Veeco

DIMENSION

icon

with ScanAsyst

Instruction Manual
004-1023-000

Document Revision History: DImension icon Manual

Revision	Date	Section(s) Affected	Ref. DC R	Approval
F	March 19, 2010	13.5.2		Vinson Kelley
E	February 11, 2010	8.1.1, 8.2		Vinson Kelley
D	January 20, 2010	NanoScope 8.10 updates		Vinson Kelley
C	July 9, 2009	Icon PT added		Vinson Kelley
B	Apr. 27, 2009	Icon-PI added		Vinson Kelley
A	Mar. 04, 2009	Initial Release		Vinson Kelley

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

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

Hardware Designs:
TrakScan™
StiffStage™

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

Software Options:
NanoScript™
Navigator™
FeatureFind™

Miscellaneous:
NanoProbe®

Table of Contents

Chapter 8   Adaptive XY Scanning   **163**

Chapter 9   ScanAsyst   **171**

Chapter 12　　　　Fluid Imaging　　**235**

Chapter 13　　　　Scanning Tunneling Microscopy (STM)　　**265**

List of Figures

Chapter 1   System Overview

The Dimension® Icon™ Scanning Probe Microscope (SPM), shown in Figure 1.0a, 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 electrodes on the piezo tube controls the vertical height of the tip. The Dimension Icon head includes X,Y and Z position sensors. The sensed XYZ position information provides the feedback for closed loop control of probe tip location, ensuring that the probe completes each move command at the intended position. Stepper motors coupled to lead screws translate slides with the sample attached. A separate motor drive controls the height of the microscope and tip relative to the sample surface.

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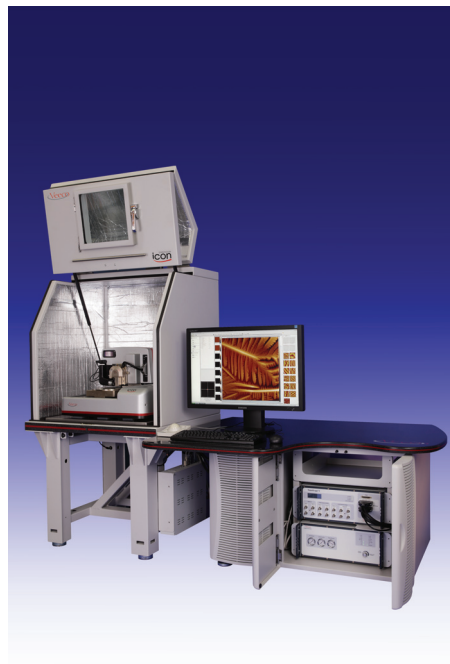
This manual details facility requirements, installation requirements and procedures, maintenance requirements and procedures, and applications used with the Dimension Icon Scanning Probe Microscope.

This chapter discusses the following:

- **Conventions and Definitions:** Section 1.1

- **System Overview:** Section 1.2

  - **Dimension Icon SPM Features:** Section 1.2.1

- **Control Station Overview:** Section 1.3

  - **Input and Display Devices:** Section 1.3.1

  - **Computer:** Section 1.3.2

  - **NanoScope V Controller:** Section 1.3.3

  - **Dimension Stage Controller:** Section 1.3.4

- **Dimension Icon SPM Overview:** Section 1.4

  - **Dimension Icon Microscope Electronics Box:** Section 1.4.1

  - **Optics and Motors Overview:** Section 1.4.2

  - **Stage System:** Section 1.4.3

  - **Dimension SPM Head:** Section 1.4.4

  - **Probe Holder:** Section 1.4.5

  - **Video Microscope:** Section 1.4.6

- **Sample Size & Handling:** Section 1.5

- **Facilities Specifications:** Section 1.6

- **Applications:** Section 1.7

- **Maintenance and Troubleshooting:** Section 1.8

1.1 Conventions and Definitions

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

- Three font styles distinguish among contexts. For example:
  Window or Menu Item / **BUTTON OR PARAMETER NAME** is set to **VALUE**.

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

1.2 System Overview

Figure 1.2a Dimension Icon SPM

There are several typical configurations of the Dimension Icon Scanning Probe Microscope (see Chapter 3 for detailed information).

- VT-1000 air table (Dimension Icon-PI).

- AVH-1000 workstation configuration including electronics cabinet, air table and acoustic hood (Dimension Icon).

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

1.2.1 Dimension Icon SPM Features

Enhanced Motorized Positioning Stage

The X-Y stage permits micron-scale positioning of samples beneath the tip. The stage has 6.06-inch (X direction) by 7.283-inch (Y direction) travel, and is offered with a 200mm (8-inch) chuck. With sample chuck rotation, a complete 200mm (8-inch) diameter sample is accessible.

The Dimension Icon's motorized Z-stage provides accurate, automatic tip engagement and approach. The rigidity of the Z-stage permits low noise, and high accuracy imaging with the instrument. The Z-stage is driven by a motorized lead screw for coarse approach of the head to the sample. This configuration permits samples of different thicknesses to be imaged with little difficulty.

Specification Summary

- Travel: X-axis 154 mm (6.06")
       Y-axis 185 mm (7.283")

- Resolution: 1µm

- Repeatability: ±2µm

Integrated Dimension Stage Controller

The Dimension Stage Controller integrates the illuminator control, power supply, and air and vacuum pumps.

Optical Microscope

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

Video Image Capture Capability

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

Computer System

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

1.3 Control Station Overview

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

1.3.1 Input and Display Devices

Input and display devices include a large monitor (Dimension Icon) or two smaller monitors (Dimension Icon-PI), keyboard, mouse, and trackball (see Figure 1.3a and Figure 1.3b). These devices convey information signals to the computer to operate the software and SPM.

CAUTION: The monitors contain a hazardous voltage of 120V.
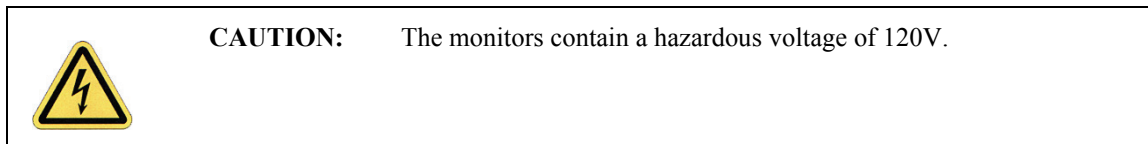
Figure 1.3a   Dimension Icon Input and Display Equipment
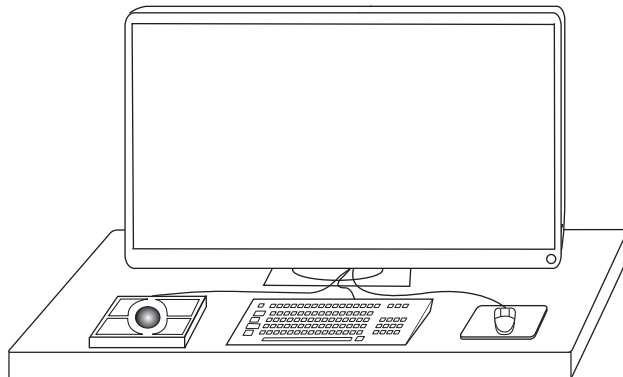
Figure 1.3b   Dimension Icon-PI Input and Display Equipment
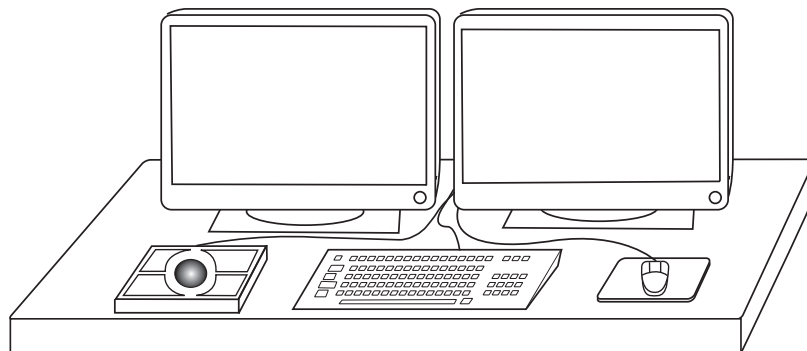
1.3.2 Computer

The computer, shown in Figure 1.3c, is the main control unit of the Dimension Icon SPM system; it includes a DVD-RW drive and, in the case of the Dimension Icon, a custom Digital Signal Processing/Interface (DSP) card. The computer receives data from the input devices, and controls external hardware via the standard ports and the input/output (I/O) bus. The computer isolates the user from direct interaction with the hardware. (See **Dimension Icon Control Station Connections:** Section 4.4.1 for a complete description of the computer connections).

Figure 1.3c Dimension Icon Computer rear view (left) and Icon-PI Computer rear view (right). Not to scale.
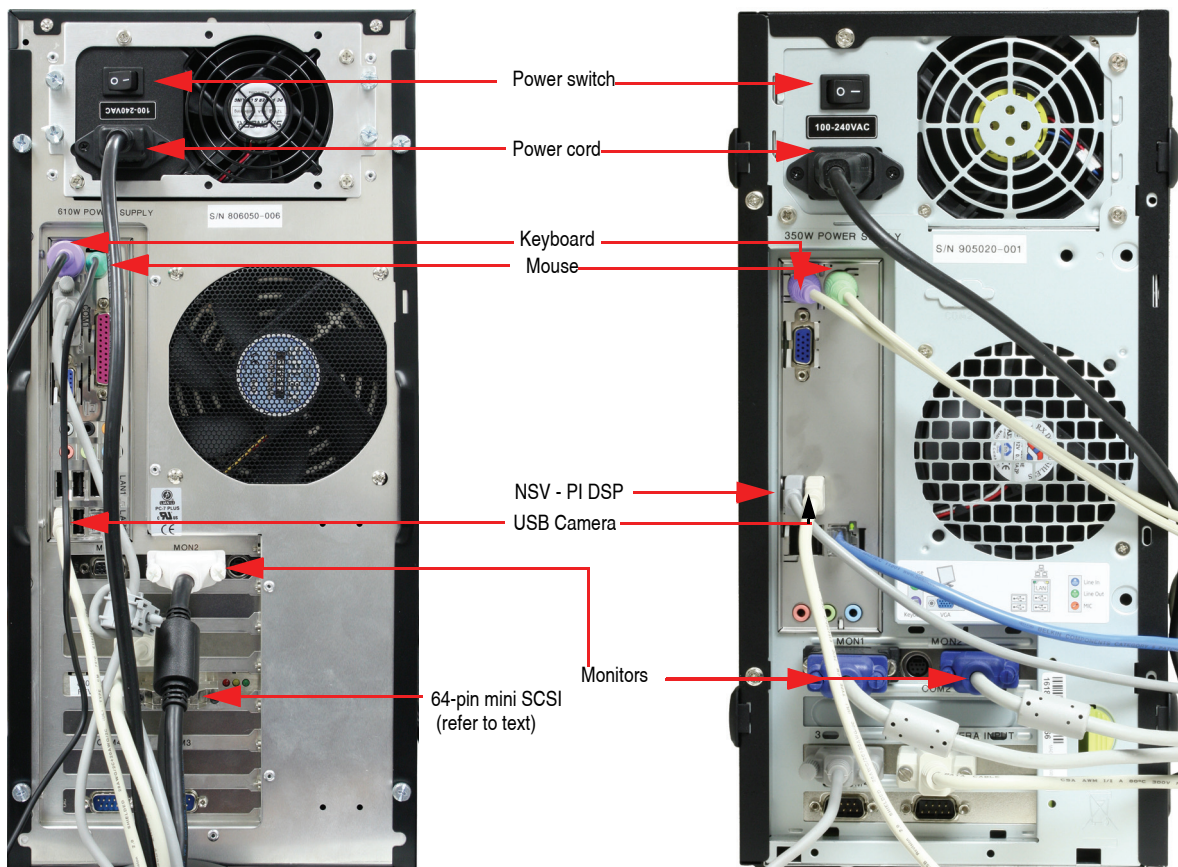
1.3.3   NanoScope V Controller

The NanoScope V Controller, shown in Figure 1.3d, controls the microscope head and scanning. The Dimension Icon NanoScope V controller is controlled via a 68-pin micro-SCSI cable connection between the rear of the controller and the computer (see Figure 1.3e and Figure 1.3f) while the Icon-PI Controller is controlled via a USB 2 cable.

Figure 1.3d   NanoScope V Controller

The controller connects to the back of the Dimension Icon microscope via two cables from the front of the controller (see Figure 1.3g).

	CAUTION:	Cables should not be removed or installed while power is applied to the system.
		Connecting cables while power is applied may damage the instrument.

Figure 1.3e NanoScope V Controller (back view)

Ventilation Fans, Outflow

Fuses

Power Switch

68-pin Micro-SCSI connector to PC

AC Input Requirements

Fuse Ratings

Power Cord
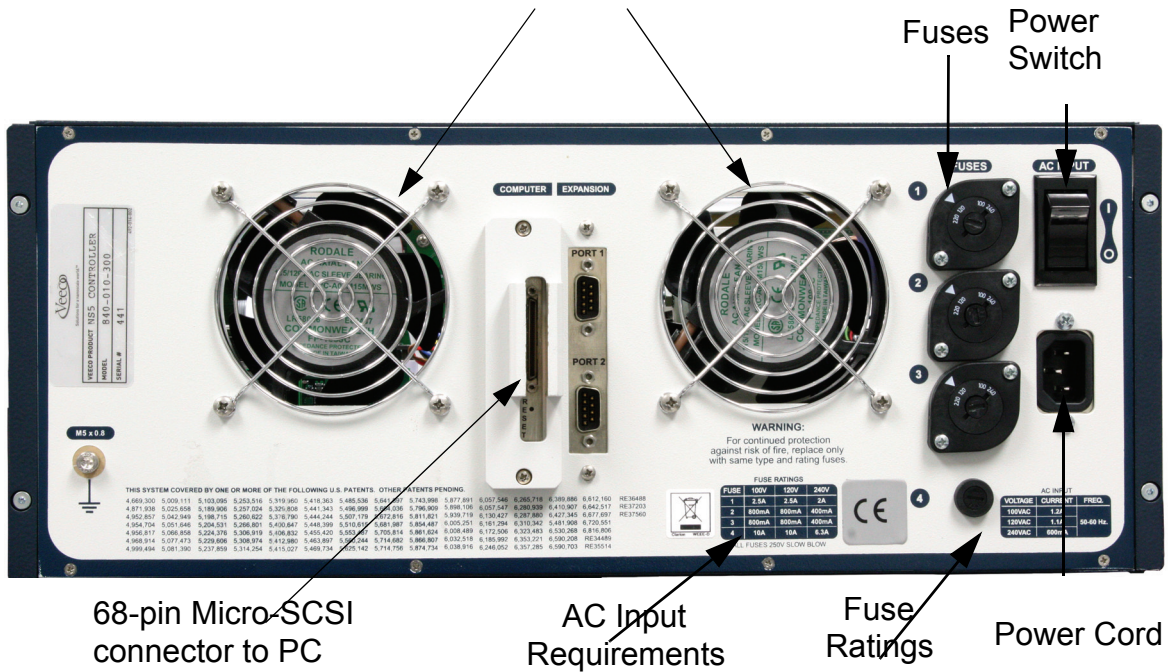
Figure 1.3f NanoScope V-PI Controller (back view)

Ventilation Fans, Outflow

Fuses

Power Switch

USB connector to PC

AC Input Requirements

Fuse Ratings

Power Cord
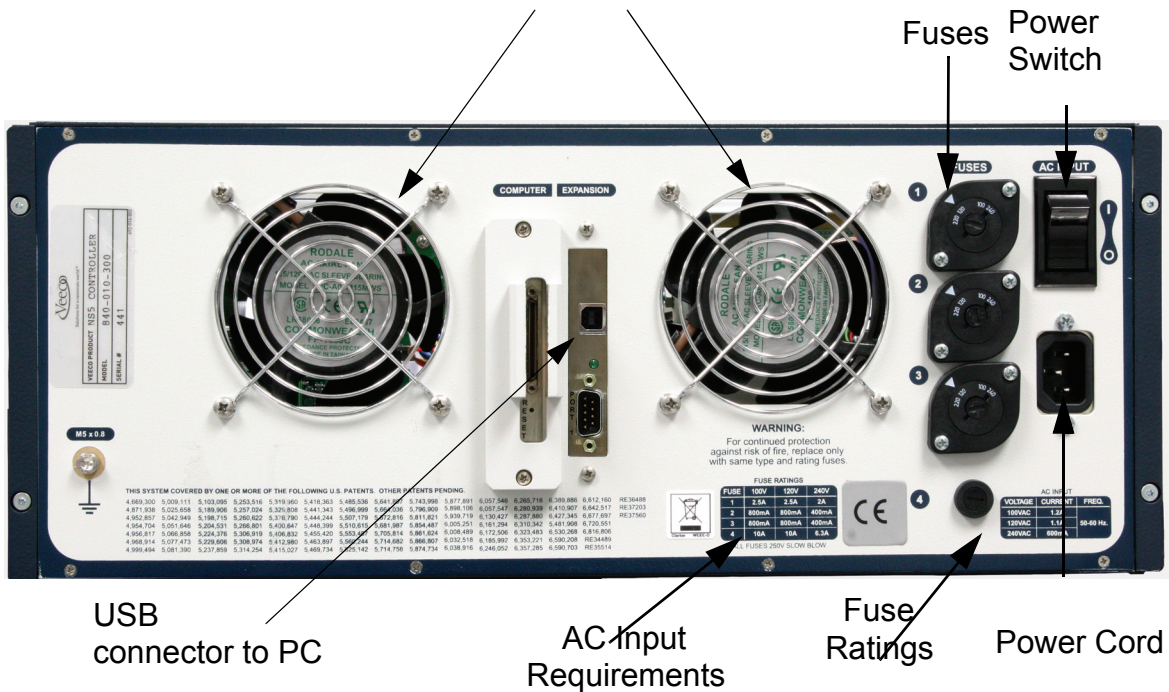
Figure 1.3g Controller (front view)

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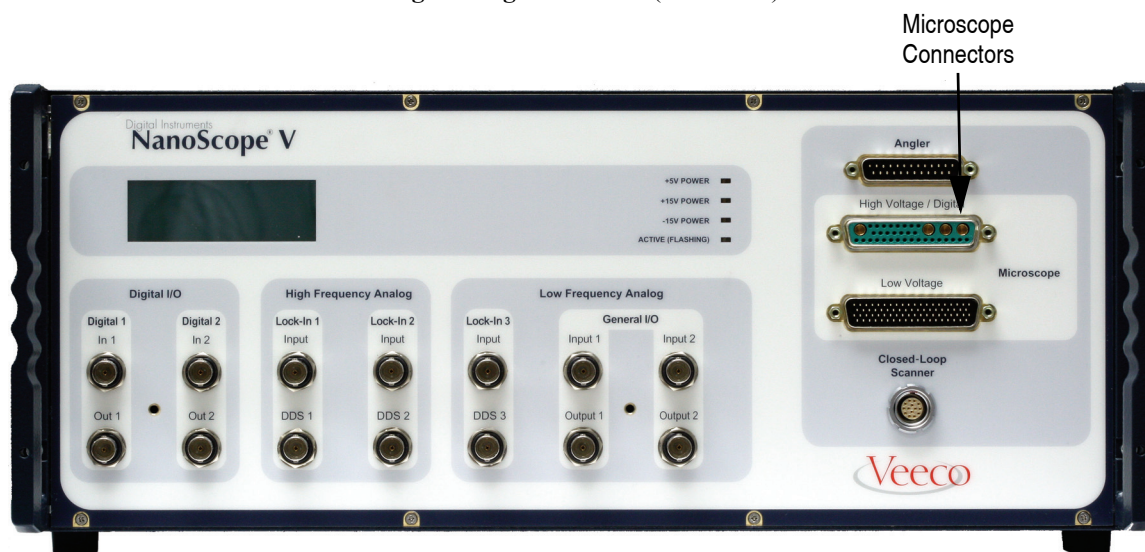
CAUTION: The NanoScope controller will overheat if the controller ventilation
holes are blocked or if the controller is exposed to heat from an
outside source.

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

1.3.4 Dimension Stage Controller

The Dimension Stage Controller, shown in Figure 1.3h, controls the vacuum and air supply and optics illumination. The Dimension Stage Controller is controlled via a serial cable connection between it and the computer. The Dimension Stage Controller features gauges on the front panel to indicate vacuum and positive pressures. The Dimension Stage Controller channels positive pressure to the underside of the chuck during X-Y movements, allowing the chuck to glide smoothly over the granite. The Dimension Stage Controller drives the motors of the Z-stage, focuses, zooms and powers the video camera. The Dimension Stage Controller incorporates status LEDs, shown in Figure 1.3h, on the upper right front panel.

Figure 1.3h   Dimension Stage Controller (front view)
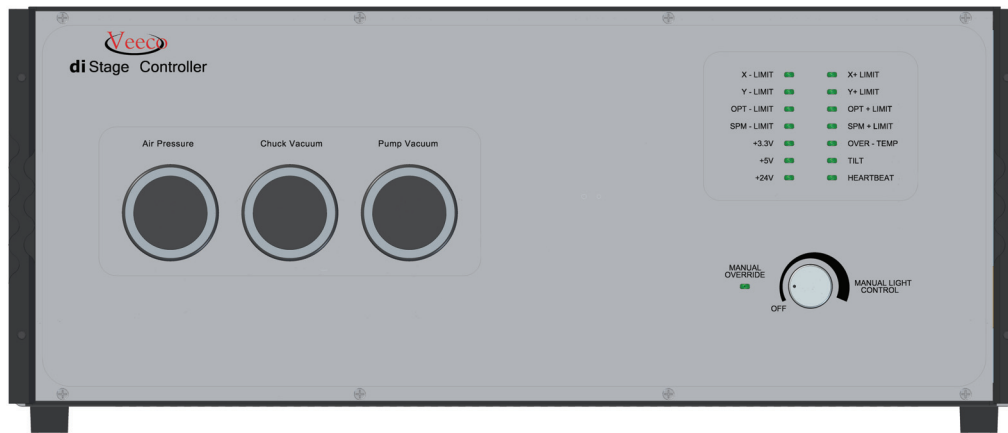
	CAUTION:	Do not turn the Dimension Stage Controller on its side. Turning the controller on its side activates an alarm and turns the vacuum pump off.
	ATTENTION:	Afin d'éviter de sérieux dommages mécaniques, ne tournez pas le boîtier de contrôle sur le côté. Tourner le boîtier sur le côté déclencheune alarme et coupe la pompe à vide.
	VORSICHT:	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.

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

The Dimension Stage 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 routes vacuum and positive pressure control from the Dimension controller to the stage and chuck.

Illumination System

Illumination can be computer or manually controlled. The knob located on the front of the Dimension controller regulates manual illumination control. For computer control, there is an "illumination" parameter in the **Other Controls** panel to control brightness. The illuminator control knob must be in the "off" position to allow software control. Sample illumination is provided by an LED in the optics assembly on the left.

1.4   Dimension Icon SPM Overview

1.4.1   Dimension Icon Microscope Electronics Box

The Dimension Icon Microscope Electronics Box, shown in Figure 1.4a, located below the microscope, switches between the different operating modes, amplifies and combines signals from the photodiodes as appropriate. (See **Dimension Icon Microscope Electronics Box Connections: Section 4.4.4** for a complete description of the Electronics Box connections).

Figure 1.4a   Dimension Icon Microscope Electronics Box

Electronics Box

The Dimension Icon Microscope Electronics Box houses circuit boards to control signals from the SPM head and (optional) applications modules.

1.4.2   Optics and Motors Overview

The optics 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 stepper motor/lead screw (focus motor) to adjust the focal point approximately 1.5mm above the cantilever to approximately 3.5mm below. The range of focus below the cantilever allows you to focus on the sample while leaving up to 3.5mm clearance between the tip and sample.

Figure 1.4b   Dimension Icon
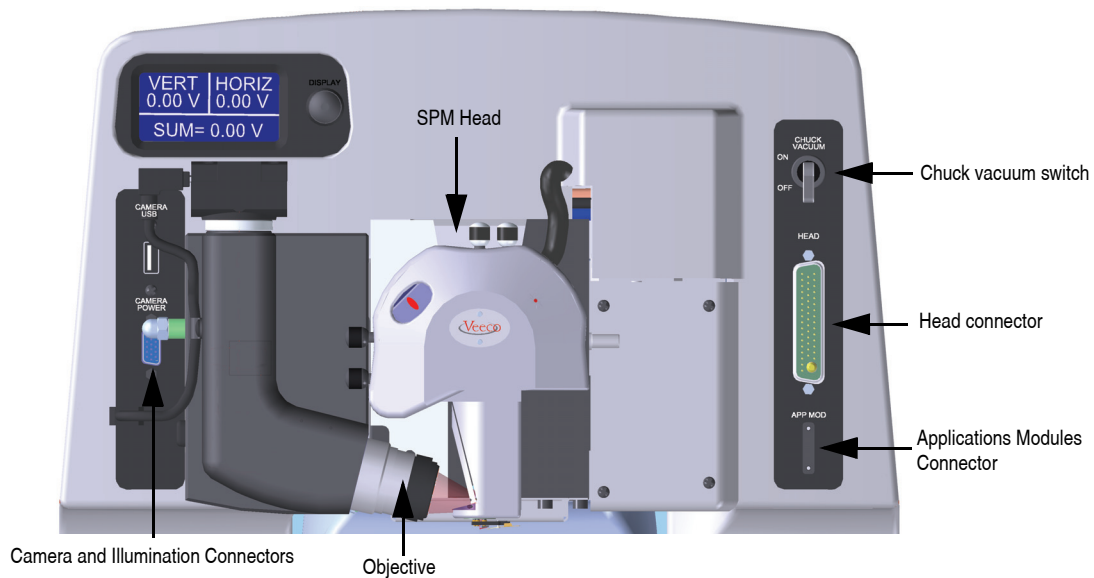
1.4.3   Stage System

The Dimension Icon X-Y stage provides positioning repeatability of 2μm. The 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 154mm (X direction) by 185mm (Y direction) travel range.

The Dimension Icon 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.

Vacuum Power Switch

The vacuum power switch at the upper right of the Dimension Icon (see Figure 1.4b) toggles with an **ON/OFF** control switch for sample vacuum capabilities.

Figure 1.4c   Dimension Icon
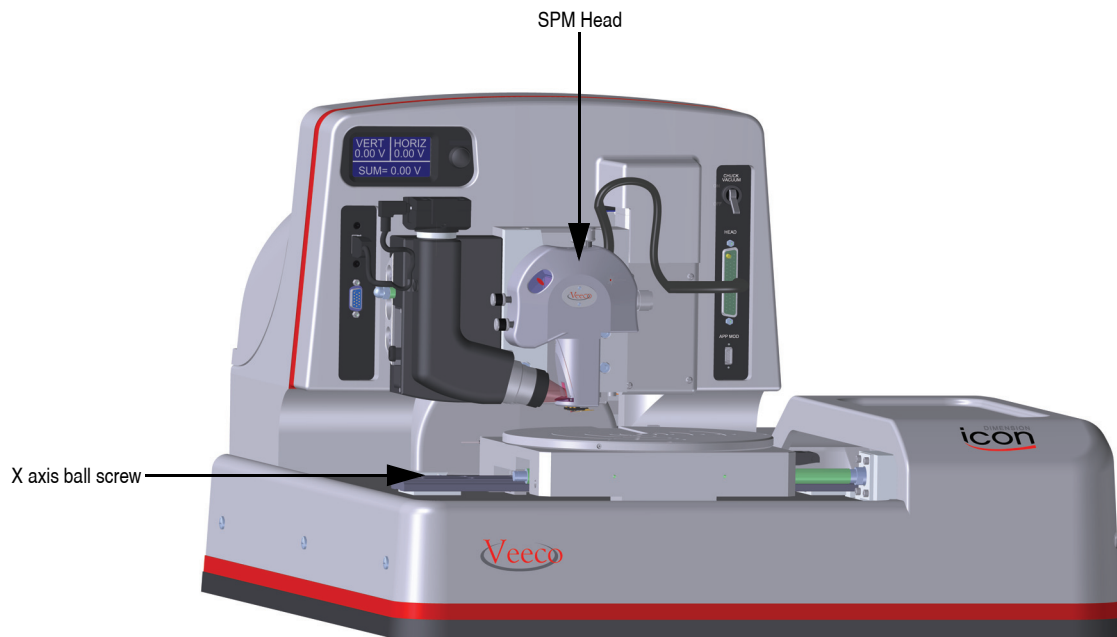
1.4.4 Dimension SPM Head

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 1.4d 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 patented technology sharply reduces bowing and attenuation artifacts due to cantilever scanning across the laser beam's otherwise stationary focal plane.

Figure 1.4d Dimension SPM Head

Laser Spot Detector Screen (dark red)

Laser aim adjustments

Position Sensitive Detector
(special photodiode)

Laser diode

Collimator

Beamsplitter
(behind screen)

Focusing lens

Detector
Mirror
positioners

Laser Light's
Beam path

Adjustable (detector)
mirror

Tracking Lens
(corrective)

Fixed mirror

Lens

Probe Tip Holder
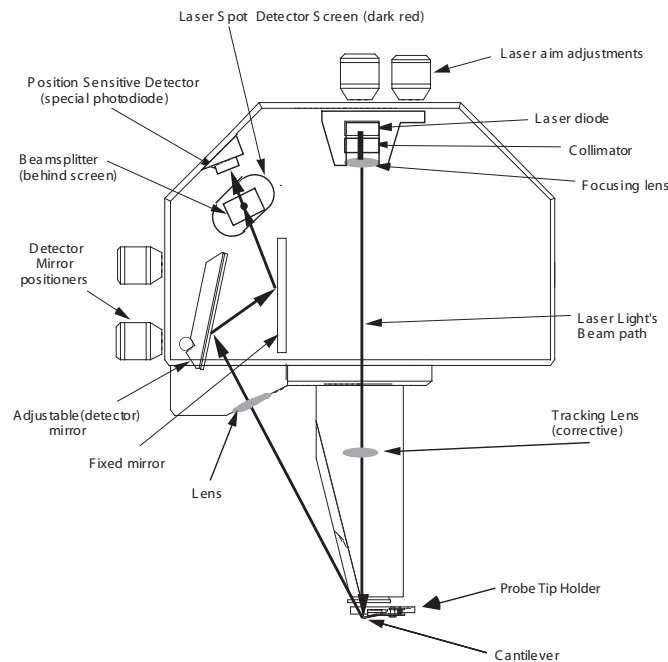
Cantilever

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 670nm) as it reflects off the cantilever. The integrated scanner head consists of the following subassemblies:

Preamp Board

The preamp board is located inside the Dimension 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 a sensor preamplifier. The head is connected to a 48-pin male connector cable plugged into the socket on the front right of the Dimension Icon microscope.

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 and TappingMode. 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 and Torsional Resonance Mode Microscopy). Figure 1.4e illustrates the arrangement of the photodiode elements in the Dimension Icon SPM head.

