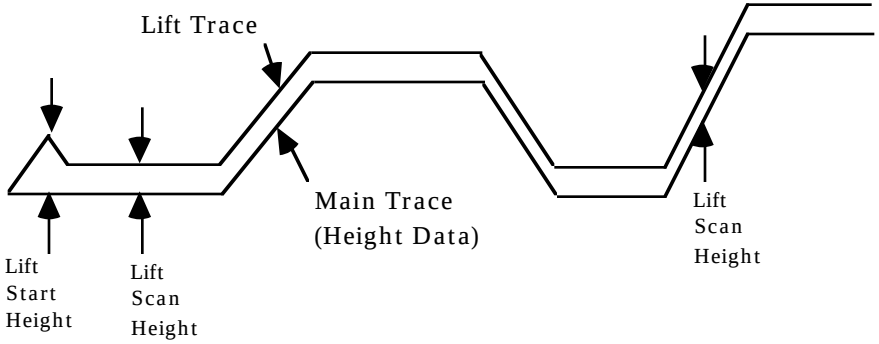
In **Interleave** mode, the system first performs a standard trace and retrace with the **Main Feedback Controls** in effect. The tip moves at half the normal rate in the slow scan direction. As shown on the right in Figure 14.3d, an additional trace and retrace are then performed with the **Interleave Feedback Controls** enabled. The frame rate halves because twice as many scan lines are performed for the same scan rate.

Four modes are possible for **Interleave scan**: **Disabled, Interleave, Lift,** and **Linear**. With **Interleave** selected, the feedback remains on during the Interleave pass with the values under **Interleave Feedback Controls** in effect. In **Lift Mode**, the feedback is instead turned off, and the tip lifts off the surface and scans at a user-selected height.

14.3.10 Lift Mode

With the **Interleave scan** option set to **Lift**, the motion of the tip during the Interleave trace and retrace is as shown in Figure 14.3e.

Figure 14.3e Lift Mode



The tip moves from the **Lift start height** to the final **Lift scan height**. The **Lift scan height** value is added point-by-point to the height data obtained during the Main topography trace and retrace. These values can be positive or negative. A large **Lift start height** pulls the tip from the surface to eliminate sticking.

14.4 Interleave Scanning/Lift Mode Operation

14.4.1 Basic Lift Mode Operation

These instructions apply to STM, Contact AFM and Tapping Mode modes. You must be familiar with using Tapping Mode or Contact AFM to obtain good images of surface topography. See Chapter 15 for specific examples using MFM.

Use of Interleave scanning requires the steps below:

1. Verify the main and interleave values are equal by clicking on the bullets to the left of the appropriate Interleave parameters to set the parameters to the **Off** (grayed condition).

   Note: When the bullets are gray, the interleave parameters default to the main feedback values. You can change these values while in the gray condition after engaging.

2. Engage and obtain a topography scan using either Contact AFM or Tapping Mode.

   Note: See Chapter 8 for Contact AFM scanning procedures and Chapter 10 for Tapping Mode scanning procedures.

ATTENTION: When using Lift Mode, adjust the gains and setpoint under **Feedback Controls** to give a true image of the surface. A poor measurement of surface height results in inaccurate measurement during the lift pass or causes the tip to strike the surface. Height data is displayed on Channel 1.

3. Adjust the **Interleave Controls** panel to desired settings. If using **Lift** mode, set the **Lift start height** and **Lift scan height**. If using other modes, set the parameters appropriate to those modes.

   Note: Certain parameter constraints are imposed: scan sizes, offsets, angles, and rates and numbers of samples per scan line are the same for the main and interleave data. The imaging context (Contact, Tapping Mode, or force modulation) must also match.

Basic Lift Mode Operation continued...

4. Choose the **Interleave mode** (**Interleave, Linear** or **Lift**) appropriate for the measurements to be performed and set the **Interleave Controls** accordingly.

   Note: When using Tapping Mode, the **Drive amplitude**, **Drive frequency**, **Gains**, and **Setpoint** can be set differently in the **Interleave Controls** panel than in the main **Feedback Controls** panel.

5. Turn on the desired parameters by clicking on the bullets to the left of the appropriate Interleave parameters to set the parameters to **On** (green condition).

6. Display the interleave data by switching **Scan line** in the **Channel 2** panel to **Interleave**.

14.5 Advanced Lift Mode Operation

14.5.1 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.

14.5.2 Tip Shape

As shown in Figure 14.3e, the tip separation in the Lift Mode 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 14.3e).

14.5.3 Scan Line Direction

The **Line direction** should be set to **Retrace** for both the main and interleaved scans. If 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**.

14.6 Lift Mode with Tapping Mode

There are additional considerations when using Lift Mode with Tapping Mode.

14.6.1 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 Lift Mode 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. However, certain parameters can be set independently for the Interleave scan.

14.6.2 Setpoint Selection

When the main and interleave **Drive amplitudes** and **Drive frequencies** are equal (the bullets under **Interleave Controls** are disabled), the cantilever oscillation amplitude increases to the free oscillation amplitude when the tip is lifted off the surface in Lift Mode. 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** is increased or the main **Drive frequency** is altered after the tip is engaged. The vibration amplitude remains at the setpoint during the main scan even if these parameters are changed. This could make the tip strike the surface in the lift scan for small **Lift scan Heights**.

14.6.3 Interleave Drive Amplitude and Frequency Selection

Interleave Drive Amplitude

The cantilever drive amplitude can be set differently in the Lift scan as compared to the main scan by toggling the flag on the left of the corresponding Interleave Control 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. You can also adjust the Interleave **Drive frequency** which may be useful if acquiring amplitude data in Lift Mode.

Interleave Drive Amplitude and Frequency Selection continued...

CAUTION: Set this parameter to a small value *before* engaging to avoid possibly of striking the surface and harming 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.

WARNHINWEIS: 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.

Amplitude Data Interpretation

When monitoring amplitude data in Lift Mode, brighter regions correspond to smaller amplitude, and darker regions to larger amplitude.

Cantilever Oscillation Amplitude

The selection of the oscillation amplitude in Lift Mode 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 measurements (MFM or EFM) decreases with distance from the surface. Typically, the resolution is limited to a value (in nm) roughly equal to the Lift scan height.

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 cantilever travel as possible should fall within the range of the force gradient.

Chapter 15 Magnetic Force Microscopy

15.1 Overview

This chapter describes how to perform Magnetic Force Microscopy (MFM) using the Interleave and LiftMode procedures discussed in Chapter 14. Please review those sections prior to attempting MFM. Best results will be obtained with the Digital Instruments Veeco Extender™ Electronics Module. This hardware unit allows phase detection and frequency modulation for optimal MFM imaging.

Specifically, this chapter discusses the following topics:

- **Overview:** Section 15.1
- **Magnetic Force Microscopy:** Section 15.2
- **Basic MFM Operation:** Section 15.3
- **Advanced MFM Operation:** Section 15.4
- **Installation of Extender Electronics Module:** Section 15.5
- **Troubleshooting:** Section 15.6

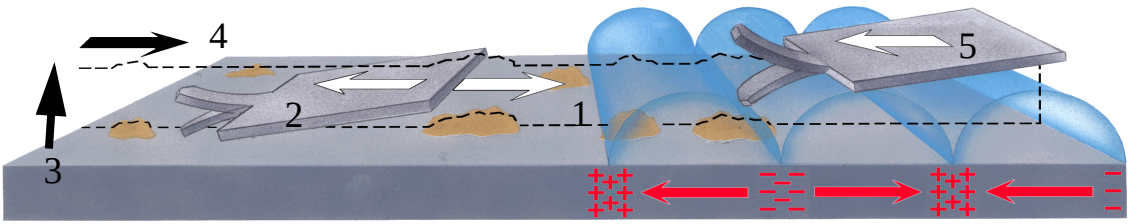
15.2 Magnetic Force Microscopy

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 15.2a, the tip then raises just above the sample surface. The surface topography is scanned and monitored for the influence of magnetic forces. MFM measures these influences using the principle of force gradient detection.

15.2.1 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 ~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 15.2a MFM LiftMode Principles



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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).

Magnetic Force Microscopy continued...

15.2.2 Amplitude Detection Techniques

All standard Dimension-series SPMs are capable of MFM imaging using amplitude detection techniques. By adding an Extender™ electronics module, the Dimension 3100 may be used for frequency modulation or phase detection, with improved results (See Figure 15.2b). Amplitude detection has largely been superseded by frequency modulation and phase detection. A more extensive discussion of force gradient detection and MFM imaging is given in the reprint *Magnetic Force Microscopy: Recent Advances and Applications*. Contact Digital Instruments Veeco to obtain a copy.

Figure 15.2b Extender™ Electronics Module

15.3 Basic MFM Operation

In the instructions below, steps specific to phase and amplitude imaging are described independently. Use the icons in the margin to locate steps specific to either frequency modulation and phase detection, or amplitude detection.

15.3.1 MFM Using LiftMode

LiftMode allows the imaging of relatively weak but long-range magnetic interactions while minimizing the influence of topography (See Figure 15.2a). 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 Tapping Mode. 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 15.3e).

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 purchase of MFM probes, and can be obtained free of charge from Digital Instruments Veeco. 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).

MFM Using LiftMode continued...

As mentioned above, the NanoScope III uses force gradient detection for MFM imaging. Within this general technique, there are three possible schemes, known as frequency modulation, phase detection, and amplitude detection. All three are discussed here. Prior to 1994, amplitude detection was the standard method for magnetic force imaging on the NanoScope III. However, hardware that allows phase detection and frequency modulation is now available for all Tapping Mode-capable microscopes in the form of Digital Instruments Veeco's Extender™ Electronics Module. (Microscopes without the Extender addition cannot utilize phase detection; for more information, contact Digital Instruments Veeco.) 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. If extensive MFM imaging is planned, the Extender Electronics module is strongly recommended.

For MFM procedures, NanoProbe™ magnetic coated tips are required. Various kinds of MFM probes are available for specific applications; contact Digital Instruments Veeco for more information. For specific information regarding Tapping Mode and Interleave Scanning please refer to Chapter 10 and Chapter 14 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.

15.3.2 Magnetic Force Microscopy Procedure

1. Magnetize a NanoProbe magnetic probe with a strong permanent magnet before installing the tip 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 due to the perpendicular component of the samples's stray field. Tip magnetizers are provided with MFM probes purchased from Digital Instruments Veeco.

2. Mount a NanoProbe magnetic probe on the scanner or tip holder.

Magnetic Force Microscopy Procedure continued...

3. Set up the AFM for Tapping Mode operation (See Chapter 10).

4. In all **Channel** panels, set the **Highpass** and **Lowpass** filters to **Off**.

5. Set the **Rounding** parameter in the **Microscope / Calibrate / Scanner** window to zero (**0**).

6. Tune the cantilever drive frequency.

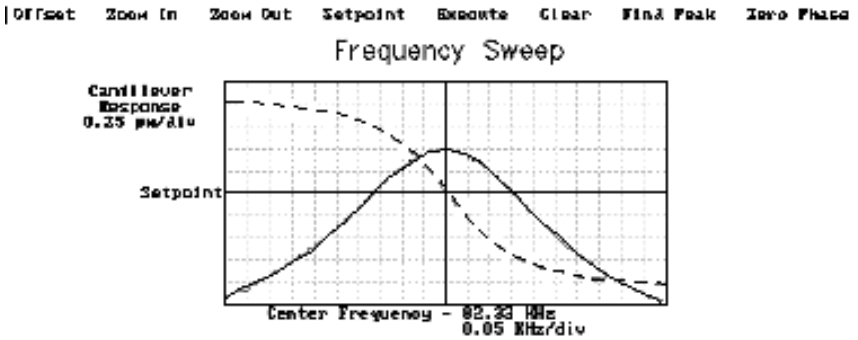
> **Note:** The procedure to tune the cantilever drive amplitude depends on whether you are using phase detection or amplitude detection. Both cases rely on automatic **Cantilever tune** just as when preparing for Tapping Mode (See Chapter 10). MFM cantilevers have resonant frequencies between 50 and 100 kHz. If using the **AutoTune** feature, these values can be used as bounds for the frequency sweep. With the Extender option, two curves appear in the **Cantilever Tune** box: the *amplitude* curve in white, and the *phase* curve in yellow (See Figure 15.3a). Microscopes without the Extender Electronics Module display only the amplitude curve.

Magnetic Force Microscopy Procedure continued...

Setting a Drive Frequency for Phase Detection

The Drive frequency should be set to the center of the cantilever resonance, as shown in Figure 15.3a. This occurs automatically if using **AutoTune.**

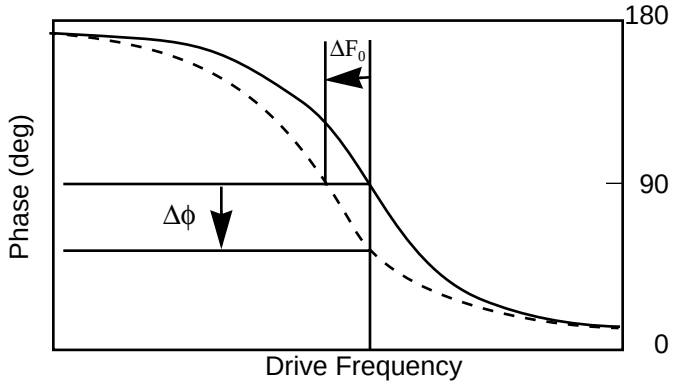
Figure 15.3a Cantilever Tune for Phase Detection and Frequency Modulation



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 menu bar above the **Cantilever Tune** frequency sweep window. The phase curve should appear as in Figure 15.3a, 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 15.3b**).**

Note: The Extender electronics give a measure of the phase lag of the cantilever oscillation relative to the piezo drive. This measurement is monotonic versus frequency as is the true phase lag in degrees. The Extender measurement has slightly different nonlinear characteristics *vs.* frequency. The measurement technique allows optimal signal-to-noise ratios; however, absolute values of phase data should be taken as approximate. Users requiring quantitative measures of force gradient are advised to use frequency modulation (See section on Frequency Modulation later in the chapter).

Figure 15.3b 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 under the **CANTILEVER TUNE** pop-down menu to manually move the drive frequency to the side of the resonance (See Figure 15.3b).

3. Set the **Drive Frequency** to the steepest part of the resonance curve for maximum sensitivity.

Setting a Drive Frequency for Amplitude Detection continued...

Note: As the tip oscillates above the sample, a gradient in the magnetic force will shift the resonance frequency f_0 (See Figure 15.3c).

Tracking the variations in oscillation amplitude while in LiftMode yields an image of the magnetic force gradients.

Figure 15.3c Cantilever Tune for Amplitude Detection

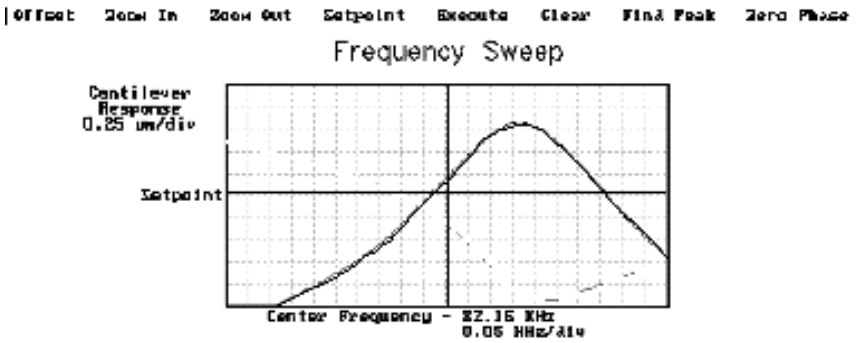
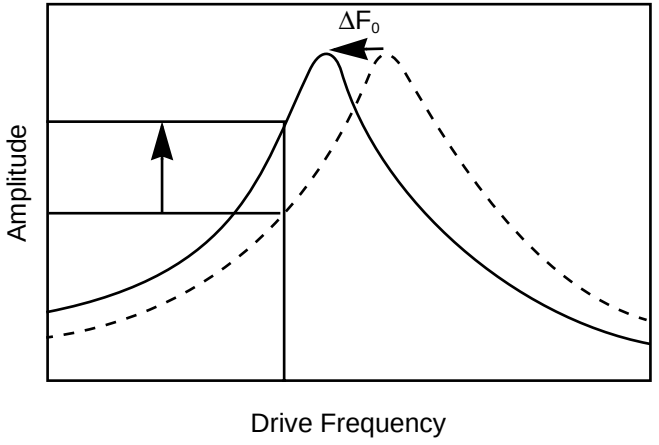


Figure 15.3d Shift in Amplitude at Fixed Drive Frequency



Setting a Drive Frequency for Amplitude Detection continued...

4. Select an appropriate **Target Amplitude** (approximately 2 volts) using **Auto Tune** to adjust the **Drive Amplitude** so that the RMS voltage response of the photodetector is approximately 2 volts before tuning. Or, exit **Cantilever Tune** and manually adjust the **Drive Amplitude** parameter under **Feedback Controls**.

   Note: Somewhat larger values may be beneficial if using amplitude detection.

5. Quit **Cantilever Tune** and return to **Image Mode**.

6. Under **Interleave Controls** set the **Lift start height** to **0 nm**, and **Lift scan height** to **100 nm**.

   Note: The lift height can be optimized later.

7. Set the remaining Interleave parameters (**Setpoint**, **Drive Amplitude, Drive frequency**, **Gains**) to the **Main Controls** values by setting the flags left of the **Interleave Control** column to **Off**.

8. Under **Scan Controls**, set the **Scan size** to **5 µm** and **Scan rate** to **1–2 Hz**.

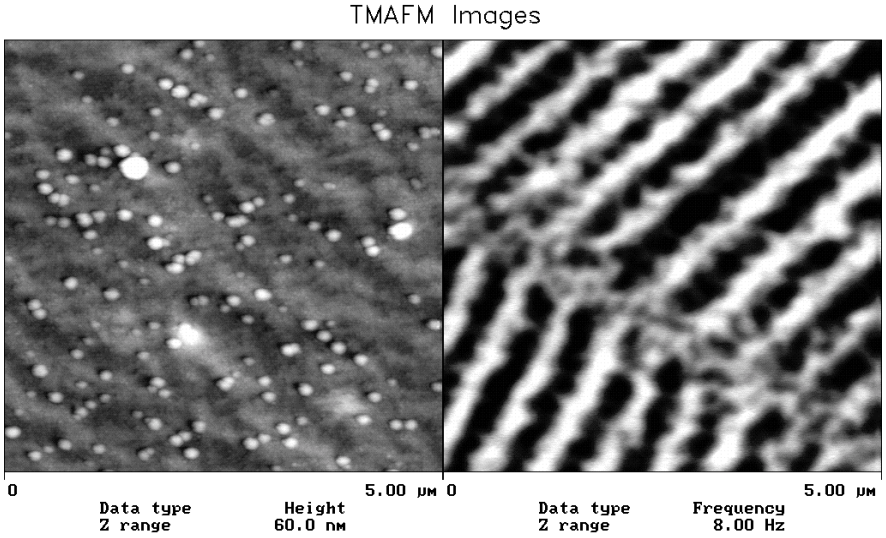
9. For the left image, set the **Z range** to **75 nm** and the **Line direction** to **Retrace**.

10. Engage the AFM and make the necessary adjustments to obtain a good topographical image while displaying height data.

ATTENTION: Use the maximum 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 15.3e. The surface is fairly flat with lubrication nodules of various sizes. A good image of the nodules indicates that the tip is sharp.

Setting a Drive Frequency for Amplitude Detection continued...

Figure 15.3e Topographic (left) and Magnetic Force Gradient Image (right)



Note: The MFM data displays in Channel 2; however, the parameter settings are different depending on whether Phase Detection or Amplitude Detection is being used

Phase Detection

1. Set the **Channel 2** image **Data type** to **Phase**; **Z range** to **3 degrees**; and **Line direction** to **Retrace**.

ATTENTION: It is important that the **Scan 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 2** image **Data type** to **Amplitude**, **Z range** to **1 nm**, and **Line direction** to **Retrace**.

2. Change **Interleave mode** to **Enable** to invoke **LiftMode**.

3. Set the **Channel 2 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 15.3e should appear as the Channel 2 image. The alternating dark and light stripes represent the recorded magnetic information, signifying a varying resonant frequency and magnetic force gradient on the tip.

ATTENTION: Keep the **Setpoint** as large as possible while consistent with a good image. Wider scans (> 25 µm) will reveal separate tracks in which the magnetic stripes are at different angles.

Frequency Modulation

With the Extender Electronics Module, it may be desirable to use frequency modulation. This activates a feedback loop which modulates the **Drive Frequency** to keep the cantilever's 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, but switch the Channel 2 image **Data type** to **Frequency**. Try a **Z range** (frequency shift) of approximately **10 Hz**. Select **Other Controls**, then adjust the frequency modulation gains. Setting both frequency modulation **Integral gain** and **Proportional gain** to **100** is a good starting point. 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. With 225-micron MFM cantilevers, gains are usually in the range of **50-150**.

15.4 Advanced MFM Operation

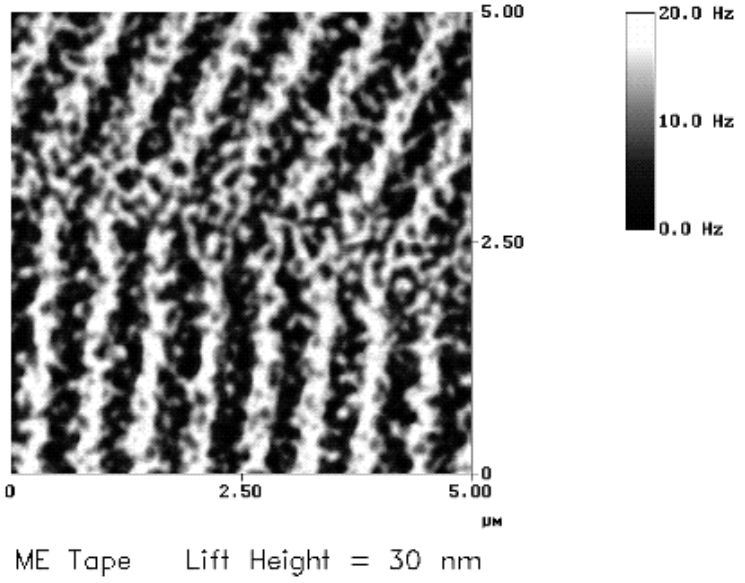
15.4.1 Lift Scan Height and Magnetic Imaging Resolution

The most important parameter affecting imaging resolution is **Lift scan height**. The range of 10–200 nm is most useful. In general, MFM resolution is roughly equal to the lift height. Smaller **Lift Scan heights** give better resolution; conversely, 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 15.3e has a resolution limited by the 100 nm **Lift Scan height**. To improve the resolution, try reducing the **Lift scan height** to ~ **25 nm**. 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 **30 nm** is shown in Figure 15.4a. Note the fine magnetic structure that is not visible in Figure 15.3e. When imaging a sample for the first time, begin with moderate **Lift scan heights** (**50 nm or greater**), then adjust downward. On relatively smooth samples (e.g., hard disks), lift heights down to 0 nm can be used, as long as the drive amplitude is adjusted accordingly. (Lift scan heights of 0 nm 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 resolution of MFM with the NanoScope III is near 20 nm. Resolution is affected by properties of the tip, including mechanical sharpness and magnetic structure. When in good condition, NanoProbe magnetically coated tips routinely give 50 nm resolution, and many achieve 30 nm or better.

Figure 15.4a High-resolution Magnetic Force Gradient Image



15.4.2 Linear Lift

Linear Lift works well for measuring topography on samples by fitting the scan line to the surface and lifting a user determined height above the sample to capture the image of the sample.

15.4.3 Fine Tuning Interleave Controls

Certain scanning parameters found under **Interleave Controls** 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 circular flag to the immediate left of the desired Interleave control, toggling its state from **Off** (gray bullet) to **On** (green bullet). 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.

Advanced MFM Operation continued...

15.4.4 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 the 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 (i.e., when the flag is set to **Off** [gray bullet]).

Drive Amplitude continued...

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.

WARNHINWEIS: 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 15.4b. With phase detection and frequency modulation, changes in amplitude produce no change in contrast, and results are thus more reproducible than with amplitude detection.

  Note: On some microscope models, there is a lowpass filter in the scanning electronics that prevents fast switching of the **Drive Amplitude** between main and Interleave scanning. This can interfere with very fast rates (> a few Hz). The filter can be disabled easily; contact Digital Instruments Veeco technical support for more information.

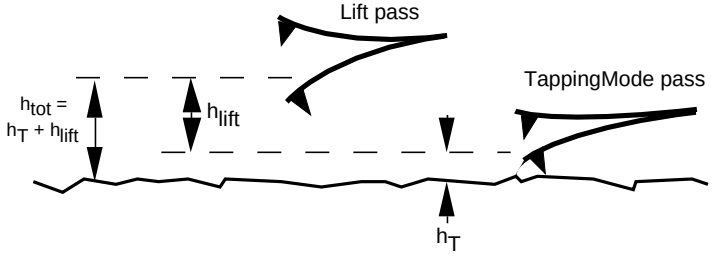
Drive Amplitude continued...

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 Tapping Mode h_T, and the lift scan height h_{lift} (See Figure 15.4b). In **Tapping Mode**, 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 25 nm/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 0 nm still gives a mean tip-sample distance of h_T in **LiftMode**.

Figure 15.4b Tip Height and Oscillation Amplitudes (Tapping Mode & LiftMode)



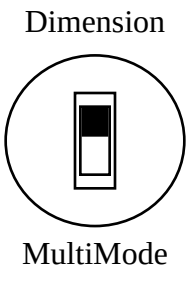
The relationship between setpoint voltage and oscillation amplitude is known as the sensitivity. Its value can be determined with **Force Calibration** (See Chapter 13). For 225 µm MFM cantilevers, the sensitivity is typically in the range 20-25 nm/V. The exact value depends on the position of the laser spot on the cantilever, the cantilever geometry, and the particular instrument. A 1V **Setpoint** typically corresponds to h_T~14-18 nm.

15.5 Installation of Extender Electronics Module

In Spring of 1994, Digital Instruments Veeco began making available its Extender Electronics Module (sometimes referred to as “phase box” or “phase extender”) for customers wanting phase detection and frequency modulation MFM. For Dimension systems, the Extender is installed using 37-to-37 pin ribbon cables between: 1) the NanoScope III or IIIA SPM controller and the Extender Electronics Module; as well as 2) from the FM / Extender to the microscope. A hardware change is also required; the main electronics backplane board at the rear of the microscope must be swapped out (this may be done by either the customer or a factory representative). Detailed installation instructions are provided in Digital Instruments Veeco *Support Note No. 205*. For more information, contact Digital Instruments Veeco.

Note: The phase box is equipped with a slider switch for switching internal electronics between Dimension-series and MultiMode SPM signals. This switch may be accessed through a hole on the underside of the box as shown below:

Figure 15.5a Phase Box



For Dimension-series SPMs, always set the switch to **Dimension**. Use a pencil to access the switch through the hole.

Installation of Extender Electronics Module continued...

CAUTION: Do not insert a conducting object (e.g., screwdriver) into the Phase Extender box while it is energized.

ATTENTION: Ne pas insérer d' objet conducteur (par exemple: un tournevis) dans le boîtier d'extension de phase (Phase Extender Box) quand celui-ci est sous tension.

WARNHINWEIS: Stecken Sie keine leitfähigen Teile (zum Beispiel Schraubenzieher) in die Phase Extender Box, während diese eingeschaltet ist.

Microscope Parameter Files

There are two parameter (par) file contexts that are used with the Extender Box: Extended AFM and Extended STM. The former of these two contexts is used for the following operational modes: Contact AFM, Tapping Mode AFM, LFM, MFM, Force Modulation, and Electric Field Measurement. The latter context is for use only with STM.

For each scanner intended to be used with the Extender, there must be a PAR file for the context required. For example, if there are three scanners, all intended for both STM and AFM use, a total of six PAR files are required.

Note: Due to the many Digital Instruments Veeco microscope systems available, the variety of scanners, when the system was purchased, and the system's history of software updates, not all systems have PAR files with the same name or naming scheme. You can find the name of the most recent PAR file in use on the line that begins with \Microscope file:.

Note: When closing the SYSTEM.PAR file after viewing it, if you are asked to SAVE the file or to SAVE CHANGES, be sure to say **No**.

To modify the PAR files on a Dimension system, locate the proper PAR file in the /SPM/EQUIP directory on your computer. Use a text editor to modify the file. The text must be saved as a plain ASCII text file.

Note: Microsoft Word® or other similar desk-top-editors normally save files with embedded formatting commands causing the par files to be unreadable by the NanoScope software.

Using the EDIT program of a text file editor, modify the file to include a new line at the bottom of the file. For example:

If the line

\Is FM: No

is present in the file, then change the line to read:

\Is FM: Yes

If the line

\Is FM: No

is not present, add the line:

\Is FM: Yes

Then locate the line:

\In Polarity: Reverse

and change it to read:

\In Polarity: Forward

Microscope Parameter Files continued...

Then locate the line:

\In Polarity: AUX D: Reverse

Change it to read:

\In Polarity AUX D: Forward

Repeat the procedure above for each scanner (i.e. PAR file) in use. After the cables are connected, power up the NanoScope controller and start the microscope control program.

Important Points

1. Extender-compatible microscope electronics are required to permit operation of the phase detection extender option. Standard electronics on these microscopes require hardware upgrades. Consult your Digital Instruments Veeco sales representative for details.

2. Turn off the power to the NanoScope controller whenever connecting or disconnecting the Extender.

3. In **LiftMode**, the best performance is obtained if the RMS amplitude is kept below 7.0 volts, the limit of the RMS output's linear operation.

15.6 Troubleshooting

15.6.1 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**, that **Interleave** is **Enabled** 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 Controls** values.

15.6.2 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.

15.6.3 Optical Interference

When using **Amplitude Detection**, 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 ~1–2 µm 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.

> **Note:** Optical interference is essentially eliminated by using phase detection or frequency modulation.

Chapter 16 Electric Techniques

16.1 Overview

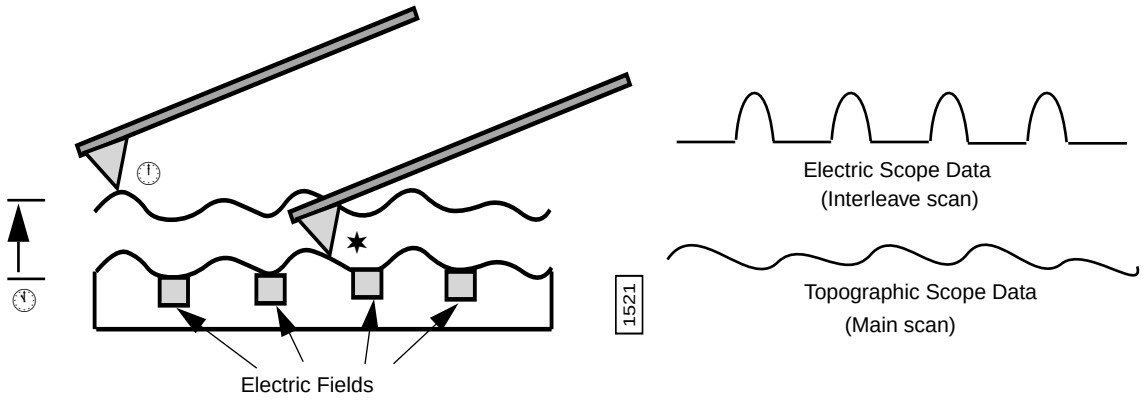
This chapter describes how to perform two electric techniques: Electric Force Microscopy (EFM) and Surface Potential. EFM is similar to Magnetic Force Microscopy (MFM) and shares many of the same procedural techniques. Both modes utilize the Interleave and LiftMode procedures discussed in previous chapters. Please read those chapters before attempting electric force measurements.

Specifically, this chapter includes:

16.2 Electric Techniques Overview

There are two types of electric techniques used with the Dimension Series system: **Electric Force Microscopy (EFM)** and **Surface Potential** imaging. Both techniques employ a two-pass **LiftMode** measurement. **LiftMode** allows the imaging of relatively weak but long-range magnetic and electrostatic interactions while minimizing the influence of topography (See Figure 16.2a). **LiftMode** records measurements in two passes (each consisting of one trace and one retrace) across each scanline. First, **LiftMode** records topographical data in Tapping Mode on one trace and retrace. The tip then raises to the lift scan height, and a second trace and retrace is performed while maintaining a constant separation between the tip and local surface topography.

Figure 16.2a 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.

Electric Techniques Overview continued...

16.2.1 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. Since 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.

All standard Dimension Series SPMs are capable of EFM imaging using amplitude detection techniques. By adding an Extender™ electronics module, you can use the Dimension system for frequency modulation or phase detection with improved results (See Figure 16.2b). Frequency modulation and phase detection has largely superseded amplitude detection. The Extender™ electronics module is required for surface potential imaging, and is strongly recommended for electric force microscopy.

Figure 16.2b Extender™ Electronics Module

If applying a voltage to the tip is desired, then the special electric cantilever holder is required (model MMEFCH).

16.2.2 Surface Potential Imaging Overview

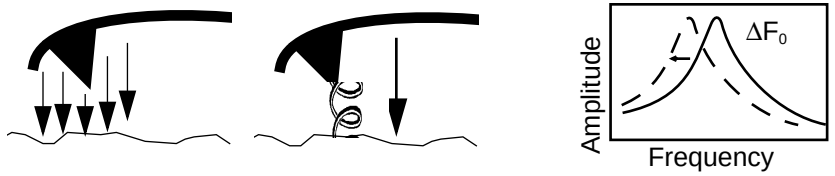
Surface potential imaging measures the effective surface voltage of the sample by adjusting the voltage on the tip so that it feels a minimum electric force from the sample. (In this state, the voltage on the tip and sample is the same.) Samples for surface potential measurements should have an equivalent surface voltage of less than ±10 volts, and operation is easiest for voltage ranges of ±5 volts. The noise level of this technique is typically 10 mV. 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. This method requires the Extender Electronics Module and version 3.1 or later of the NanoScope III software.

16.3 Electric Force Microscopy

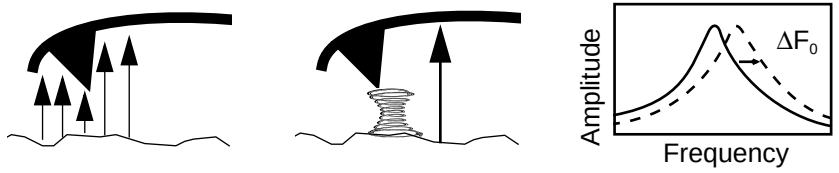
16.3.1 Electric Force Microscopy Theory

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 16.3a.

Figure 16.3a Comparison of Attractive and Repulsive Forces



Attractive gradient equivalent to additional spring in tension attached to tip, reducing the cantilever resonance frequency.



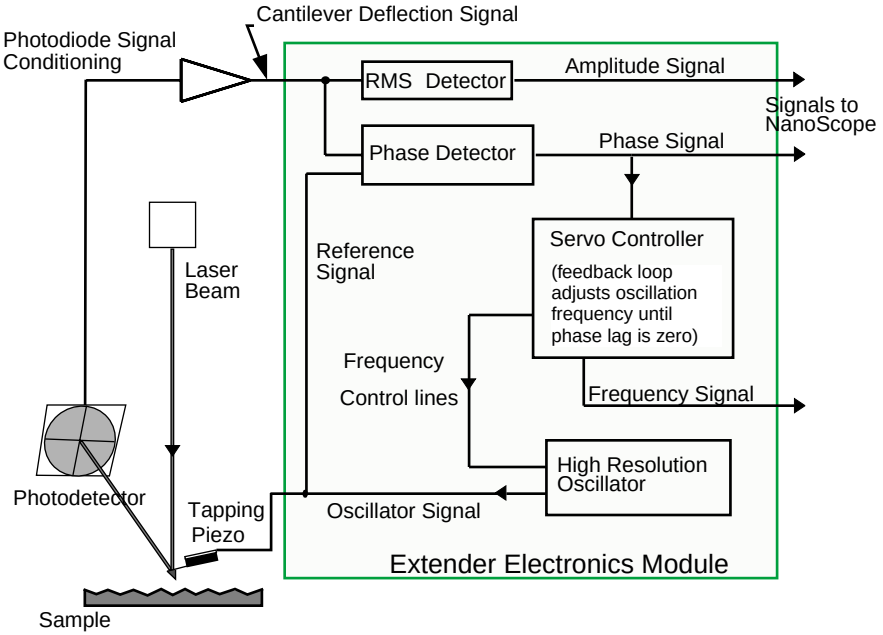
Repulsive gradient equivalent to additional spring in compression attached to tip, increasing the cantilever resonance frequency.

Changes in cantilever resonant frequency are detected in one of the following ways:

- Phase detection (with Extender Electronics Module only)
- Frequency modulation (with Extender Electronics Module only)
- 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 16.3b shows a diagram of how the Extender electronics module 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 16.3b Diagram of Extender Electronics Module 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.

Electric Force Microscopy continued...

16.3.2 Electric Force Microscopy Preparation

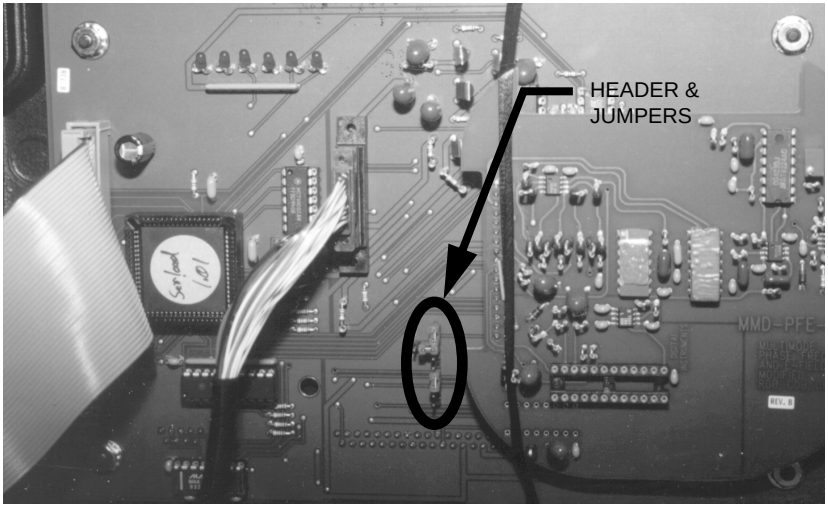
This section explains how to apply a voltage to the tip or sample to generate electric fields. If the sample has a permanent electric field which does not require the external application of voltage, the steps below are not required and you can proceed to Section 16.3.3.

Setting Jumper Configurations

To apply voltage to the tip or sample, you may need to make minor changes to the jumpers on the microscope's backplane and the toggle switches on the Extender Electronics Module (if equipped). Original jumper configurations and jumper changes are dependent on the microscope and the desired measurements. Section provides jumper configuration instructions for basic microscope models operating with and without the Extender Electronics Module.

The backplane board used with Dimension Series SPMs is shown below in Figure 16.3c. There is a header supplied with jumpers at the center of the board. For non-EFM applications and Surface Potential operation, leave or return jumpers to their original positions.

Figure 16.3c Microscope Backplane



Instructions for Reconfiguring Jumpers

Carefully examine the jumper configuration figures in the following pages and identify which jumper configuration is correct for your application. If the configuration you choose differs from the configuration as shipped from the factory, follow the instructions below. Refer to Figure 16.3e and Figure 16.3j for examples of factory jumper configurations.

1. Power-down the NanoScope controller and unplug the power cable from the microscope's electronic box.

2. Remove the back panel on the microscope's electronic box.

3. Locate header and jumpers per Figure 16.3c on the main electronics backplane.

   Note: Jumper systems without the Extender Electronics Module should appear as shown in Figure 16.3e; whereas jumper systems with the Extender Electronics option should appear as in Figure 16.3j.

4. Reconfigure jumpers on the backplane header using the appropriate jumper configuration.

5. After correctly configuring the backplane jumpers, replace the cover on the electronics box.

6. Plug the power cable back into the microscope.

7. Power-up the NanoScope controller.

Setting Analog 2 in the Software

If you have chosen a configuration where Analog 2 applies to the tip or sample, you must enable **Analog** in the software. The Analog 2 parameter appears in both the **Feedback Controls** and **Interleave Controls** panels.

Note: For Version 4.23 and lower, Analog 2 appears only in the **Feedback Controls** panel.

Electric Force Microscopy Preparation continued...

Instructions for Enabling Analog 2 in Software

1. Select **Di** / **Microscope Select** / **Edit** / **Advanced**, and set Analog 2 to **User defined**.

2. Click **OK** to exit both dialog boxes.

Note: For software versions 4.23 and lower, select **Microscope / Calibrate / Detector** to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Disallow**.

Instructions for Disabling Analog 2 in Software

1. Select **Di** / **Microscope Select** / **Edit** / **Advanced**, and set Analog 2 to **Atten switch**.

2. Click **OK** to exit both dialog boxes.

Note: Remember to set **Allow in attenuation** to **Allow** when finished. For all other configurations it should be left on **Allow**.

Setting the Extender Electronics Box

1. For systems with an Extender Electronics Box, locate the two toggle switches on the backside of the Extender Electronics Box (See Figure 16.3d).