Figure 1.4e Quad Photodetector Arrangement
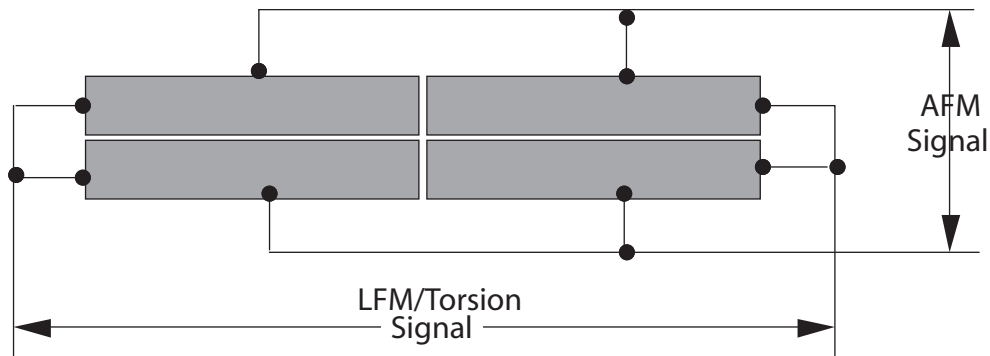
Beamsplitter and Laser Spot Detector Screen

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 position relative to the photodetector.

Scanner Piezo Tube

Figure 1.4f 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 Icon 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 1.4f Dimension Head Scanner
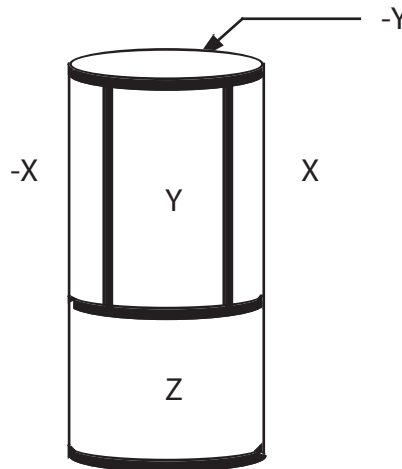
The end of the scanner allows easy removal of the imaging probe assembly. This permits loading the tip on a separate fixture.

Table 1.4a Scanner Piezo Tube Specifications

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

1.4.5   Probe Holder

The cantilever holder is a small printed circuit card or acrylic block that holds the cantilever firmly at the proper angle. The standard cantilever holder is used for operation in air, while a cantilever holder with a clear glass window is used for fluid cell operations. Cantilever holders include the cantilever piezoelectric stack and the electrical contacts to the drive circuits. 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 1.4b). The standard cantilever holder shown in Figure 1.4g contains a piezoelectric stack to oscillate the cantilever when operating in TappingMode. The same cantilever holder is used for Contact AFM but no voltage is applied to the piezo stack.

Figure 1.4g   Standard Cantilever Holder

SIDE VIEW

tip is installed

Cantilever Probe Tip
(tip faces down)

Spring Loaded Probe Clip

Electrical Mounting Sockets (4 plcs)

Cantilever
Mounting
Groove
(no tip
installed)

BOTTOM VIEW
(TIP SIDE)

no tip installed
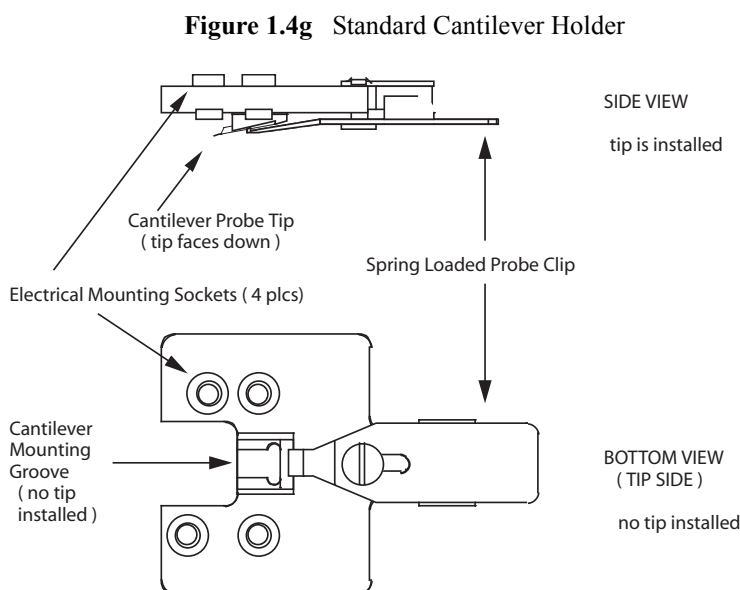
The fluid cell cantilever holder shown in Figure 1.4h provides an optically transparent cover over the back of the cantilever to maintain the optical path of the laser beam constant when the tip is submerged in a fluid medium. If the fluid medium was not covered by the fluid cell, imaging with the tip 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 1.4b   Cantilever Holder Specifications

Holders	Specifications
Standard	TappingMode, Contact AFM, MFM
Optional	STM and Force Modulation
Fluid Cell	Fluid Contact AFM, Fluid TappingMode
TrMode	Torsional Resonance

Figure 1.4h   Fluid Cell Cantilever Holder
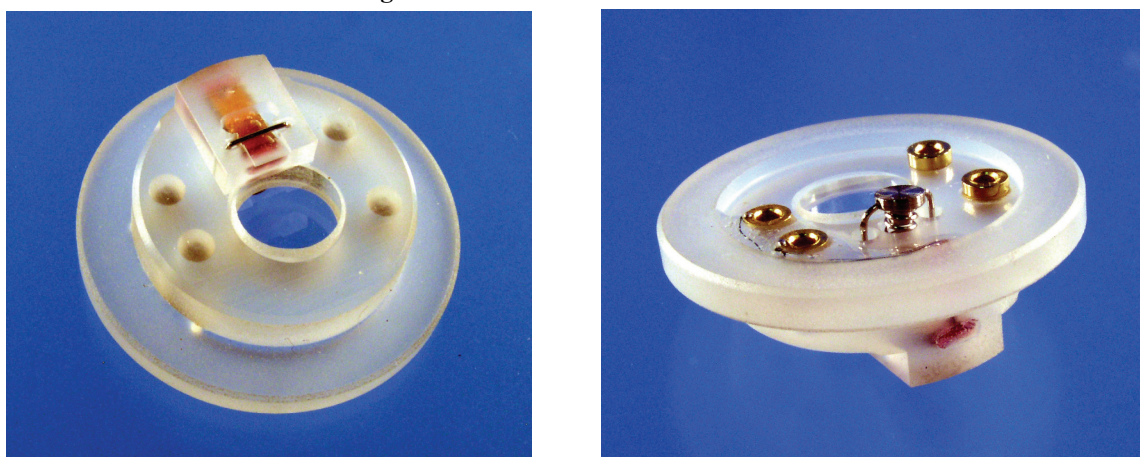
1.4.6   Video Microscope

The integrated optical microscope features:

- 10x objective (long working distance)

- Color video camera, 2560 x 1920 resolution

- Digital zoom

- 508-4010 x magnification range with 30-inch monitor and corresponding field of view of 190—1500 μm

- Motorized focus

- Through-the-lens illumination

- Focus tracking and automated engagement

1.5   Sample Size & Handling

Samples may be up to 200mm (8 inch) diameter x 17mm (0.67 inch) thick.

Table 1.5a   Sample Specifications

	Specifications
Max Sample Size	(Wafers and disk media) 8-inch dia. x 0.65" thick
Max. Sample Weight	1.0 lb (454 grams)
Inspectable Area	154mm x 185mm 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 upper right rear of the microscope.

1.6   Facilities Specifications

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

1.7   Applications

The Dimension Icon SPM can be used for several applications. 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 Icon SPM:

- Contact AFM

- Contact AFM in Fluids

- TappingMode

- TappingMode in Fluids

- Lateral Force Mode

- Force Imaging

- Interleave Scanning

- Magnetic Force Imaging

- Electric Techniques

- Calibration

1.8   Maintenance and Troubleshooting

Refer to Chapter 20 for detailed information regarding maintenance and troubleshooting issues for the Dimension Icon SPM.

Chapter 2   Safety

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

Specifically, this chapter discusses the following areas of information:

- **Safety Requirements:** Section 2.1

- **Safety Precautions:** Section 2.2

    - **General Operator Safety:** Section 2.2.1

    - **Microscope:** Section 2.2.2

    - **Sample Safeguards:** Section 2.2.3

- **Ergonomics:** Section 2.3

- **Environmental Conditions:** Section 2.4

- **Hazard Labels:** Section 2.5

    - **Laser Explanatory Label:** Section 2.5.1

    - **Laser Warning Label:** Section 2.5.2

    - **Aperture Warning Label:** Section 2.5.3

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

2.1   Safety Requirements

Figure 2.1a   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.
	This symbol identifies a corrosive material hazard. Corrosive material can cause serious damage to the equipment.
	Ce symbole indique un risque de la matière corrosive. La matière corrosive peut causer des dommages sérieux au matériel.
	Dieses Symbol bedeutet "Gefahr durch Korrosive Materialien". Korrosive Materialien können erhebliche Schäden am Gerät oder anderen Gegenständen verursachen.

2.2 Safety Precautions

Because the Dimension Icon 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.

2.2.1 General Operator Safety

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

WARNING:	Follow company and government safety regulations. Keep unauthorized personnel out of the area when working on equipment.
AVERTISSEMENT:	Il est impératif de suivre les prérogatives imposées tant au niveau 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.

	CAUTION:	Please contact Veeco before attempting to move the Dimension Icon SPM system.
	ATTENTION:	Il est impératif de contacter Veeco avant de déplacer le Dimension Icon SPM.
	VORSICHT:	Bitte kontaktieren Sie Veeco bevor Sie das Dimension Icon 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. (Veeco service personnel, *only*).
	AVERTISSEMENT:	Les tensions utilisées dans le système sont potentiellement dangeureuses et peuvent blesser les utilisateurs. Avant toute intervention électrique, ne pas oublier de débrancher le système. (Réservé au personnel de 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 Veeco Personal.)

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

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

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

2.2.2   Microscope

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

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

	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 Icon 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, mais 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 Icon SPMs erfaßt wird, kann leicht ernsthaft verletzt werden.

	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 Veeco for repair or replacement.
	AVERTISSEMENT:	Les parties électroniques du microscope, du controleur et des équipements périphériques comportent des équipements fonctionnant avec de hauts voltages. Ne pas essayer d'effectuer de réparations, aucune de ces parties n'étant concue pour être réparée par l'utilisateur. Déconnecter les équipements défectueux et les envoyer à Digital Instruments/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 Veeco.

	WARNING:	Do not attempt repairs on electrical components. If it is necessary to enter the electrical chassis for any reason (e.g., to replace a computer card), power-down the entire system and disconnect it from its power source.
	AVERTISSEMENT:	Ne pas essayer de réparer les parties électroniques. Si il est nécessaire d'accéder au boitier électronique (pour remplacer une carte dans l'ordinateur par exemple), éteindre tout le système et le déconnecter.
	WARNUNG:	Versuchen Sie nicht, elektrische Komponenten selbst zu reparieren. Falls es aus irgend einem Grund notwendig sein sollte, ein Gehäuse mit elektrischen Bauteilen zu öffnen (z.B., um eine Computer-Karte auszutauschen), schalten Sie das gesamte System ab, und trennen Sie es von der Spannungsquelle.

	CAUTION:	Avoid spilling fluids onto the microscope stage or into electrical assemblies, particularly the SPM head. If it is necessary to use fluids, apply only small amounts as needed.
	ATTENTION:	Eviter d'éclabousser la platine du microscope et les assemblages électriques, en particulier la tête du microscope. Si il est nécessaire d'utiliser des liquides, ne les employer qu'en faibles quantités.
	VORSICHT:	Vermeiden Sie es, Flüssigkeiten auf dem Probentisch oder über elektronische Bauteile, insbesondere den Mikroskopkopf, zu verschütten. Wenn es notwendig ist, Flüssigkeiten zu verwenden, benutzen Sie dem Bedarf entsprechend nur geringe Mengen.

	CAUTION:	Cables should not be removed or installed while power is applied to the system. Connecting cables while power is applied may damage the instrument.

2.2.3   Sample Safeguards

	CAUTION:	**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. Dispose of wipes in an appropriately labelled solvent-contaminated waste container.
	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.
	VORSICHT:	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.

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

2.3   Ergonomics

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

2.4   Environmental Conditions

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

2.5   Hazard Labels

The Dimension Icon 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 Icon SPM:

2.5.1 Laser Explanatory Label

The **Laser Explanatory Label** (see Figure 2.5a) 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.

2.5.2 Laser Warning Label

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

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

2.5.3   Aperture Warning Label

The **Laser Aperture Warning Label**, shown in Figure 2.5b, is located on the bottom of the head where the laser light exits.

Figure 2.5b   Aperture Warning Label

2.5.4 Head Serial Number and Contact Information Labels

The head serial number and contact information labels, shown in Figure 2.5c, are located on the back of the Dimension Icon head,

Figure 2.5c Serial number and contact information labels
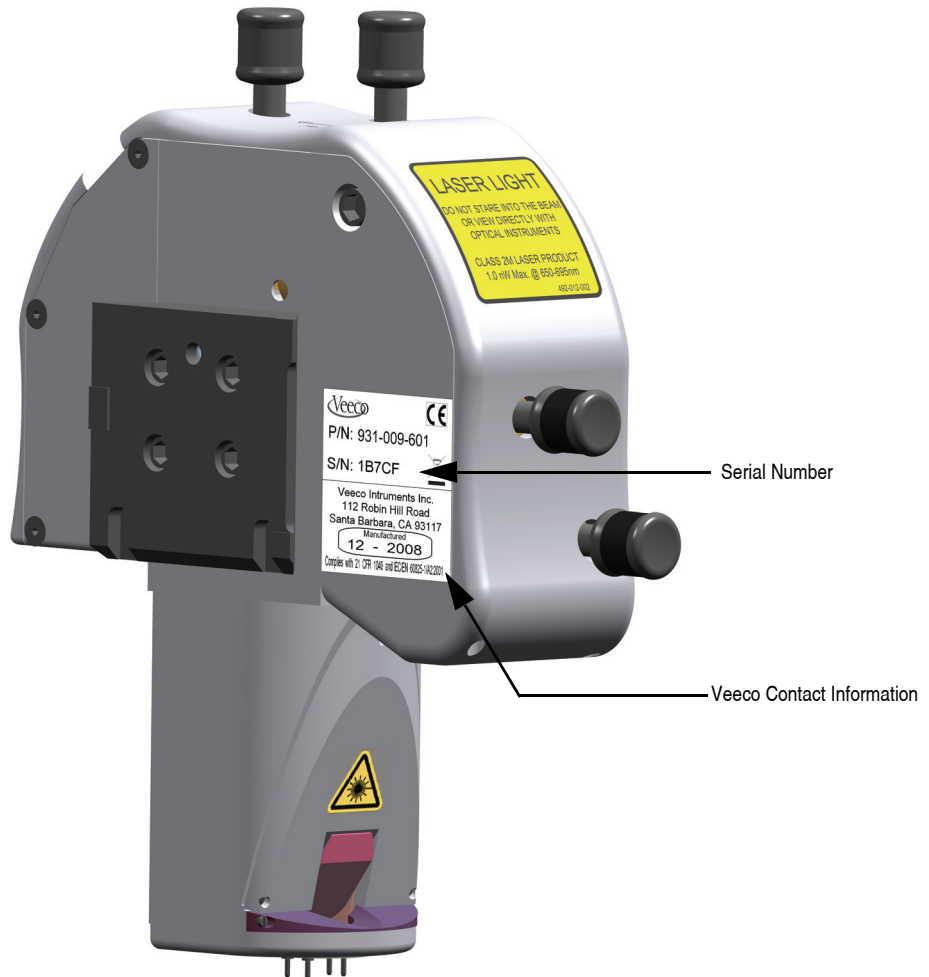
Chapter 3   Facilities Requirements

This chapter details facility site requirements, safety requirements, and configuration options for the Dimension Icon 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.

3.1   Product Configurations

The following are typical configurations for the Dimension Icon 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.

3.1.1   Dimension Icon-PI

This configuration consists of two basic elements: the VT-1000 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-1000 is a compact air table on which the Dimension icon microscope rests and does not include an acoustic hood. The electronics box ("e-box") is bolted to a shelf on the side of the VT-1000. Figure 3.1a 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, 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 monitor, or placing computer on the floor, which may save some space and change the facilities requirements.

Figure 3.1a   Typical Dimension Icon-PI Configuration
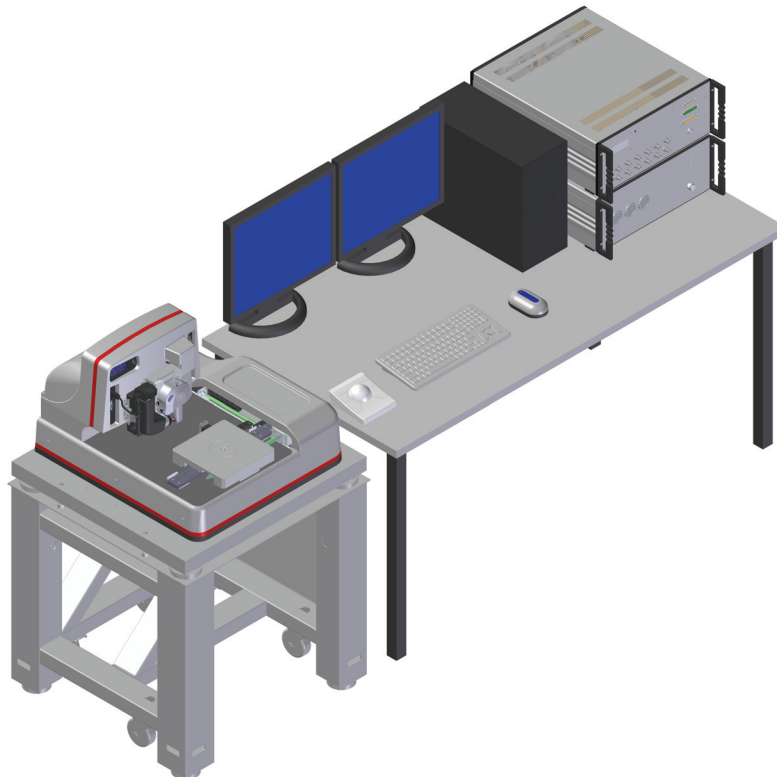
The AVH-1000 air table with integrated hood, discussed on Page 39 and in Section 3.3.2, can be ordered as an option on the Dimension Icon-PI. Note that, because of the hood, the footprint of the AVH-1000 is somewhat larger than the footprint of the VT-1000.

3.1.2 Dimension Icon-PT

This configuration consists of two basic elements: the VT-1000 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-1000 is a compact air table on which the Dimension icon microscope rests and does not include an acoustic hood. The electronics box ("e-box") is bolted to a shelf on the side of the VT-1000. Figure 3.1b 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 monitor, 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 monitor, or placing computer on the floor, which may save some space and change the facilities requirements.

Figure 3.1b Typical Dimension Icon-PT Configuration
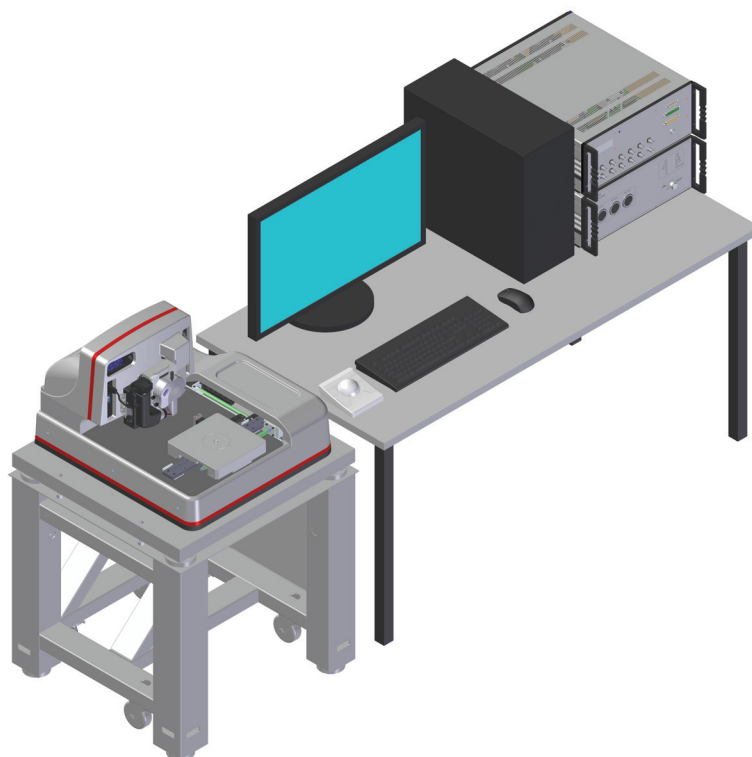
The AVH-1000 air table with integrated hood, discussed on Page 39 and in Section 3.3.2, can be ordered as an option on the Dimension Icon-PT. Note that, because of the hood, the footprint of the AVH-1000 is somewhat larger than the footprint of the VT-1000.

3.1.3   Dimension Icon

The Dimension Icon configuration, shown in Figure 3.1c, includes the AVH-1000, a cabinet that holds the NanoScope V Controller, the Dimension Stage Controller, the computer, monitor, keyboard, mouse and trackball that control the Dimension Icon SPM.

Figure 3.1c   Dimension Icon workstation configuration
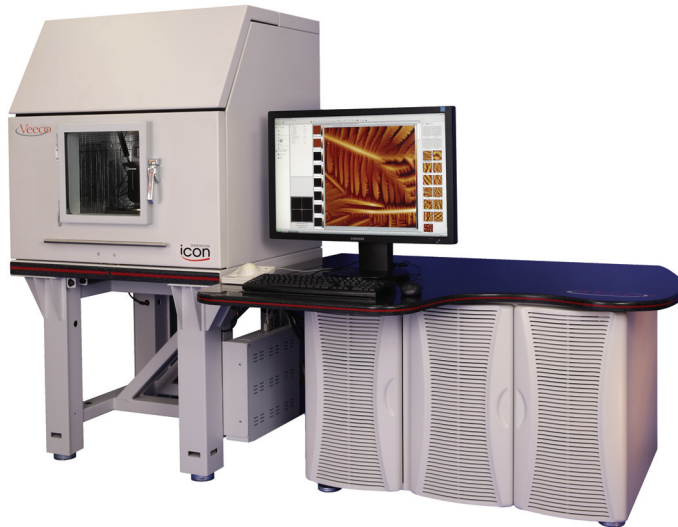
AVH-1000

The AVH-1000, shown in Figure 3.1d, is comprised of an integrated air table, on which the Dimension Icon rests and an acoustic hood which can be opened to access the sample stage or raised to access the instrument and lowered to seal the instrument against vibration. The AVH-1000 is ***strongly recommended*** (see Section 3.9) to provide increased acoustic isolation and vibration isolation.

Figure 3.1d   The AVH-1000
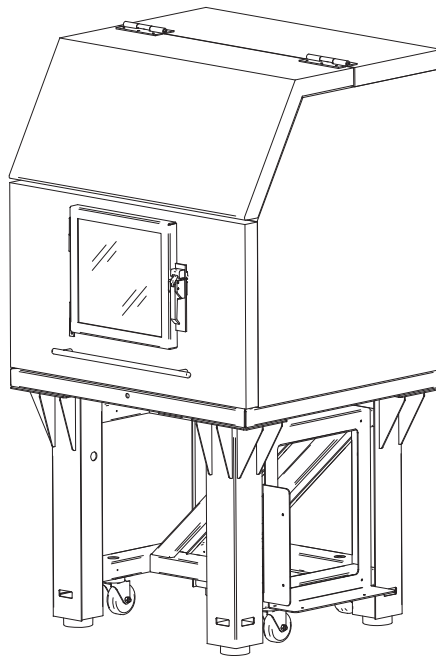
3.2   Microscope Dimensions

The Dimension Icon SPM, shown in Figure 3.2a, measures 23.4" wide by 25.1" deep by 16.0" tall and weighs 275 lbs. The Dimension Icon sits on a silicone vibration isolation pad that is 1" thick. A vibration isolation table (AVH-1000 or VT-1000) is required.

Figure 3.2a   Dimension Icon Dimensions

3.3 Acoustic/Vibration Isolation Systems

3.3.1 VT-1000

The VT-1000, shown in Figure 3.3a, is a counterweighted air table measuring 32" (including shelf for e-box) wide, 28" deep and 30" tall.

Figure 3.3a VT-1000 Dimensions
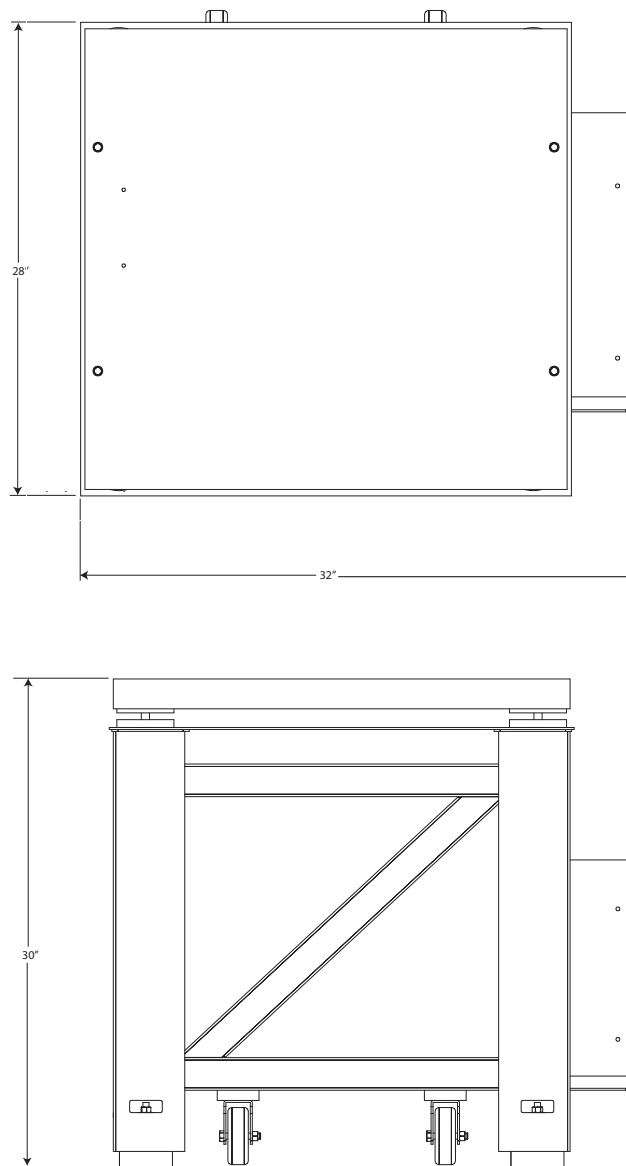
3.3.2   AVH-1000

The AVH-1000, shown in Figure 3.3b, Figure 3.3c, Figure 3.3d and Figure 3.3e, is ***strongly recommended*** (see Section 3.9) to provide increased acoustic and vibration isolation. The acoustic hood weighs approximately 900 lbs.

Figure 3.3b   AVH-1000
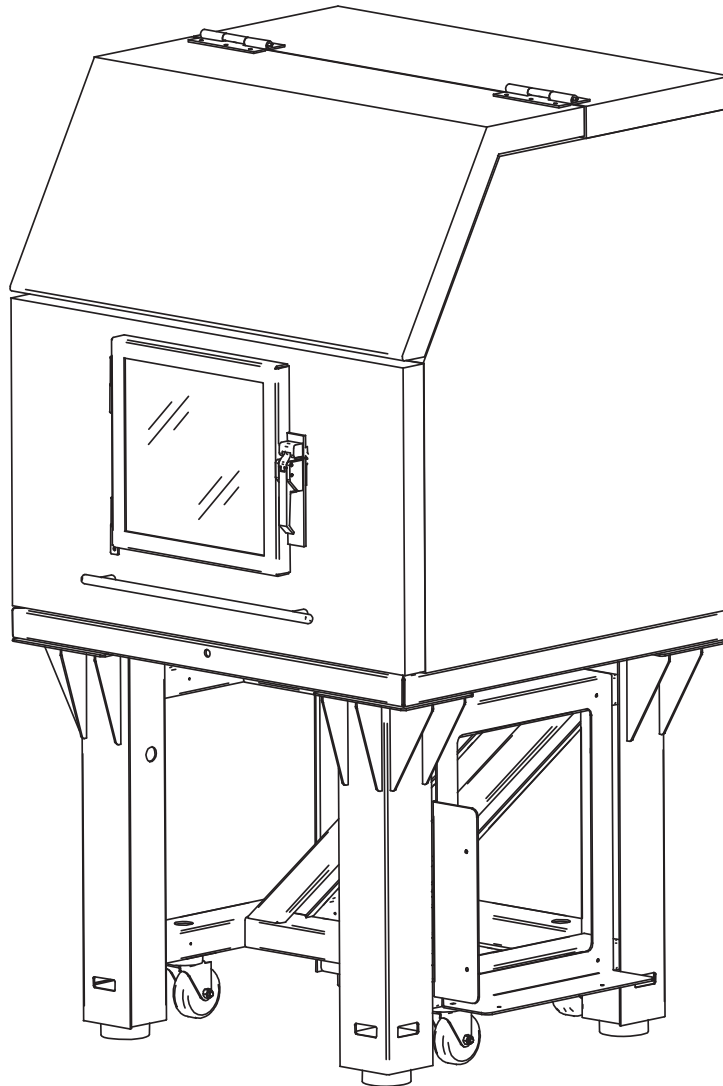
Figure 3.3c   AVH-1000 Front View
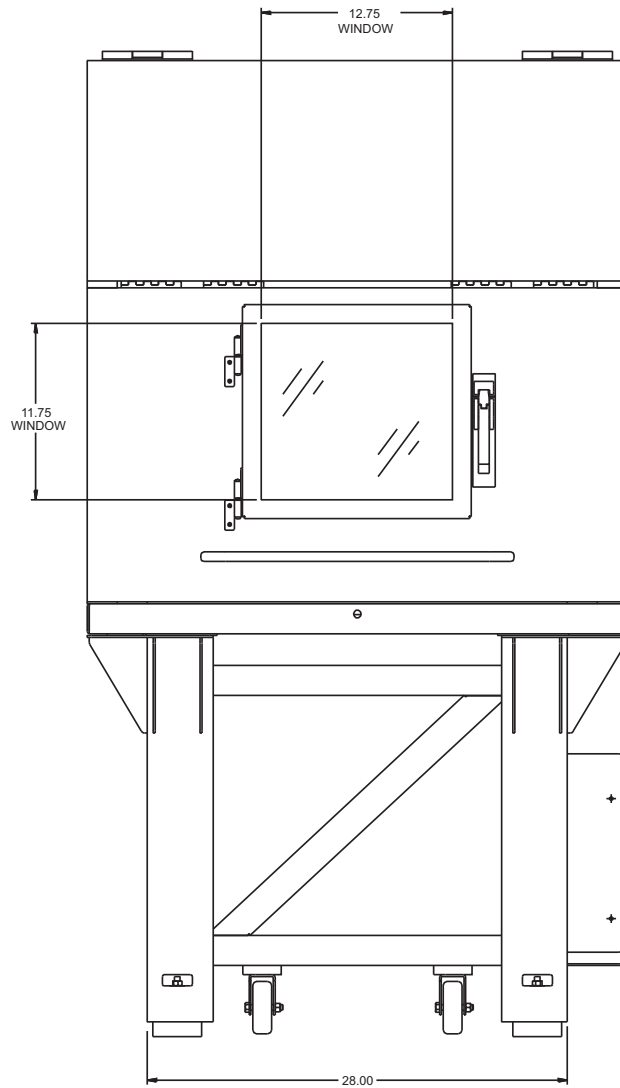
Figure 3.3d AVH-1000 Side View
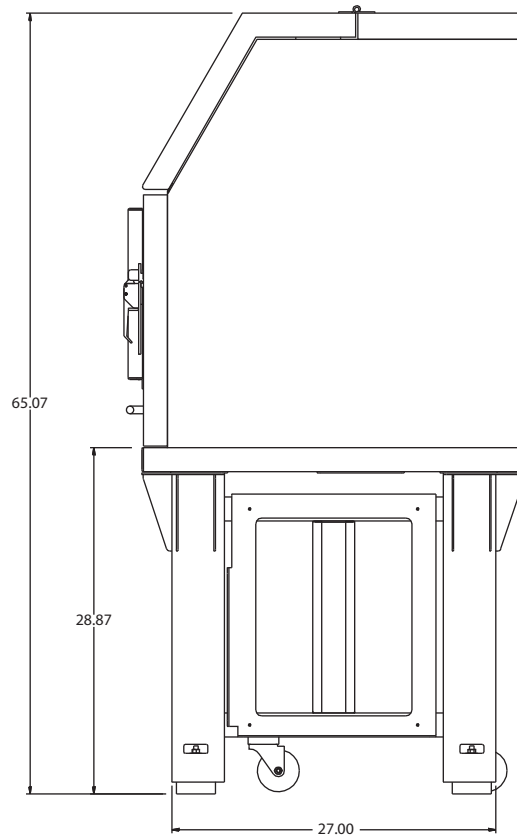
Figure 3.3e AVH-1000 Top View
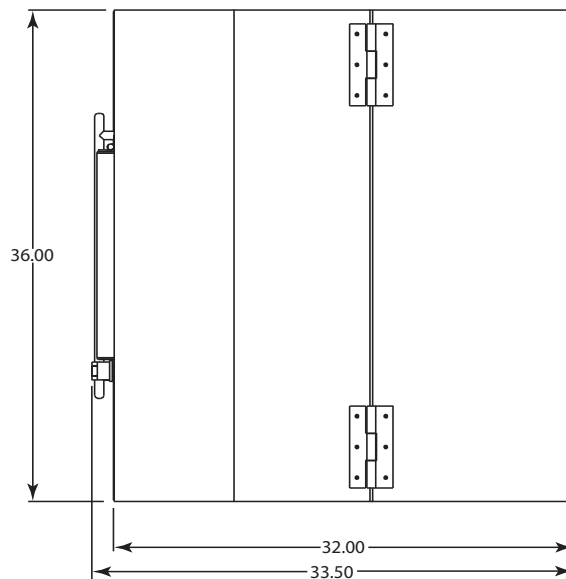
3.4   Dimension Icon Workstation

The Dimension Icon workstation, shown in Figure 3.4a, holds the NanoScope V Controller and Dimension Stage Controller in slide-out racks. The Dimension Icon computer is located to the left of the controllers.

Figure 3.4a   Dimension Icon system
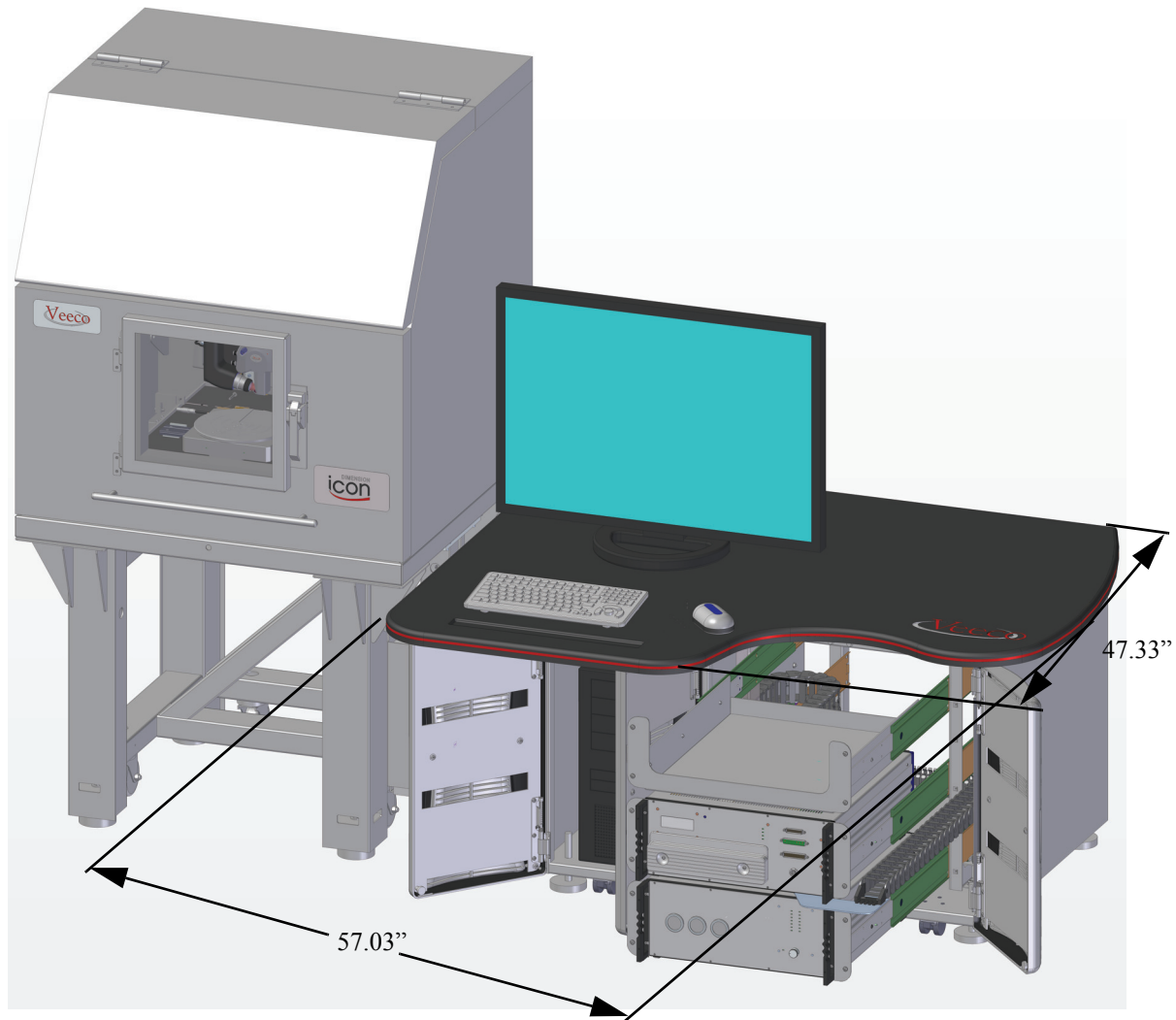
The Corian^{TM} workstation top measures 57.03" wide by 47.33" deep. When the Corian top is removed, the workstation base measures 41.5" wide x 34" deep. The workstation with computer, monitor and controllers weighs approximately 560 lbs.

3.5   Footprint

3.5.1   Dimension Icon

Several possible Dimension Icon microscope/hood and workstation configurations are shown below. Dimensions are in inches.

Figure 3.5a   Recommended configuration
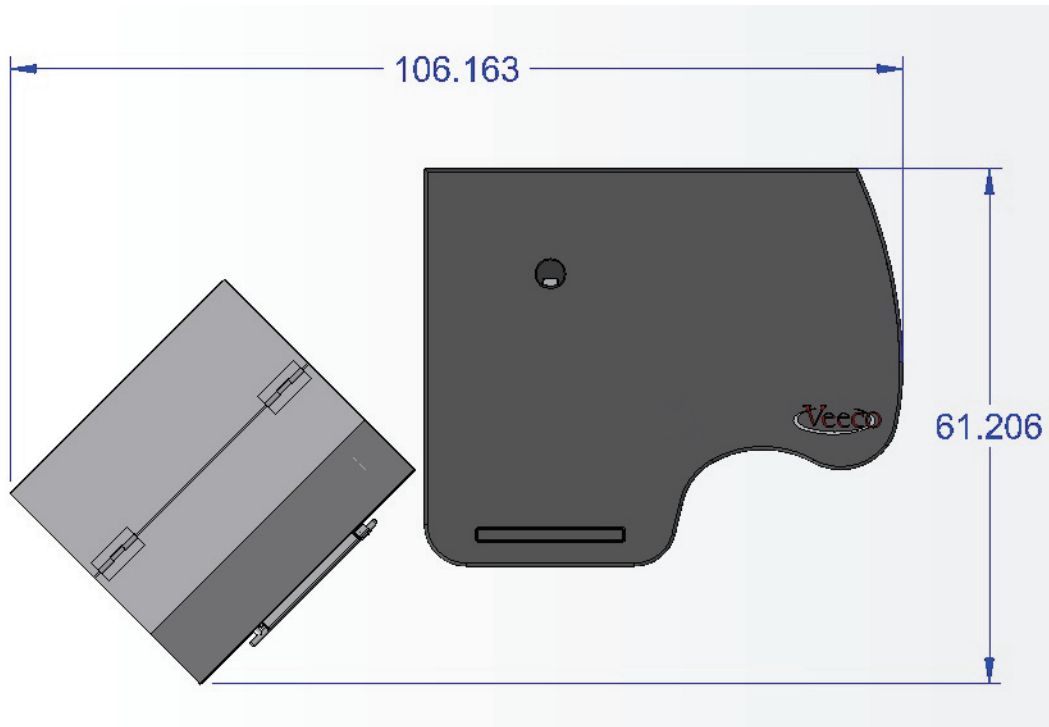
Figure 3.5b Linear configuration
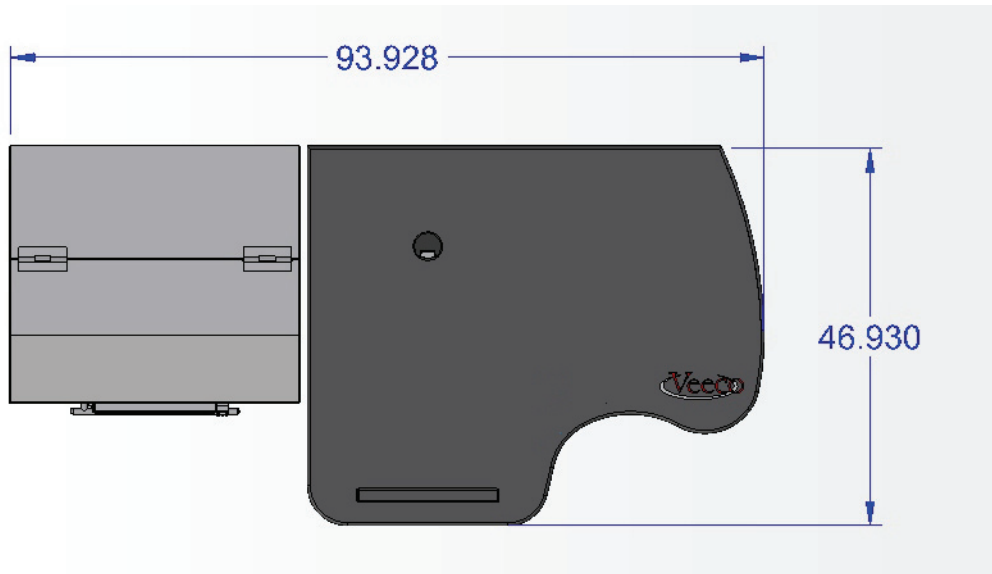
Figure 3.5c Perpendicular configuration
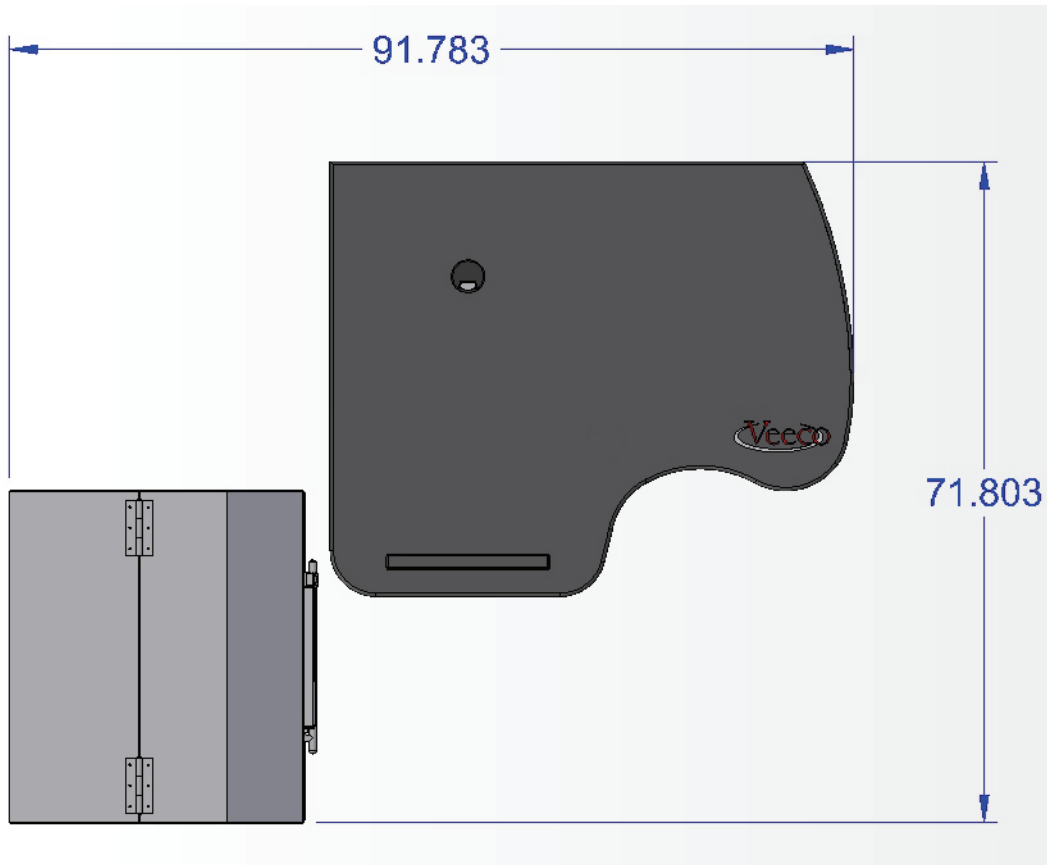
3.5.2   Dimension Icon-PI

Figure 3.5d shows the footprint of a typical Dimension Icon-PI installation.

Figure 3.5d   Dimension Icon-PI Footprint
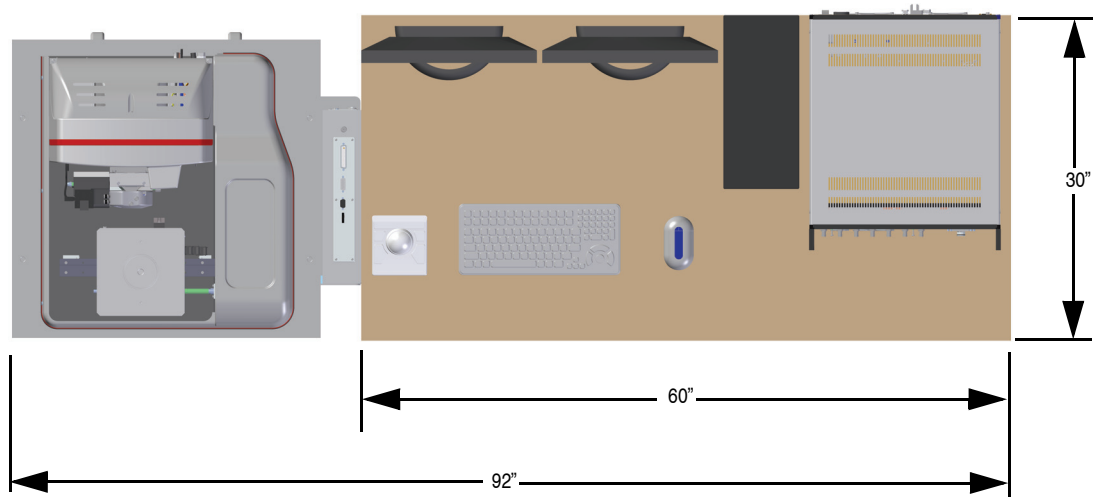
3.5.3   Dimension Icon-PT

Figure 3.5e shows the footprint of a typical Dimension Icon-PT installation.

Figure 3.5e   Dimension Icon-PT Footprint
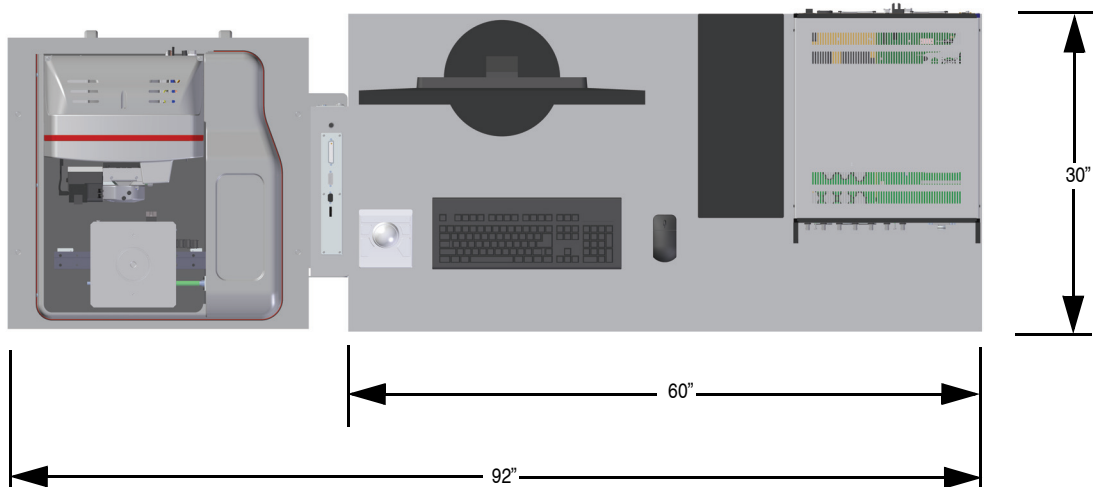
3.6   Computer/Controller Facility Requirements

For non-workstation (see **Dimension Icon-PI:** Section 3.1.1 and **Dimension Icon-PT:** Section 3.1.2) configurations, the customer must supply a computer and controller table.

Figure 3.6a   Dimension Icon-PI Control Electronics Footprint
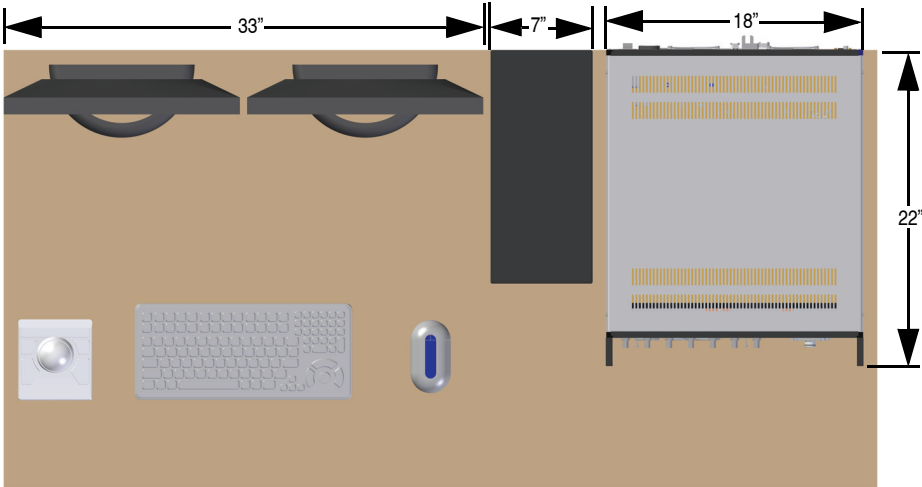
Figure 3.6b Dimension Icon-PT Control Electronics Footprint
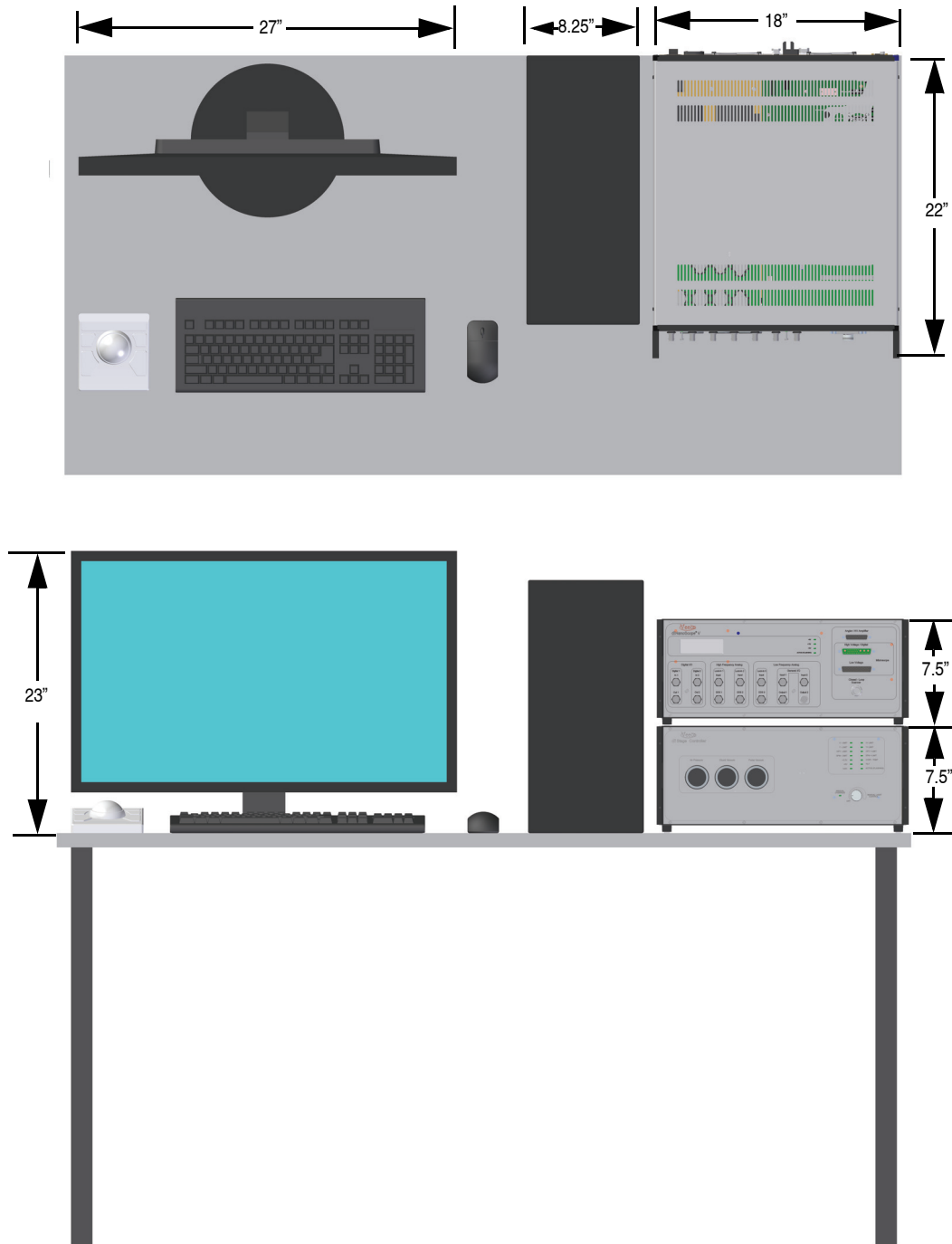
Weight: Approximately 125 lbs. (56 kg) on table top.

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

3.7   Customer Provided Assistance

Due to the size and weight of the Dimension Icon system, some user assistance will be required on the first day of installation. In particular:

1. Uncrating

2. Rolling the acoustic hood or vibration isolation table from the crate

3. Pushing the Dimension Icon microscope onto the vibration isolation table

4. Removing and replacing the workstation table top if required.

A pallet jack (maximum fork spacing 28"/71cm, maximum fork thickness 3.5"/9cm) is required to move the crate containing the Dimension Icon microscope to the acoustic hood.

> **Note:**   Veeco is not liable for injuries sustained if Veeco's instructions are not followed.

3.8   Facilities Requirements Summary

If the Dimension Icon system is used in a clean room with raised floors a pedestal must be provided to support leveling feet on the air table. 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 0.250 OD flexible house.

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

Table 3.8a   Dimension Icon Facilities Requirements Summary

Utility With	Dimension Icon with AVH-1000 Hood/Table	Dimension Icon-PI/PT with VT-1000 Table
Electrical	600W; single phase; 100V, 120V or 240V duplex outlet; dedicated circuit, 50/60Hz	600W; single phase; 100V, 120V or 240V duplex outlet; dedicated circuit, 50/60Hz
Disconnect or Shutoff	None required	None required
Clean Dry Air for Sample Chuck	Supplied with Dimension Icon	Supplied with Dimension Icon
Clean Dry Air for Air Table	Requires user supplied air 5.5 bar (80 PSI), 28 liters/min. (1 CFM)	Requires user supplied air 5.5 bar (80 PSI), 28 liters/min. (1 CFM)
Vacuum for Sample Holddown	Supplied with Dimension Icon	Supplied with Dimension Icon
Exhaust	None required	None required
Lan Connection	None required but network support card included	None required but network support card included
Approximate Footprints	915mm (36") x 813mm (32.0") hood 1448mm (57") x 1219mm (48") workstation	813mm (32.0") x 711mm (28.0") air table 1524mm (60") x 762mm (30") computer table
Hood and Table Including SPM	approx. 590 kg (1,300 lbs) evenly distributed	approx. 392kg (860 lbs) evenly distributed
Workstation with computer, monitor and controllers	approx. 255kg (560lbs)	57kg (125lbs) + customer table
Min. Door Width	864mm (34") minimum	711mm (28") minimum
Environment/Noise	See Section 3.9.	See Section 3.9.
Crated weight and dimensions of hood/air table	550kg (1200lbs) 1194mm (47") wide x 1041mm (41") deep x 1880mm (74") high. See Figure 3.8d.	281kg (617lbs) 914mm (36") wide x 864mm (34") deep x 940mm (37") high. See Figure 3.8e
Crated weight and dimensions of electronics and workstation	390kg (850lbs) 1605mm (63.25") wide x 1350mm (53") deep x 1100mm (43.25") high. See Figure 3.8b.	approx. 200kg (450lbs) 1524mm (60") wide x 1156mm (45.5") deep x 838mm (33" high). See Figure 3.8c.
Crated weight and dimensions of the microscope	250kg (550lbs) 950mm (37.25") wide x 840mm (33") deep x 1315mm (51.75") high. See Figure 3.8a.	250kg (550lbs) 950mm (37.25") wide x 840mm (33") deep x 1315mm (51.75") high. See Figure 3.8a.