2. Verify that they are toggled as shown in Table 16.3a.

Figure 16.3d Toggle Switches on Extender Electronics Module

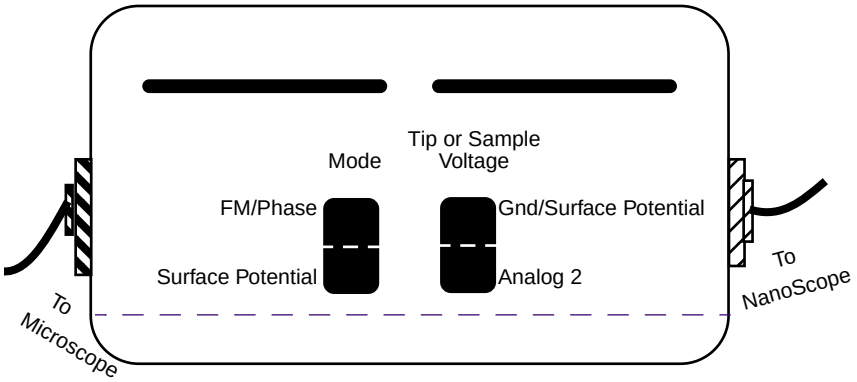


Table 16.3a: Extender Electronics Module Toggle Switch Settings

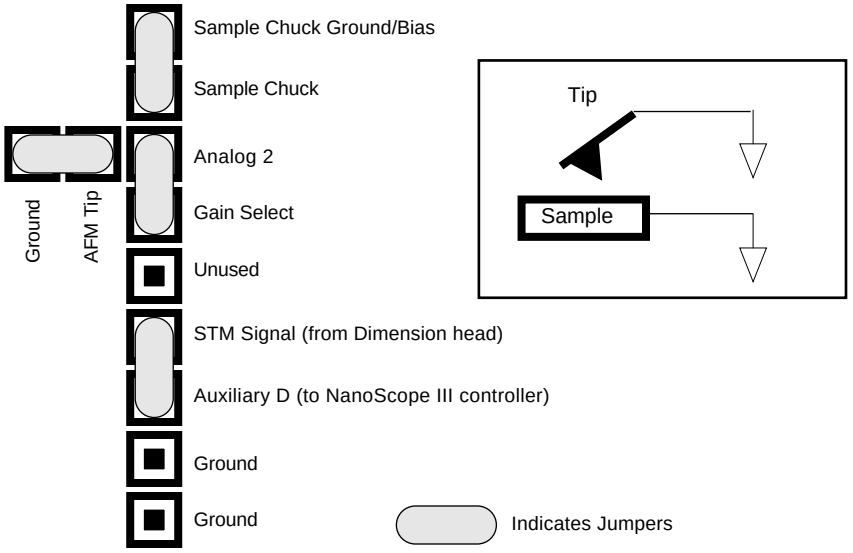
	Mode		**Tip or Sample Voltage**	
	FM/Phase	**Surface Potential**	**GND/ Surface Potential**	**Analog 2**
EFM with Analog 2 biasing tip or sample	**X**			**X**
EFM in all other configurations	**X**		**X**	
Standard Operation	**X**		**X**	

Electric Force Microscopy Preparation continued...

Jumper Configurations Without Extender Electronics

As shipped from the factory, the jumper configuration on a Dimension Series system without the Extender Electronics Module appears as shown in Figure 16.3e below. This configuration connects both tip and sample to ground.

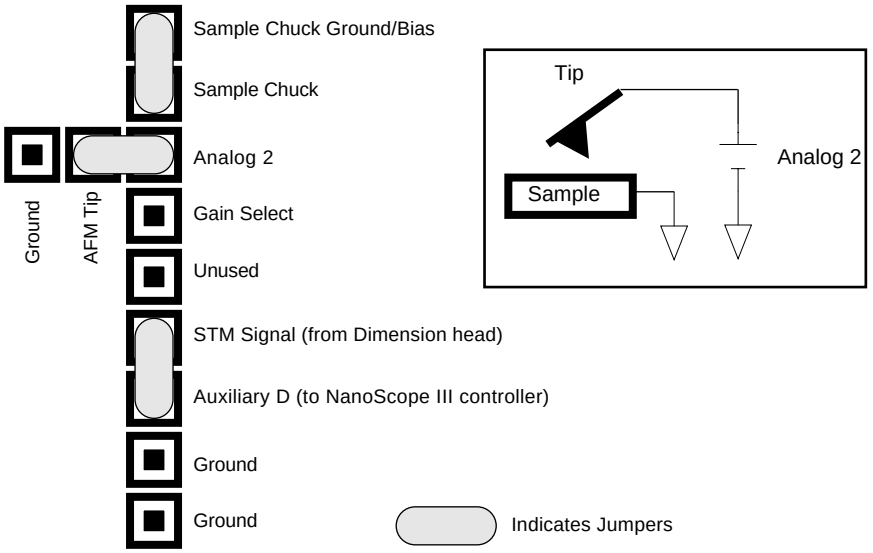
Figure 16.3e Normal Jumper Configuration



Analog 2 Voltage Applied to the Tip (No Extender Electronics)

The jumper configuration in Figure 16.3f connects the Analog 2 signal from the NanoScope III controller (± 12 VDC range) to the tip. Remember to enable the Analog 2 voltage line as described in Section 16.3.2.

Figure 16.3f Jumper Configuration for Application of Analog 2 Voltage to Tip

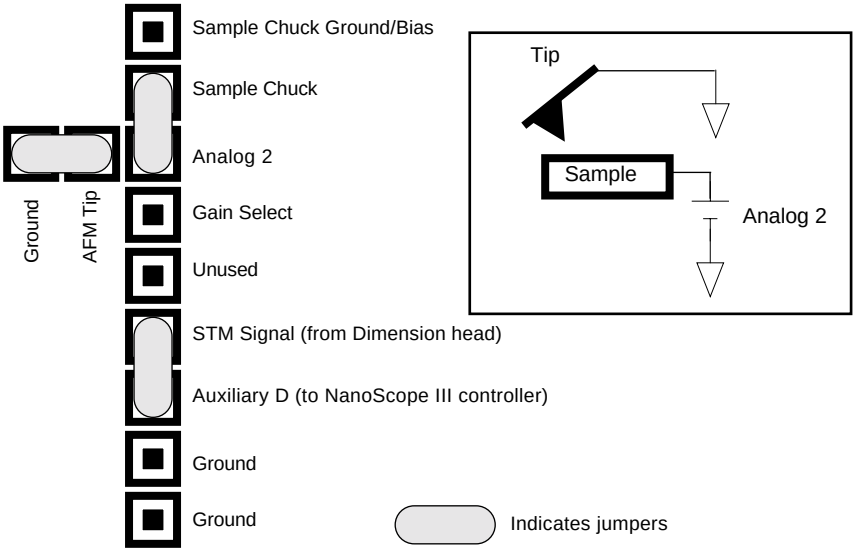


Jumper Configurations Without Extender Electronics continued...

Analog 2 Voltage Applied to the Sample (No Extender Electronics)

The jumper configuration in Figure 16.3g connects the Analog 2 signal from the NanoScope III controller (± 12 VDC range) to the sample chuck. Remember to enable the Analog 2 voltage line as described in Section 16.3.2.

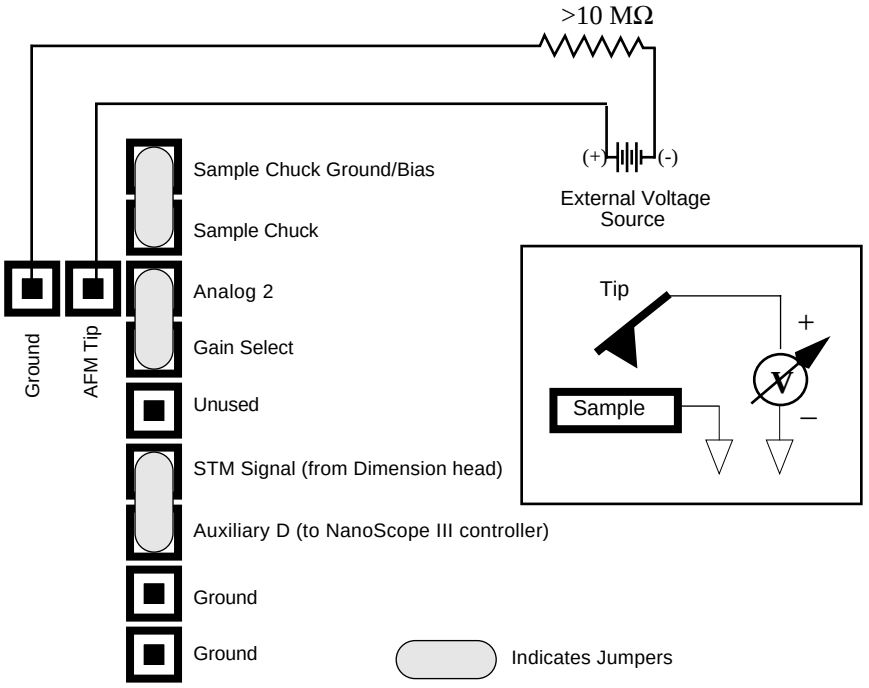
Figure 16.3g Jumper Configuration (Application of Analog 2 Voltage to Sample**)**



External Voltage Source Applied to the Tip (No Extender Electronics)

In some cases, it may be advantageous to use voltages greater than 12 V, or to use a pulsed power supply. If an external source of voltage is to be applied to the tip, configure jumpers as shown in Figure 16.3h.

Figure 16.3h Jumper Configuration for Applying External Voltage to Tip

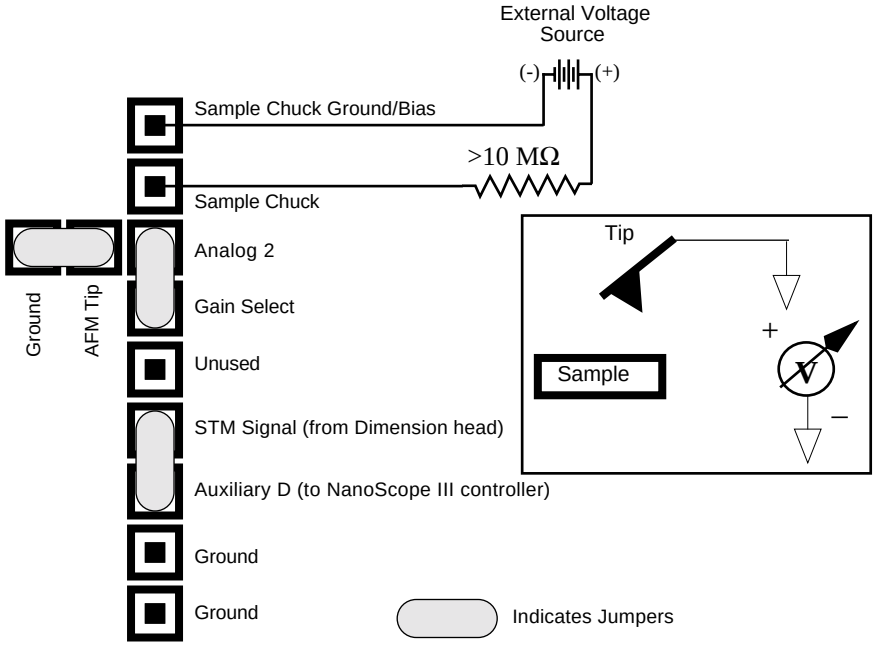


Place a current-limiting resistor (e.g., 10–100 MΩ) in series with the external voltage supply as shown to protect the tip and sample from damage. You may also use current-limited power supplies. Connect voltage leads to pins on the header using soldered, push-on connectors. Do not solder leads directly to the header pins, as the heat may cause damage and/or make jumpering the pins difficult.

External Voltage Source Applied to Sample (No Extender Electronics)

In some cases, it may be advantageous to use voltages greater than 12 V, or to use a pulsed power supply. If an external source of voltage is to be applied to the sample, configure jumpers as shown in Figure 16.3i.

Figure 16.3i Jumper Configuration for Applying External Voltage to Sample



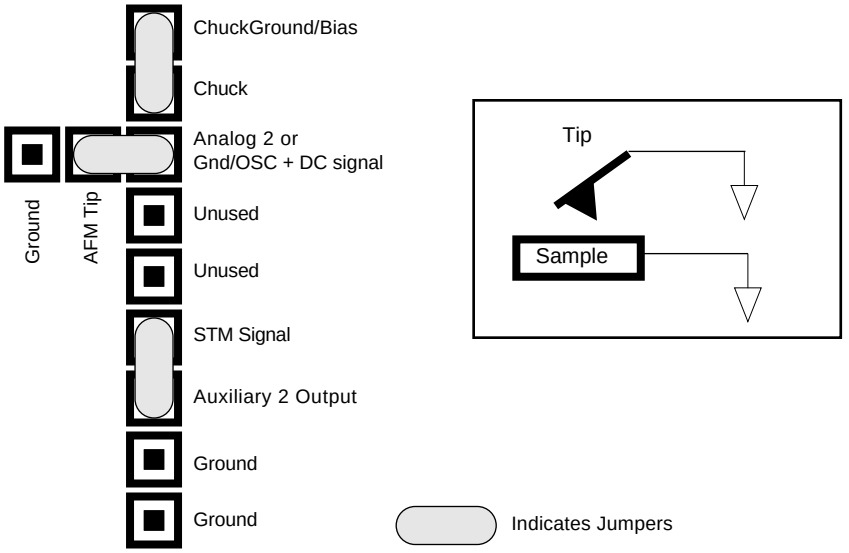
A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to pins on the header using soldered, push-on connectors. *Do not* solder leads directly to the header pins, as the heat may cause damage and/or make jumpering the pins difficult.

Jumper Configurations With Extender Electronics

CAUTION: Power down the NanoScope controller and unplug the power cable from the microscope electronic box before attempting to adjust jumper configurations.

As shipped from the factory, systems with the Extender Electronics option should have an original backplane jumper configuration as shown in Figure 16.3j. This configuration connects both the tip and sample to the ground.

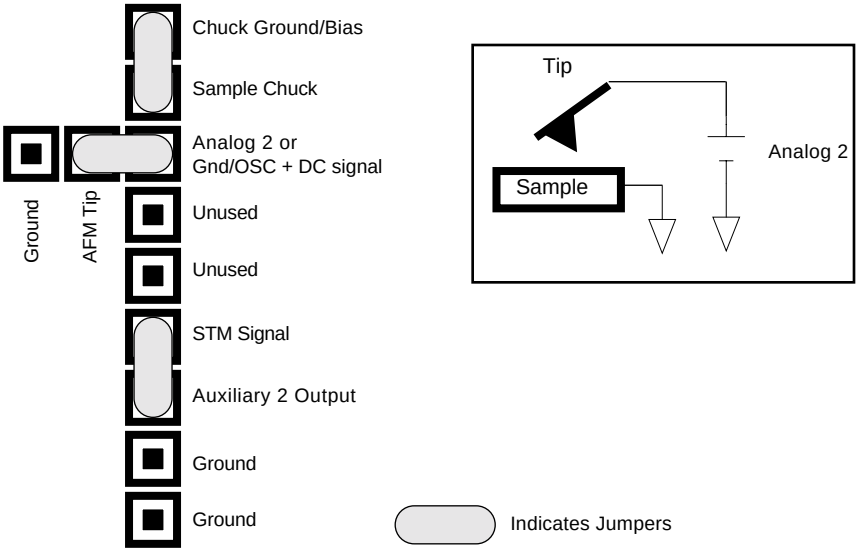
Figure 16.3j **N**ormal Jumper Configuration with Extender Electronics Module



Analog 2 Voltage Applied to the Tip (With Extender Electronics)

Notice that the jumper configuration in Figure 16.3k connects the Analog 2 signal from the NanoScope III controller (± 12 V range) to the tip, and is **exactly the same** as the jumper configuration shown in Figure 16.3j, the standard configuration as shipped from the factory. Remember to enable the Analog 2 voltage line as described in Section 16.3.2.

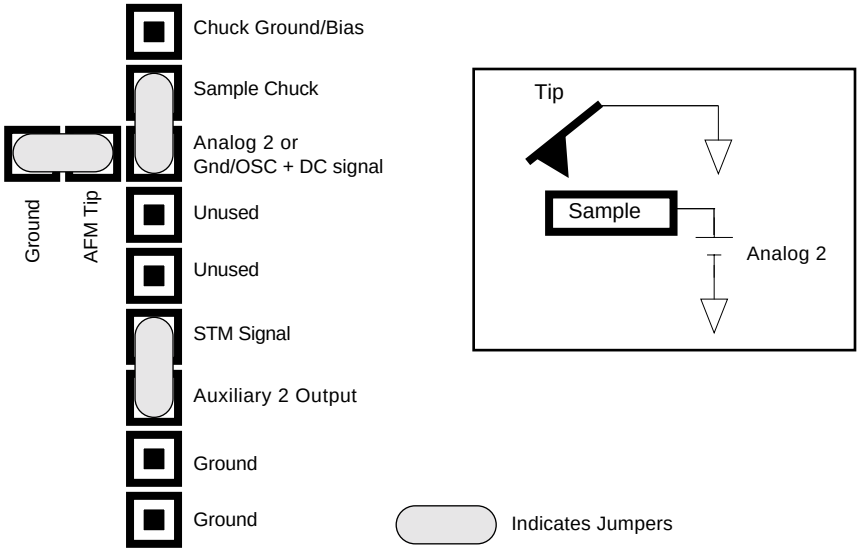
Figure 16.3k Jumper Configuration for Application of Voltage to Tip



Analog 2 Voltage Applied to Sample (With Extender Electronics)

The jumper configuration in Figure 16.3l connects the Analog 2 signal from the NanoScope III controller (± 12 V range) to the sample. Remember to enable the Analog 2 voltage line as described in Section 16.3.2.

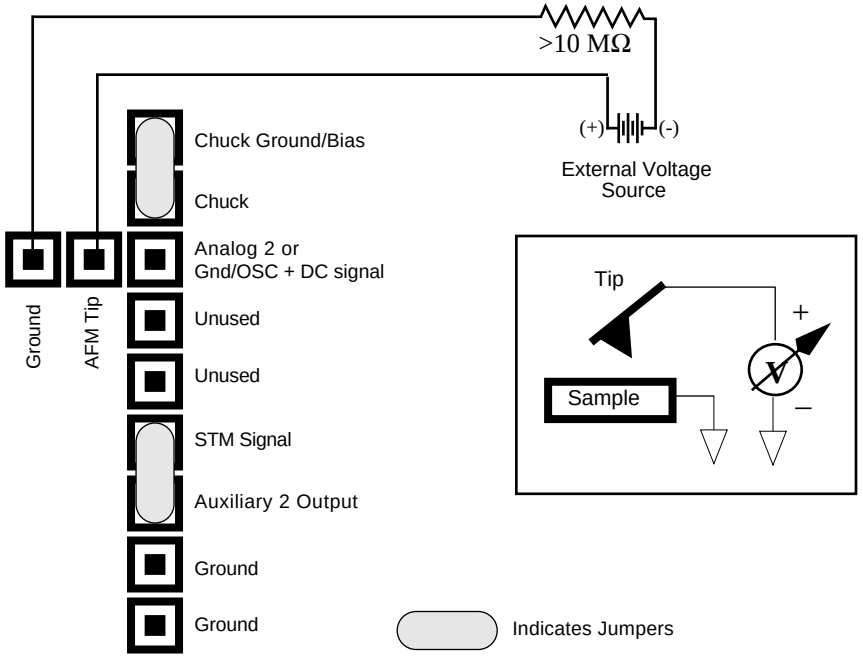
Figure 16.3l Jumper Configuration for Application of Voltage to Sample



External Voltage Source Applied to Tip (With Extender Electronics)

In some cases, it may be advantageous to use voltages greater than 12 V, or to use a pulsed power supply. If an external source of voltage is to be applied to the tip, configure jumpers as shown in Figure 16.3m.

Figure 16.3m Jumper Configuration for Applying External Voltage to Tip

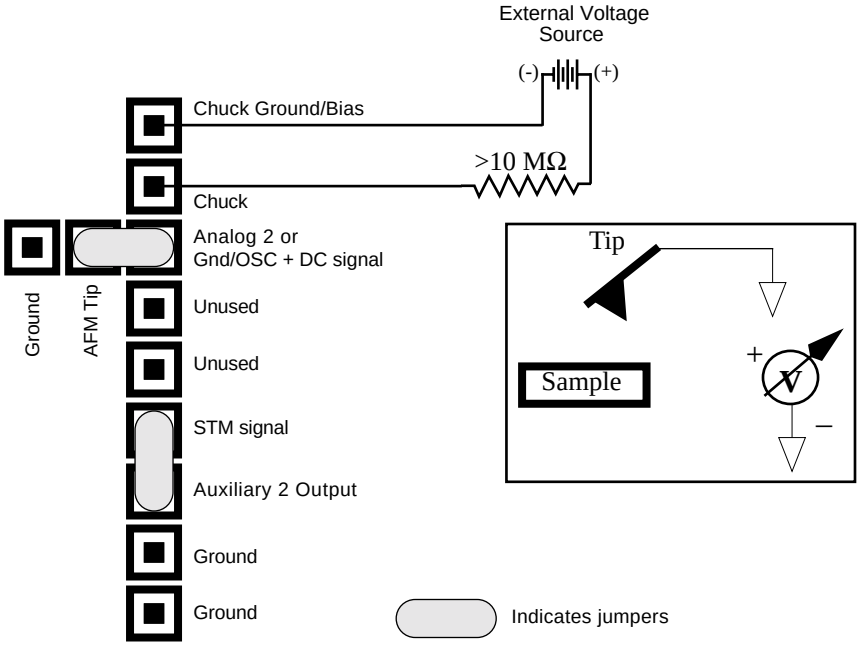


Place a current-limiting resistor (e.g., 10–100 MΩ) in series with the external voltage supply as shown to protect the tip and sample from damage. You can also use current-limited power supplies. Connect voltage leads to pins on the header using soldered, push-on connectors. Do not solder leads directly to the header pins as the heat may cause damage or make jumpering the pins difficult.