Figure 3.8a Microscope crate, weight 250kg (550lbs)
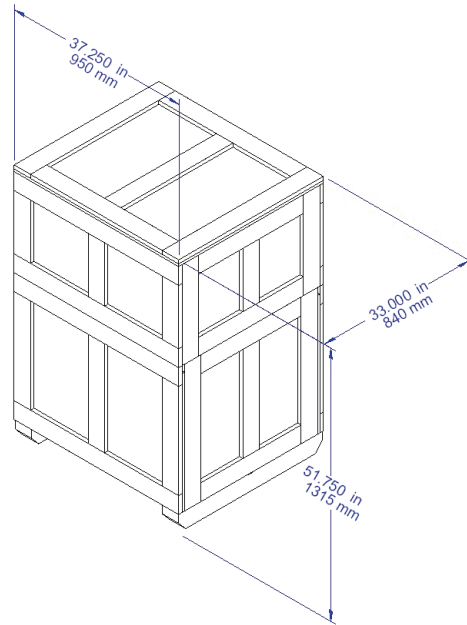
Figure 3.8b Workstation crate 390kg (850lbs). Lid is slightly larger than base.
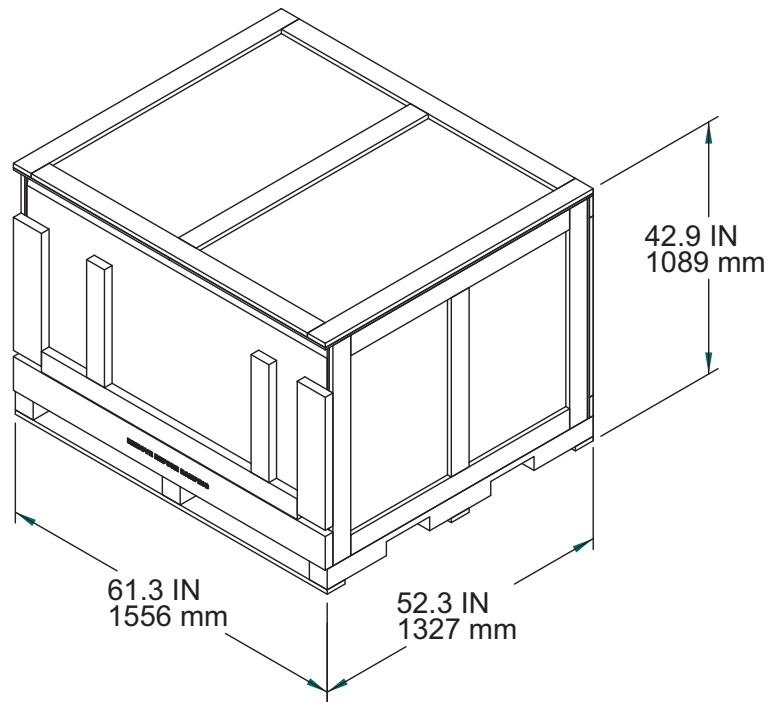
Figure 3.8c Icon-PI/PT electronics crate 200kg (450lbs)

33 IN
838 mm

60 IN
1524 mm

45.5 IN
1156 mm
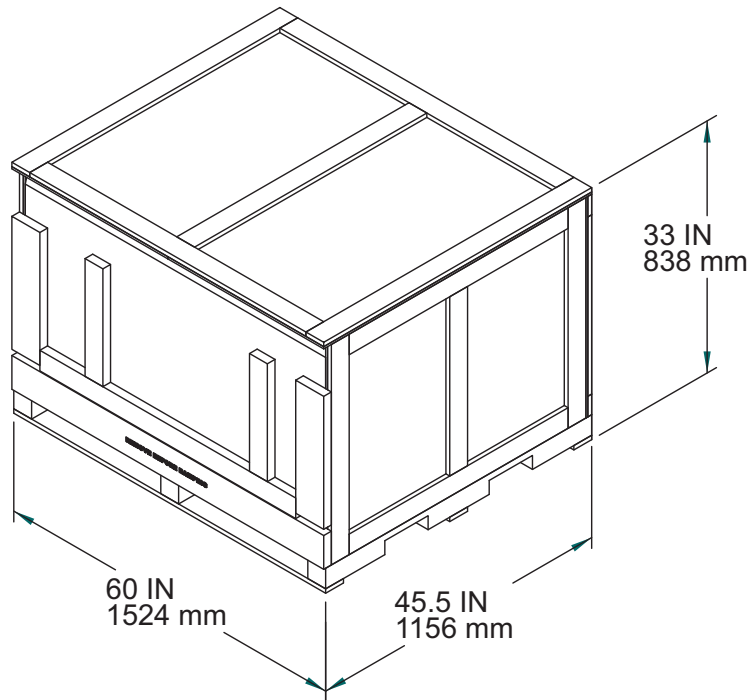
Figure 3.8d   AVH-1000 Acoustic hood crate 550kg (1200lbs)
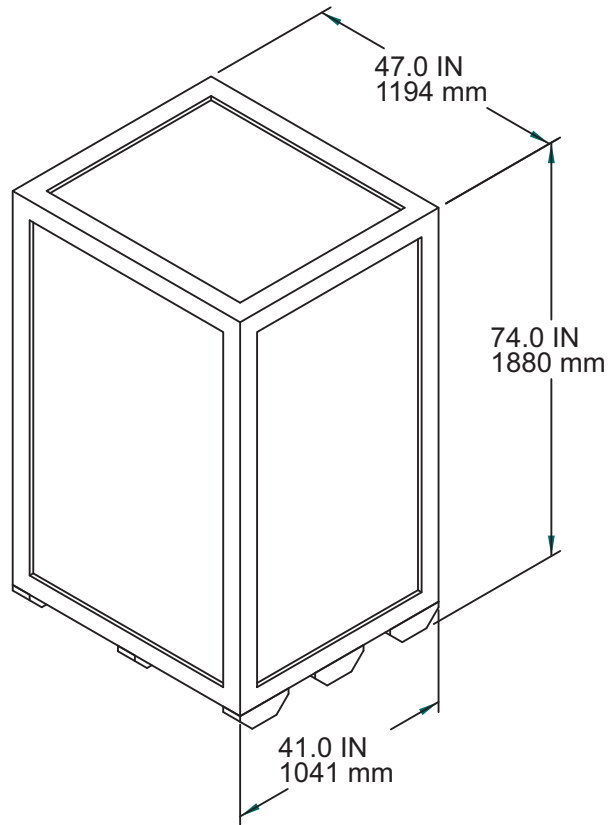
Figure 3.8e   VT-1000 air table crate (620 lbs)
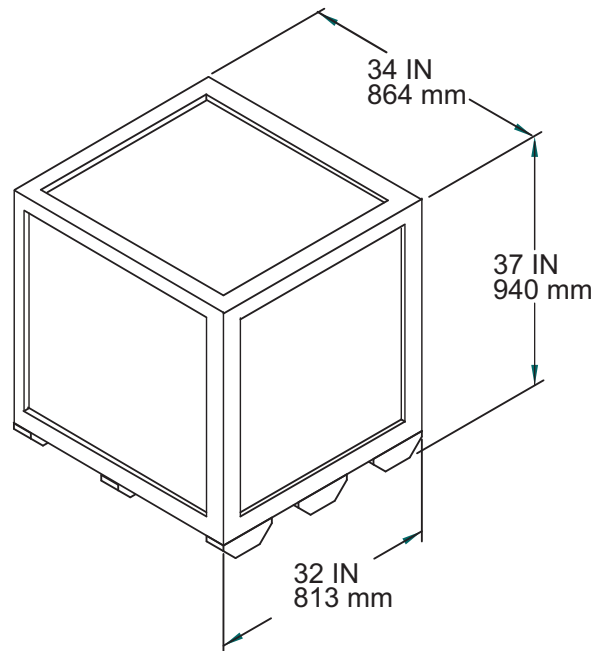
3.9   Acoustic/Vibration Specifications

The following conditions must be met in order to achieve 30 pico meters RMS (45 pico meters - Dimension Icon-PI/PT) noise specifications:

- •   Acoustic: Acoustic noise should not exceed 85dBA (Note "A" weighting).

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

Figure 3.9a   Vibration Criteria Plot
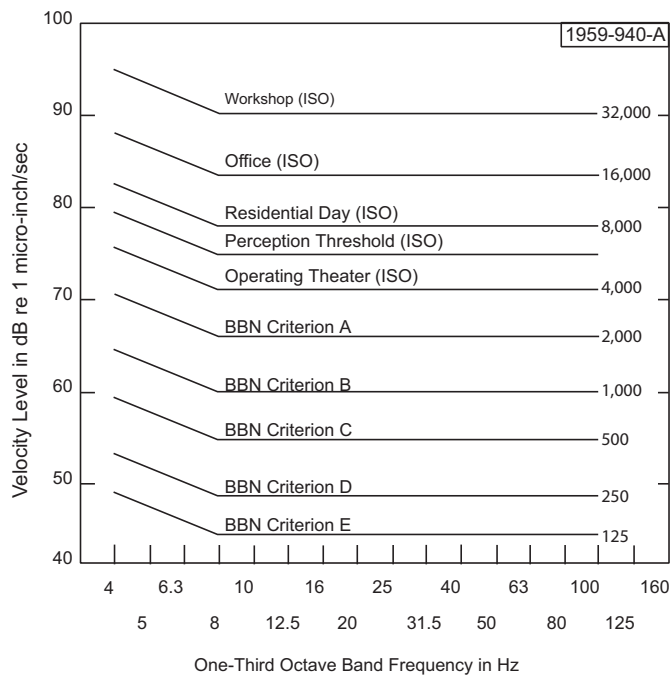
BBN Criterion A - Probe Test Equipment. 100X Microscopes
BBN Criterion B - 500X Microscopes. Aligners, Steppers to 5m Geometries
BBN Criterion C - 1000X Microscopes. Aligners, Steppers to 1.5m Geometries
BBN Criterion D - Steppers, E-Beams to 0.3m Geometries, CD Inspection Equipment.
            Most SEMs to 50,000X
BBN Criterion E - Anticipated Adequate for Future Fabrication and Test Equipment
            for Low Submicron Geometries

3.10   General Facilities Guidelines

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

- For the AVH-1000 and VT-1000 vibration isolation systems, you must provide either clean dry air or nitrogen with a pressure capacity of 80psi to float the air tables. You must provide the regulator for adjusting the pressure from the source. The source can be either a house air system or a tank.

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

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

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

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

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

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

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

- Power should be in dedicated 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

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

- **Software Power-up:** Section 4.6.3

- **Log into Windows XP:** Section 4.6.4

- **Log On:** Section 4.6.5

- **Start the NanoScope Software:** Section 4.6.6

- **Select Realtime:** Section 4.6.7

- **Begin Stage Initialization:** Section 4.6.8

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

4.1 Shipping and Receiving

4.1.1 Component List

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

Equipment Received

- Electronic copy of the Dimension Icon Instruction Manual (this document)

- Training Manual

- Dimension Stage Controller

- Dimension Icon Microscope

- Dimension Icon Microscope Head. Do not remove the SPM head from the foam padded shipping box yet.

- NanoScope V Controller

- Computer

- 30" LCD Monitor (Dimension Icon or Dimension Icon-PT) or two 19" LCD Monitors (Dimension Icon-PI)

- Hard Drive Back-up on DVD

- Mouse, Keyboard and Trackball

- Dimension Accessories Kit

- Cantilever Stand

- Probe Holders (2)

- 20 Silicon Nitride probes (model DNP-20) and 10 TappingMode probes.

- Wafer Handling Tool Kit

- Calibration Standard

- Vibration Isolation Pad. The optional integrated vibration isolation table/acoustic enclosure is *strongly* **recommended** (Model AVH-1000).

- Power distribution box

- 8" Sample Chuck (option)

Cables Received

- NS V Controller-to-Dimension Icon Cables

- Dimension Icon DC Power Cable

- Power Cords (3)

- Serial Cable, 9-pin D, 12'

- Serial Cable, 9-pin D, 6' (3)

- Vacuum Hose Assembly

- SCSI cable (NanoScope V Controller to PC)

- USB cable (NanoScope V-PI Controller to PC)

Tools Received

- Ratchet wrench, 1/4" hex

- Insert bit, #2 Phillips 1/4" hex

- Screwdriver, #1 Phillips

- L-key ball hex set english, 10 pieces

- 1/2" open end wrench

- Screwdriver, 3/16" slotted blade

- Miniature screwdriver, Phillips

- L-key, 4mm hex

- L-key, 5mm hex

- Foam tape, 2-sided, black

4.2   Uncrating the System

The Dimension Icon system typically ships in three crates:

1. Dimension Icon workstation crate. Approximate dimensions: 1605 mm wide (63.25 inches) by 1100 mm high (43.25 inches) by 1350 mm deep (53 inches). Approximate weight: 390 kg (850 lbs). The following equipment is inside this crate:

    a. Dimension Icon workstation with top installed. The NanoScope V Controller and the Dimension Stage Controller are bolted on slides inside the workstation table.

    b. 30" LCD monitor in a cardboard box

    c. Dimension Icon computer in a cardboard box

    d. X-Y stage cover in a cardboard box

<div align="center">OR</div>

2. Dimension Icon-PI/PT electronics crate. Approximate dimensions: 1524 mm (60 inches) wide by 1156 mm (45.5 inches) deep by 838 mm (33 inches) high. The following equipment is shipped inside this crate:

    a. NanoScope V Controller

    b. Dimension Stage Controller

    c. Two 19" LCD monitors in cardboard boxes

    d. Dimension Icon computer in a cardboard box

    e. X-Y stage cover in a cardboard box

3. Dimension Icon/Dimension Icon-PI/PT microscope crate. Approximate dimensions: 950 mm wide (37.25 inches) by 1315 mm high (51.75 inches) X 840 mm deep (33 inches). Approximate weight: 250 kg (550 lbs). The following equipment is inside this crate:

    a. Dimension Icon microscope

    b. Dimension Icon Electronics box ("E-box")

    c. Dimension Icon head in a foam-padded plastic carrying case

    d. Chuck base and chuck in a cardboard box

    Note: The packaging in these two boxes may vary slightly depending on accessories purchased.

4. Isolation table options:

    a. Dimension Icon: AVH - 1000 acoustic hood crate: Approximate dimensions: 1040 mm wide (41 inches) by 1880 mm high (74 inches) by 1194 mm deep (47 inches). Approximate weight: 550 kg (1,200 lbs). This crate contains the AVH-1000 acoustic hood.

    b. Dimension Icon-PI/PT: VT - 1000 vibration isolation table: Approximate dimensions: 914 mm wide (36 inches) by 940 mm high (37 inches) by 864 mm deep (34 inches). Approximate weight: 281 kg (617 lbs). This crate contains the VT-1000 vibration isolation table.

Due to the size and weight of the Dimension Icon system, some user assistance will be required for installation.

A power screwdriver for removing crate screws is required.

4.2.1 Uncrate the Dimension Icon SPM System

1. Cut and remove the shipping bands encircling the Dimension Icon shipping crate.

2. Undo the clamps connecting the crate cover to the crate bottom. See Figure 4.2a.

Figure 4.2a Undo the crate clamps

3. Remove the crate cover. See Figure 4.2b. This is a two person job.

Figure 4.2b   Remove the crate cover

4. Remove the eight screws, marked in red, and drop the front panel of the crate.

5. Remove the two bolts and washers holding the crossbar that secures the Dimension Icon microscope to the crate base.

6. Remove the wood crossbar.

7. Remove the wood side pieces from the crate. See Figure 4.2c.

Figure 4.2c   Remove the wood side pieces from the crate

8. Remove the envelope labeled tools.

9. Cut the zip tie closing the plastic drop cloth and unwrap the Dimension Icon microscope.

10. Unpack all system components. Each of the items below are shipped in separate boxes:

   • Dimension Icon Microscope. Do not remove this from the shipping crate.

   • NanoScope V-PI Controller if you have a Dimension Icon-PI.

- NanoScope V Controller if you have a Dimension Icon-PT.

- Dimension Stage Controller if you have a Dimension Icon-PI/PT.

- Dimension Electronics Box ("E-Box")

- Dimension Microscope Head

- Microscope Accessories (Dimension accessories box)

- Chuck Base Hardware (packed with Dimension Icon microscope)

- X-Y stage cover

11. 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.

CAUTION: Do not remove the plastic wrap from the microscope 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.

4.2.2   Workstation[1]

1.   Unpack all system components. Each of the items below are shipped in separate boxes:

   •   LCD Monitor

   •   Computer and Keyboard

Because the Dimension Icon workstation table top is fairly deep (47.3", 1202mm), it may need to be removed for the workstation to clear most doors. Remove the workstation top, including the two "L" brackets attached to its bottom, by unscrewing the three allen head button screws on each side of the workstation, shown in Figure 4.2d.

Figure 4.2d   Workstation base
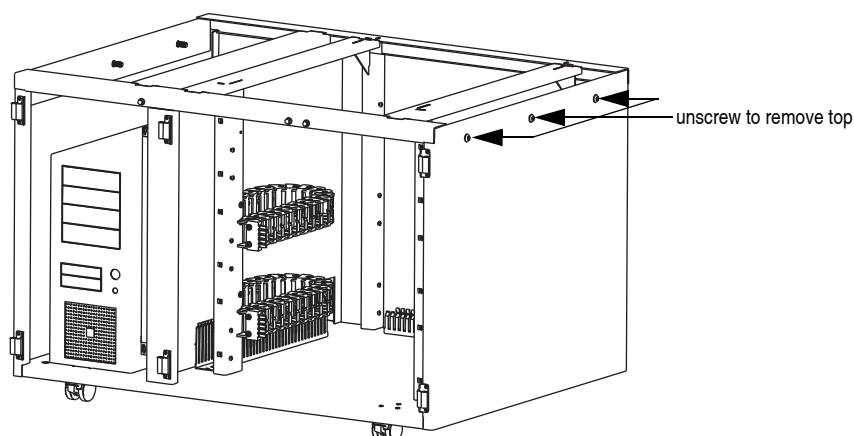
The Corian[TM] workstation top is also rather heavy (~100lbs/45kg) and requires three people to lift it. Use a four wheeled dolly to transport the top.

4.2.3   Non-Workstation

1.   Unpack all system components. Each of the items below are shipped in separate boxes:

   •   LCD Monitor(s)

   •   Computer and Keyboard

   •   Dimension Stage Controller

   •   NanoScope V Controller

---

1. The workstation configuration is not available with the Dimension Icon-PI or Dimesnion Icon-PT.

4.3   Installing the Dimension Icon System

4.3.1   Install the Dimension Icon SPM Unit

1. Move the acoustic hood/vibration isolation table to the desired location.

2. Using a pallet jack (maximum fork spacing 28"/71cm, maximum fork thickness 3.5"/9cm), move the crate containing the Dimension Icon microscope to the front of the acoustic hood.

3. Remove the red air and vacuum plugs, shown in Figure 4.3a, from the back of the Dimension Icon microscope.

Figure 4.3a   Remove the red air and vacuum plugs

4. Uncoil and unwrap the cables and hoses.

5. Attach the air and vacuum hoses to appropriate fittings, shown in Figure 4.3a, on the back of the Dimension Icon microscope.

6. Place the vibration isolation pad on the isolation table.

7. Slide the Dimension Icon microscope to the vibration isolation table.

> **Note:**   The distance from the left and front edges should equal 7" and 1" respectively.

	CAUTION:	The Dimension Icon 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 Icon microscope. Improper lifting may cause muscle strain or back injury.

	CAUTION:	Do not place your fingers under the Dimension Icon granite base when moving the microscope. They can get crushed when the granite base settles on the vibration isolation pad.

8. Carefully remove the plastic wrap from the stage.

9. Cut the zip-tie, shown in Figure 4.3b, holding the air hose and barbs to the X-axis stage.

Figure 4.3b   Cut the zip-tie

10. Using an allen wrench, remove the red shipping bracket, shown in Figure 4.3c, securing the X-axis linear bearing to the granite base.

Figure 4.3c   Remove the red shipping bracket
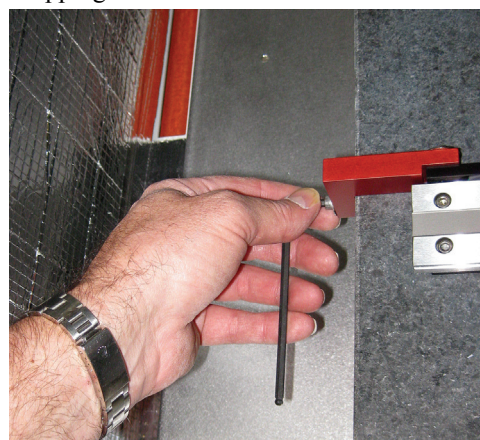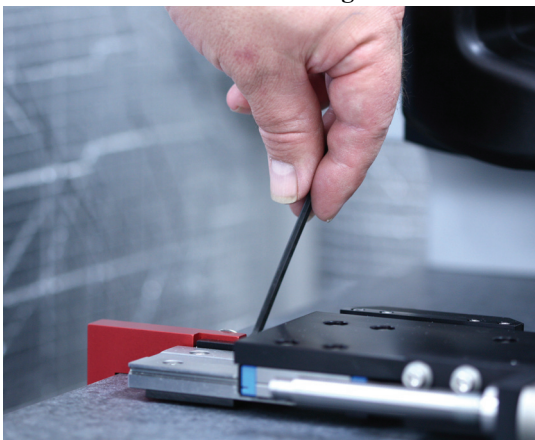
11. Wipe down the granite base with isopropyl alcohol.

12. Remove two #10 cap screws from the back of the X-Y flexure, shown in Figure 4.3d.

Figure 4.3d   Remove the cap screws from the back of the X-Y flexure
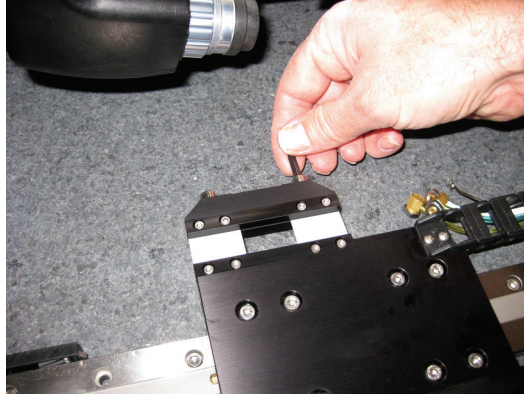
13. Attach the chuck base to the X-axis flexure using the same cap screws.

14. Attach the right angle barbs to the chuck base.

> **Note:**   A nut is attached to each barb to retain the o-ring. Remove the nuts before attaching the barbs.

15. Connect the color-coded vacuum and air hoses to the chuck base.

16. Attach the sample bias wire to the chuck base.

4.3.2 Install the Dimension Electronics Box on the base of the vibration isolation table

1. Slide two bolts through the front of the bracket attached to right legs of the AVH-1000 or VT -1000 vibration isolation table and screw them into the captured nuts on front side of the E-box. See #1 in Figure 4.3e.

2. Slide two bolts through the bottom of the bracket attached right legs of the AVH-1000 or VT-1000 vibration isolation table and screw them into the captured nuts on bottom of the E-box. See # 2 in Figure 4.3e.

Figure 4.3e   Attaching the E-box to the AVH-1000 or VT-1000 frame. AVH-1000 shown.
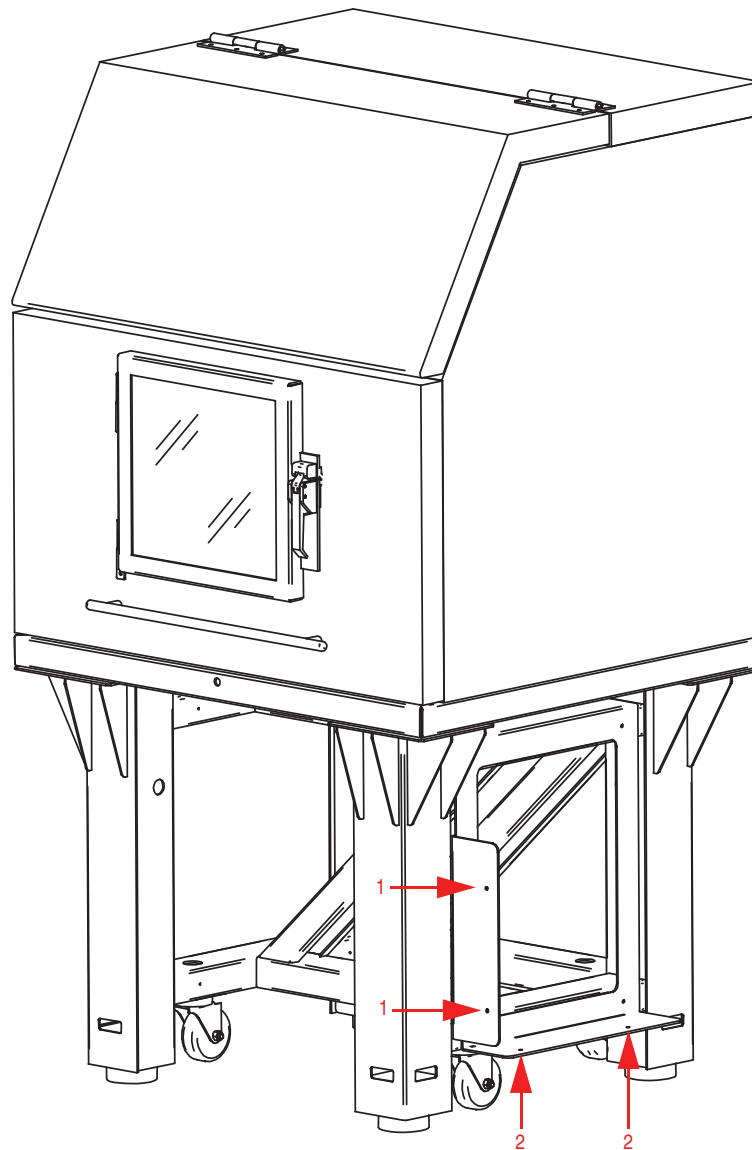
4.3.3 Route the Air Table Cabling

1. Route all cables connected to the Dimension microscope through the slot, shown in Figure 4.3f, located in the far right side of the AVH-1000 vibration isolation table. See Figure 4.3g.

Figure 4.3f AVH-1000 Air Table showing slot for cables
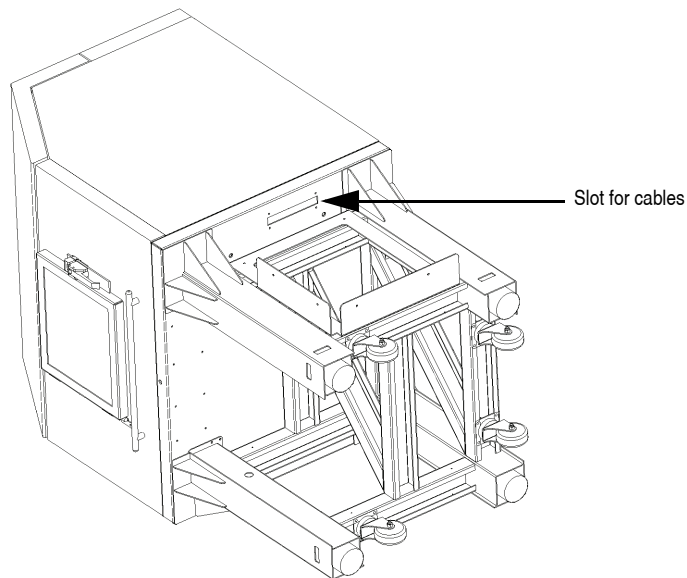
Figure 4.3g Route the cables through the slot in the vibration isolation table
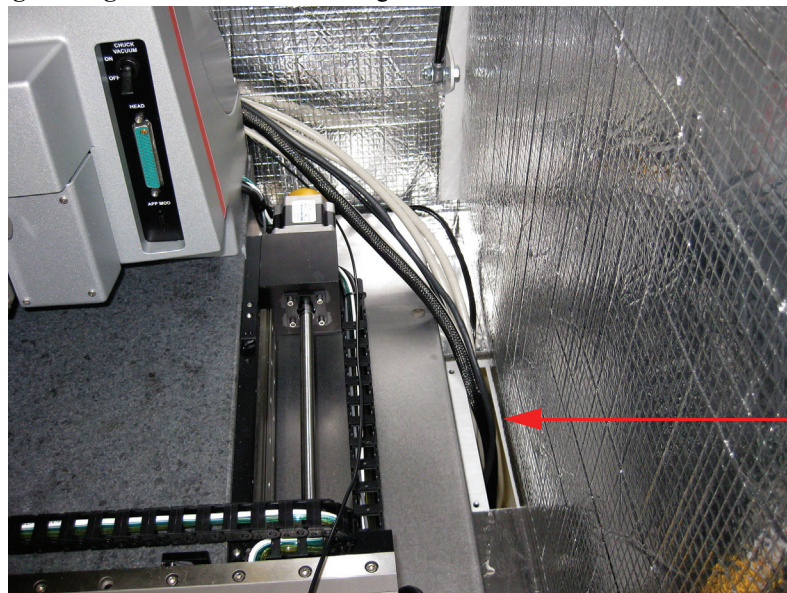
2. Bolt the inside half of the cable clamp, shown in Figure 4.3h, to the holes adjacent to the cable slot on the underside of the air table. See Figure 4.3i.

Figure 4.3h Cable Clamp
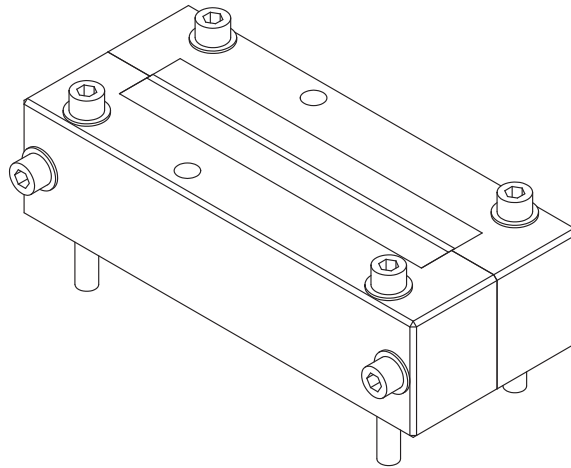
Figure 4.3i Install the inside half of the cable clamp

3. Unpack the X-Y stage cover and place it over the microscope as shown in Figure 4.3j.

Figure 4.3j   Place the cover over the microscope

4. The cables should have a rounded stress relief as shown in Figure 4.3k.

Figure 4.3k   Rounded corner for cables

5. Place two foam cable routing pieces, shown in Figure 4.3l, between the cables and the hood. These will prevent the cables from touching the acoustic hood wall.

Figure 4.3l   Foam pads to route cables
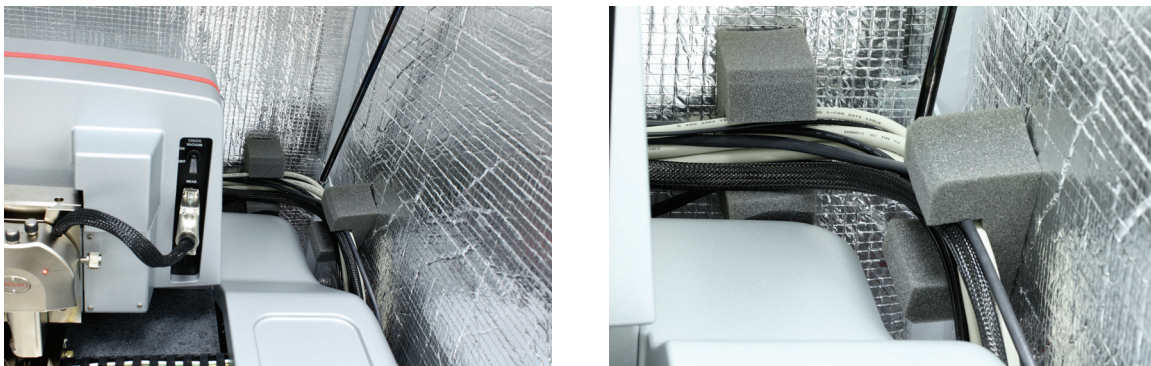
6. Use a phillips head bit in a right angle ratchet wrench to tighten the two screws on the right side of the cover. Push the front of the cover towards the granite base to tighten the right front screw.

Figure 4.3m   Push the front of the cover in to tighten the right front screw.

> ⚠️ **CAUTION:**   Do not attempt to move the Dimension Icon by pushing on the covers.

7. Tighten the three screws of the left side of the stage cover.

8. Loosely bolt the other half of the cable clamp to the half attached to the AVH-1000 vibration isolation table.

9. Ensure that there is slack in the cables between the cable clamp on the vibration isolation table and the cable clamp at the rear of the Dimension Icon microscope.

10. Tighten the halves of the cable clamp attached to the vibration isolation table.

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

4.3.4   Install the Control Station

User-supplied workstation

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

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

2. Place the following input and display equipment on the control station (see Figure 4.3n and Figure 4.3o):

    •   Monitors

    •   Keyboard

    •   Mouse and Mouse Pad

    •   Trackball

Figure 4.3n   Dimension Icon-PT Input and Display Equipment
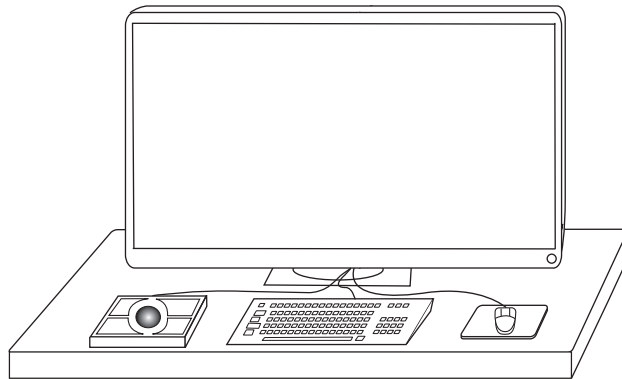
Figure 4.3o   Dimension Icon-PI Input and Display Equipment
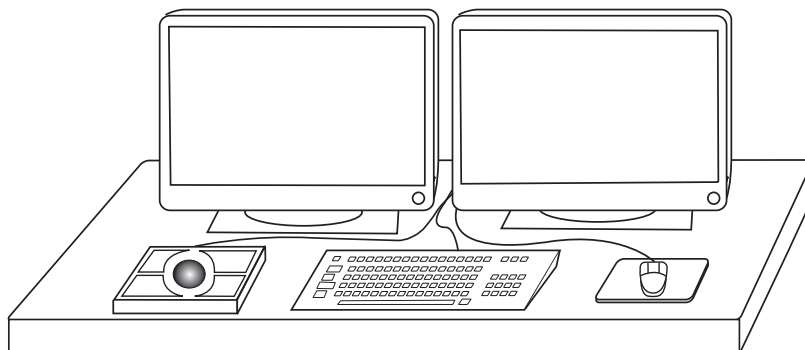
3. Place the computer on the side of the input and display devices closest to the Dimension icon microscope.

4. Place the NanoScope V controller and Dimension Stage Controller next to the computer. See Figure 4.3p for a typical Dimension Icon-PT configuration and Figure 4.3q for a typical Dimension Icon-PI configuration.

⚠	**CAUTION:**	Do not set the Dimension Stage Controller on its side.
	CAUTION:	Do not set the Dimension Stage Controller on the same table as the Dimension icon microscope. Vibrations from the vacuum pump affect imaging performance. You may stack the NanoScope V controller atop the Dimension Stage Controller.
	CAUTION:	Do not obstruct the ventilation slots on the top of the NanoScope V or Dimension Stage Controllers.

Figure 4.3p Typical Dimension Icon-PT Configuration
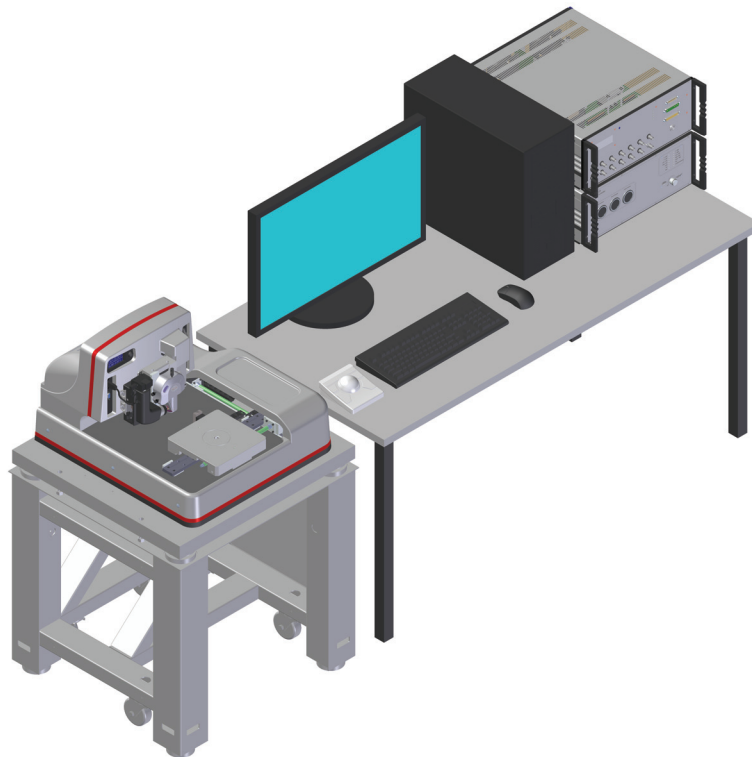
Figure 4.3q Typical Dimension Icon-PI Configuration
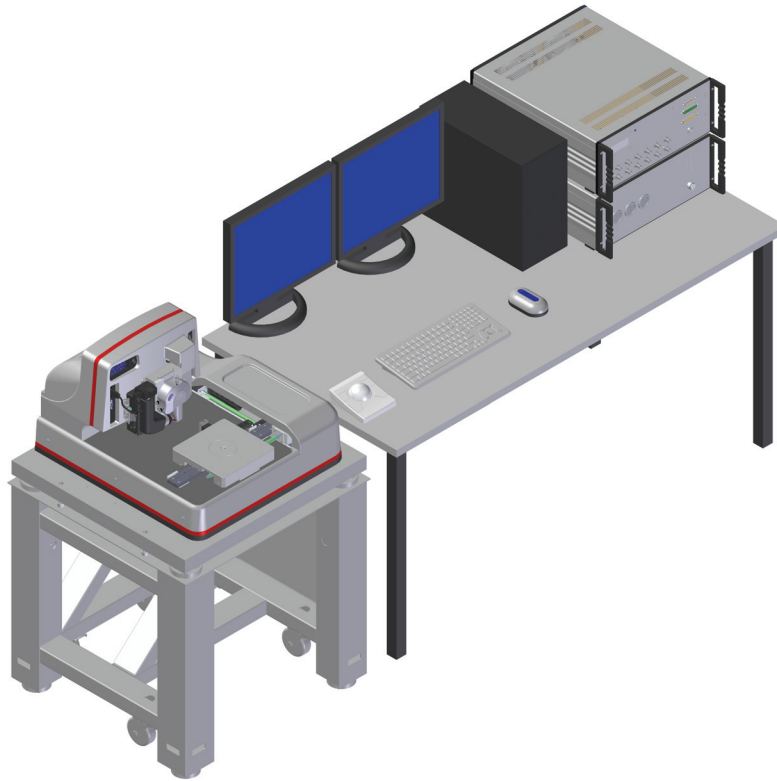
AVH-1000 Workstation Configuration

1. Set the workstation table on the right side of the Dimension Icon microscope.

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

2. Place the following input and display equipment on the control station table top (see Figure 4.3r):

   • Monitor

   • Keyboard

   • Mouse and Mouse Pad

   • Trackball

Figure 4.3r Dimension Icon Input and Display Equipment

Trackball   Keyboard   Mouse
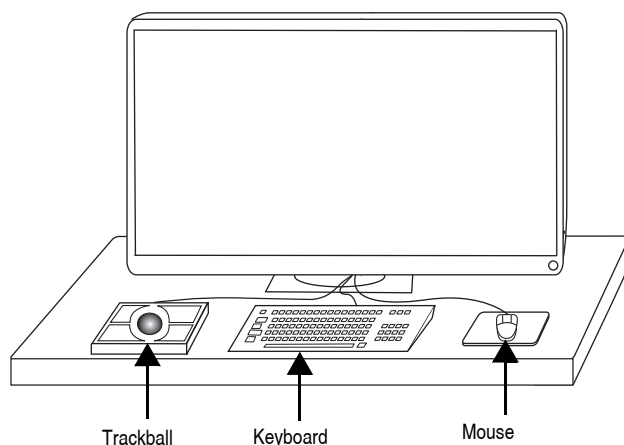
3. Place the computer in the left bay of the workstation cabinet.

4. Attach the Dimension Stage Controller to the bottom slide on the right of the workstation cabinet.

5. Attach the NanoScope V Controller to the middle slide on the right of the workstation cabinet.

6. Attach the empty shelf to the top slide in the workstation.

7. Route the cables

CAUTION:	Do not set the Dimension Stage Controller on its side.	
CAUTION:	Do not set the Dimension Stage Controller on the same table as the Dimension Icon microscope. Vibrations from the vacuum pump affect imaging performance. You may stack the NanoScope V controller atop the Dimension Stage Controller.	
CAUTION:	Do not obstruct the ventilation slots on the top of the NanoScope V or Dimension Stage Controllers.	

4.4 Connecting the Dimension Icon System

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

4.4.1 Dimension Icon Control Station Connections

Connect the Display and Input Devices

1. Connect the monitor power cord to the power strip, but do not initiate power.

2. Connect the monitor video cable to the computer (see #5 in Figure 4.4a and Table 4.4a).

3. Connect the following input devices to the computer (see Figure 4.4a and Table 4.4a):

   • Keyboard (#2)

   • Mouse (#2)

   • Trackball (COM 1 - #8)

   > **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.

Connect the Computer

	CAUTION:	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 4.4a Dimension Icon Computer (rear view)
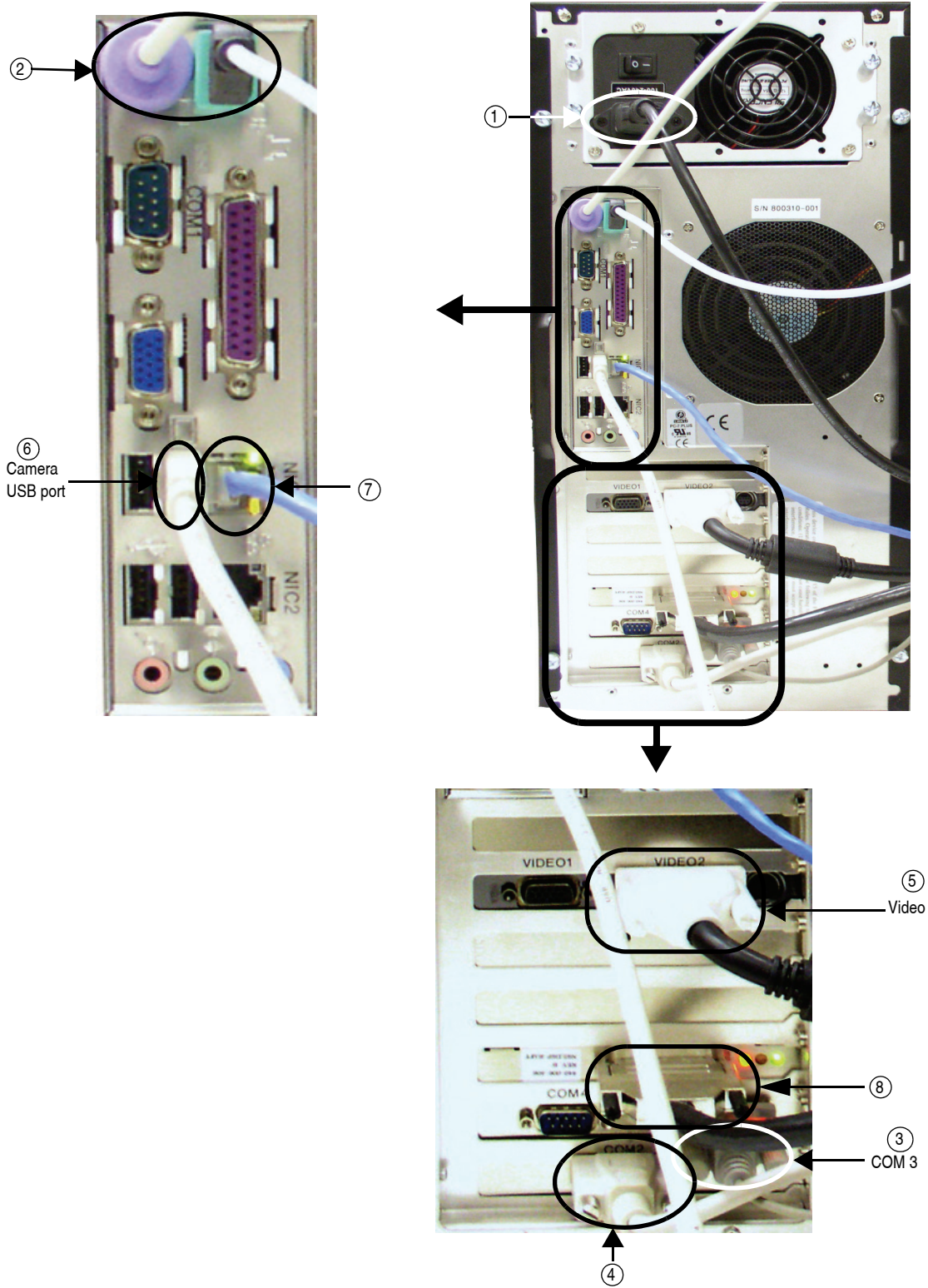
Figure 4.4b Dimension Icon-PI Computer (rear view)
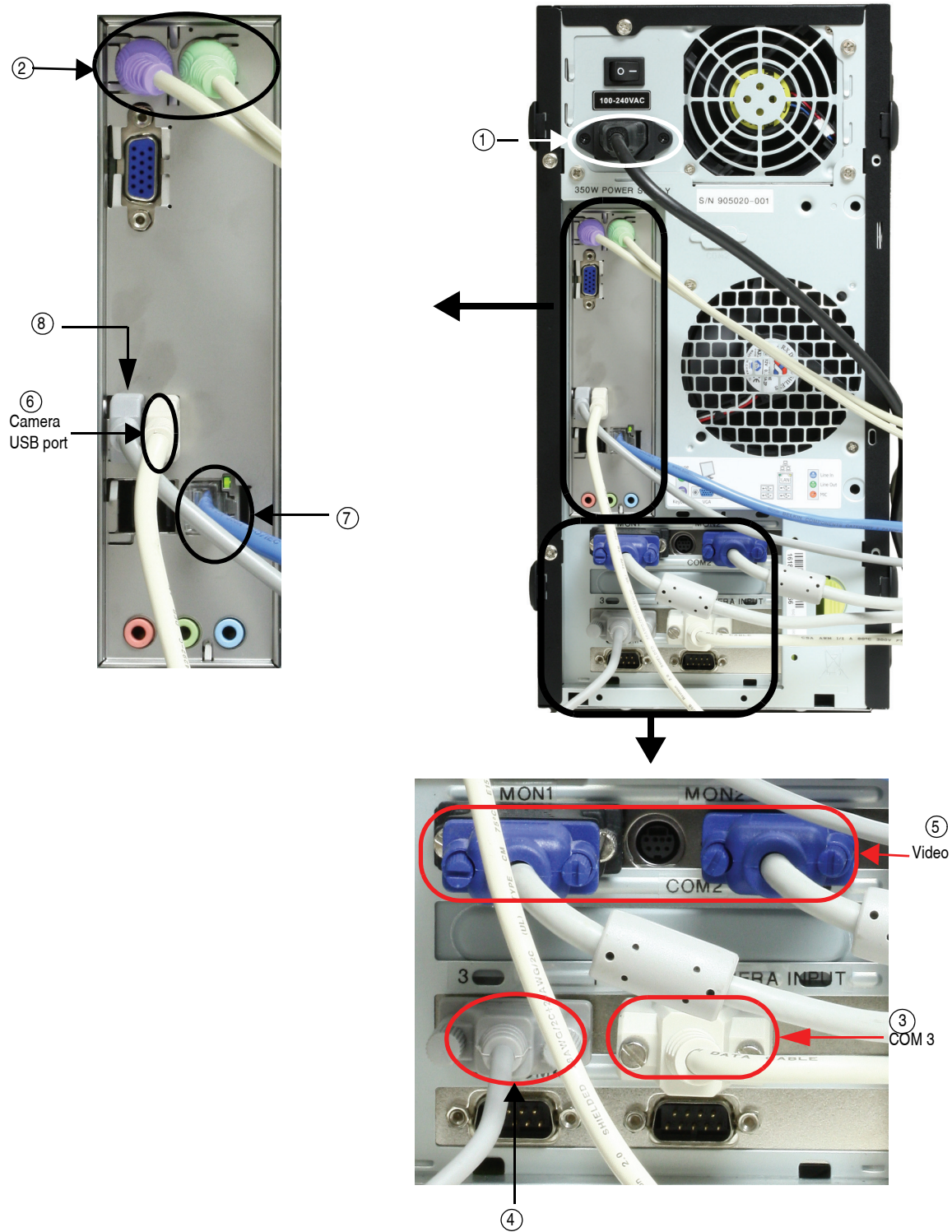
> **Note:** The computer ships with the network board disabled to avoid error messages for computers not used on a network.

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 4.4a, Figure 4.4a and Figure 4.4b for list of extensions, connection information and connection location.

Table 4.4a   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 (COM1)
4	Serial Cable (12')	464-000-010	Dimension Back Panel	Motor control for stage (COM2)
5	Monitor Cable	n/a	Monitors	Video signals
6	USB Cable	n/a	Dimension Back Panel	Optical image signal from camera
7	Cable, RJ45 LAN	n/a	Host	Network connection
8	Cable, 68-pin micro-SCSI		NanoScope V Controller	Computer to NS V controller electronic interface
8	Cable, USB		NanoScope V-PI Controller	Computer to NS V-PI controller electronic interface

4.4.2 Connect the NanoScope Controller

Connections to the microscope are made through connectors on the front of the NanoScope V Controller. See Table 4.4b for descriptions of the main front panel connectors.

Plug the cables from the connectors labeled **NS5 - High Voltage/Digital** and **NS5 - Low Voltage** on the rear of the E-box (see Figure 4.4f) into the NanoScope V Controller connectors labelled **MICROSCOPE** (**High Voltage/Digital** and **Low Voltage Analog**) at the right front of the controller (see Figure 4.4c).

Connect a cable from the connector labelled **NS5 - Closed Loop Scanner** on the rear of the E-box (see Figure 4.4f) to the **Closed Loop Scanner** connector at the right front of the NSV controller (see Figure 4.4c).

If you have a PicoAngler, connect it to the **Angler** connector at the right front of the NSV controller (see Figure 4.4c).

Table 4.4b   NanoScope V Controller, Main Connections

Connector Label	Connector Type	Purpose
High Voltage/Digital	36-pin combo D-sub, female	Piezo drive/digital control between controller and E-box/SPM
Low Voltage	78-pin DB, male	Differential low voltage analog/digital (LVDS) signals between controller and E-box/SPM
Angler	25-pin DB, female	Communicates with PicoAngler
Computer (back panel)	68-pin micro-SCSI, female	data/control between NS V Controller and computer
Expansion (back panel)	USB type B	data/control between NS V-PI Controller and computer
Closed Loop Scanner	14-pin Lemo, female	input/output to the XYZ Closed Loop Scanner Head
Digital I/O	BNC, female	TTL compatible synchronization signals (i.e. EOL and EOF)
High Frequency/ Analog	BNC, female	Used as either a lock-in input or for HSDC.[a] Sample rate is 50 MS/s.
Low Frequency Analog	BNC, female	Used for external measurement. Can bring up as a data type in NanoScope software.[b]

a.This option is not available on the Dimension Icon-PI.
b.This is an optional feature on the Dimension Icon-PI and requires a software key.

Figure 4.4c NanoScope V Controller (front view)
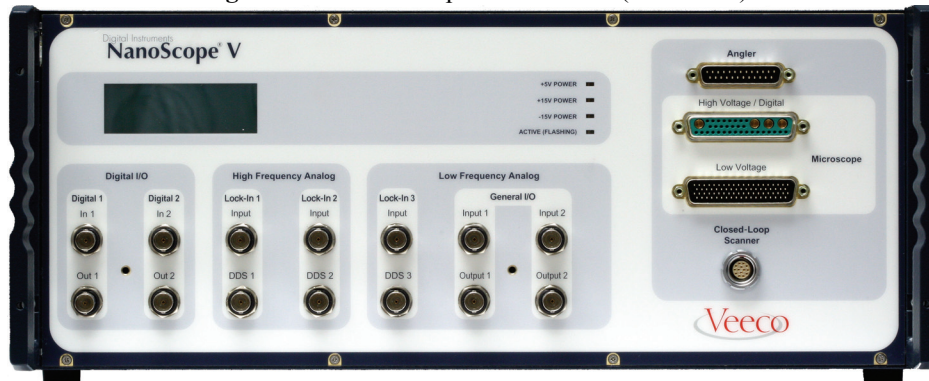
Figure 4.4d NanoScope V Controller (rear view)

Figure 4.4e NanoScope V-PI Controller (rear view)

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

CAUTION: Do not remove or install the cables while the NanoScope V Controller is powered-up or in operation.

Figure 4.4f Dimension Microscope Electronics Box (rear view)

To NS V Front Panel

HV AMPLIFIER -
HIGH VOLTAGE

NS5 -
HIGH VOLTAGE
/ DIGITAL

NS5 -
LOW VOLTAGE

RS-232

NS5 -
CLOSED
LOOP
SCANNER

CLH to NS V Front Panel
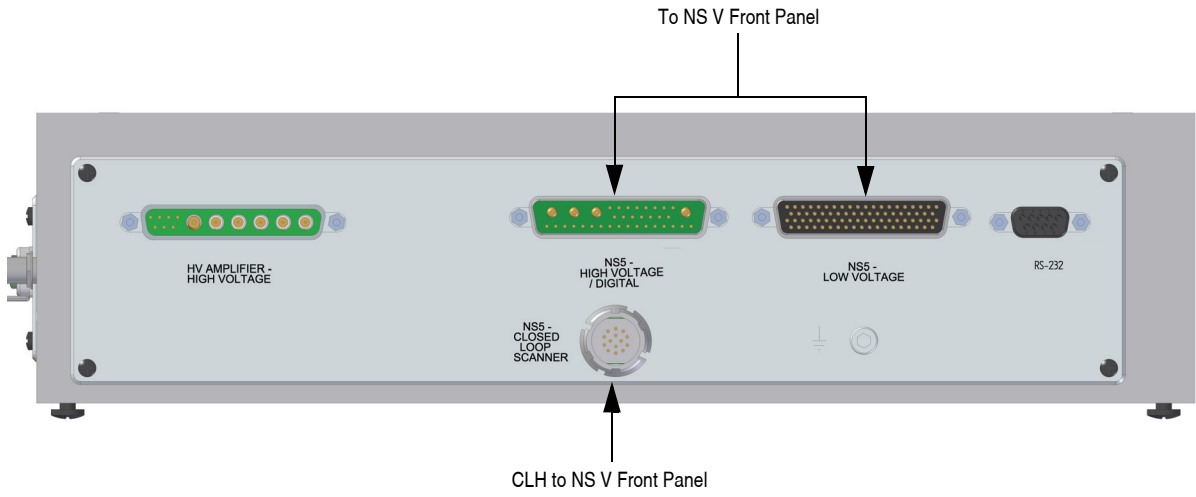
4.4.3   Connect the Dimension Stage Controller

1. Connect cables to the **Dimension Stage Controller**. See Table 4.4c and Figure 4.4g for list of cables, connection information and connection location.

> **Note:**   The Dimension Icon cables are bundled through the cable clamp provided if the system includes an acoustic hood. (See section titled **Route the Air Table Cabling:** Section 4.3.3).

Table 4.4c   Dimension Controller Connections

Fig Ref	Cable	Part Number	Box	Function
1	AC Power	466-000-004	Power Strip	Dimension controller Power
P6/ P7	Vacuum Hose Assembly	860-000-012	Dimension Icon	Vacuum and air supply
P1	Serial Cable (6')	464-000-024	Computer	Serial communication w/ computer
P3	Serial Cable		NS V Controller	Crash protection
P4	37 pin Cable		Dimension Icon	Motion Control
P5	37 pin Cable		Dimension Icon	Motion Control

Figure 4.4g   Dimension Controller (rear view)
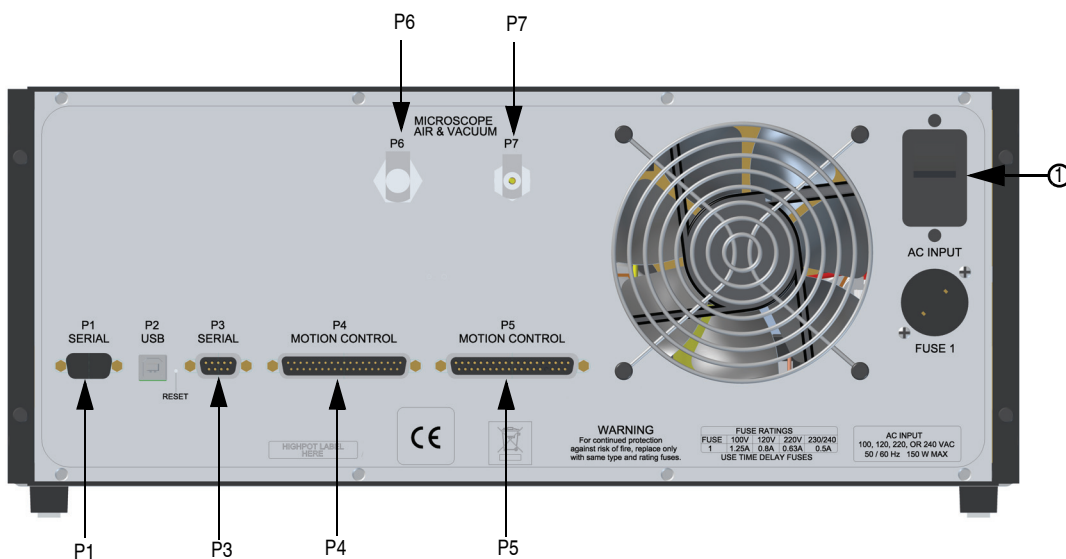
Figure 4.4h Dimension Controller (front view)
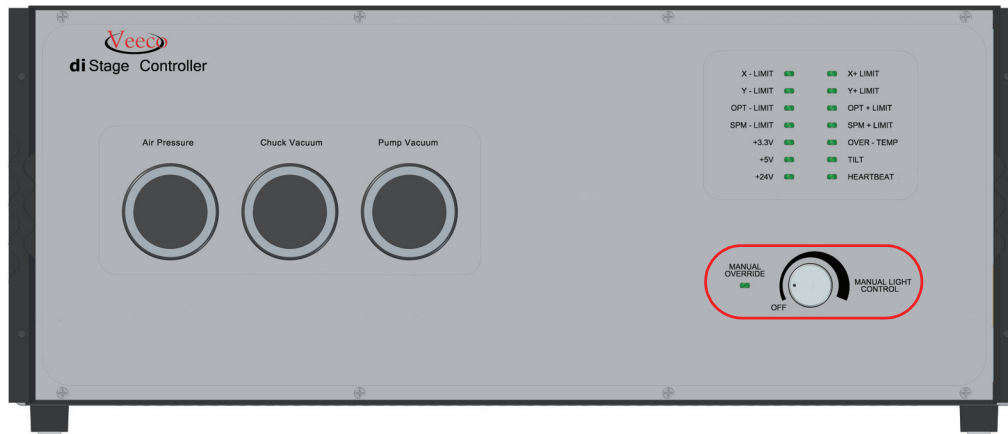
2. To set illumination, rotate the manual light control switch on the lower right front of the Dimension controller to **ON** for manual control or **OFF** for computer control (see Figure 4.4h).

> **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 in the **Align** and **Navigate** windows.

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

4. Toggle the chuck vacuum switch, located on the front of the microscope unit, to **OFF** (see Figure 4.4i).

Figure 4.4i Chuck vacuum switch

4.4.4 Dimension Icon Microscope Electronics Box Connections

1. Connect the following cables from the back of the Dimension Icon microscope to the top of Electronics Box, shown in Figure 4.4j. See Table 4.4d and Figure 4.4j for list of extensions, connection information and connection location.

Table 4.4d   Dimension Icon Microscope Electronics Box Connections

Fig Ref	Cable	Part Number	Box	Function
1	Application Module Sensor Cable, 15-pin D	465-000-015	Application Module	Application Module control
2	Heater/Cooler Serial Cable, 15-pin D	820-017-606	Heater/Cooler	Heater/Cooler control
3	LCD Serial Cable, 15-pin D		Dimension Icon LCD	LCD control
4	Microscope Head cable, 47-pin		Dimension Icon head	Microscope head control
5	Sample bias cable, coax-SMA		Application Module	Sample bias control

Figure 4.4j   Dimension Microscope Electronics Box (top view)
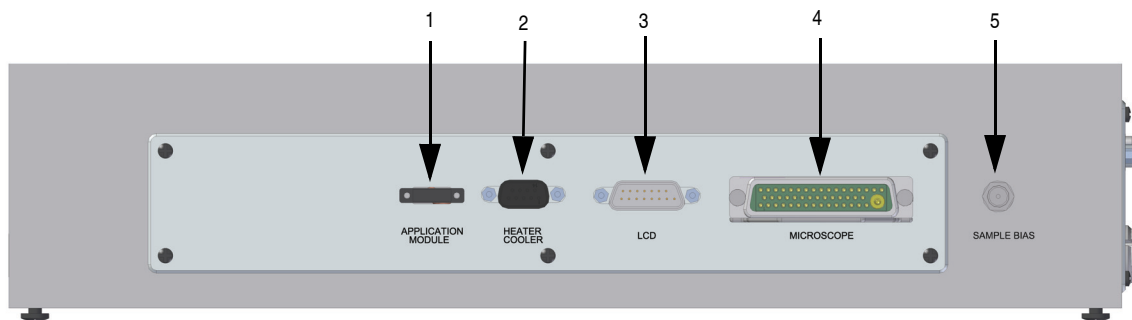
4.5   Float the Air Table

After you have connected the air table, slowly adjust the pressure regulator until all isolators start to float. Then adjust the regulator to 5-10 PSI above that point.

4.6   System Power-up

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

4.6.1   Prepare the System for Power-up

1. Plug the PC, monitor(s), NanoScope V Controller and Stage Controller power cables into the outlets on the power distribution box, shown in Figure 4.6a.

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

3. Verify that all cables are connected properly, especially the NanoScope V SPM controller to the Dimension 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 cable that connects the microscope head to the microscope front panel.

4.6.2   Power-up Sequence

1. Turn on the system power switch, shown in Figure 4.6a, located near the PC.

2. Turn on the monitor(s) using the push-button switch located on the front of the monitor(s).

3. Turn on the computer using the push-button switch located on the front of the computer.

4. Turn on the NanoScope V controller using the power switch located on the rear of the NanoScope V controller.

5. Turn on the Dimension controller, using the power switch on the rear of the Dimension controller.

Note: Because the NanoScope V Controller and the Dimension Stage Controller power switches are on the back of their cabinets, leave their power switches on when you power-off or power on your Dimension Icon system. Normal power-on consists of simply turning on the system power switch.

Figure 4.6a Dimension Icon power distribution box and system power switch
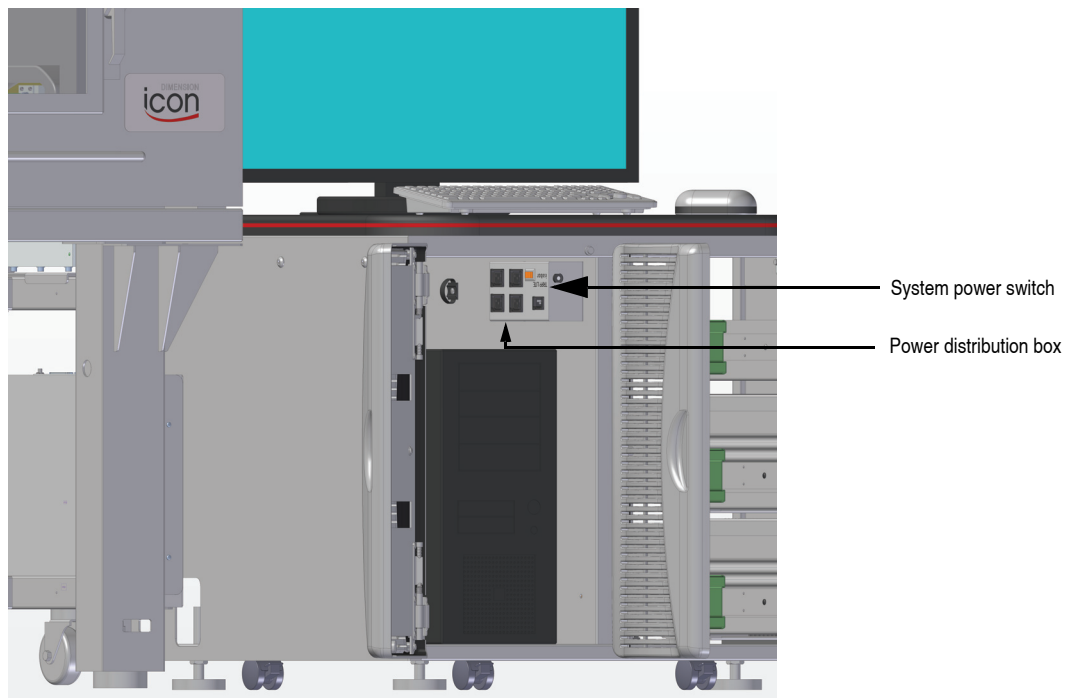
The Dimension Icon-PI system uses the same power distribution switch as the Dimension Icon (workstation) system; hence the same power-up sequence.