External Voltage Source Applied to Sample (With Extender Electronics)

In some cases, it may be advantageous to utilize voltages greater than 12 V, or to utilize a pulsed power supply. If an external source of voltage is to be applied to the sample, configure jumpers as shown in Figure 16.3n.

Figure 16.3n Jumper Configuration for Applying External Voltage to Sample



Place a current-limiting resistor (e.g., 10-100 MΩ) in series with the external voltage supply as shown to protect the tip and sample from damage.You can also use current-limited power supplies. Connect voltage leads to pins on the header using soldered, push-on connectors. Do not solder leads directly to the header pins, as the heat may cause damage and make jumpering the pins difficult.

Electric Force Microscopy continued...

16.3.3 Electric Force Microscopy Procedures

This section details procedures for conducting EFM for each of the three techniques: phase detection, frequency modulation, and amplitude detection.

> **Note:** Amplitude detection is the only procedure described here that can be done without the Extender Electronics Module; however, this method is no longer recommended.

General Procedures

1. Verify that the following electric force microscopy preparation is complete (See Section 16.3.2):
    - Jumper Configurations
    - Extender Settings
    - Analog 2 Setting in Software
2. Connect the sample with electricity by mounting it to a standard sample disk using conducting epoxy or silver paint. Hold the disk down to the chuck with either vacuum or the magnetic mount. In most cases it is important that the sample and sample chuck are electrically connected.
3. Verify that the connection is good; a poor connection introduces noise.
4. If an external power supply connects directly to leads on the sample itself, it is important to electrically isolate the sample from the sample chuck. A piece of Kapton tape covering the bottom of a sample disk works well.
5. Mount a metal-coated NanoProbe cantilever into the cantilever holder. MFM-style cantilevers (225 micron long, with resonant frequencies around 70 kHz [Model MESP]) usually work well. It is also possible to metal-coat standard Tapping Mode cantilevers. Make sure that any deposited metal you use adheres strongly to the silicon cantilever.

6. Set up the AFM as usual for Tapping Mode operation (See Chapter 10).

7. Select **View / Cantilever Tune.**

8. Follow the instructions below for the type of electric force imaging desired, **Phase Detection, Frequency Modulation,** or **Amplitude Detection**.

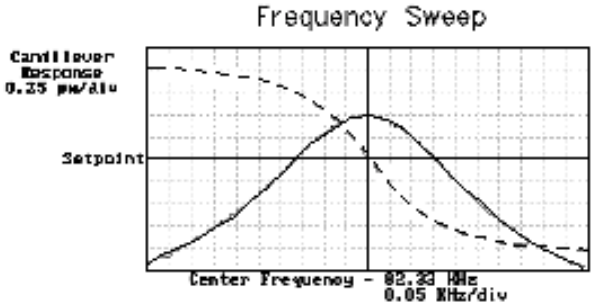
Phase Detection

Phase Detection is only available when the Extender Electronics Module has been correctly configured into the system.

1. In the **Auto Tune Controls** window, set **Start frequency** and **End frequency** to appropriate values for your cantilever (e.g., MESP cantilevers, set **Start frequency** to 40 kHz and **End frequency** to 100 kHz). Select **Auto Tune**.

   Note: Two curves appear on the Cantilever Tune graph: the amplitude curve in white and the phase curve in yellow. (In Figure 16.3o, the phase curve is the dashed line and the amplitude curve is the solid line.)

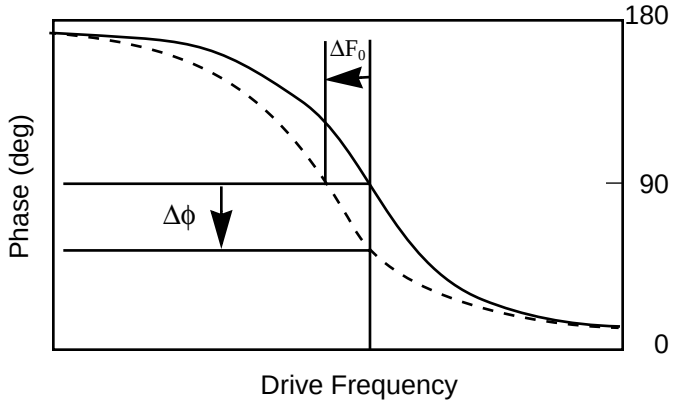
Figure 16.3o Phase Detection Cantilever Tune



Phase Detection continued...

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 voltage and the cantilever response. Gradients in the electric force will cause a shift ΔF_0 in the resonance frequency. Resonance shifts give rise to the phase shifts $\Delta\phi$ used to generate an image of the electric force gradients (See Figure 16.3p).

Figure 16.3p Shift in Phase at Fixed Drive Frequency



2. Quit **Auto Tune** and return to **Image Mode.**

3. Engage the AFM and make the necessary adjustments to obtain a good topography (**Height**) image on Channel 1.

4. In the **Interleave Controls** panel set the **Lift start height** to 0 nm, and **Lift scan height** to 100 nm.

   Note: You can optimize the lift height later.

5. Set the remaining Interleave parameters (**Setpoint**, **Drive amplitude**, **Drive frequency**, gains, etc.) to the main Feedback Controls values by clicking the flags to the left of each parameter to "off" (grayed bullets).

6. Set the Channel 2 **Data type** to **Phase,** and choose **Retrace** for the scan **Line direction** on both Channel 1 and 2.

7. In the **Interleave Controls** panel set **Interleave mode** to **Lift**.

Phase Detection continued...

Note: For software versions 4.23 and lower set **Interleave scan** to **Lift** and switch **Interleave mode** to **Enable in** the Interleave Controls panel.

8. Set the Channel 2 **Scan line** to **Interleave** to display interleave data.

   Note: For software versions 4.23 and lower, there are no separate Analog 2 setting in the **Interleave Controls** panel. Set Analog 2 in the **Feedback Controls** panel to **0 volts**.

9. In the **Interleave Controls** panel set **Analog 2** to the desired voltage.

   Note: Applying a voltage during the main line can make Tapping Mode operation difficult.

10. Optimize the **Lift scan height**. For high-resolution, set the **Lift scan height** as small as possible without crashing the tip into the surface. There are two ways to determine if the tip is touching the surface:

    - If the tip crashes into the surface, it typically creates bright or dark specks or streaks in the image. Also, if the **Lift scan height** is set extremely low, the tip may continuously "tap" on the surface during the LiftMode scan.
    - Toggle between the **Interleave** and **Main scan lines** for the phase image. If the two images appear similar this indicates the tip is continuously tapping on the surface during the LiftMode scan. Increase the **Lift scan height** until the **Interleave** scan image changes indicating that the tip is oscillating above the surface and not continuously tapping.

11. Adjust the sample or tip voltage to confirm that contrast is due to electrical force gradients.

Phase Detection continued...

Note: On very rough samples, contrast in LiftMode images may be from air damping between the tip and surface. Look at the phase data in Scope Mode while adjusting the tip or sample voltage up and down. Contrast due to electrical force gradients increases or decreases as the tip-sample voltage changes.

Frequency Modulation

Use Frequency Modulation (FM) for quantitative results. This technique directly measures the change in resonant frequency felt by the cantilever. A feedback loop keeps the phase at 0 degrees by adjusting the drive frequency. The software records the change in drive frequency to create an image of the electric field gradient. In the **Interleave Controls** panel set the **Input feedback** to **Frequency**. Switch the **Data Type** for Channel 2 to **Frequency**. Optimize the FM gains in the Other Controls panel to properly track the shifts in resonant frequency (starting values: FM **igain** = 40 and FM **pgain** = 60).

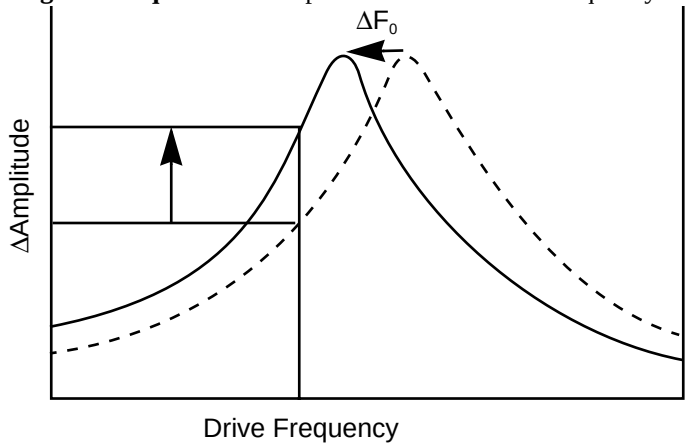
Note: For Versions 4.23 and lower, there is no **Input feedback** setting. Switch the **Data Type** for Channel 2 to **Frequency**.

Amplitude Detection

Note: This imaging method, although described here, is not recommended due to the presence of artifacts.

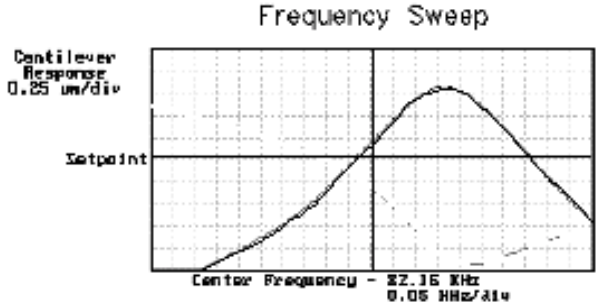
Amplitude Detection, unlike **Phase Detection**, is available with or without the optional Extender Electronics Module. This section describes the differences in set up for **Amplitude Detection**. Changes in the cantilever amplitude provide an indirect measure of shifts in the cantilever resonance frequency as shown in Figure 16.3q.

Figure 16.3q Shift In Amplitude at Fixed Drive Frequency



Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 16.3r.

Figure 16.3r Amplitude Detection Cantilever Tune



For maximum sensitivity, set the **Drive frequency** to the steepest part of the resonance curve. As the tip oscillates above the sample, a gradient in the electric force shifts the resonance frequency F_0 (See Figure 16.3r).

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 slightly better results are obtainable on the low side, as shown in Figure 16.3r.

Amplitude Detection continued...

When using **Amplitude Detection**, optical interference may appear in the lift (electric force) image when imaging highly reflective samples. Optical interference appears as evenly spaced, 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 and reflecting off the sample) interferes with laser light reflecting from the cantilever. You can alleviate interference by moving the laser beam spot up the cantilever away from the tip; about one-third of the cantilever length from the tip works well. On the Dimension head, you can refine the adjustment by carefully moving the beam spot laterally on the cantilever while scanning to minimize interference fringes.

To eliminate optical interference, use Phase Detection or Frequency Modulation, available only with the Extender Electronics Module.

16.4 Surface Potential Detection

16.4.1 Surface Potential Detection Theory

The Extender Electronics Module allows measurement of local sample surface potential. This is similar to techniques called Scanning Maxwell Stress Microscopy and Kelvin Probe Microscopy. Surface potential detection is a two-pass system where the first pass obtains surface topography and the second pass measures surface potential. The two measurements are interleaved, that is, they each measure one line at a time with both images displaying simultaneously on the screen.

Note: Surface potential detection EFM is only possible using the Extender Electronics Module. This section does not apply to microscopes not equipped with the Extender Electronics Module.

See Figure 16.4a for a block diagram of the surface potential measurement system. On the first pass, standard Tapping Mode measures the sample topography. In Tapping Mode the cantilever mechanically vibrates near its resonant frequency by a small piezoelectric element in the cantilever holder called the drive piezo. On the second pass, the drive piezo is turned off. Instead, to measure the surface potential, apply an oscillating voltage

$$V_{ac}(\sin\omega t)$$

directly to the cantilever tip. This creates an oscillating electric force at the frequency ω on the cantilever. The oscillating force has the following amplitude:

$$F = \frac{dC}{dz}\Delta V_{dc} V_{ac}$$

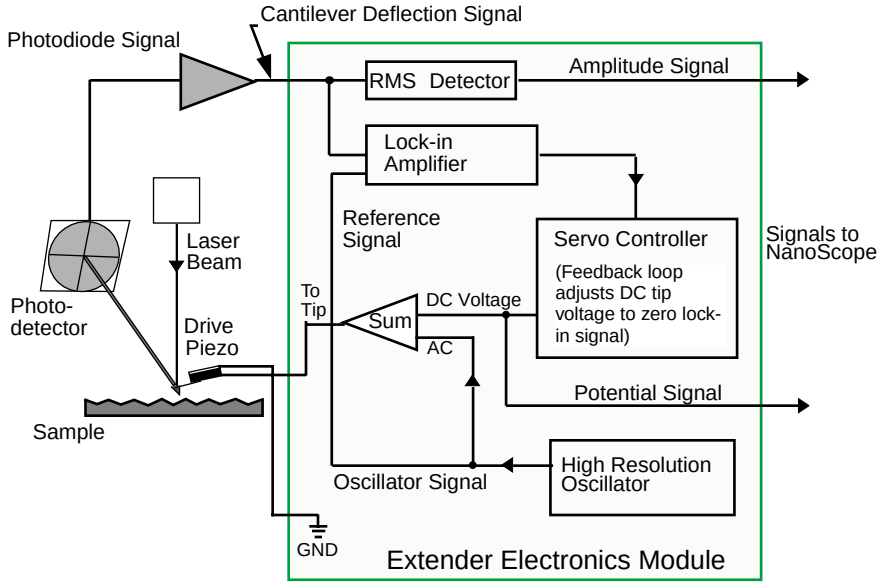
where $\frac{dC}{dz}$ is the vertical derivative of the tip/sample capacitance.

$\Delta V_{dc} = V_{tip} - V_{sample}$ is the DC voltage difference between the tip and the sample,

and V_{ac} is the amplitude of the oscillating voltage applied to the cantilever tip.

Surface Potential Detection Theory continued...

Figure 16.4a Simplified Block Diagram of Extender Electronics Module



The force on the cantilever depends on the product of the AC drive voltage and the DC voltage difference between the tip and the sample. When the tip and sample are at the same DC voltage ($\Delta V_{dc}=0$), the cantilever experiences no oscillating force. Therefore, the Extender Electronics Module determines the effective local surface potential on the sample (V_{sample}) by adjusting the DC voltage on the tip (V_{tip}) until the oscillation amplitude equals zero. At this point the tip voltage remains the same as the unknown surface potential. The NanoScope III records the voltage applied to the cantilever tip (V_{tip}) to construct a voltage map of the surface.

16.4.2 Surface Potential Detection Preparation

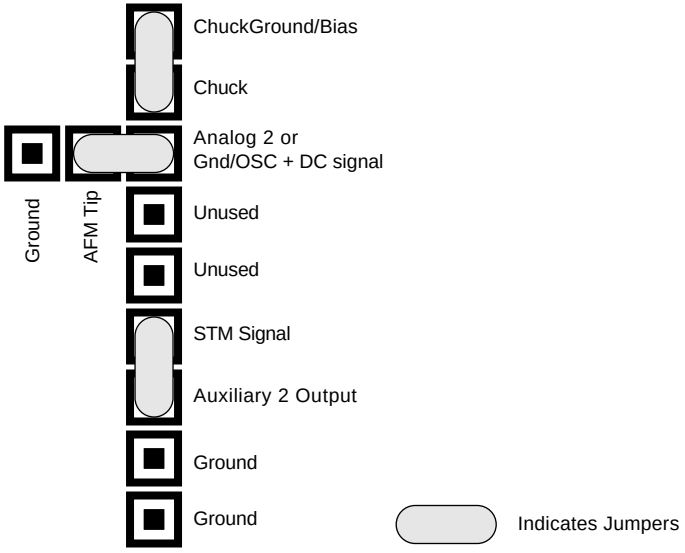
It is often desirable to apply a voltage to one or more areas of a sample. This may be done in two ways: by connecting a voltage to the sample mounting chuck, or by making direct contact to the sample. In both cases, jumper configurations on the backplane of the microscope are changed to match the environment desired.

Note: In addition to any reconfigured jumpers, connect the common or negative terminal of an external power supply to the Dimension system ground. Remove the back panel of the electronics box to access the ground and other Dimension signals (See Figure 16.4b).

1. Power-down the NanoScope controller, and unplug the NanoScope power cable from the microscope electronic box.

2. Remove the back panel on the microscope's electronic box and locate the jumpers on the main electronics backplane per Figure 16.4b. As shipped from the factory, the backplane jumpers appear as shown in Figure 16.4b.

Figure 16.4b Normal Jumper Configuration



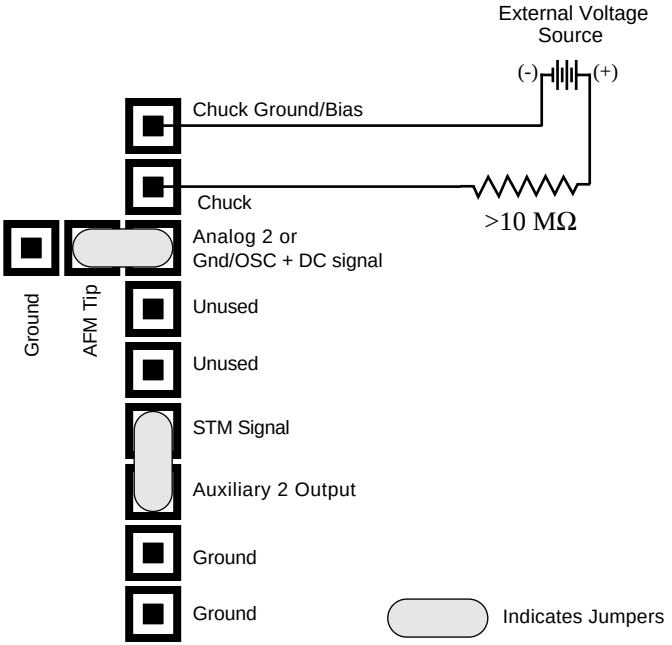
Surface Potential Detection Preparation continued...

3. Depending upon whether voltage is applied to the sample directly or indirectly, reconfigure jumpers on the backplane header according to either Figure 16.4c or Figure 16.4e.

Applying Voltage to Sample Through the Sample Chuck

When you apply an external voltage to the sample via the sample chuck, configure the jumpers as shown in Figure 16.4c.

Figure 16.4c Jumper Configuration for Application of Voltage to Sample



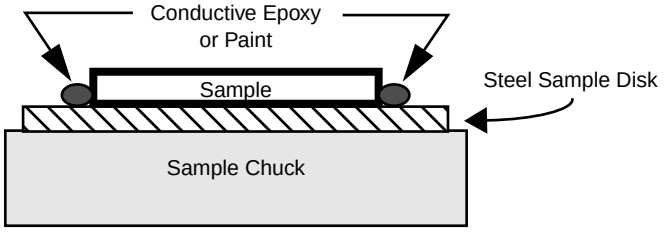
Place a current-limiting resistor (e.g., 10–100 MΩ) in series with the external voltage supply to protect the tip and sample from damage. You may use current-limited power supplies. Connect voltage leads to pins on the header using soldered, push-on connectors.

CAUTION: Do not solder leads directly to the header pins. Heat may cause damage and make jumpering the pins difficult.

Applying Voltage to Sample Through the Sample Chuck continued...

1. You must electrically connect the sample to the chuck. Attach the sample to a standard steel sample disk using conductive epoxy or silver paint (See Figure 16.4d) or hold the disk down on the sample chuck using vacuum or the magnetic mount.

Figure 16.4d Separating Sample from Piezo Cap Using Steel Sample Puck



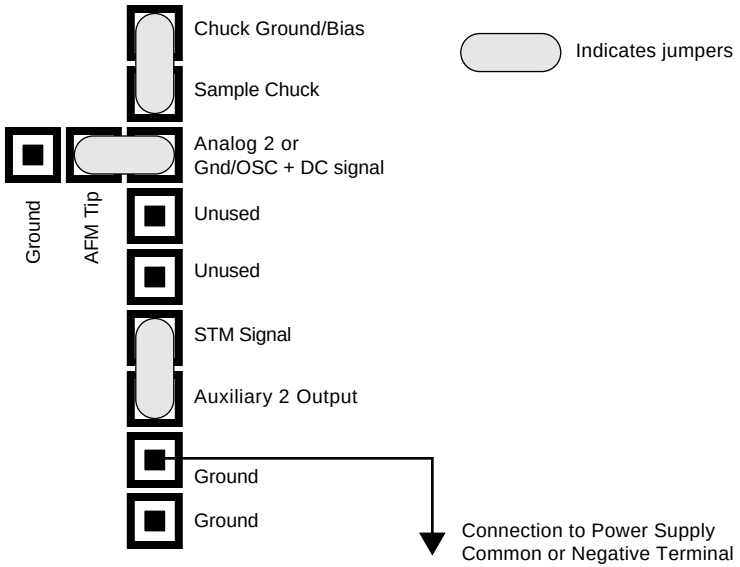
2. Verify that there is a good connection between the sample and the chuck.