4.6.3 Software Power-up

⚠	**CAUTION:**	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.
	VORSICHT:	An dieser Stelle müssen die „Pre Power-up" und „Power-up" Checklisten komplett sein, bevor Sie fortfahren.

4.6.4 Log into Windows XP

1. Press **CTRL-ALT-DELETE** to log into Windows.

4.6.5 Log On

In the **Logon Information** window, enter the default settings into the User Name and Password fields.

1. **User Name:** `Nanoscope`

2. **Password:** (Leave *blank*)

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

4.6.6 Start the NanoScope Software

Go to the desktop and click on the NanoScope icon or select the **D:\SPM\z.exe** file to start the NanoScope software (see Figure 4.6b).

Figure 4.6b Select the NanoScope Icon

4.6.7 Select Realtime

Click **EXPERIMENT** > **SELECT EXPERIMENT** or the **SELECT EXPERIMENT** icon, in the top left of the NanoScope software window. See Figure 4.6c. The system automatically initializes for approximately one minute.

Figure 4.6c Click the **SELECT EXPERIMENT** icon to start
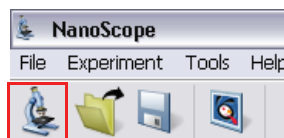
The stage may need to be initialized any time the system or one of its components has been powered-down. This allows the stage controller and computer to locate home positions and reset the coordinate system of the stage. If the **Engage** and **Withdraw** icons are grayed out and/or the messages shown in Figure 4.6d are displayed, the stage needs to be initialized.

Figure 4.6d   Stage Not Initialized
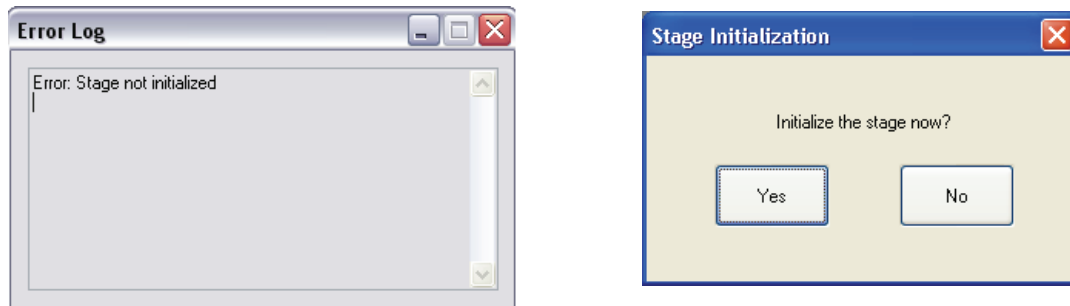
4.6.8   Begin Stage Initialization

- Select **STAGE** > **INITIALIZE**.

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

Chapter 5   Stage System

The Dimension Icon Scanning Probe Microscope (SPM) features a large sample stage capable of high-precision positioning for such materials 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:

- **Mounting of Samples:** Section 5.1
  - **Vacuum Chucks:** Section 5.1.1
  - **Magnetic Pucks:** Section 5.1.2
  - **Axis Orientation:** Section 5.1.3
- **Stage Menu Commands:** Section 5.2
  - **Align:** Section 5.2.1
  - **Navigate:** Section 5.2.2
  - **Load/Unload Sample:** Section 5.2.3
  - **Move To (X,Y):** Section 5.2.4
  - **Set Reference:** Section 5.2.5
  - **Programmed Move:** Section 5.2.6
  - **Initialize:** Section 5.2.7
- **SPM Parameters:** Section 5.3

5.1 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.

5.1.1 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.

⚠️	**CAUTION:**	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. Dispose of wipes in an appropriately labelled solvent-contaminated waste container. When cleaning dust from the stage area, use a vacuum cleaner with a soft brush. DO NOT clean stages using compressed air.

5.1.2 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.

5.1.3   Axis Orientation

Motorized X-Y Stages

When viewing the Dimension Icon 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 5.1a).

Figure 5.1a   Stage X-Y Axis Orientation
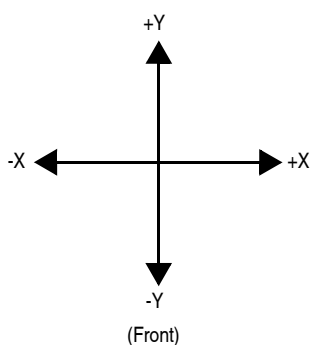
5.2   Stage Menu Commands

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

- **Align:** Section 5.2.1

- **Navigate:** Section 5.2.2

- **Move To (X,Y):** Section 5.2.4

- **Set Reference:** Section 5.2.5

- **Programmed Move:** Section 5.2.6

- **Initialize:** Section 5.2.7

- **SPM Parameters:** Section 5.3

5.2.1 Align

Once you've created the workflow and scan parameters, prepare the system to scan. This includes aligning the laser, adjusting the photo detector, locating the cantilever tip with the optical microscope, and focusing the optical microscope on the surface.

1. Mount the probe into the cantilever holder

2. Mount the cantilever holder onto the end of the scanner head.

3. Click the ALIGN icon in the **Workflow Toolbar**. This opens the **Align** window, shown in Figure 5.2a.

Figure 5.2a   The **Align** Window
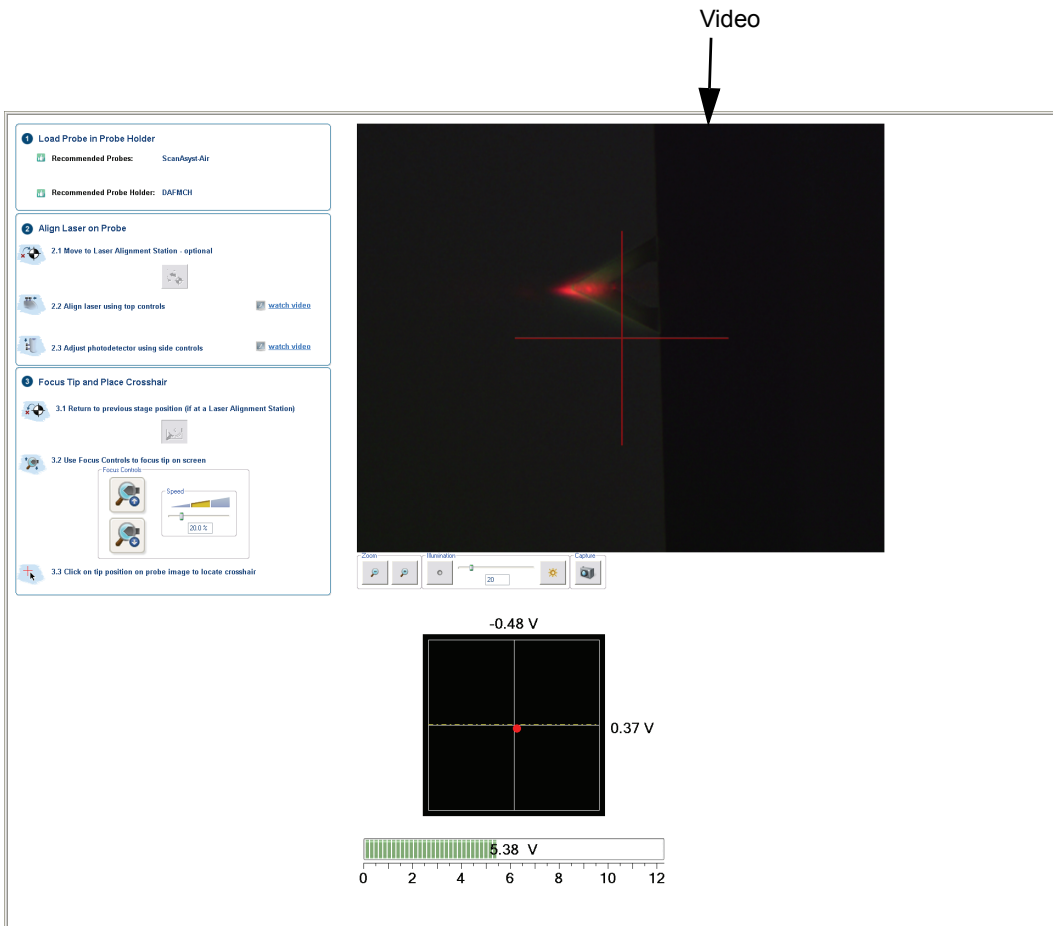
Several panels in the **Align** window help you through the process:

- The **Video** panel, shown in Figure 5.2a, displays an image of the area around the cantilever.

- The **Meter** panel, shown in Figure 5.2b, displays the signals on the quad photo detector and the laser sum signal.

Figure 5.2b   The Meter panel in the **Align** window
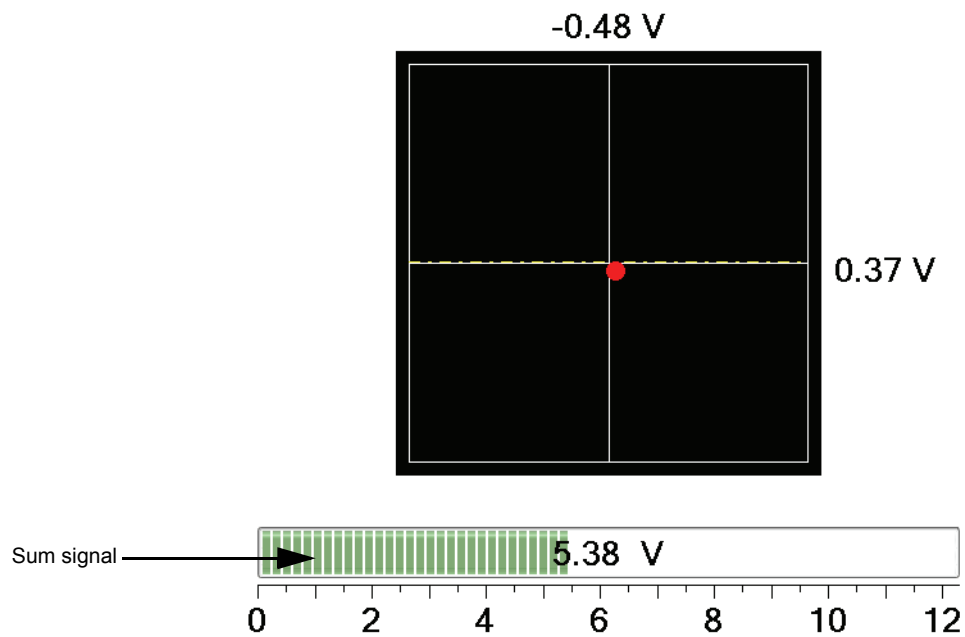
- The align instruction video, accessible through the **Align Laser on Probe** panel (Fig Figure 5.2c) and shown in Figure 5.2d, plays a short instructional movie showing the effects of moving the laser and photo detector alignment knobs.

Figure 5.2c   Watch Video Commands in the Align Panel
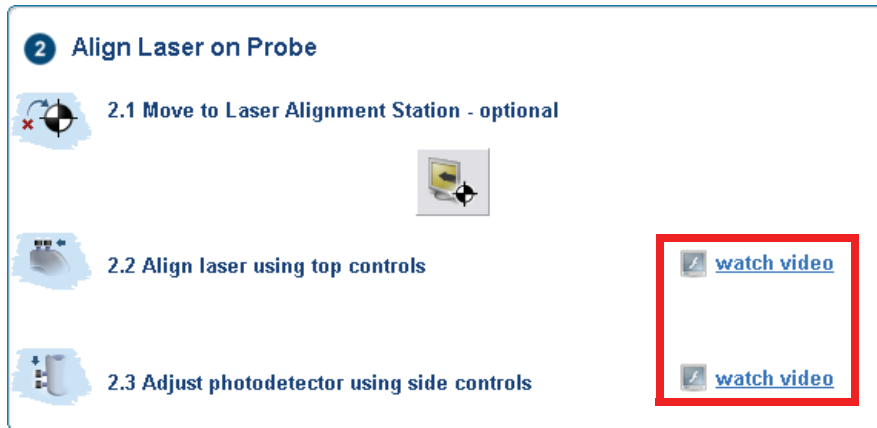
Figure 5.2d   The align instruction movie window
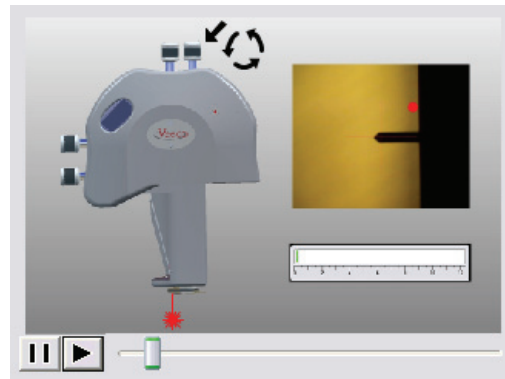
Refer to Chapter 7 for additional detail about adjusting the laser and photo detector mirror on your Dimension Icon head.

- The **optics** panel, shown in Figure 5.2e, has buttons to zoom or unzoom the camera and buttons to adjust the sample illumination LED. Zooming out may aid in locating the tip.

Figure 5.2e   The optics panel

- The **Focus Controls** panel, shown in Figure 5.2f focuses the optics on the probe tip and thus knows the Z position of the probe tip. This knowledge is needed to successfully engage the probe tip onto your sample.

Figure 5.2f   The **Focus** panel
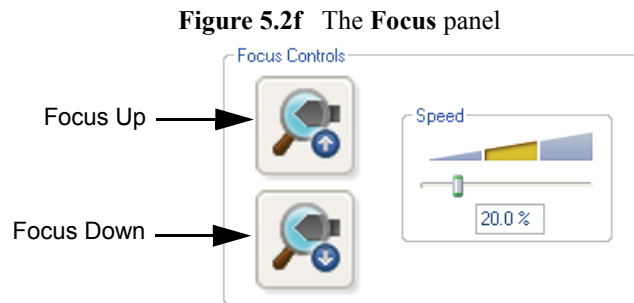
- The **Alignment Station** panel, shown in Figure 5.2g, moves the stage to position the head over a mirrored surface, shown in Figure 5.2h, mounted to the Dimension Icon stage. In many cases finding the laser reflection is easier if a mirrored surface replaces the sample.

Figure 5.2g The **Alignment Station** panel

Figure 5.2h The Dimension Icon alignment station
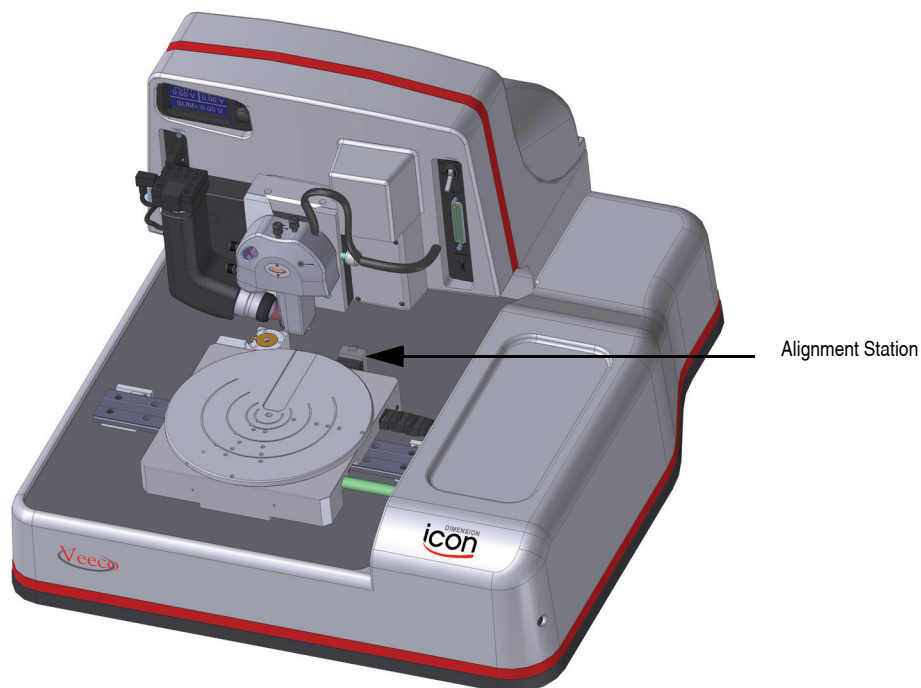
Alignment Station

4. New Dimension users will probably find the laser alignment procedure easier by clicking **ALIGNMENT STATION** icon. This will move the stage so that the Dimension Icon head is positioned over the Alignment Station mirror and select the **TIP REFLECTION** option in the **Focus Selec**t panel.

> **CAUTION:**   Do not use the Alignment Station when using the Direct Drive Fluid Cantilever Holder (see **Fluid Probe Holder:** Section 12.2.1) as it will hit the Alignment Station and knock it off its magnetically secured base. If you have selected a fluid experiment, the Alignment station button will be grayed out.

5. Using the video image as a guide, adjust the laser control knobs on the top of the Dimension head to place the laser spot near the probe tip.

6. Adjust the laser control knobs on the side of the Dimension head to center the laser diode on the quad photo detector and maximize the sum signal.

7. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Controls** panel, shown in Figure 5.2f, move the focus up or down. The focus **Speed** is controlled by the sliding bar or by typing a value from 0 to 100.

    When you leave **Locate Tip** mode, the optics move to a focus position, typically 1mm below the tip. To set this distance, select **Microscope** > **Engage Settings** > **General** > **Sample clearance**.

5.2.2   Navigate

1. When the tip is in focus and centered in the field of view, click the **NAVIGATE** icon in the **Workflow Toolbar**. This opens the **Navigate** window, shown in Figure 5.2i.

Figure 5.2i   The **Navigate** Window

Video
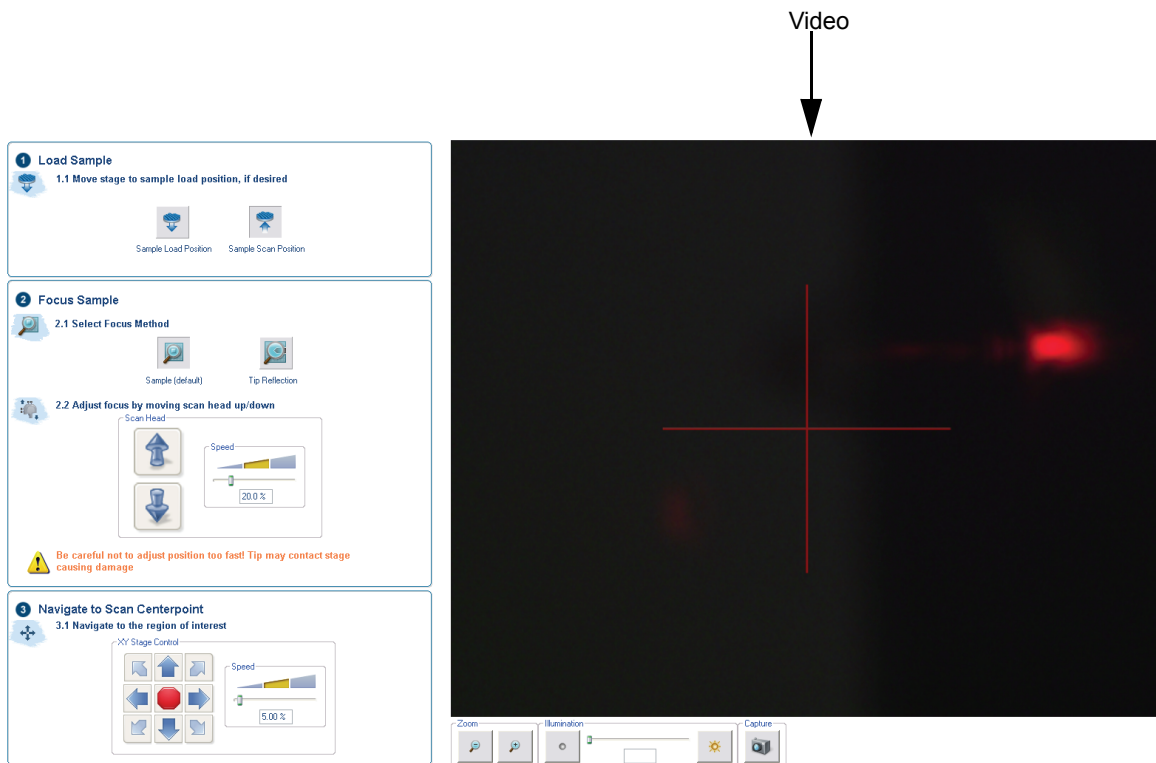
2. Focus the optics on the sample surface using either the trackball or the **Scan Head** arrows, shown in Figure 5.2j, in the **Navigate View**. If you have used the Alignment station, the stage will prompt you before moving back to the sample position.

Figure 5.2j Focus: Z Motor Controls
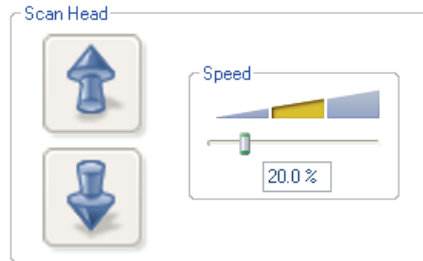
3. To use the track ball, roll the trackball up or down while holding down the bottom-left button. To use the **Z Motor** arrows, click and hold them down. You can use the speed controls in the **Z Motor** area to adjust the speed. This adjustment raises or lowers the Z stage on which the SPM and optics are mounted.

4. To focus on the sample **SURFACE** (normal operation) or the **TIP REFLECTION** (for extremely clean samples), change the **Focus Select** parameter accordingly.

Figure 5.2k The **Navigate Focus Select** Panel

Focus Surface   Focus Tip Reflection

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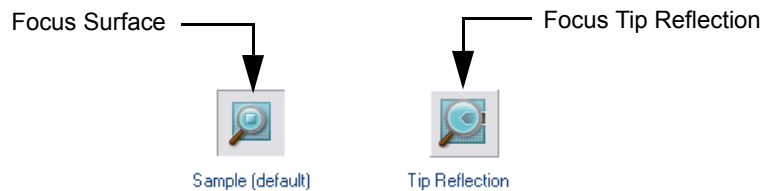
Note: For reflective or semi-reflective samples, the tip reflection is easier to bring into focus than the surface, especially if the sample is very flat or clean.

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 1mm (1000µm) above the surface.

---

5. 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.

6. Move the X-Y stage to align the desired location on the sample under the crosshairs either by using the trackball without holding down any buttons or by using the **XY Stage Control** arrows, shown in Figure 5.2l, in the **Navigate View**.

Figure 5.2l   XY Stage Controls
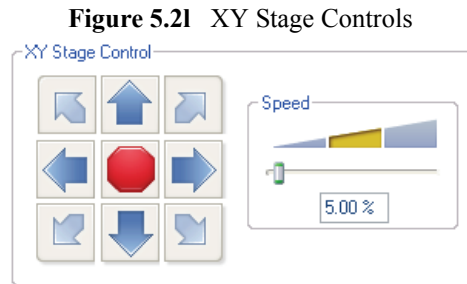
7. Verify that the surface remains in focus.

5.2.3   Load/Unload Sample

The **Load/Unload Sample** function, available in the **Navigate** window, first lifts the Dimension Icon head then indexes the stage to a preprogrammed location (usually front-center) where samples may be rapidly reloaded.

1. To load a sample from the stage, click the **SAMPLE LOAD POSITION** icon.

2. This raises the head to the **Load/Unload** height (see **Stage Parameters:** Section 7.3.1), the stage indexes to the front-center position and displays a corresponding message, shown in Figure 5.2m.

Figure 5.2m   Moving the Stage to Load/Unload position

3. To release the vacuum chuck's hold on the sample, use the toggle switch at the upper-right corner of the microscope, shown in Figure 1.4b. When the vacuum is released, remove the sample.

4. Verify that the stage and vacuum chuck are clean and free of debris. If debris is present, clean the stage using a lint-free wipe and isopropyl alcohol. Dispose of wipes in an appropriately labelled solvent-contaminated waste container.

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 chuck vacuum switch.

8. Click the **SAMPLE SCAN POSITION** icon.

9. The stage moves back to the sample analysis position and displays a corresponding message, shown in Figure 5.2n.

Figure 5.2n   Moving the Stage to Load position

5.2.4   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 References** panel, the stage automatically defaults to the last origin that was previously used. All subsequent X-Y moves are done from the origin.

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 5.2o).

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

Figure 5.2o   Move To Prompt
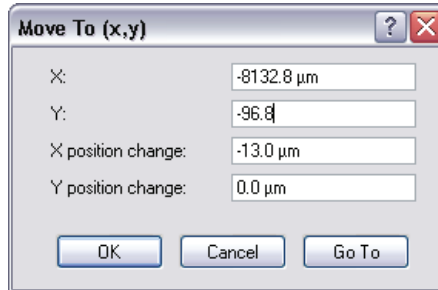
3. Enter the X and Y coordinates of the desired move to position, then click **OK**.

4. The stage moves to the new position. Should it become necessary to halt movement, click the **Abort** button or press the **Pause** key (see Figure 5.2p).

Figure 5.2p   Abort Motion Prompt
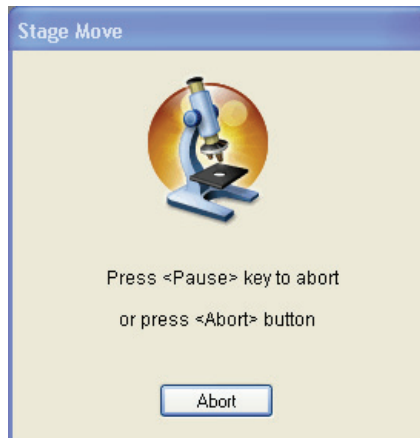
CAUTION:	Always verify that the tip is off the surface before attempting stage movements. If manual stage movements are attempted during engagement (by turning the leadscrew knobs on the stage's X-Y slide assemblies) the tip and/or sample may be damaged.
ATTENTION:	Il est impératif de toujours s'assurer 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.
ATTENTION:	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 **Cancel** button.

5.2.5 Set Reference

The **Set Stage 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 **Navigate** menu to focus on the sample and move the sample to the desired origin.

2. Select the **SET REFERENCES** option under the **STAGE** menu. This opens the **Set Reference** panel, shown in Figure 5.2q, in the **Navigate** window.

Figure 5.2q   Set Reference Panel
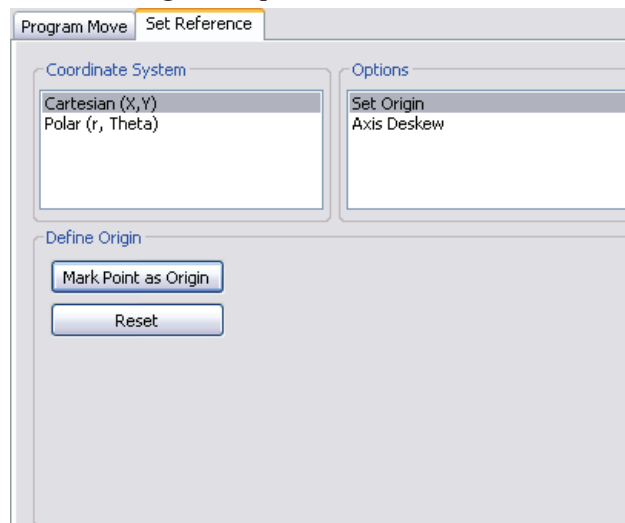
3. Select a coordinate system, either **CARTESIAN (X,Y)** or **POLAR (R, THETA)**. See Figure 5.2q.

4. Select the **SET ORIGIN** option.

5. Make a stage move using either the trackball or the **Move To X,Y** panel(i.e. enter **X** and **Y POSITION** or **POS**ition **CHANGE** and click **OK**). See **Move To (X,Y):** Section 5.2.4 for additional details.

6. Click **MARK POINT AS ORIGIN** to set the current stage position as the new origin; this is the first of two points to be used for defining a reference line on the coordinate grid.

7. Use the trackball to either move the stage right or upward as necessary.

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. Click the **X - Axis** button, shown in Figure 5.2r. This establishes the (Y = 0) reference line.

Figure 5.2r   Cartesian Axis Deskew
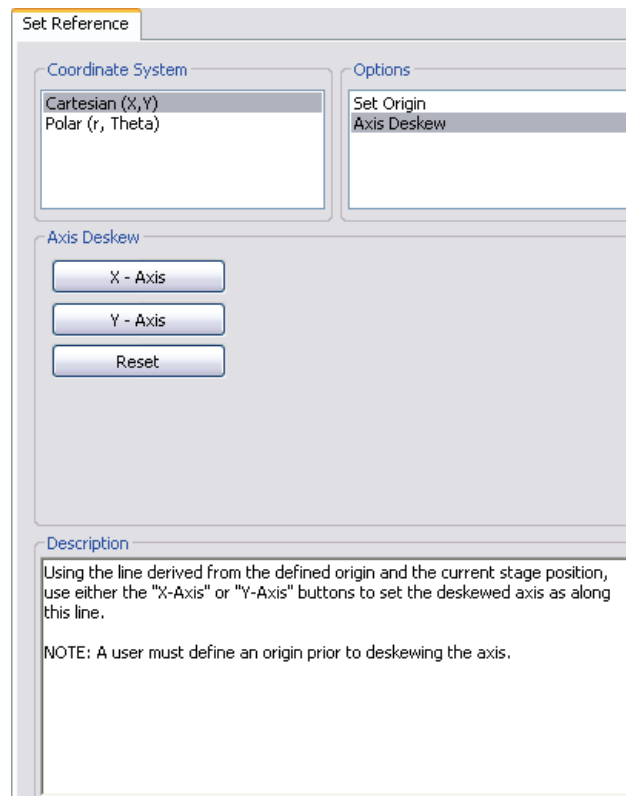
Move Stage Upward

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. Click the **Y - Axis** button, shown in Figure 5.2r. 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 5.2s). 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 5.2s Defining the X-Axis
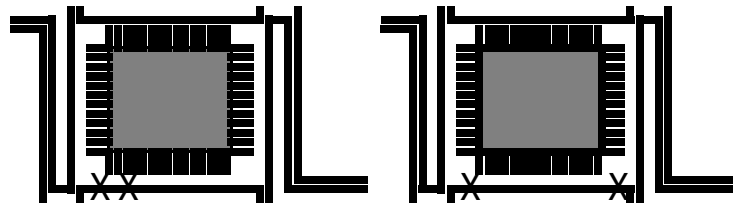
Figure 5.2t Resultant Reference Line
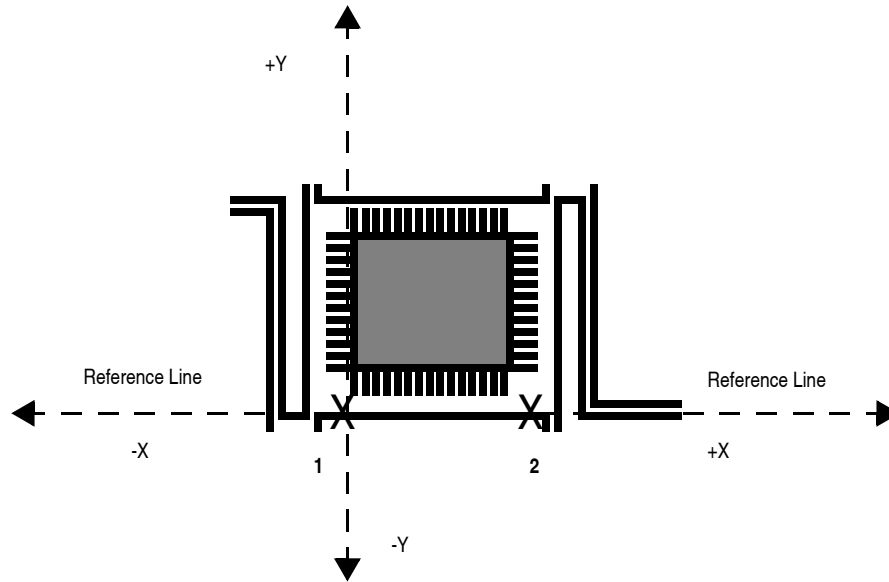
5.2.6   Programmed Move

The **STAGE > 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 access this option, select **STAGE > PROGRAMMED MOVE,** shown in Figure 5.2u.

> **Note:**   **Programmed Move** is available in the **Navigate** view only.

Figure 5.2u   Selecting Programmed Move
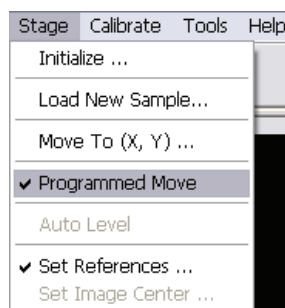
This opens the **Programmed Move** panel, shown in Figure 5.2v, in the **Navigate** window.

Figure 5.2v The **Programmed Move** Panel
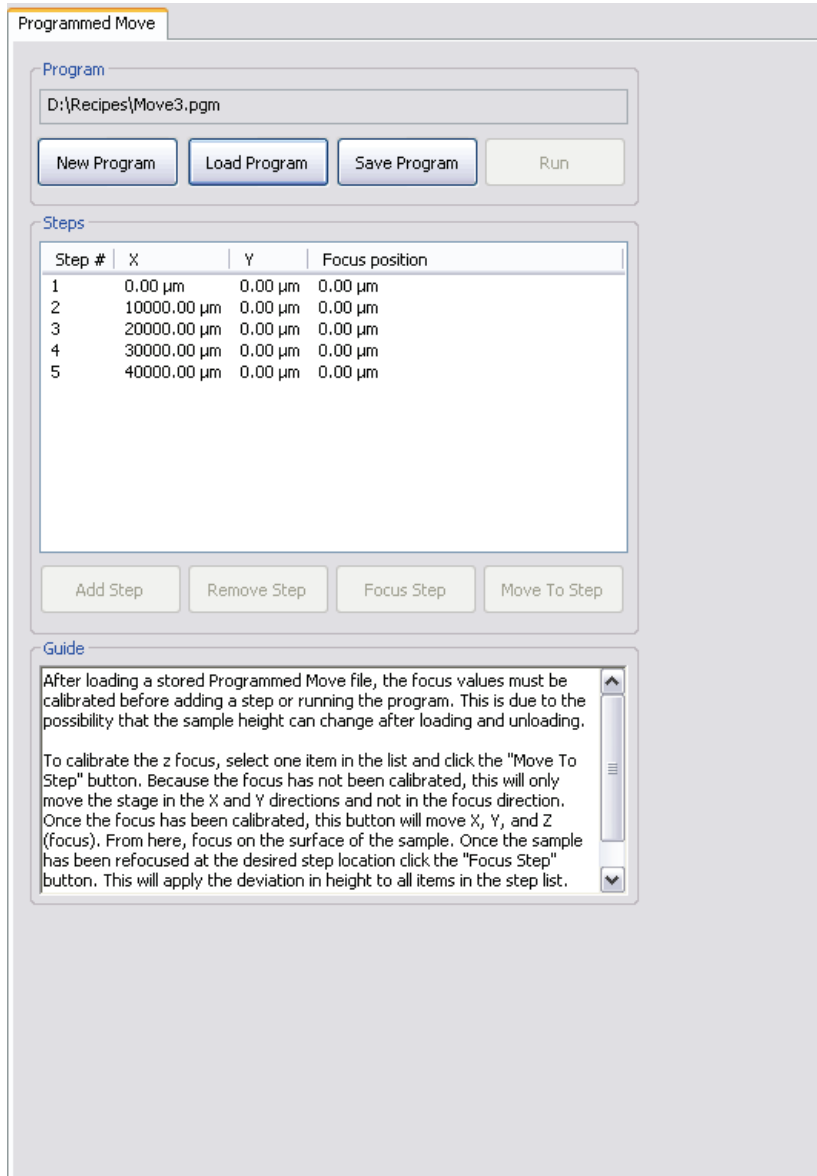
The name of the move program currently loaded is displayed in the **Program** name panel (in the above example, the program is called "Move3"). Moves are first programmed using the **Teach** function. Later they may be executed using the **Run** function.

Definitions

- **Program Name Field**—Provides the name of the move program currently loaded (in this example, the program is called "move"). Moves are first programmed using the ADD and REMOVE STEP functions. Later they may be executed using the **Run** option.

- RUN (button)—Executes the specified program name.
  Once a program sequence has been taught to the computer, the program can be run from the **Programmed Move** panel. The stage will move to each position in the same order it was taught, relative to the current origin point. At each program position, the sample will be scanned for 1.5 frames, captured, then indexed to the next position. Captured data from each position is stored on the hard disk; there should be sufficient memory to record 100 frames at 256 samples each.

- *Origin Points—Current origin at the time of programming.*
  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 will be necessary to reestablish the original origin point to locate the same positions on the sample. When generating maps of programmed moves, the origin point should always be indicated. This is done in the **Set Reference** panel.

Teaching a Programmed Move

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

1. Click **NEW PROGRAM** or **LOAD PROGRAM** to start.

2. Select a file name for a new or existing **Programmed Move** file. See Figure 5.2w. Enter a new **File name** for a new program.

Figure 5.2w   Programmed Move File Selection
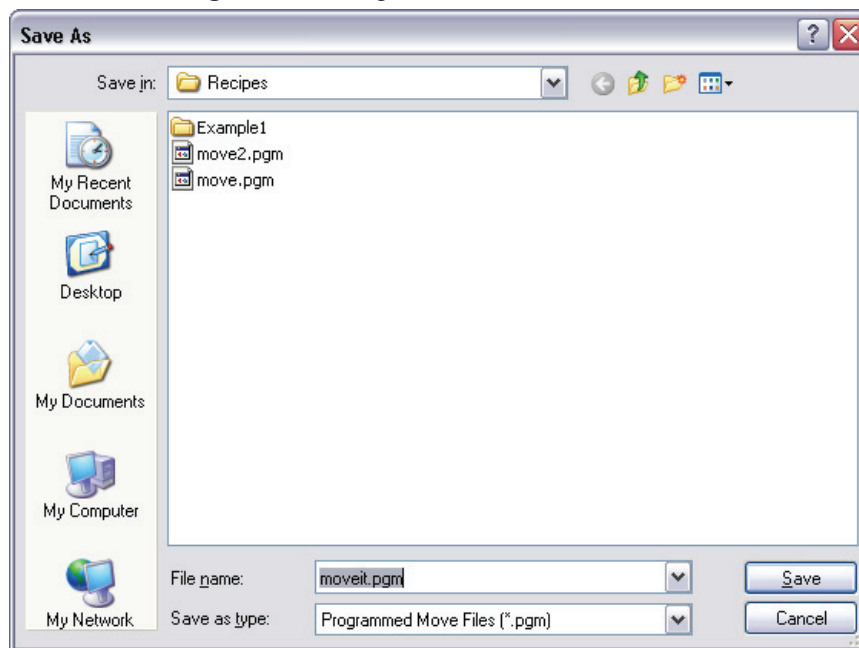
3. Click **SAVE** to proceed with programming moves. A **Programmed Move** window, shown in Figure 5.2v, will be displayed.

4. Make a stage move using either the trackball or the **Move To X,Y** panel (i.e. enter **X** and **Y POSITION** or **POS**ition **CHANGE** and click **OK**). See **Move To (X,Y):** Section 5.2.4 for additional details.

5. Click **ADD STEP**.

6. This procedure may be repeated until all the desired positions have been programmed up to a maximum of 100 steps. Each position will be automatically assigned a program step number.

It may prove helpful to draw a simple map of the sample, along with each programmed position, before programmed moves are entered. If reducing cycle time is important, position order should be optimized to reduce stage travel. Also, note the origin point at the time of programming.

7. When all stage positions have been entered into the program, click **SAVE PROGRAM**.

8. Additional programs are entered in the same manner, using a separate program name for each.

Removing a Programmed Option

To remove a step from the programmed sequence:

1. Click **LOAD PROGRAM** and select a file name for an existing **Programmed Move** file. See Figure 5.2x.

Figure 5.2x   Open an existing Programmed Move file
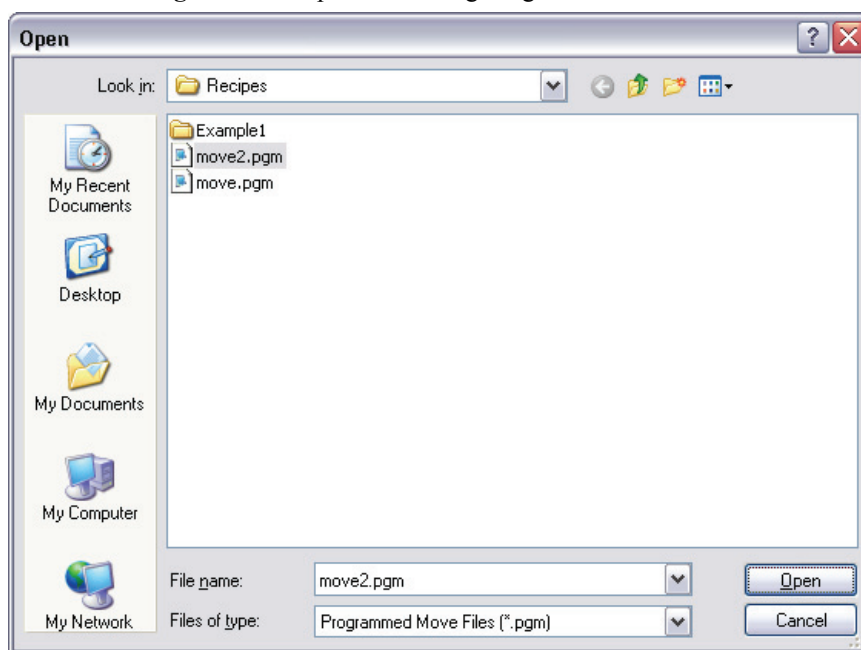
2. Click **OPEN** to proceed with programming moves. The program will be loaded into the **Programmed Move** panel.

3. Highlight the **Step #** in the **Steps** sub-panel. See Figure 5.2y.

Figure 5.2y   Highlight the **Step #**
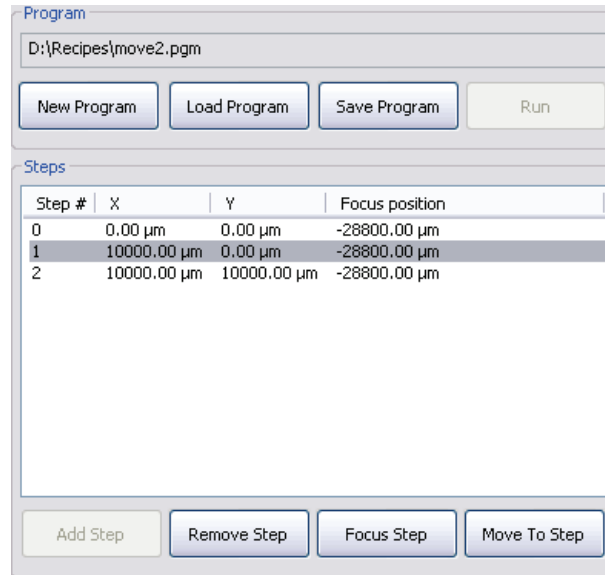
4. Click the REMOVE STEP button. When individual program steps are removed, all subsequent steps are "moved up" by one count. (For example, after removing step #2 from a program, step #3 would become the new step #2, step #4 would become the new step #3, etc.)

5.   Click the SAVE PROGRAM button to save the edited version of program.

Inserting a Step into an Existing Program Sequence

To add a step from the programmed sequence:

1. Click **LOAD PROGRAM** and select a file name for an existing **Programmed Move** file. See Figure 5.2x.

2. Click **OPEN** to proceed with programming moves. The program will be loaded into the **Programmed Move** panel.

3. Highlight the **Step #** in the **Steps** sub-panel. See Figure 5.2z.

Figure 5.2z Highlight the **Step #**
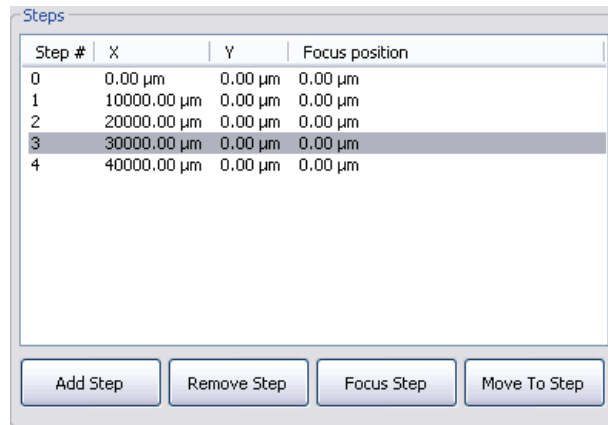
4. Make a stage move using either the trackball or the **Move To X,Y** panel (i.e. enter **X** and **Y** **POSITION** or **POS**ition **CHANGE** and click **OK**). See **Move To (X,Y):** Section 5.2.4 for additional details.

5. Click **ADD STEP**.

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

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

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 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 **STAGE > PROGRAMMED MOVE**.

2. Select a file name for an existing **Programmed Move** file. See Figure 5.2aa.

Figure 5.2aa   Open an existing Programmed Move file
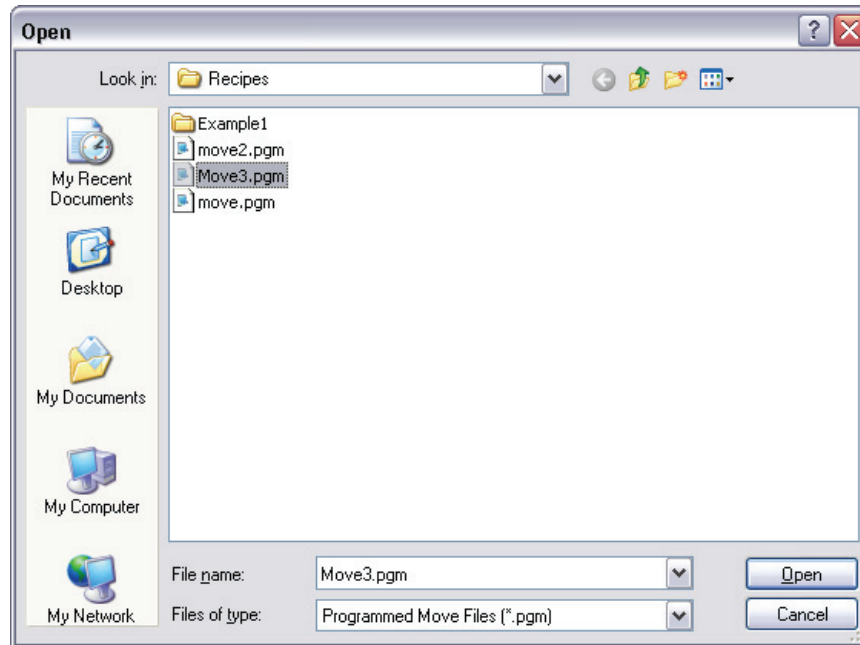
3. Click **OPEN**. The program will be loaded into the **Programmed Move** panel.

Because the sample height may have changed, you must calibrate the Z position. To do this:

4. Select one step in the list and click **MOVE TO STEP**. See Figure 5.2ab. This will move the stage to that (X,Y) location.

Figure 5.2ab Highlight a **Step** and click **MOVE TO STEP**.
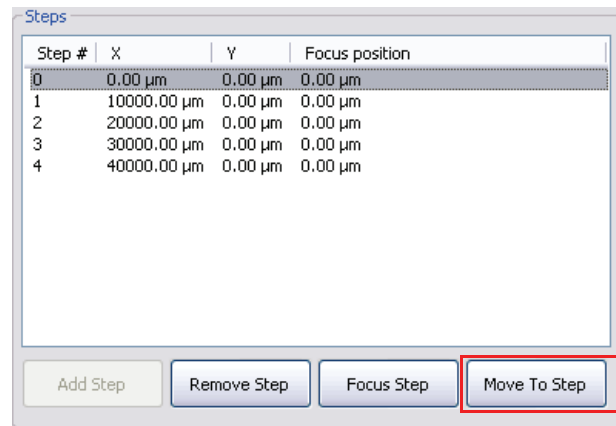
5. Click **FOCUS STEP** and focus on the sample surface using the **Focus Z Motor** controls, shown in Figure 5.2ac.

Figure 5.2ac Focus: Z Motor Controls
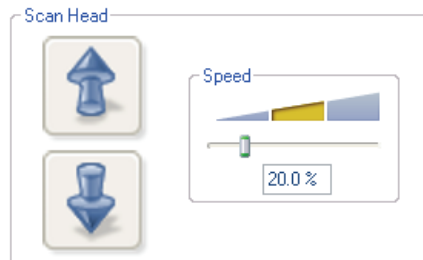
6. Click **SAVE PROGRAM**.

7. Click the **RUN** button.

5.2.7 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.

2. Select **Stage** > **Initialize**. The dialog box offers two options (see Figure 5.2ad).

Figure 5.2ad   Stage Initialize/Cancel Prompt
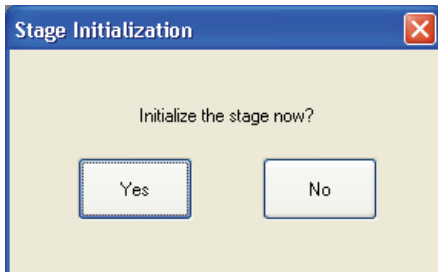
3. To begin initialization, click **Yes**. As the stage begins a series of motorized movements, the screen indicates operating status (see Figure 5.2ae).

Figure 5.2ae   SPM Move to Lower Limits Prompt
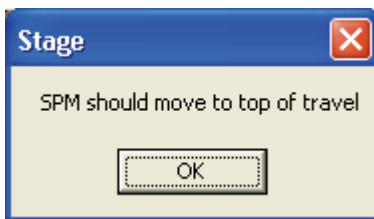
Note: The initialization sequence may be aborted at any time by clicking the **Abort** button or pressing the **Pause** key (see Figure 5.2af).

Figure 5.2af   Stage Initializing Prompt
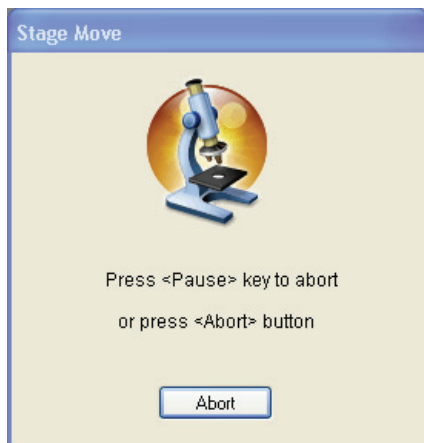
4. The microscope Z-axis moves to the top of its travel. When this is achieved, click **Ok** to continue.

5. The microscope focuses its camera optics to the extent of its travel. When this is achieved, click **Ok** to continue (see Figure 5.2ag).

Figure 5.2ag Optics Move to End of Travel Prompt
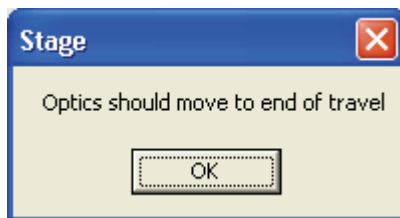
6. Next, the camera zoom optics assembly zooms. When the camera zooms in to the limit of its travel, click **Ok** to continue (see Figure 5.2ah).

Figure 5.2ah 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 5.2ai). The dialog box removes itself from the screen.

Figure 5.2ai Stage Zoom Out Prompt

5.3   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

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

This chapter addresses the following:

- **Silicon Cantilever Substrates:** Section 6.1

  - **Wafer Tool Kit:** Section 6.1.1

  - **Cantilever Preparation:** Section 6.1.2

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

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

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

6.1   Silicon Cantilever Substrates

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

6.1.1   Wafer Tool Kit

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

- Wafer tweezers

- Flat, substrate tweezers

- Regular tweezers

- Curved, sharp-pointed tweezers

- Flat, L-shaped tweezers

6.1.2   Cantilever Preparation

To prepare the cantilever for imaging:.

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

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

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

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

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

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

Note: Cover the petri dish when not in use.

Figure 6.1a Silicon Cantilever Substrates in Wafer

Cantilever

Press Here
To Break
Out Substrate

Substrate
Supporting
Arm

Cantilever
Substrate
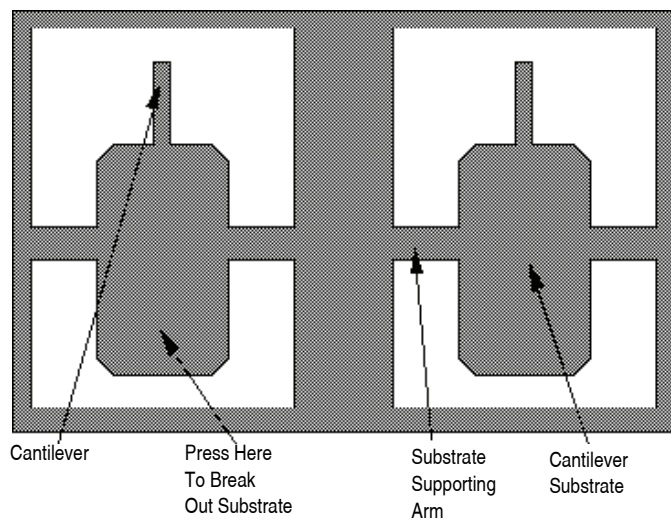
5. Use the curved, sharp-pointed tweezers to remove the cantilever substrate from the wafer container, grasping the sides of the substrate away from the lever and probe tip.

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

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

6.1.3   Tip Shape of Etched Silicon Probes

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

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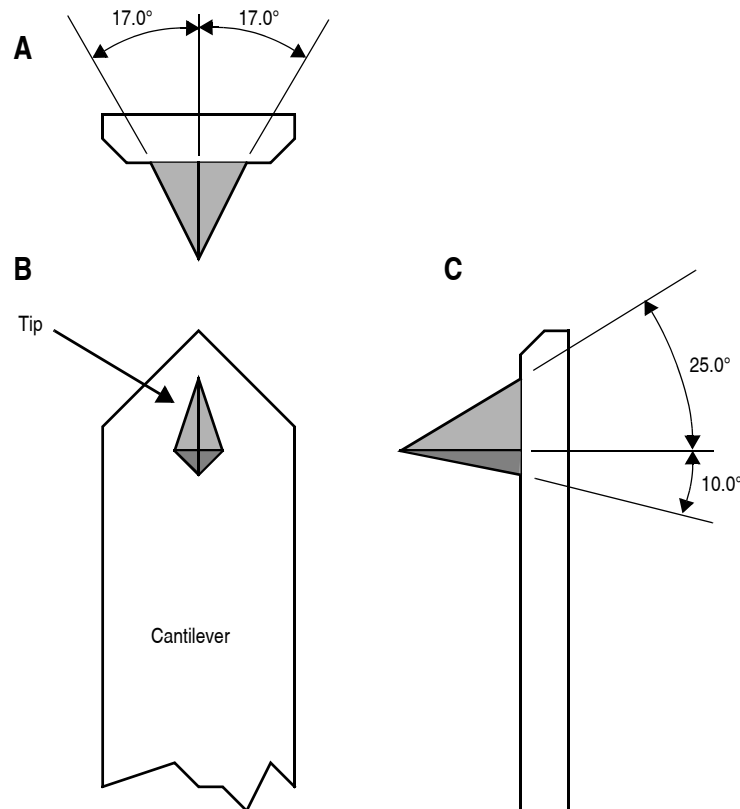
The present process creates a tip which is symmetric from side-to-side with a 17 ± 2 ° half cone angle (see A of Figure 6.1b) and asymmetric from front-to-back, along the length of the lever (see C of Figure 6.1b).

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

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

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

Scan direction = 0 deg.

Scan Line Profile

55°   80°   10°

1 µm - 2 µm Deep Trench

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

To measure sidewall angles, the best orientation of the sample uses the back edge of the tip (that which faces back towards the cantilever substrate) to measure step angles (see Figure 6.1c). Using the back edge, step angles approaching 90 degrees can be measured routinely, depending on the step height.

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

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

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

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

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

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

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

6.2   Silicon Nitride Cantilever Substrates

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

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

2.   Inspect the wafer with an optical microscope.

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

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

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

Figure 6.2a   Silicon Nitride Cantilevers in a Wafer

Press here to break out strip

Position glass slide underneath for support
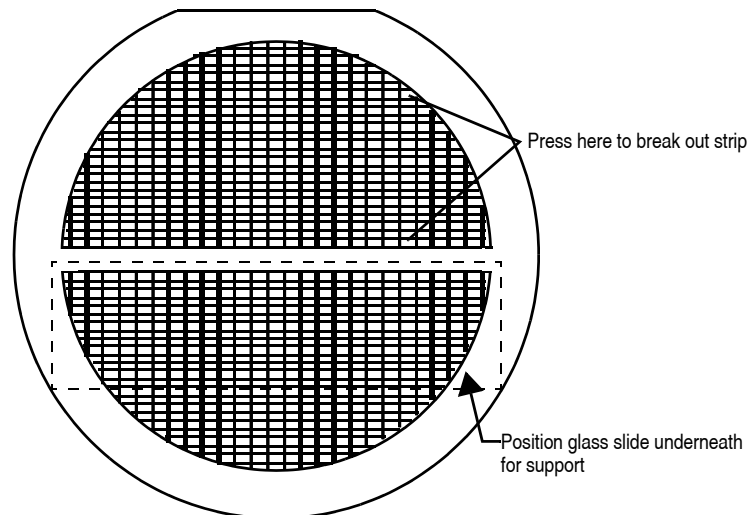
	CAUTION:	Be careful to avoid pushing strips together as the cantilevers are between the strips. All cantilevers on one side of both strips could break off if the strips are inadvertently pushed together.

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

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

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

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

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

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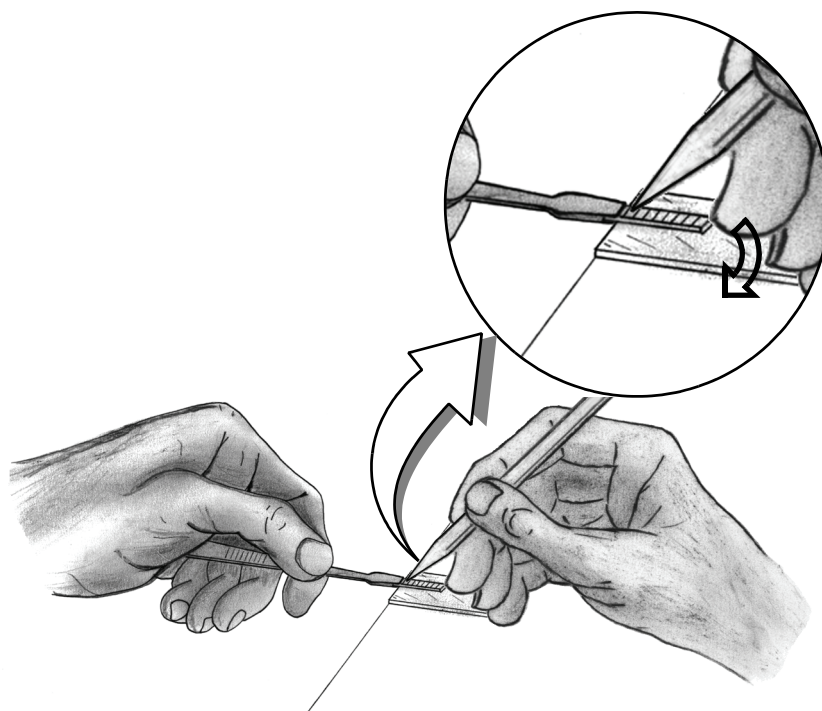
> **Note:** Extra substrates are easily stored in a covered petri dish. The shipped substrates are secured with X0-grade, GEL-PAK™ adhesive strips. The strips are used to permit easy removal of the substrates. If GEL-PAK adhesive strips cannot be found, a simple substitute is the adhesive area from a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

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

6.2.1   Tip Shape of Silicon Nitride Probes

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

Figure 6.2c   Silicon Nitride Cantilevers

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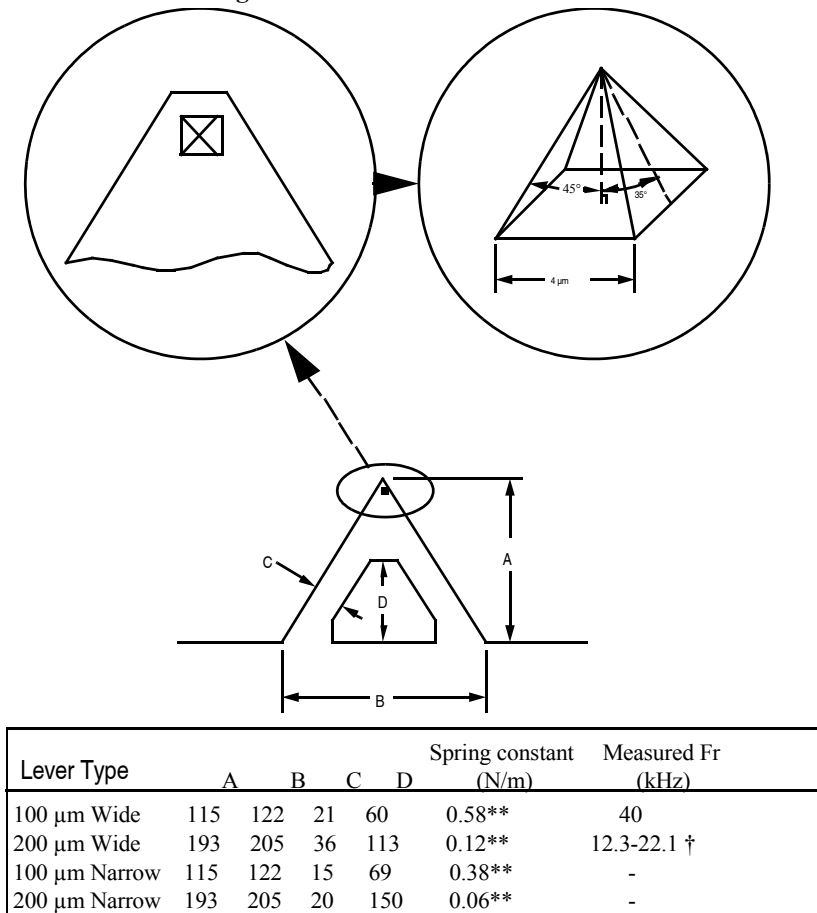
Because the Silicon Nitride probe tips have lower aspect ratios than single-crystal etched silicon probes, the steepest measurable step wall angle is appreciably lower. The highest measurable angle using silicon nitride probes is approximately 65° (see Figure 6.2d) using the inner face of the tip (towards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

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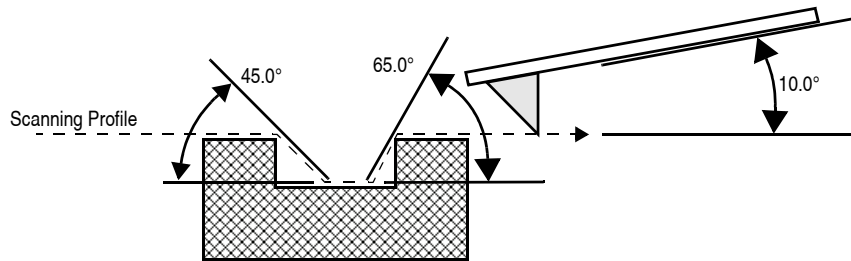
There are two types of silicon nitride cantilever probes available: standard and oxide-sharpened tip processes. The standard devices have the nitride deposited directly into the etched silicon mold pit formed by the intersecting <111> planes, and have points that are slightly rounded with respect to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

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

Chapter 7   Head, Probe, & Sample Preparation

This chapter includes information regarding the Dimension Icon Scanning Probe Microscope (SPM) setup and operation procedures for Contact Mode and TappingMode. 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 11 discusses SPM operation in TappingMode in more detail, while Chapter 10 reviews SPM operation in Contact Mode AFM. This chapter contains the following:

- **System Information:** Section 7.1

    - **Mouse versus Trackball:** Section 7.1.1

    - **Motor Interlock:** Section 7.1.2

    - **Laser Requirements:** Section 7.1.3

- **Basic AFM Operation:** Section 7.2

    - **Select the Microscope:** Section 7.2.1

    - **Select Mode of Operation:** Section 7.2.2

    - **Prepare the Cantilever Holder:** Section 7.2.3

    - **Load the Cantilever Holder:** Section 7.2.4

    - **Remove the Dimension SPM Head:** Section 7.2.5

    - **Install the Cantilever Holder:** Section 7.2.6

    - **Replace the Dimension SPM Head:** Section 7.2.7

    - **Connect the Dimension Head:** Section 7.2.8

    - **Align Laser:** Section 7.2.9

    - **Adjust Photodetector:** Section 7.2.10

    - **Locate Tip:** Section 7.2.11

- • **Load the Sample:** Section 7.2.12

- • **Focus Surface:** Section 7.2.13

- • **Cantilever Tune (TappingMode only):** Section 7.2.14

- • **Set Initial Scan Parameters:** Section 7.2.15

- • **Engage:** Section 7.2.16

- • **Withdraw:** Section 7.2.17

- • **Advanced AFM Operation:** Section 7.3

  - • **Stage Parameters:** Section 7.3.1

7.1   System Information

7.1.1   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 commands are controlled by the trackball and its buttons. Motor operation is permitted only when the appropriate menu enables movement.