Surface Potential Detection Preparation continued...

Applying Voltage to the Sample Directly

If you apply voltage directly to the sample, there is no need to reconfigure the jumpers. The jumpers remain in the same position as shipped from the factory (See Figure 16.4e).

Figure 16.4e Jumper Configuration for Application of Voltage

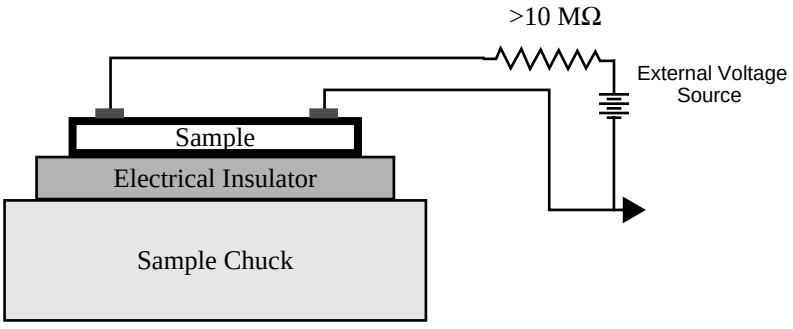


1. The sample must be electrically insulated from the chuck. Connect the external voltage source directly to the sample by attaching fine gauge wire to appropriate contacts (e.g., on integrated circuits, connect electrical leads directly to pads).

2. For normal operation, the sample chuck is held at ground. Insulate any electrical connections from the sample chuck using an electrical insulator (See Figure 16.4f).

Applying Voltage to the Sample Directly continued...

Figure 16.4f Electrical Insulator



Surface Potential Detection continued...

16.4.3 Surface Potential Imaging Procedure

1. Locate the two toggle switches on the backside of the Extender Electronics box (See Figure 16.4g).

2. Verify that they are toggled as shown in Table 16.4a.

Figure 16.4g Toggle Switches on Back of Extender Electronics Module

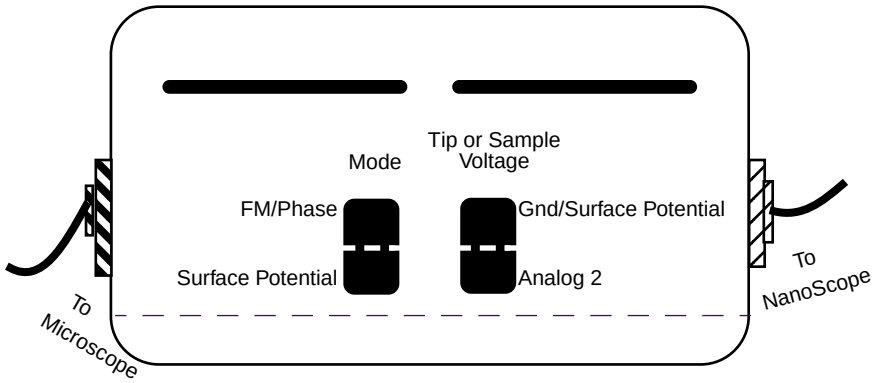


Table 16.4a: Extender Electronics Module Toggle Switch Settings

	Mode		**Tip or Sample Voltage**	
	FM/Phase	**Surface Potential**	**GND/ Surface Potential**	**Analog 2**
Surface Potential		**X**	**X**	
Std. Operation	**X**		**X**	

Note: The toggle switch combination of Surface Potential = ON and Analog2 = ON is **not recommended** and can produce erratic and undefined results.

3. Mount a sample onto the Dimension 3100 stage. Make necessary external electrical connections for the sample.

Surface Potential Detection Procedure continued...

4. Mount a metal-coated NanoProbe cantilever into the cantilever holder.

   Note: MFM-style cantilevers (225 micron long, with resonant frequencies around 70 kHz, Model MESP) typically work well. You may also deposit custom coatings on model FESP silicon Tapping Mode cantilevers.

5. Verify that all deposited metal adheres strongly to the silicon cantilever.

6. Set up the AFM as usual for Tapping Mode operation.

7. Use **Cantilever Tune** and **AutoTune** to locate the cantilever's resonant peak. Two curves should appear in the Cantilever Tune box—the amplitude curve in white and the lock-in curve in yellow.

   Note: In Surface Potential it is important that the resonant peak is symmetric. If the peak is unsatisfactory, you can change its shape by readjusting the position of the cantilever chip in the holder. The laser and photodiode may require readjustment after moving the cantilever.

8. Engage the AFM.

9. Make adjustments to obtain a good Tapping Mode image while displaying height data in Channel 1.

   Note: You must have a good height image before proceeding. Any measurement using LiftMode, including Surface Potential, depends on accurate height information. Go to Scope Mode (**View** / **Scope Mode**) and verify that the Trace (white) and Retrace (yellow) match. There may be an offset between them, but the shape and slopes of features should be the same.

Surface Potential Detection Procedure continued...

10. Under the **Panels** menu, select **Interleave Controls** to view the set of scan parameters used for measuring Surface Potential in the interleaved scan. You may enter different values from those on the main scan for any of the interleaved scan parameters.

11. Set the interleave **Drive frequency**, **Amplitude setpoint** and **SPM Feedback** to the main feedback values (grayed buttons).

12. To start choose a **Drive amplitude** of 6 V.

    Note: The Interleave Controls **Drive amplitude** equals the AC voltage applied to the AFM tip. Higher **Drive amplitudes** produce larger electric forces on the cantilever, resulting in more sensitive potential measurements. Conversely, the maximum total voltage (AC + DC) applied to the tip is ±10 V. A large **Drive amplitude** reduces the range of the DC voltage applied to the cantilever. If the potentials are very large, choose a small **Drive amplitude** (do not use less than 2 V). Small surface potentials are imaged more successfully with large **Drive amplitudes**.

13. Set the **Drive phase** in the **Interleave Controls** panel. For MESP cantilevers (resonant frequency ~60-80khz) enter a **Drive phase** of –90 degrees.

    Note: For Versions 4.23 and lower, you must enter the **Drive phase** in the **Feedback Controls** panel because there is no separate setting available in the **Interleave Controls** panel.

    Note: Cantilevers with significantly different resonant frequencies may require a different phase setting.

14. Choose a **Lift start height** of 0 nm and a **Lift scan height** of 100 nm. You may readjust the **Lift scan height** later.

15. Set the **Input feedback** to **Potential**.

Surface Potential Detection Procedure continued...

16. Set **Interleave mode** to **Lift**.

17. Set the Channel 2 image **Data type** to **Potential** and select **Interleave** for the **Scan line**.

18. For both data channels (height and potential) set the **Scan line** direction to **Retrace**. Choose the retrace direction because the lift step occurs on the trace scan.

    Note: For Versions 4.23 and lower, set **Interleave scan** to **Lift**, set **Interleave mode** to **Enabled**, and switch the **Data type** for Channel 2 to **Potential**.

When the microscope completes a topographic scan line (trace and retrace), the system turns off the drive piezo and switches the oscillator signal to the cantilever. The cantilever is driven electrically according to the interleave **Drive amplitude** selected. Also, a feedback circuit enables in the Extender box to adjust the DC voltage on the tip to maintain the cantilever oscillation amplitude at zero. To do this, the feedback circuit uses the lock-in signal of the cantilever oscillation to keep this value at zero volts. When the cantilever oscillation amplitude returns to zero, the DC voltage on the tip and sample are equal. The NanoScope records the DC voltage applied to the tip and this signal displays in the **Potential** data type.

19. Adjust the **FM gains**.

    Note: The feedback loop used by the Extender Electronics Module for Surface Potential measurements is the same as the one used in Frequency Modulation for magnetic and electric force gradient detection.

20. Select the **Other Controls** panel and set the **FM igain** to **15** and **FM pgain** to **100**.

Surface Potential Detection Procedure continued...

Note: As with topography gains, you can optimize the scan by increasing the gains to maximize feedback response, but not so high that oscillation sets in.

21. Optimize the Lift scan heights by choosing the smallest **Lift scan height** value that does not cause the tip to crash into the sample surface.

Note: If the tip crashes into the surface during Potential measurement, dark or light spots or streaks appear in the Potential image. Increase the **Lift scan height** to minimize these streaks. Because the tip does not oscillate during the Potential measurement (the feedback loop works to keep the amplitude zero), the lift height is generally smaller than with other LiftMode techniques. **Lift scan heights** down to –5nm are possible on smooth samples. Sample roughness, scan speed, and target amplitude used during tuning affects the lower limit of the **Lift scan height**.

16.4.4 Open Loop Operation

At times it is useful to run Potential in the “open-loop” configuration which disables the Potential feedback loop and generates only qualitative data. The AC voltage is applied to the cantilever as in the standard Potential operation (the drive piezo used for mechanical driving of the tip is disabled). Because the feedback is disabled, there is no adjustment of the DC voltage on the tip, so the oscillating electrical forces drives the cantilever into motion. You can monitor this motion by observing the lock-in signal (the input to the feedback loop), called “phase” in the software.

Open Loop Operation continued...

For Open Loop Operation, set up the system as described above in Section 16.4.3 with the following changes:

1. Set the **FM igain** and **FM pgain** to **0**.

   Note: Turning the FM gains to zero stops further changes to the DC voltage on the tip but does not set the tip voltage back to zero.

2. Select **Phase** as the **Data type** for Channel 2.

   Note: For Versions 4.23 and lower, run the Open loop operation before the feedback loop is turned on. Set **Data type** to **Phase** in the Channel 2 panel.

16.5 Troubleshooting

The Surface Potential feedback loop can be unstable. This instability may cause the Potential signal to oscillate or remain at either +10 V or -10 V. Below are suggestions for verifying that the feedback loop is working properly with no oscillation.

1. Go into Scope Mode and look at the Potential signal. If oscillation noise is evident in the signal, reduce the FM gains. If oscillations persist even at very low FM gains, increase the **Lift scan height** or reduce the **Drive amplitude** until oscillation stops.

2. If the tip crashes into the surface, the Lock-in signal unstabilizes and causes the feedback loop to malfunction. To prevent this problem, increase the **Lift height** and reduce the **Drive amplitude**. Once oscillation stops, you may increase the FM gains for improved performance.

3. In Scope Mode, if the Potential signal is perfectly flat and shows no noise even with a small Z-range, the feedback loop is probably stuck at ±10 V. Verify this by changing the value of **Real-time planefit** to **None** in the Channel 2 panel and increasing the **Data scale** to the maximum value (20V). If the scope trace is at one of the limits the potential signal is railed. Common reasons for this include:

    - **Inappropriate Drive phase:** For an MESP cantilever verify that the **Interleave Drive** phase is set to –90 degrees. For Versions 4.23 and lower, set the Feedback Control Drive phase to –90 degrees.
    - **Incorrectly set toggle switches on the Extender Module:** Verify toggle switches are set to Surface Potential and GND/Surface Potential.
    - **Incorrect electrical connection:** Verify that the sample is connected properly to ground or a power supply. Verify that the jumpers in the base of the MultiMode are set properly for your configuration.

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Chapter 17 Calibration

17.1 Overview

This chapter provides detailed instructions for the fine calibration of Digital Instruments Veeco Dimension SPMs. Additionally, the latter part of the chapter focuses on problems commonly encountered during operation of the microscope and then concludes with maintenance procedures for the Dimension SPM.

Specifically this chapter includes the following topics:

17.2 SPM Calibration Overview

Digital Instruments Veeco employs a software-guided calibration procedure for all its 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 Digital Instruments Veeco. Calibration theory, however, is straightforward and consists of the following steps:

1. Scan a calibration reference having surface features of precisely known dimensions.

2. Compare known dimensions on the calibration reference's surface with those estimated by the SPM software.

3. Adjust calibration parameters until the SPM's dimensions accord with the true dimensions of the reference.

The **Capture Calibration** and **Autocalibration** routines are designed to optimize measuring accuracy over the entire measuring range of the scanner. In order to obtain the finest accuracy possible for a given scan size, the scanner calibration parameters can be manually fine-tuned. This may prove useful in applications where measuring accuracy must be better than 1 percent.

Digital Instruments Veeco recommends that you adhere to the following Calibration schedule (See Table 17.2a). After initial installation, perform the **Fine-Tuning X-Y Calibration** (See Section 17.7) 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 17.6). For most applications it is sufficient to perform the Z calibration with the same frequency. For critical height measurements we recommend monthly Z calibration.

> **Note:** After initial execution, only perform the **Full X-Y Calibration Routine** if you find that calibration measurements are more than 10% off at any point during **Fine-Tuning Calibration**. For more advanced users, calculate the simple **Critical Height Measurements** monthly.

SPM Calibration Overview continued...

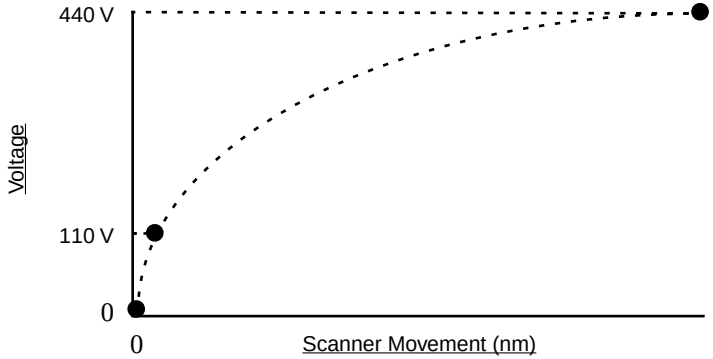
Table 17.2a: Calibration Schedule

Calibration Routine	Time Frame	Frequency
Fine-Tuning Calibration (or Full X-Y Calibration if required)	First Year	Every 3 months
	Subsequent Years	Every 6 months
Z Calibration (for General Applications)	First Year	Every 3 months
	Subsequent Years	Every 6 months
Critical Height Measurements	All Years	Monthly

17.2.1 Theory Behind Calibration

For purposes of calibration, 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 17.2a.

Figure 17.2a Scanner Sensitivity Curve

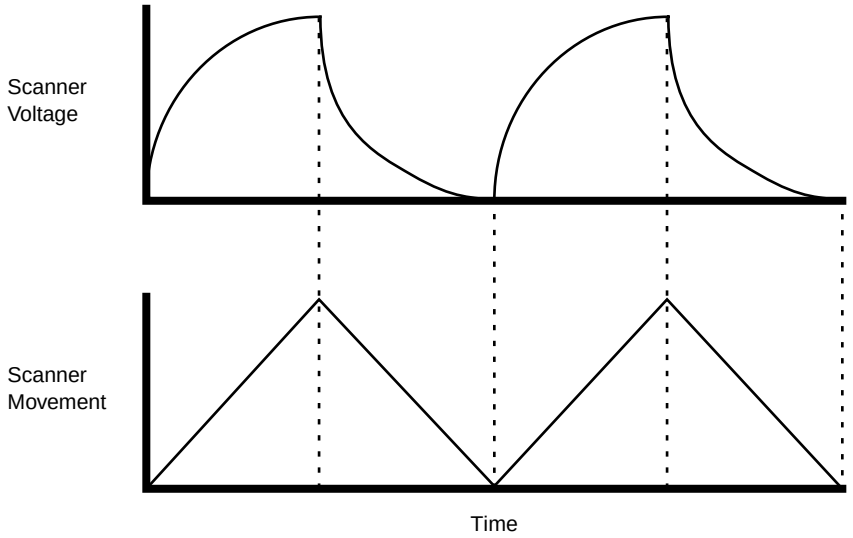


This curve typifies scanner sensitivity across the full range of movement. The vertical axis denotes voltage applied to the scanner. The horizontal axis denotes scanner movement. At higher voltages, the scanner's sensitivity increases (i.e., more movement per voltage applied). At zero volts, the scanner is motionless.

Theory Behind Calibration continued...

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 (See Figure 17.2b).

Figure 17.2b Scanner Voltage and Movement



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 17.2b. 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).

Theory Behind Calibration continued...

Through rigorous quality control of its scanner piezos, Digital Instruments Veeco has achieved excellent modeling of scanner characteristics. Two calibration points are typically used for fine-tuning: at 150 and 440 volts. A third point is assumed at 0 nm/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 the scanner's movements. 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 Section 17.5). Otherwise, calibrations will be undone by the linearity adjustments.

17.3 Sensitivity and Scanner Calibration

ATTENTION: 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.

17.3.1 Scanner Properties

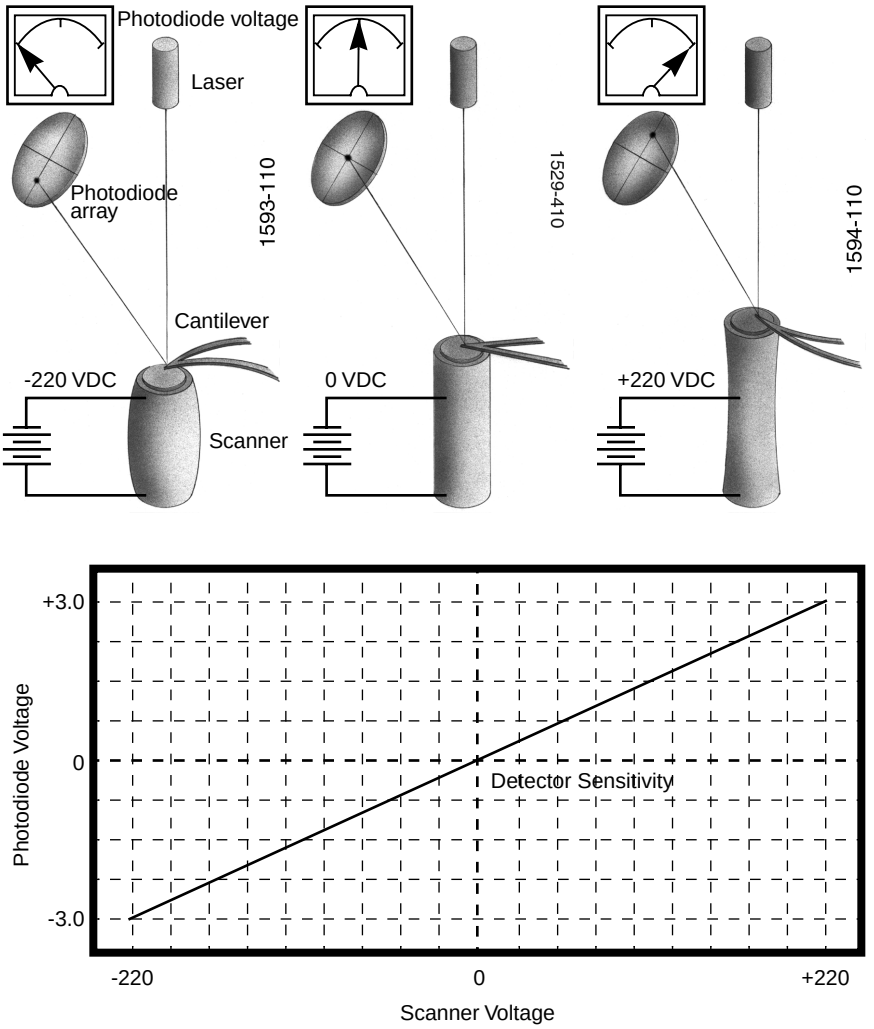
Scanners typically consist of a hollow tube made of piezoelectric material such as PZT (lead zirconium titanate). Piezo materials contract and elongate when voltage is applied, according to whether the voltage is negative or positive, and depending upon the orientation of the material's polarized grain 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 voltage-to-piezo movement. 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.

Scanner Properties continued...

The diagram below depicts scanner crystal voltage versus photodiode voltage (See Figure 17.3a). In this instance, detector sensitivity is given as volt per volt, a parameter provided in the **Force Calibration** screen.

Figure 17.3a Scanner Crystal Voltage and Photodiode Voltage



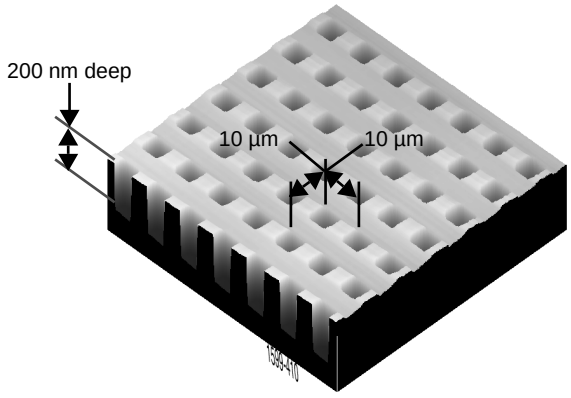
Scanner Properties continued...

The **Microscope / Calibrate / Scanner** 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, other parameters measure the scanner's X-Y coupling and other effects.

17.4 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 17.4a).

Figure 17.4a Silicon Calibration Reference



A calibration reference consists of a silicon substrate having a regular series of pits, each 200 nm deep, which is plated with platinum. Pits are spaced apart on 10 µm 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.

17.5 Small Scan Size Calibration

If using scan sizes of 5 µm or smaller, Digital Instruments Veeco recommends calibrating the scanner for small scan sizes. Contact Digital Instruments Veeco for further instructions.

17.6 Full X-Y Calibration Routine

17.6.1 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 (such as the “J” and “K”).

Check Scanner Parameter Values

1. If the system's original scanner parameters are deleted, copy the scanner parameters from the software CD shipped with every system. 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, fax or call Digital Instruments Veeco for scanner calibration records.

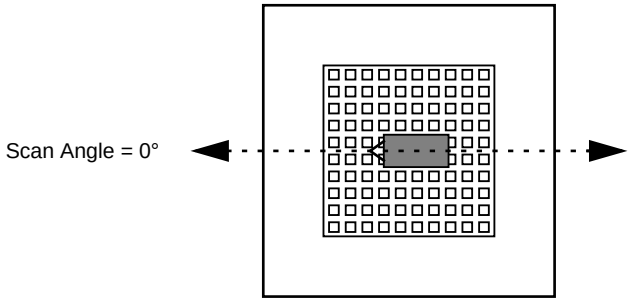
Linearity Correction continued...

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 17.6a).

Figure 17.6a 0 Degrees Scan Angle



3. Use a 1-micron pitch reference for D and E scanners. Use a 10 micron pitch height reference for G, J, and K scanners.

4. Align the reference within approximately 2 degrees of perpendicularity to the scan axes.

Set Real Time 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
	Number of samples	256
	Slow scan axis	Enabled
	Z limit	440 V
Other Controls	Units	Volts
Channel 1	Data type	Height
	Z range	~ 20 V[a]

a. Adjust the **Z range** parameter to obtain the best contrast.

Set Up Contact AFM

1. Set the **AFM 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 Real-time parameters to obtain a good-quality, maximum **Scan size** image. Set the **Scan rate** to **2.44 Hz** and the **Number of samples** parameter to **256**.

4. With the sample engaged, check the scanning line relative to the reference's features; the scan should be orthogonal to the pits on the reference.

Set Up Contact AFM continued...

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.

Check Sample Orthogonality

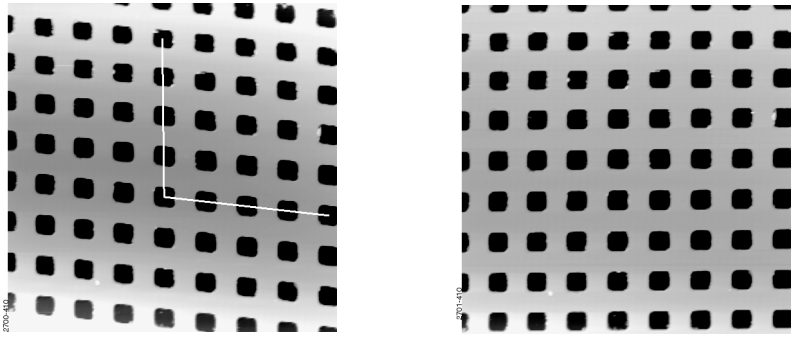
1. Check the sample scan for orthogonality along both the X- and Y-axes.

2. If the scan is aligned along one axis of the scan but not another, adjust the microscope's **Orthogonality** parameter in the **Scanner Calibration** panel.

3. To measure a captured image's orthogonality, view it using the **Off-line / View / Top View** function.

4. On the display monitor, select the **Angle** command and use the mouse to draw a cursor between the edges (or centers) of widely spaced pits (See Figure 17.6b).

Figure 17.6b Non-Orthogonal and Corrected, Orthogonal Image



Check Sample Orthogonality continued...

> **Note:** In Figure 17.6b, 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. The angle displays on the white status bar at the bottom of the display monitor.

5. Use the mouse to drag the cursor until it is oriented correctly then read the angle off the status bar. If the angle differs by more than a half degree, a correction is required; otherwise, move on to the next step.

Adjust Sample Orthogonality

1. Click on **Real Time / Microscope / Calibrate / Scanner** to access the **Scanner Calibration** panel. The **Orthogonality** parameter is displayed on the bottom-right corner of the panel.

2. Enter the difference between 90 degrees and the angle measured in the **Top View** image.

> **Note:** For example, if the angle measured in the **Top View** image was 92.5 degrees, enter a value of **-2.5** degrees in the **Orthogonality** parameter.

3. Click **OK** to exit the **Scanner Calibration** panel

4. Capture another image and re-measure the angle.

5. Repeat correction of **Orthogonality** until the scanned image shows less than 0.5 degrees of error.

> **Note:** After a major change to the orthogonality parameter, you may need to physically realign the calibration standard to the image frame.

Linearity Correction Procedure continued...

Adjust Mag0 and Arg0

1. Select **Microscope / Calibrate / Scanner** to display the **Scanner Calibration** dialog box.

2. 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 display monitor screen itself is not linear, use the Real-time **Zoom** box to check **mag0** and **arg**.

ATTENTION: 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. After engaging, click on **Microscope / Calibrate / Scanner** to open the **Scanner Calibration** window. As parameters values are changed, the effects will be seen on the display monitor.

2. Move the mouse cursor to the display monitor and select **Zoom Out** to produce a box whose size and position can be changed by alternate clicks on the left mouse button.

3. Adjust the box until it is about one-third the size of the scan.

4. Click **once** on the right mouse button to set the box, free the cursor, and move to **Execute**.

   Note: Clicking twice will execute a **Zoom**, which you do not want to do.

5. Select **Fast mag0,** the first scanner parameter to modify.

6. Move the zoom box to the start of the fast scan (on the left if the **Line direction** is set to **Trace**).

Adjust Fast Mag0 continued...

7. 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.

8. Move the zoom box to the end third of the scan.

9. Align one side of the zoom box with desired features and observe how the other side aligns with the features under it.

10. Compare the beginning third of the scan to the features in the end third of the scan. 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.

11. 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.

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.

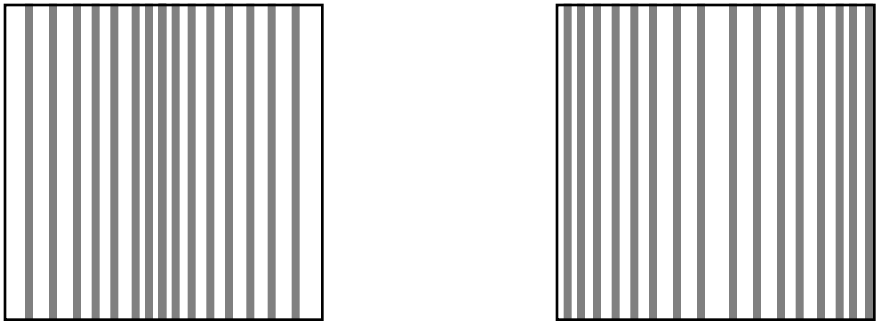
4. After an adjustment in **Fast arg** is made, **Fast mag0** may need readjusting. Repeat **Fast mag0** adjustment procedure until the rulings are evenly spaced across the Fast-axis.

Adjusting Fast Arg continued...

5. If using a one-dimensional reference, repeat the procedure for adjusting **Fast mag1** before proceeding with the next step.

   Note: Adjusting the slow linearity requires the one-dimensional reference to be physically rotated 90 degrees.

Figure 17.6c X Scan Linearization



Center Compressed Center Expanded

6. 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.

Adjusting Slow Mag0 continued...

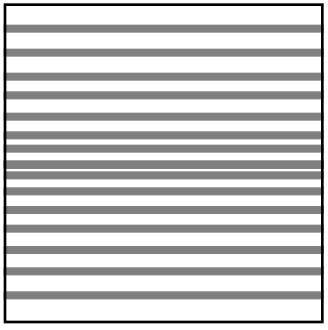
ATTENTION: Be careful not to confuse scan top and bottom with beginning and ending, as the scan direction alternates.

4. Adjust the zoom box to fit the beginning third of the scan and check against the end third.

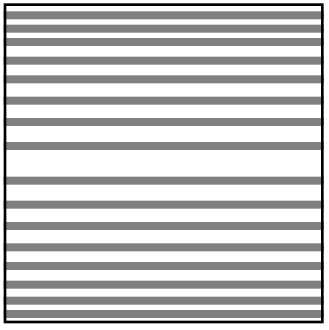
5. If the features of the end third are too large for the box, decrease the parameter. If the features are too small, increase the parameter.

ATTENTION: Compare only parts of the current scan, not the previous scan.

Figure 17.6d Y Scan Linearization



Center Compressed



Center Expanded

Adjusting Slow Arg

1. Follow the same instructions for **Fast mag0**.

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.

Linearity Correction Procedure continued...

Adjust Fast Mag1

1. For initial adjustment, click **OK** to close the **Scanner Calibration** window.

   Note: Selecting **Restore** resets parameters to default values when the box is opened.

2. Change the **Scan size** to **150 volts**.

3. Select **View / Scope Mode**.

4. On the display monitor, select **Dual Trace**. If the two scope traces do not overlap, **Fast mag$_1$** needs adjusting.

5. On the control panel, select **Slow scan axis**. When tall features appear on the scope trace, press the keyboard right or left arrow key to toggle **Slow scan axis** to **Disabled**.

6. Select **Microscope / Calibrate / Scanner** to open the **Scanner Calibration** box.

7. Select **Fast mag1**.

8. Use the left and right arrow keys to change the value until the two traces align.

   Note: The yellow retrace line will shift in the same direction as the arrow.

9. When done, click **OK** to close the **Scanner Calibration** window.

10. Set **Slow scan axi**s back to **Enabled**.

11. Select **View / Image Mode**.

Adjust Fast Mag1 continued...

Initial adjustment is usually adequate; however, if more precision is desired, use the following fine adjustment techniques to adjust **Fast mag1**.

12. Use the same procedure for adjusting Fast mag0.

13. 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.

14. 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**.

Adjust Slow Mag1

1. With the **Scan size** set to **150 volts**, select **Microscope / Calibrate / Scanner**.

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 **Off-line / Modify / Zoom** window. The scanner is now ready for calibration of the X and Y parameters.

Full X-Y Calibration Routine continued...

17.6.2 X-Y Calibration using Capture Calibration

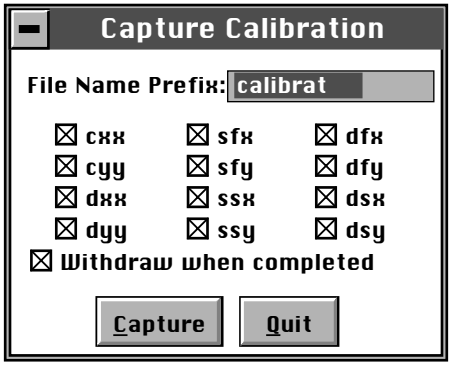
The **Capture Calibration** command calibrates scanners using set parameters in the NanoScope software. The basic calibration procedure using version 4.42 software and a 10-micron calibration reference (See Figure 17.4a) is described below. Note that D and E scanners require a 1-micron cross-ruling.

1. With the **Scan rate** set to **2.44 Hz** and **Number of samples** parameter to **256**, a full **Capture Calibration** requires approximately 70 minutes.

   Note: Increasing the **Number of samples** or decreasing the **Scan rate** significantly increases the required time.

2. Using the mouse, click on **Real-time / Capture / Calibration**. The **Capture Calibration** dialog box displays, listing twelve parameters used in the calibration procedure (See Figure 17.6e).

Figure 17.6e Capture Calibration Prompt



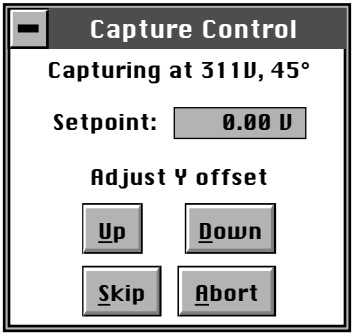
3. If this is a first-time calibration, or if the microscope's calibration has not been checked within the last three months, verify that all parameters are selected.

4. Click on **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.

5. 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 17.6f).

Figure 17.6f 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.

6. If the scan has not settled out by the time the capture status changes to **On**, click on **SKIP** to increment the capture to skip 1, skip 2 or skip 3.

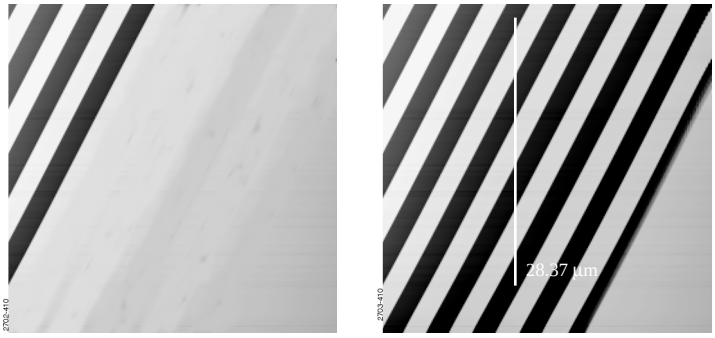
X-Y Calibration Using Capture Calibration continued...

Note: Do not click on **Abort** unless you want to stop the entire **Capture Calibration** program.

7. If portions of features are missing, or if the image is blank, click repeatedly on **ADJUST Y OFFSET / UP** or **DOWN** 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 17.6g).

8. 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.

Figure 17.6g 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.

9. Go to the **Capture** directory where all **Capture Calibration** files are saved.

10. Select **Off-line / File / Browse** or click on **Browse** to review all **Capture Calibration** files.

11. Verify that all calibration images contain features spanning the full width and height of the image frame (See Figure 17.6g).

X-Y Calibration Using Capture Calibration continued...

12. Recapture all images unsuitable for calibration. Record the file name extensions for all unusable files (e.g., .cxy, .dyy), then delete the files.

13. Re-engage the reference surface and select **Capture Calibration**.

14. Verify that the file name prefix is identical to that of the usable files, then deselect all usable file name extensions from the last capture. Click on their respective boxes to remove the X.

15. Finally, click on **CAPTURE** to recapture the selected files.

Full X-Y Calibration Routine continued...

17.6.3 Off-line / Utility / Autocalibration

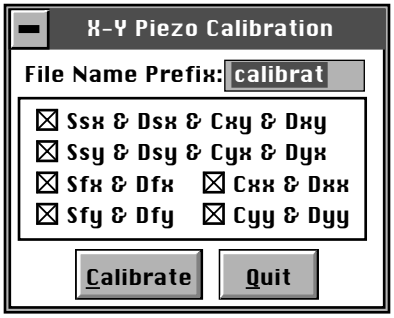
After the **Capture Calibration** routine is completed, the user measures surface features contained within each image and enters their dimensions into the software. The software compares its estimates with the actual (user-entered) dimensions to make final corrections. This portion of calibration is carried out using the **Off-line / Utility / Autocalibration** command.

To utilize the **Off-line / Utility / Autocalibration** command, do the following:

1. Select the desired captured calibration images in the **Capture** directory.

2. Select the **Off-line / Utility / Autocalibration** command. The control monitor will display the **X-Y Piezo Calibration** dialog box (See Figure 17.6h).

Figure 17.6h X-Y Piezo Calibration Prompt



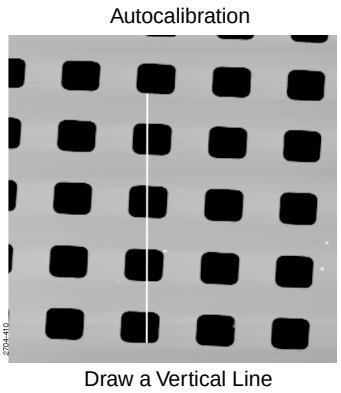
3. Verify that the file name prefix assigned to the captured files from the **Capture Calibration** routine is correct.

4. For normal calibration, verify that all parameters are selected in the dialog box, then click on **Calibrate** to execute the routine.

Note: The software sequentially presents various calibration images on the display monitor while prompting the user to draw either a vertical line or a horizontal line. The control monitor simultaneously displays various dialog boxes (one for each image), requesting the user to enter a distance.

5. Use the mouse to draw a line on the image. Draw the line to span as many features as possible, preferably connecting like edges (See Figure 17.6i).

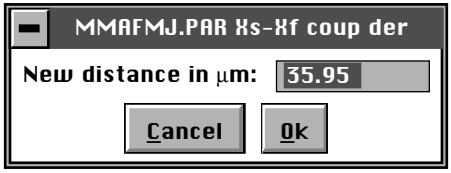
Figure 17.6i Calibration Line



In this example illustrated in Figure 17.6i, a line is drawn from the bottom edge of one feature to the bottom edge of another feature four rows away—a distance of 40 microns. The control monitor simultaneously displays a dialog box for entering the distance indicated by the white line (See Figure 17.6j). The distance displayed in the box (in this example, 35.95 µm) is the software's estimate of the length of the line drawn on the image based on current calibration values. If the line length is different from the value shown, change the value to reflect the correct line length. (In this example, the user would enter a value of **40**.)

Off-line/Utility/Autocalibration continued...

Figure 17.6j Calibration Line Distance Prompt



6. Enter the distance covered by the white line drawn on the image. If a 10-micron reference is employed, like portions of features are spaced 10 microns apart (e.g., between bottom edges, left sides, etc.).

ATTENTION: Measure features without regard to how they appear in calibration images. Features are represented with stretched, distorted, or angled appearances due to the unusual movements employed during **Capture Calibration** scanning. Regardless, features are separated by the same (e.g., 10-micron) spacings.

7. Continue drawing lines and entering measured distances until all **Capture Calibration** images are measured. When the software is finished, it prompts the user that it is done.

If **Capture Calibration** and **Autocalibration** routines are completed correctly, the SPM is 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 measuring accuracy. This is accomplished through the use of calibration parameters discussed in Section 17.7.

17.7 Fine-tuning for X-Y Calibration

Fine-tuning is usually performed at two **Scan size** settings: **150** and **440** volts. 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.

> **Note:** If you are using an A scanner to image atomic-scale features, substitute graphite or mica for the silicon calibration reference.

17.7.1 Prepare System for Fine-tuning

1. Set the **Scan size** parameter on the **Scan Controls** panel to the maximum value (440 volts).
2. Verify that the **Scan angle** is set at **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.**

17.7.2 Measure Horizontally at 440V Scan Size

1. Set the **Scan size** parameter on the **Scan Controls** panel to the maximum value (**440** volts).
2. Verify that the **Scan angle** is set to **0.00** degrees.
3. **Engage** the surface.

Measure Horizontally at 440 Scan Size continued...

4. Select two widely-spaced features on the sample image of known separation. Use the mouse to draw a horizontal line between them. (For example, on a 10-micron, 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.

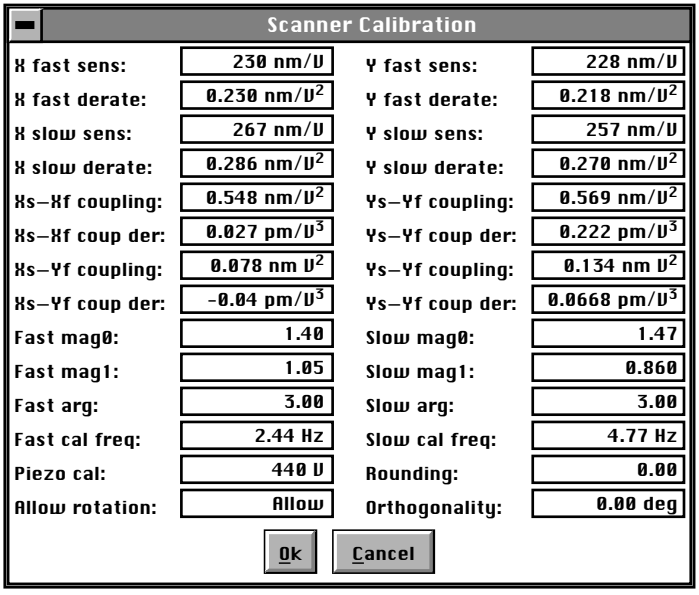
5. Verify that the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to Section 17.7.4.

6. Based upon the results in the step above, 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 **Real time / Microscope / Calibrate / Scanner** option. The **Scanner Calibration** dialog box displays (See Figure 17.7a).

Figure 17.7a Scanner Calibration Panel



Measure Horizontally at 440 Scan Size continued...

8. Multiply the quotient obtained above by the **X fast sens** value shown on the **Scanner Calibration** panel.

9. 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.

10. To save it to the computer's hard disk, click on **OK**. This closes the **Scanner Calibration** panel.

17.7.3 Measure Vertically at 440V Scan Size

1. Return to the image of the calibration reference.

2. Clear the horizontal line drawn earlier and click the right mouse button, or click on **CLEAR**.

3. Draw a vertical line between features.

4. Wait for at least three full scans to allow the piezo to stabilize then select two widely spaced features and draw a line connecting like portions of features (top edge-to-top edge, etc.). The SPM displays the calculated distance between features.

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 Section 17.7.4.

6. Based upon the results from the step above, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

Measure Vertically at 440 Scan Size continued...

7. Select the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box.

8. Select the **Y slow sens** parameter.

9. Record the **Y slow sens** value shown on the **Scanner Calibration** panel and multiply the quotient obtained earlier by the **Y slow sens** value shown on the **Scanner Calibration** panel.

10. Enter the new value to adjust the scanner's slow axis to more closely match calculated distances with actual feature distances.

11. To save the new parameter value, click **OK**.

17.7.4 Measure Horizontally at 150 V Scan Size

1. Set the **Scan size** parameter on the **Scan Controls** panel to one-third the maximum (**150** volts).

2. Verify that the **Scan angle** is set to **0.00** degrees, and that **Units** (**Other Controls** panel) is set to **Volts**.

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 10-micron, 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 will be required; go to the next step. If the displayed distance agrees with the known distance, skip to Section 17.7.5.

6. Perform fine-tuning adjustments using either trial and error or calculate the precise correction (See Calculation Method).

Measure Horizontally at 150V Scan Size continued...

Trial and Error Method

1. Select the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box.

2. Select the **X fast derate** parameter or **Y fast derate** for Y-axis adjustment.

3. If the measured distance is less than the actual distance, decrease the **X fast derate** parameter slightly or **Y fast derate** for Y-axis adjustment and re-measure image features.

4. Adjust deratings up or down until measurements accord with known feature distances.

Calculation Method

1. Perform the following calculation where *s* is the **Sens** value; *a* is the actual distance; *d* is the derating value; *m* is the measured distance; and, *v* is the **Scan size** in volts:

$$\frac{s - \left(\frac{a}{m} \cdot [s - d(440 - v)]\right)}{440 - v}$$

2. Select the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box.

3. Select the **X fast derate** parameter.

4. Record the **X fast derate** value shown on the **Scanner Calibration** panel and multiply the value obtained in an earlier step by the **X fast derate** value shown on the **Scanner Calibration** panel.

5. Reenter the new **X fast derate** value to adjust the scanner's fast axis to more closely match calculated distances with actual feature distances.

6. To set the new parameter value, click **OK**.

Fine-tuning for X-Y Measuring Accuracy continued...

17.7.5 Measure Vertically at 150 V 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 10-micron, 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. If the displayed distance does not agree with the known distance, perform the calculation illustrated above to make a correction.

5. Select the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box.

6. Select the **Y slow derate** parameter.

7. Record the **Y slow derate** value shown on the **Scanner Calibration** panel and multiply the value obtained in an earlier step by the **Y slow derate** value shown on the **Scanner Calibration** panel.

8. Enter the new value to adjust the scanner's slow axis to more closely match calculated distances with actual feature distances.

9. To set the new parameter value, click on **OK**.

Fine-tuning for X-Y Measuring Accuracy continued...

17.7.6 Change Scan angle and Repeat Calibration Routines

1. Change the **Scan angle** on the **Scan Controls** panel to **90** degrees.

2. 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 both the X- and Y-axis.

17.8 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 Digital Instruments Veeco have 200 nm 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 metrology head employed with a Digital Instruments Veeco Dimension Series microscope.

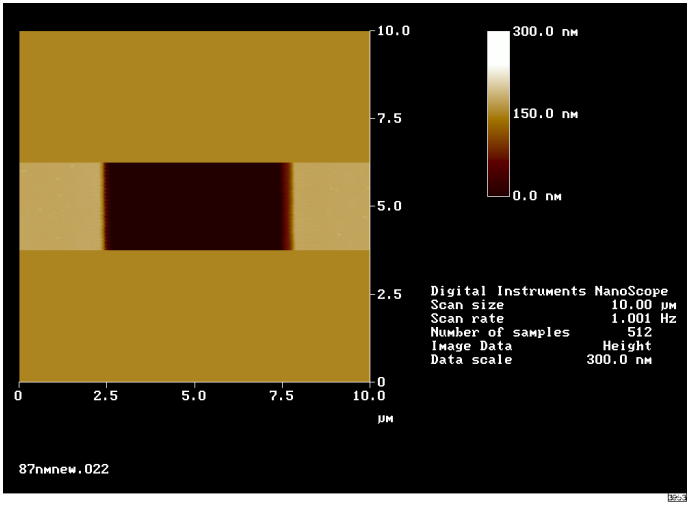
ATTENTION: Refer to the label on your calibration reference sample to verify the measurement employed is 200 nm. Older systems may have samples with a different Z value.

17.8.1 Engage

1. Set up the microscope for **Tapping Mode** imaging.
2. Select **Engage** under the **Motor** pop-down menu or click on the **ENGAGE** icon.
3. Find a square pit and center the pit in the image using a **Scan size** of approximately **10 µm**.

4. Change the aspect ratio to 4:1, and verify that the image includes the pit along with portions of the surrounding flat area (See Figure 17.8a).

Figure 17.8a Z Calibration Image



5. Verify that the **Z Center Position** value shown next to the image display is close to **0 volts (**±5 volts).

6. If the **Z Center Position** value is non-zero, use the **Real-time / Motor / Tip Up** and **Tip Down** buttons to adjust.

17.8.2 Capture and Correct an Image

1. Capture an image by selecting **Capture** in the **Real-time** menu, or click on the **CAPTURE** icon. When the image is captured, go to the **Off-line** screen.

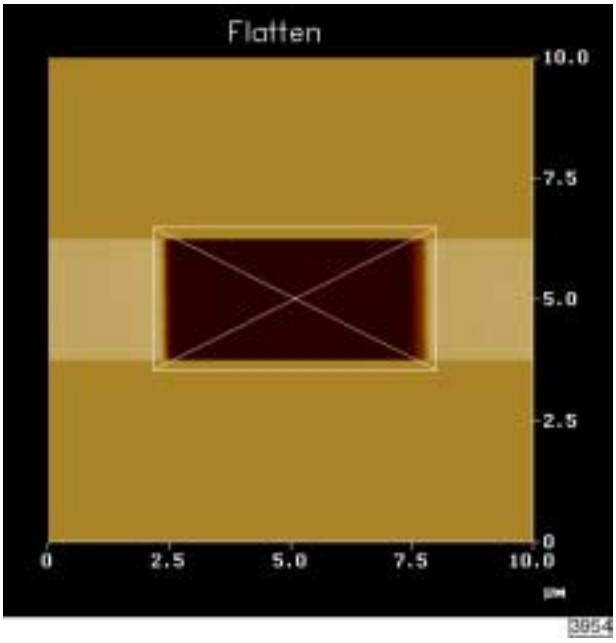
2. Remove all tilt and scan line errata from the image before continuing by selecting **Off-line / Modify / Flatten Manual**.

3. Set the **Flatten order** parameter in the dialog box to **1**.

Capture and Correct and Image continued...

4. Go to the display screen and draw a stopband over the pit as shown in Figure 17.8b.

Figure 17.8b Draw a Stopband



5. Click on **Execute** to complete the flattening procedure.

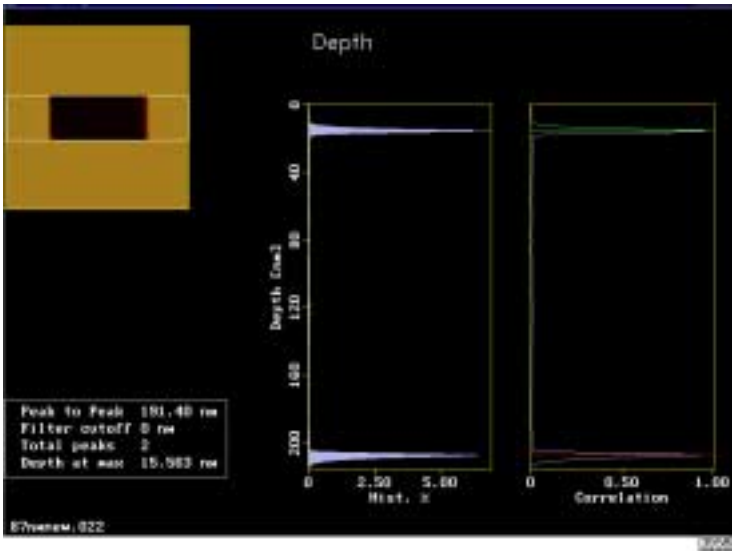
6. **Quit** the dialog box.

17.8.3 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.

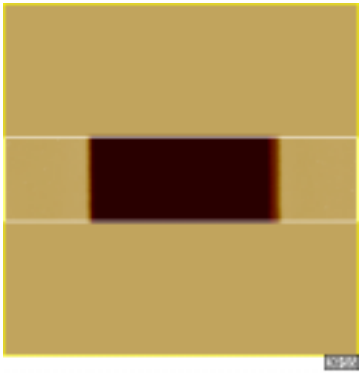
1. Select the **Off-line / Analyze / Depth** command.

Figure 17.8c Depth Analysis Screen



2. Go to the display screen and draw a cursor box surrounding the entire image (See Figure 17.8d).

Figure 17.8d Draw a Cursor Box



Measure Vertical Features continued...

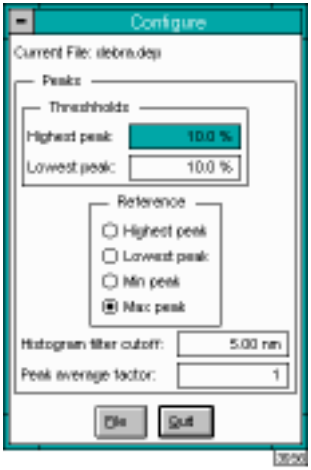
3. Click on **EXECUTE** in the display monitor's top menu bar.

   Note: Height data within the drawn cursor box displays on the monitor, showing two, prominent peaks. These peaks correspond to two elevations on the surface: the bottom of each 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 **Configure** dialog box to **2-5 nm**.

5. Open the **Configure** dialog box by clicking on the **CONFIGURE** button in the **Depth** dialog box (See Figure 17.8e).

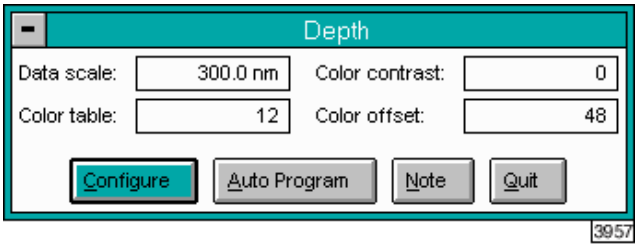
Figure 17.8e Z Calibration Configure Dialog Box



6. In the **Data result** dialog box located under the image on the display screen, check the **Peak to Peak** value and record it for future reference.

7. Click on **QUIT** to exit the **Depth** dialog box**.**

Figure 17.8f Z Calibration Depth Dialog Box



17.8.4 Correct Z Sensitivity

If the depth of the pit on the 10-micron silicon calibration reference deviates significantly from 200 nm, correct the **Z sensitivity** parameter in the **Z Calibration** dialog box.

1. Transfer to the **Z Calibration** dialog box by selecting **Real-time / Microscope / Calibrate / Z**.

2. Write the value indicated in the **Z sensitivity** parameter and divide the actual depth of features (200 nm for the 10-micron calibration reference) by the measured depth (indicated in **Depth** analysis by the **Peak to Peak** value):

$$\frac{200\ \text{nm}}{\text{Peak to Peak value}}$$

3. Multiply this quotient by the **Z sensitivity** value in the **Z Calibration** dialog box, and replace the value with the result.

4. Click on **OK** to enter the new **Z sensitivity** value.

Note: The numerator value above (200 nm) is for Digital Instruments Veeco 10-micron silicon reference. For other calibration references, set the numerator equal to the depth of features measured by **Depth** analysis. Ideally, calibration references should have features with heights comparable to those being imaged and measured on samples.

Correct Z Sensitivity continued...

17.8.5 Recheck Z-axis Measuring Accuracy

1. 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%.

17.8.6 Calculate Retracted and Extended Offset Deratings

Piezoelectric materials exhibit greater sensitivity at higher voltages. In the steps outlined above, the Z-axis calibrates while scanning near the middle of its voltage range (i.e., **Z Center Position ~ 0 V**). In this section, the Z-axis piezo calibrates while extended and retracted to offset the increased sensitivity.

1. Select **Engage** under the **Motor** pop-down menu or click on the **ENGAGE** icon.

2. Use the **Real-time / Motor / Tip Up** button until the **Z Center Position** reads **100 V (**±5 volts).

   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 to determine the measured depth of the calibration standard with a 100 volt **Z Center Position**.

4. Record the measured depth. If the depth measured by the extended piezo is off by more than two percent, adjust accordingly.

   Note: The measured depth should read 200 nm on a Digital Instruments Veeco 10 µm silicon calibration reference.

5. Select the **Real-time / Microscope / Calibrate / Z** option to display the **Z Calibration** panel

6. Click on the **Extended offset der** parameter.

Calculate Retracted and Extended Offset Deratings continued...

7. Perform the following calculation:

$$(1 + \text{current offset der}) \frac{200\ \text{nm}}{\text{meas. depth}} - 1$$

For example, if the current offset equals 4% and the measured depth equals 175 nm, then:

$$(1 + .04) \frac{200\ \text{nm}}{175\ \text{nm}} - 1 = 0.19$$

8. Enter the percent value for the **External offset derating%** menu item.

> **Note:** 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** by 100 V.

9. Repeat the steps above using the **Motor Control** panel and the **Tip Down** button to retract the piezo to a **Z Center Position** of **-100 Volts**.

Chapter 18 Maintenance and Troubleshooting

18.1 Overview

Few maintenance procedures should be necessary for the continued operation of the Dimension SPM microscope. Procedures include cleaning and adjusting angular alignment of the reflecting mirror for the laser beam.

Specifically, this chapter details the following:

- **Overview:** Section 18.1
- **Maintenance:** Section 18.2
- **Troubleshooting:** Section 18.3

18.2 Maintenance

18.2.1 Returning Components for Repair

None of the D3100 SPM components are customer serviceable. If you experience trouble with any component of the instrument, please review the troubleshooting section of this manual, then call Digital Instruments Veeco. If the Dimension head is in need of repair, return the head to Digital Instruments Veeco. Call to obtain a Return Materials Authorization (RMA) and pack the components in the original box used for shipping the head. Return other components in adequately strong boxes with at least four inches of foam peanuts cushioning each bubble-wrapped component. Ship return items directly to Digital Instruments Veeco.

Digital Instruments Veeco
112 Robin Hill Road
Santa Barbara, CA 93117
(805) 967-2700

18.2.2 Cleaning the Cantilever Holder

The cantilever holder is fragile. Gently wipe the cantilever holder clean. Digital Instruments Veeco recommends a camera lens cleaning kit. The rest of the microscope requires only an occasional dry wipe.

18.2.3 Cleaning the Cantilever Holder Contacts

Periodically clean the electrical contacts for the Tapping Mode cantilever holder using a Q-tip dipped in alcohol and a slight scrubbing motion to both the cantilever holder sockets and the pins on the end of the scanner. Perform this procedure every three-to-four months, or whenever the contacts are contaminated.

18.2.4 Clean the Sample Holder and X-Y Stage Surfaces

To maintain proper operation of the X-Y Sample Stage, clean the top surface of the X-Y chuck base and both sides of the sample chuck weekly with a lint-free wipe soaked in alcohol.

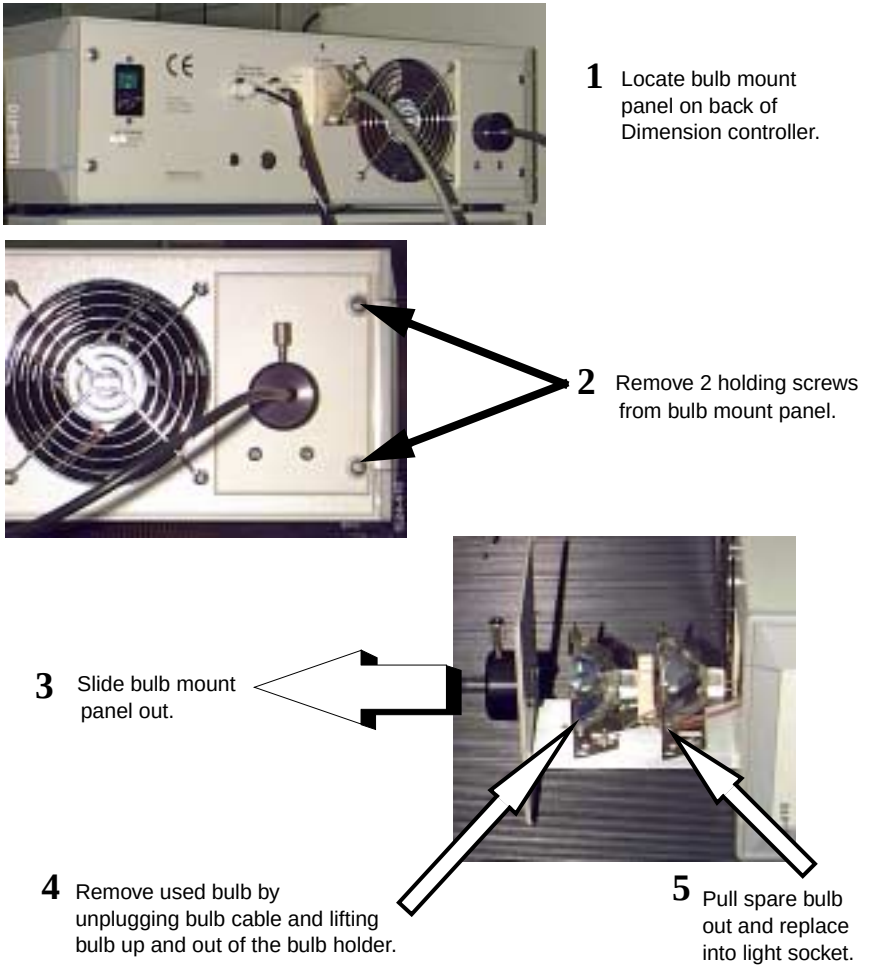
Maintenance continued...

18.2.5 Changing the Illuminator Light Bulb

ATTENTION: Illuminator light bulb is the only user serviceable part inside the Dimension control box.

Change the illuminator light bulb when it burns out. Dimension 3100 systems ship with a spare light bulb mounted within the Dimension control box back panel. Order a new illuminator bulb from Digital Instruments Veeco when the spare bulb burns out, as availability and shipping time may vary.

Figure 18.2a Change the Illuminator Bulb



Changing the Illuminator Light Bulb continued...

1. Turn the Dimension control box off and unplug it from a power source.

2. Locate the bulb mount panel on the back of the control box. Allow the system to cool before attempting to open the bulb mount panel.

3. Unscrew the two holding screws.

4. Slide the bulb mounting panel out.

5. Remove the used bulb by unplugging the bulb cable and lifting the bulb up and out of the bulb holder.

6. Pull the spare bulb (back bulb) out of the mount and place into the holder.

7. Plug in the light bulb cable.

CAUTION: Do not touch the inside of the bulb.

8. Slide the bulb mount panel back and replace the holding screws.

9. Order another spare bulb from Digital Instruments Veeco.

18.3 Troubleshooting

18.3.1 Software Main Menu Items

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

Scan Size, X-Y Offset

These controls adjust the lateral scan area and the center of the scan area.

Scan Angle

Mixes the X and Y drive voltages, causing the piezo to scan the sample or tip at varying X-Y angles.

Scan Rate

The number of lines scanned per second in the fast scan (X) direction.

Number of Samples

Sets the number of pixels displayed per line and the number of lines scanned per frame.

Slow Scan Axis

Starts and stops the slow scan (Y). This control checks for lateral mechanical drift in the microscope or assist in tuning the feedback gains.

Note: 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 to drive an amplifier capable of outputting voltages from +220V to -220V. 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 D or J scanner if scanning samples with relatively small Z features (less than 200 nm peak-to-valley). For example, setting the Z limit to 55V means that 55 volts divided by the same 16-bit digital control gives eight times finer control over the Z direction of the scanner.

Integral Gain and Proportional Gain

The Integral and Proportional gains control the response time of the feedback loop. The feedback loop keeps the output of the SPM equal to the setpoint reference chosen by moving the piezo in Z to keep the SPM output on track with 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 which causes the computer to think that the SPM output is further away from the setpoint reference than it is in reality. The computer overcompensates for this error by sending a larger voltage to the Z piezo than is truly needed. This causes the piezo scanner to move faster in Z to compensate for the mechanical hysteresis of the piezo element. The effect is smoothed out due to the fact that the piezo adjusts up to four times the rate of the display rate.

The displayed 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 tune the feedback response to the particular sample topology. This sets the response time of the system so that there is no difference between the SPM signal and the setpoint reference during scanning.

- **Proportional Gain**

  The computer multiplies this number times the value read from the comparison circuit every time the A/D converter is read. It is the high frequency feedback control.

Software Main Menu Items continued...

- **Integral Gain**

  The computer multiplies this number times an accumulated average of A/D readings. This is the low frequency feedback control.

The easiest way to set the gains properly is to view the input of the feedback loop. Display the STM current, the AFM deflection, or the TMAFM amplitude signal and raise the gain values until the input of the feedback minimizes.

> **Note:** This results in an image that shows only large transitions in Z; this is normal. There is always a time lag between the input and the output (Height data) of the feedback loop.

Typically, the Integral gain is the most sensitive control. Raise the gains together until the input signal (current, deflection or amplitude) is minimum. Do not set them so high as to cause distortions in the image. Distortions are an indication of too much feedback correction voltage sent to Z. This is generally known as feedback oscillation. Although not required, you can set the Proportional gain about 1-2 times the value of the Integral gain.

User Example

Try this experiment with an easy sample, such as a diffraction grating or the calibration standard supplied with the system.

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

2. Engage the microscope and reduce the gains until they are close to zero. The input display data becomes larger in Z, and the Height data blurs or smears. Raise the gains using the arrow keys until the input voltage is minimized.

3. Try increasing and decreasing the scan rate parameter. This increases or decreases the traveling velocity of the tip. You must increase the gain settings at faster scan speeds and decrease the gains at slower scan speeds.

18.3.2 Contact Mode AFM

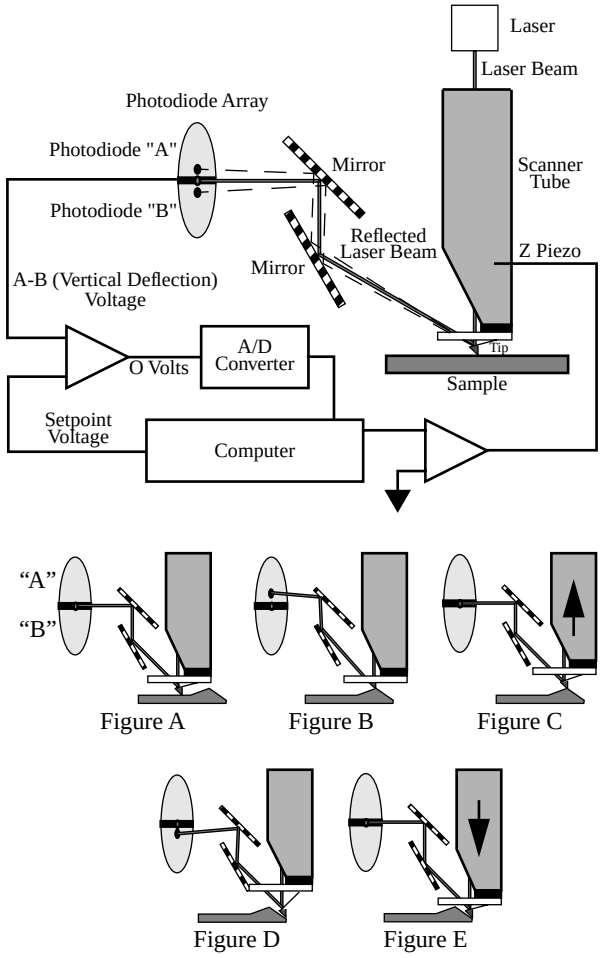
General Operating Concepts

The AFM system is comprised of two main components: 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 that generates a spot of light that reflects off of a microfabricated cantilever onto a mirror and finally into a pair of photodiodes (See Figure 18.3a). The circuitry that generates a voltage from the difference between the two photodiodes (A-B) determines the position of the laser spot. The circuit outputs a voltage ranging from +10V to -10V depending on the position of the spot on the two photodiodes.

The AFM system keeps the tip in contact with the sample surface. The sample scans 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. The feedback loop reads the position change. The feedback loop moves the sample in Z to restore the spot to its original position. Refer to Figure 18.3a.

In Figure A, the tip scans a flat portion of the sample surface left-to-right, maintaining the laser beam at the center of the photodiode array. As the tip encounters a raised feature in Figure B, the cantilever pushes up, deflecting the laser beam upward onto the A portion of the array. The A photodiode voltage increases as it receives an increased portion of the laser light, while the B photodiode voltage decreases (A > B). In Figure C, the feedback electronics sense the Vertical Deflection (A-B) voltage differential, causing a dropped voltage to the Z piezo crystal and the piezo to retract. As the Z piezo retracts, the cantilever recenters the laser beam onto the photodiode array (A = B). As the tip encounters a decline in the sample topology in Figure D, 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 the A photodiode voltage decreases (A < B). In Figure E, the feedback electronics senses the Vertical Deflection (A-B) voltage differential, increasing voltage to the Z piezo crystal and causing the piezo to extend. As the Z piezo extends, the tip pushes down until the laser beam recenters on the photodiode array (A = B).

Figure 18.3a Contact Mode AFM Concepts



The AFM first engages in the repulsive region of its operating range. In other words, the cantilever must exert a positive pressure on the sample surface. The AFM block diagram shows the relationship between cantilever movement and the laser spot on the photodiode array. The diagram shows that the spot moves up (more on A) when the cantilever is pushed up. The initial setup is to have the **Vertical Deflection** (A-B) voltage about **2-3 volts** more negative than the **Setpoint** voltage. Digital Instruments Veeco recommends starting with the **Setpoint** voltage set to **0 volts** and the **Vertical Deflection** (A-B) set to **-2 volts** because **0 volts** designates the middle of the control range. A distinct jump of about **1V** from the **Vertical Deflection** (A-B) voltage to the **Setpoint** voltage indicates a good engagement.

18.3.3 Problems with Contact Mode AFM

False engagement

False engagement occurs up to 50 percent of the time. 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 the reflective gold coating on the cantilever, allowing some of the laser spot through and onto the sample surface, or 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 with large steps or topography. These effects scatter light 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 lowers to the sample during engagement.

If a false engagement occurs, you can detect it easily by adjusting the **Setpoint** and observing the **Z Center Position** change. Increasing the **Setpoint** voltage by **1 volt** causes the **Z Center Position** to change by **1-5 volts**. If the **Z Center Position** changes by a large amount (10—20 volts), the system has falsely engaged.

Watch the **Vertical Deflection** (A-B) differential voltage while the tip engages. The voltage should ideally jump (not drift slowly) from a negative value to the setpoint voltage. If the voltage drifts slowly this indicates that reflected light is affecting the photodiode voltage. If this is the case, abort the engagement process with the **Esc** key. Adjust the photodiode position to make the **Vertical Deflection** voltage more negative than the setpoint voltage before trying the engage sequence again.

Problems with Contact Mode AFM continued...

Other sources of false engagement are:

- Photodiodes are adjusted such that the **Vertical Deflection** (A-B) voltage is more positive than the setpoint voltage. Use the photodiode adjustment knobs to adjust the **Vertical Deflection** voltage to a more negative value.
- The optical alignment of laser spot on cantilever is incorrect. This alignment causes more laser light to reflect off the sample.
- The sample has a region on it that touches the cantilever before the tip does.
- Foreign material stuck on the cantilever beam is lower than the tip.
- Discontinuity in the microscope signals. Check the cabling between the computer and the controller and between the controller and the microscope.
- The **Setpoint** is set more negative than the **Vertical Deflection** (A-B) voltage (this applies only to Contact Mode AFM). This false engagement is immediate. The computer does not show any motor travel or time delay after executing the **Engage** command. Select **Withdraw** and verify that the **Vertical Deflection** voltage reads a voltage more negative than the setpoint voltage.

Head Does Not Engage

In addition to false engagement, the system may not engage at all. Some common causes of this problem include:

- The tip begins too far from the sample. Check the **Stage / SPM Parameters** settings, then repeat the engagement procedure.
- Laser misalignment or the cantilever is damaged. Replace the cantilever substrate, realign the laser, and repeat the engagement procedure.

Problems with Contact Mode AFM continued...

Displacement of Material

Too much tracking force typically causes displacement of material. Use the **Force Calibration** window after engaging for setting the correct tracking force.

To operate in the attractive region of the force curve, tune the AFM. This takes advantage of the fluid layer that captures and holds the tip to the sample, effectively reducing the tracking force. Be aware that the tip is pulling away from the sample. This operating condition may cause the tip to pop off of the sample surface.

Lines in the Image

Lines in the image are typically caused by:

- **AFM tip picking up contamination:** The tip becomes longer and causes the feedback loop to raise the tip to keep the same tracking force. The contamination may fall off the tip and cause another level shift in the image. This condition appears as large bands in the captured image.
- **Friction:** Some samples have a stronger frictional interaction with the tip than others. The cantilever bends and straightens due to the tip sticking and slipping as it drags 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 invert from each other if the friction is high enough. Use the scope image mode in dual trace display to verify that the trace and retrace directions are close to each other. Trace and retrace can invert if there is friction present between the tip and the sample surface.

Problems with Contact Mode AFM continued...

Problems with Silicon Nitride Cantilevers

Silicon nitride cantilevers may 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 engages the sample. Warping or torquing of the cantilever is associated with older cantilever wafers. If engagement is unsuccessful the first time, try changing the actual lever used. Usually, the wide 200 µm-long lever has the most nitride 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) experience difficulty with the laser optics due to laser beam spillage over the side of the cantilever. This effect is more pronounced for highly reflective substrates.

Using a microscope with an interferometric objective lens, it is possible to observe five or more contour lines following the length of the legs of the cantilever on a warped cantilever probe. Cantilever probes that are not warped will have contour lines parallel to the substrate edge. Nikon sells interferometric objective lenses for their Optophot and similar microscopes (such as the MPlan 40(x) DI 0.5 210/0). This is a recommended lens for observing contour lines on the cantilever for diagnostic purposes. Use a bandpass color filter with this lens. Please consult Nikon for further information.

Incorrect Image Vertical Dimensions

When invoking the **Highpass filter**, height information is not accurate. The **Highpass filter** removes the DC component of scan information. This invalidates the height information in the image.

Calculate the **Sensitivity** (See Chapter 13) or deflection data will be inaccurate.

> **Note:** Set the gains low to reduce movement of the Z piezo when acquiring deflection data used in height measurements.

Problems with Contact Mode AFM continued...

Z Center Position Goes Out of Range

The Z center voltage is a measure of the average voltage to the Z electrode. The image disappears when the Z center position reaches either the fully extended or the fully retracted ends of the Z center indicator. The following conditions may cause the Z center positions to go out of range:

- Z limit too low
- Tilted sample (the sample must be as level as possible, particularly for larger scans)
- Mechanical drift causes the sample-to-tip distance to change slowly, bringing them apart completely, or too close together (if this is the case, the Z center shows either +220 [extended] or -220 [retracted], respectively)
- Drift in the optical path

Problems with Contact Mode AFM continued...

Differentiating between optical path and mechanical drift is the first step in eliminating this problem. The force curve is useful 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 important. Watch the force curve and determine whether the curve drifts vertically or horizontally. Vertical drift is indicative of optical path drift, while horizontal drift is due to a mechanical change in tip-to-sample separation.

Use the following as first steps in correcting drift:

1. Verify that the sample, tip and stage are stabilized. There should be no free movement between any of these components
2. Verify that the cantilever is securely in place and that there are no dirt particles wedged underneath it. There may still be some drift. Use the **Tip Up** and **Tip Down** command to bring the sample back into range.

3. Check for thermal stability. Do not place the SPM directly in the path of heating or air conditioning ducts. Avoid locating the SPM near large windows which trap solar heat.

   Note: Thermally caused drift due to thermal expansion of SPM components is the most common cause of mechanical drift.

18.3.4 Tapping Mode AFM

General Operating Concepts

One advantage of Tapping Mode AFM is the absence of lateral forces which exert torque on the cantilever. Unlike traditional Contact Mode AFM, the feedback loop keeps a vibrating cantilever at a constant amplitude, rather than keeping a cantilever at a constant deflection. The tip on the cantilever modulates through mechanical excitation at its resonance. A laser beam reflects off of a microfabricated cantilever onto a mirror, then reflects onto a photodiode array. The laser spot oscillates vertically across the array as a result of the vibrating cantilever. The signal from the photodiodes rectifies, then lowpass filters into a DC voltage (**RMS Ampl.**). The magnitude of 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 amount of cantilever movement. The sample surface is in close proximity to the cantilever. The distance is such that the tip touches the surface only at the lowest point of its oscillation. The RMS voltage reduces to the setpoint voltage when the feedback loop moves the tip into the sample. The sample restricts cantilever movement until the desired RMS voltage is reached. The damping of the cantilever is held constant by moving the tip in Z as it is simultaneously translated in X and Y.

Engaging in Tapping Mode AFM requires the setpoint voltage to be smaller than the RMS voltage. The tip lowers until the RMS reaches the setpoint.

Tapping Mode AFM continued...

Figure 18.3b illustrates the relationship between the RMS and the setpoint voltage during the engage cycle. The computer, not the user, determines the initial setpoint voltage. The computer sets the setpoint equal to 95 percent of the RMS amplitude. The tip then lowers until the RMS matches the setpoint. The computer tests for true engagement as follows:

1. The motor halts the tip's descent.

2. The setpoint lowers slightly.

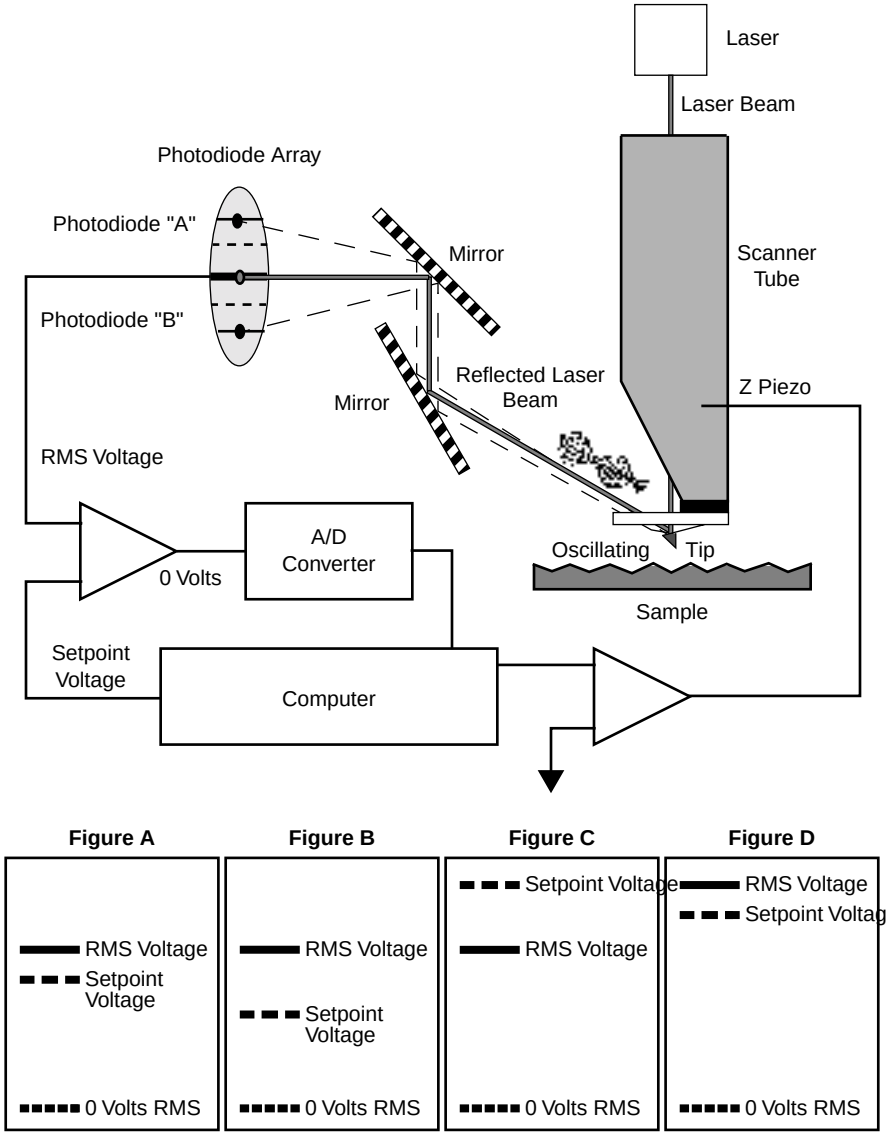
3. The feedback control monitors movement of the Z piezo. Depending upon the tip's relationship to the sample, one of the two following conditions will result:

    a. **Small Z piezo movement:** This indicates that the cantilever is truly engaged with the sample surface.

    b. **Large Z piezo movement:** This indicates that the cantilever is damped by air trapped between the cantilever and sample surface (not in contact with the actual, solid surface). This is a false engagement condition (See False engagement).

Tapping Mode AFM continued...

Figure 18.3b Tapping Mode AFM Concepts



Optimizing Tapping Mode AFM Signal After Engagement

The figures on the bottom of Figure 18.3b illustrate the relationship between the RMS and the setpoint voltages.

There are some basic rules to remember:

- The setpoint voltage is always lower than the RMS voltage (Figure A).
- 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 (Figures A and B).
- The RMS voltage controls the amount of energy that is in the cantilever (Figures A and D). This is important to note because some samples are stickier than others. The tip may stick and hold to the sample surface if the RMS amplitude is too small.

The initial setup for Tapping Mode AFM includes the following steps:

1. Tune the cantilever at its resonance.
2. Quit the **View / Cantilever Tune** software routine after finding the resonance.
3. Set the desired **RMS voltage**. This is usually about **2-3V**.
4. Engage the microscope.

Troubleshooting continued...

18.3.5 Problems with Tapping Mode AFM

Some of the problems associated with Contact Mode AFM are also relevant to Tapping Mode AFM (See Section 18.3.3).

Unusual Resonance Peaks

The most common problem encountered while operating in Tapping Mode is unusual resonance peaks in the **Cantilever Tune** window. In general, bad cantilevers produce strange resonance curves. Improper seating of the cantilever or a fracture in the cantilever cause double peaks in the resonance curve.

1. When an unusual resonance curve occurs, clean the groove on the cantilever holder and reseat the cantilever.

2. If this does not improve the resonance curve, install a new cantilever.

Streaks on Surface Features

Streaks on surface features indicate the tip is not tracking the surface properly. The following conditions or a combination of these factors may apply:

a. Insufficient tapping force.

b. An excessively fast scan rate.

c. Gain values are set too low.

Problems with Tapping Mode AFM continued...

Try the following procedures to eliminate this condition:

1. Reduce the **Setpoint** voltage to increase the amount of tapping force on the surface. Be careful when reducing the **Setpoint** voltage on soft samples. The sample surface can still be disturbed even though the forces are very small.

2. Reduce the **Scan rate**. The **Scan rate** must be slower in Tapping Mode than in Contact Mode AFM (around **1-3Hz**).

3. Increase the **Integral** and **Proportional Gains** to speed up the response time of the Z piezo transducer.

Lines Across the Image

The tip sticking to the sample surface, results in lines oriented in the X scan direction. Increase the **RMS voltage**. Working with higher **RMS voltage** gives the tip more energy to pull off the surface. To correct this condition, try the following approach:

1. Use the keyboard arrow keys 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 **2 volts**.

4. Use the arrow keys to increase the **Drive amplitude** (press 2-3 times). This increases the **RMS voltage** output from the microscope. Increasing the **RMS voltage** means the cantilever is oscillating harder, making it less subject to stick to the sample surface.

5. Use the arrow keys to reduce the **Setpoint** voltage until the **Z Center Position** voltage moves away from the retracted position.

6. Continue to reduce the **Setpoint** voltage until the topographic image on the display monitor pops into clear view.

Problems with Tapping Mode AFM continued...

Rings Around Features on the Surface

Operating with a drive frequency too close to cantilever resonance causes rings to appear around features on the surface.

1. Use the arrow keys to increment the drive frequency a little lower while watching the Real-time scan. Be aware that the **RMS voltage** might also reduce.

2. Repeat the steps in the Lines Across the Image troubleshooting section to adjust the **RMS voltage**.

Multiple or Repeating Patterns

If you experience multiple or repeating image patterns, the tip is probably chipped. Too much tapping force on the surface, or the tip encountering a feature too high to successfully traverse often chips the tip. Change the tip.

> **Note:** Operating with a smaller difference between the **RMS voltage** and the **Setpoint** voltage means that less tapping force is used which may damage the tip.

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 Off-line **Planefit** sub-command on the control monitor. Off-line **Planefit** is normally set to **Full**. For a full description of the Off-line **Planefit** command, see the *Command Reference Manual*.

18.3.6 Initialize

If you cannot access **Focus Surface** or **Locate Tip** or if the corresponding icons are grayed out, you must re-initialize the system. To initialize the system, complete the following steps.

1. Select **Initialize / Execute.**

   Note: It is not necessary to initialize more than once unless you have turned the SPM off or moved the stage manually. The **Stage / Initialize** command drives each stage motor to the limits of its travel. At the end of the **Initialize** sequence, the X-Y stage resides at the load/unload position. The Z-stage raises, positioning the microscope head far away from the surface at the end of **Initialize**.

2. If any of the motors do not actuate as described, or if you suspect any problems, verify that all connectors are firmly attached.

3. Verify that power is **On**. As a last resort, leave all power on and exit the SPM program.

4. Restart the SPM program, and select **Stage / Initialize**.

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