7.1.2   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 except for 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.

7.1.3 Laser Requirements

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

	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. 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.
	AVERTISSEMENT:	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.

7.2 Basic AFM Operation

7.2.1 Select the Microscope

1. Start the NanoScope software.

2. Set the microscope configuration by selecting **Tools > SELECT MICROSCOPE...** from the menu bar. See Figure 7.2a.

Figure 7.2a SELECT MICROSCOPE from the Tools menu
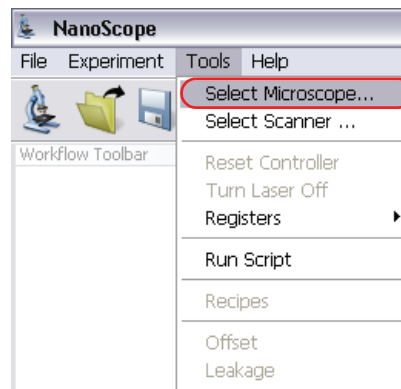
3. This opens the **Microscope Select** window, shown in Figure 7.2b.

Figure 7.2b The **Microscope Select** window
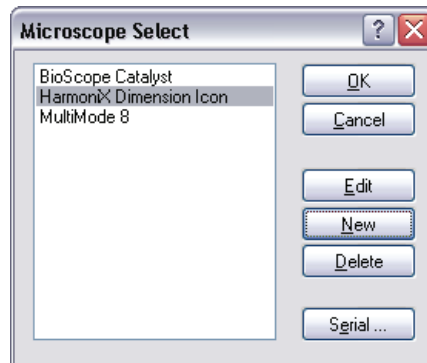
4. Click **NEW** if this is a new microscope, **EDIT** if you wish to modify a configured microscope, **DELETE** to remove a microscope configuration or **CANCEL** to exit.

5. **NEW** and **EDIT** open the **NEW EQUIPMENT** window, shown in Figure 7.2c.

Figure 7.2c Select the appropriate microscope from the **New Equipment** window
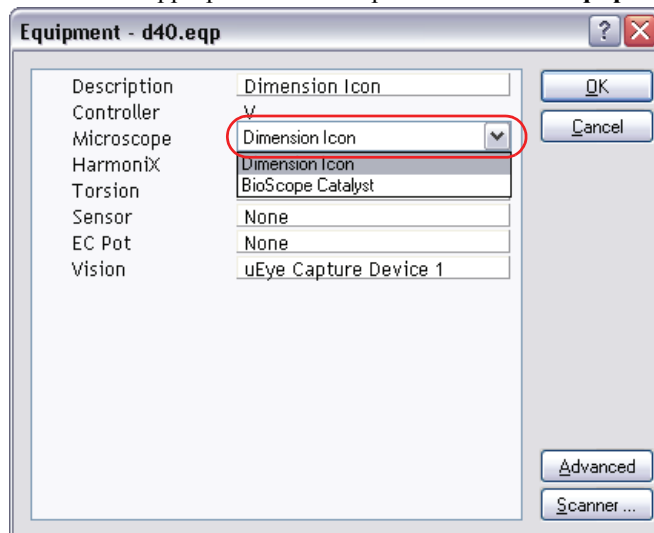
6. Select **DIMENSION ICON** or **DIMENSION ICON-PI** from the drop down **Microscope** menu.

7. Select the **SCANNER** and click OK. This returns you to the **Microscope Select** window, shown in Figure 7.2b.

8. Configure the **SERIAL** ports by clicking **SERIAL...** This opens the **Serial Port Configuration** window, shown in Figure 7.2d.

Figure 7.2d Configure the Serial ports
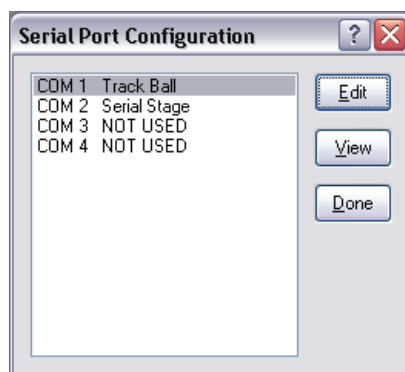
9. Select **COM 1** and click **EDIT** to open the **Edit Port Setup** window, shown in Figure 7.2e.

10. Select **TRACK BALL** from the **COM 1 EQUIPMENT TYPE** drop-down menu options.

11. Click **OK** to close the **COM 1 Port Setup** window.

Figure 7.2e   Edit the serial ports
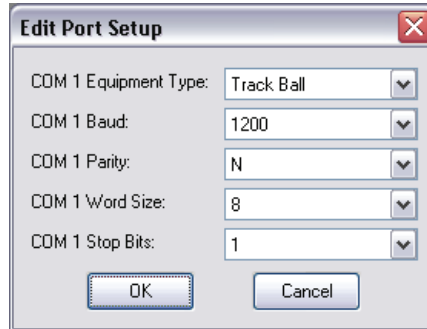
12. Select **COM 2** and click **EDIT** to open the **Edit Port Setup** window, shown in Figure 7.2e.

13. Select **SERIAL STAGE** from the **COM 2 EQUIPMENT TYPE** drop-down menu options.

14. Click **OK** to close the **COM 2 Port Setup** window.

15. Click **DONE** to close the **Serial Port Configuration** window.

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

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

7.2.2 Select Mode of Operation

1. Click **EXPERIMENT** > **SELECT EXPERIMENT** or the **SELECT EXPERIMENT** icon in the top left of the NanoScope software window. This opens the **Select Experiment** window, shown in Figure 7.2f.

Figure 7.2f The **Select Experiment**, Contact Mode, window
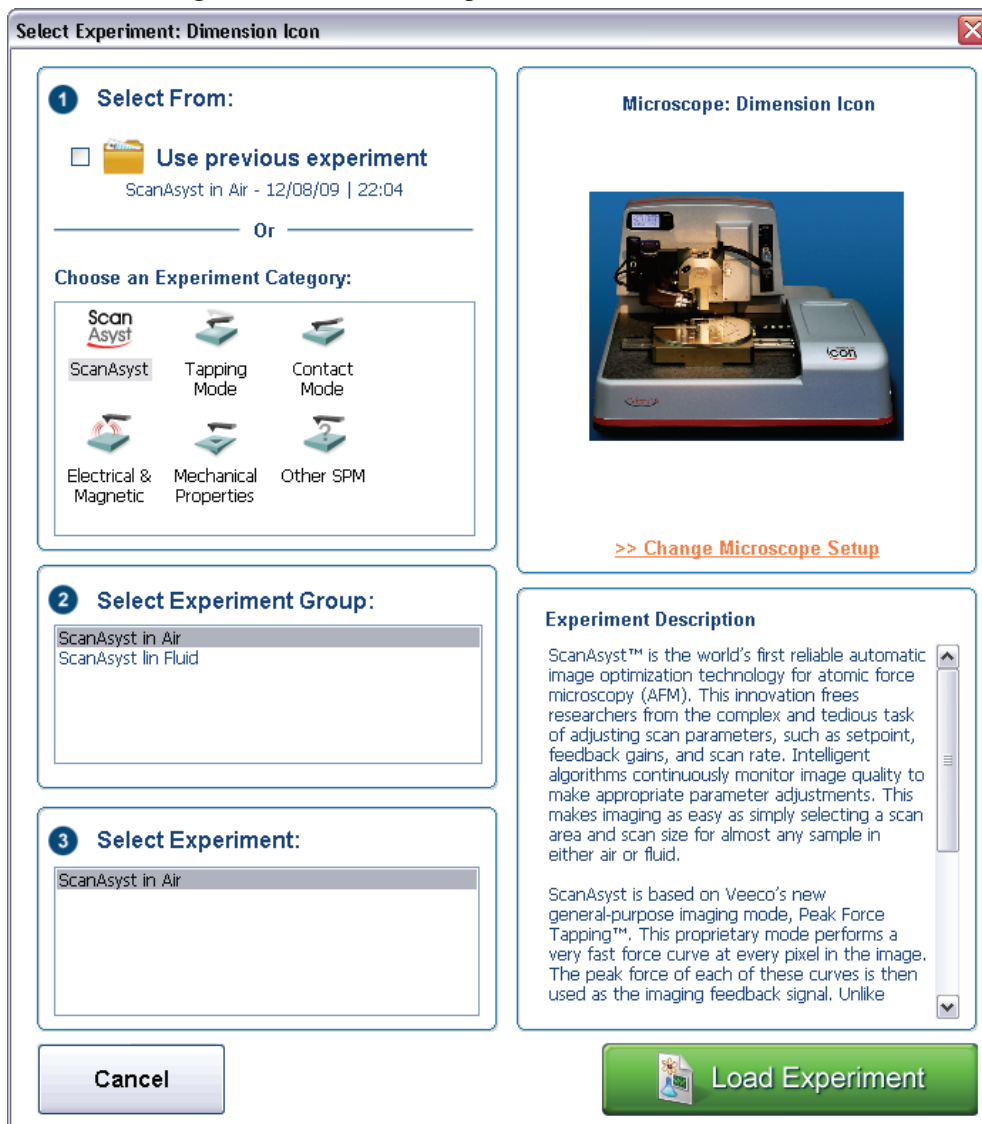
Note: Modes shown in Figure 7.2f are for a Dimension Icon. Some modes are unavailable or optional on the Dimension Icon-PI.

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

3. Click **LOAD EXPERIMENT**.

7.2.3   Prepare the Cantilever Holder

Before loading the substrate onto the cantilever holder, place the cantilever holder on the cantilever holder stand (see Figure 7.2g). The cantilever holder stand is a 2.5-inch diameter black anodized aluminum cylinder that ships with the Dimension Icon SPM for easy installation of the substrate onto the cantilever holder.

The cantilever holder stand has three stations for different tip holders: 1) standard AFM, 2) fluid imaging AFM and 3) STM. A small, raised block in the center of the gold connector identifies the standard AFM load station.

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 7.2g).

Figure 7.2g   Cantilever Holder Stand
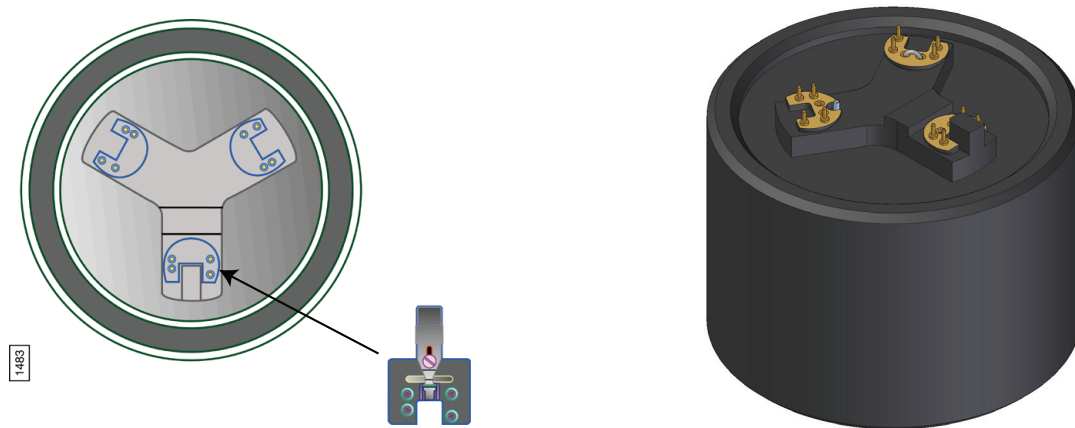
> **Note:**   You may install the AFM cantilever holder in only one orientation because the pins are asymmetrical.

7.2.4   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. In the gel-pack boxes the tip ends are pointing upward and do not need to be turned over before being placed in the cantilever holder.

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

2. Many silicon nitride substrates have cantilevers on both ends of the substrate. If this is the case, place the substrate under an optical microscope or magnifier and locate the tip you want to use.

3. Press down and slide back the spring clip of the standard AFM cantilever holder (see Figure 7.2h).

Figure 7.2h   Standard AFM Cantilever Holder
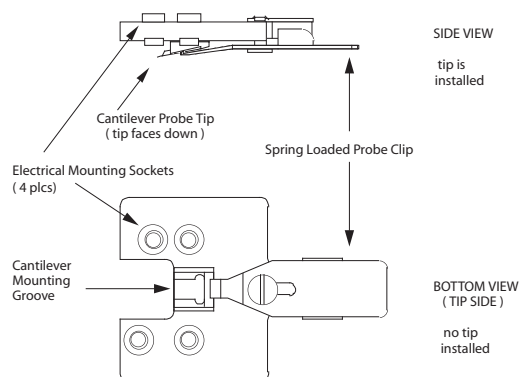
4. Orient the cantilever intended for imaging away from the cantilever holder, and place the substrate in the AFM cantilever holder groove.

5. Carefully maneuver the substrate until the substrate is flush against the back edge 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.

    Note: Always orienting the substrate to one side and the back of the cantilever holder groove will improve repeatability of tip location between runs. This will make aligning the laser onto the cantilever quicker and easier when installing new cantilevers.

TappingMode

The procedure for installing TappingMode, single crystal silicon substrates is the essentially the same as the procedure for installing silicon nitride substrates in Contact Mode AFM. However, one difference is that silicon substrates usually have only one cantilever per substrate, which can often be identified without an optical microscope.

> **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.

7.2.5 Remove the Dimension SPM Head

1. Loosen (by turning clockwise) the screw located on the right side of the Dimension SPM head dovetail to release the Dimension SPM head (see Figure 7.2i).

Figure 7.2i SPM Head Dovetail and Signal Connector
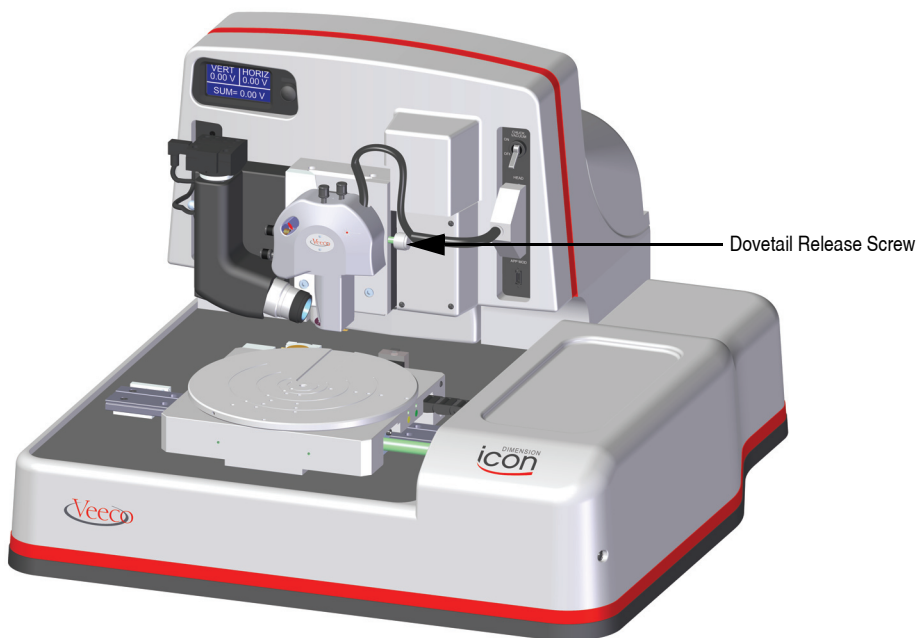
> **Note:** Dovetail engagement actuates with a spring. Failing to engage the spring-loaded dovetail causes a large increase in image noise due to reduced rigidity of the mechanical support of the SPM head.

2. Unplug the connector wire that runs from the SPM head to the Dimension base (see Figure 7.2j).

Figure 7.2j   Dimension Icon Head and connector
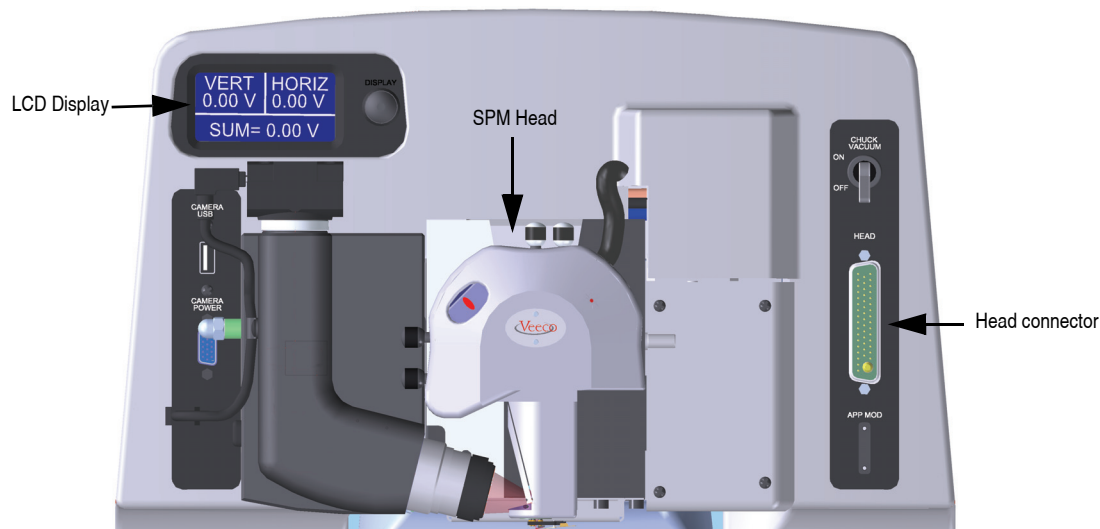
3. Carefully slide the Dimension SPM head up and out of the dovetail groove.

7.2.6   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 end of the cantilever 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.

7.2.7   Replace the Dimension SPM Head

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

2. When sliding the SPM head into the dovetail, make sure that the tip will not touch the sample. If it appears that the tip may touch when the SPM head is fully inserted in the dovetail, raise the Z-stage by selecting **Microscope** > **Withdraw** several times, or by selecting **Navigate** > **Focus Surface** and raising the Z-stage upward.

3. Tighten (by turning counterclockwise) the screw located on the right side of the Dimension SPM head dovetail to lock the head in place (see Figure 7.2i).

7.2.8   Connect the Dimension Head

Insert the Dimension SPM head black 47-pin connector plug into the socket to the right of the SPM head (see Figure 7.2j).

7.2.9   Align Laser

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

For both TappingMode 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 7.2k 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 clockwise.

Figure 7.2k   Dimension Head Laser Control Knobs
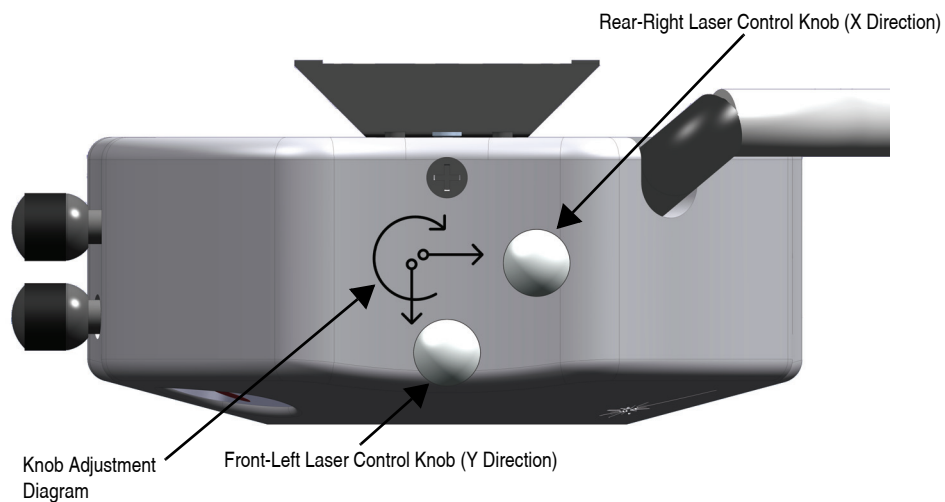
The X direction runs along the major axis of the substrate (parallel to the length of the cantilever). The right-rear laser control knob, atop the SPM head, controls the laser beam movement along the X direction. The front-left laser knob, atop the SPM head, moves the beam along the Y direction perpendicular 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 TappingMode 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 (TappingMode)

To place the laser reflection at the end of the cantilever, you must shine a laser beam on the alignment station mirror, the granite base or a piece of paper.

1. You can view the laser spot several different ways:

   a. If a cantilever is being replaced, first select **Stage** > **Load New Sample** to reflect the laser off the of the granite base while it is locked in the Z-stage dovetail (as discussed in Section 7.1.2).

   b. If the cantilever has already been removed, you may need to raise the Z-stage by selecting **Navigate** > **Focus Surface** or **Microscope** > **Withdraw**.

   c. You can see the laser by moving the Dimension Icon head over the alignment station. See Figure 5.2h.

   d. You can see the laser spot by removing the SPM head from the dovetail (without unplugging the cable between the SPM head and the Dimension base) and holding the SPM head over the granite or piece of paper.

⚠️	**CAUTION:** Use extreme caution when removing the SPM head. Hold the head firmly and do not stress the wire between the SPM head and the Dimension base. Dropping the head will likely result in damage and Veeco factory repair.

---

2. Verify that 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 7.2l).

Figure 7.2l Etched Silicon Tip Laser Alignment
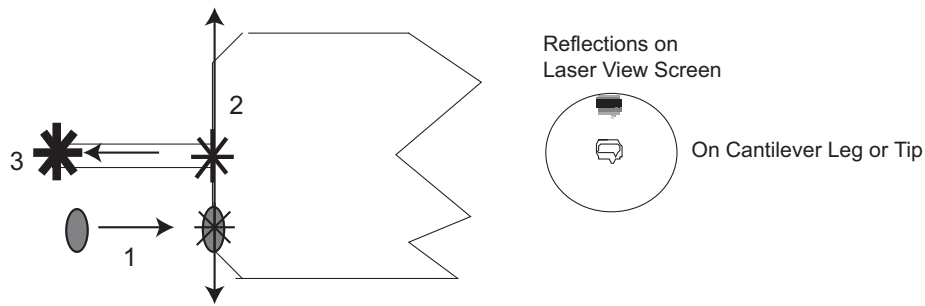
4. Turn the front-left laser control knob clockwise or counter-clockwise to move the laser in the Y direction (parallel to the substrate edge and perpendicular to the cantilever) until the beam crosses the cantilever and a shadow appears over the laser spot on the surface below. The laser is now positioned over the cantilever (see Point 2 in Figure 7.2l).

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 7.2l).

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 7.2m).

Figure 7.2m Photodetector Adjustment Knobs
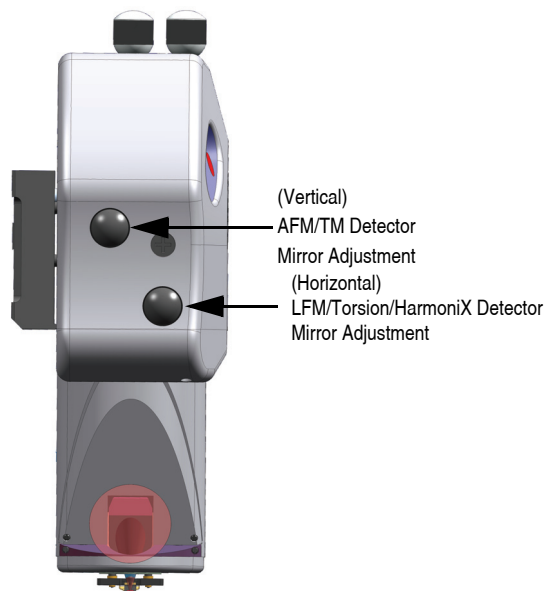
> **Note:** You may now restore the illuminator lamp to increased intensity for improved video image quality.

Silicon Nitride Tips (Contact Mode AFM)

In order to place the laser reflection at the end of the cantilever, you must shine a laser beam on either the granite base or a piece of paper.

1. You can view the laser spot several different ways:

   a. If a cantilever is being replaced, first select **Stage** > **Load New Sample** to reflect the laser off the of the granite base while it is locked in the Z-stage dovetail (as discussed in Section 7.1.2).

   b. If the cantilever has already been removed, you may need to raise the Z-stage by selecting **Navigate** > **Focus Surface** or **Microscope** > **Withdraw**.

   c. You can see the laser by moving the Dimension Icon head over the alignment station. See Figure 5.2h.

d. You can see the laser spot by removing the SPM head from the dovetail (without unplugging the cable between the SPM head and the Dimension base) and holding the SPM head over the granite or piece of paper.

CAUTION: Use extreme caution when removing the SPM head. Hold the head firmly and do not stress the wire between the SPM head and the Dimension base. Dropping the head will likely result in damage and Veeco factory repair.

---

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 and stop turning the knob. The laser is now positioned at the edge of the substrate (see Point 1 in Figure 7.2n).

Figure 7.2n   Silicon Nitride Laser Alignment
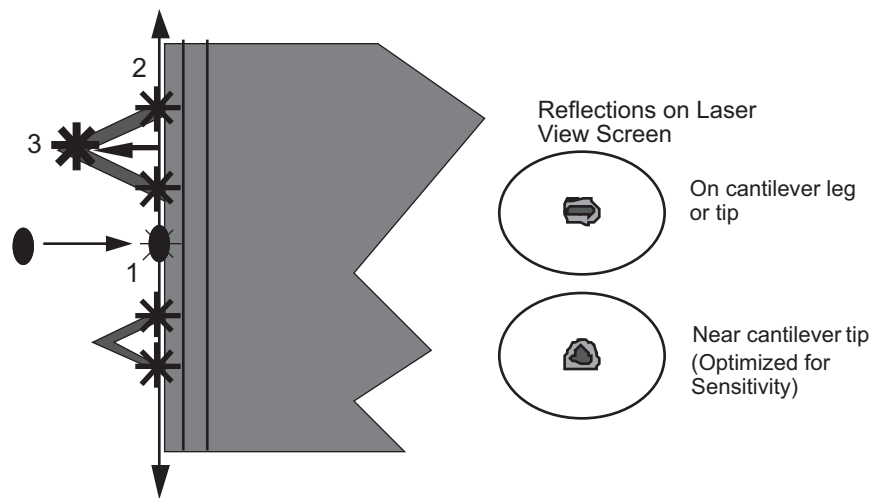
4. Once the laser is positioned just off the edge of the substrate, use the front-left laser control knob to move in the Y direction (parallel to the edge of the substrate) until the laser crosses a maximum of four legs on the two V-shaped cantilevers (see Point 2 in Figure 7.2n).

    Note: This occurs when moving in one direction (either up or down from end to end). If the laser is close to the substrate, 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 7.2n).

6. Verify the laser is located on the portion of the cantilever connecting the two lever arms near the tip location by moving the laser on and off the cantilever, repeatedly, with an 1/8 turn of the back-left control knob. Then, place the laser back on the cantilever.

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 7.2n).

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. The laser is now on one of the two cantilevers. To determine which cantilever the laser is on:

   Note: During this step do not adjust the back-right laser knob, or you may accidentally return to the original cantilever. Only use the back-right knob when you need to place the laser on the other cantilever.

   a. Rotate the back-left laser control knob in either direction until the laser disappears and does not reappear with an 1/8 turn.

   b. Use the same back-left laser control knob to rotate the laser the opposite direction, past the original cantilever.

   c. Determine which cantilever you are on:

      • If the laser was on the longer cantilever at then beginning of this step, then the laser will not cross any other cantilever legs during this movement.

      • If the laser was on the small cantilever, then the laser will cross the two longer cantilever legs.

   d. Select the cantilever for imaging by returning to the original cantilever, or locate the other end of this cantilever.

10. 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.

7.2.10 Adjust Photodetector

1. Now that the laser is aligned on the cantilever, verify that there is a spot visible on the Dimension head filter screen.

2. Verify that you have an appropriate laser sum signal (greater than 0.5V), displayed in the Dimension Icon LCD, shown in Figure 7.2j, or the **Real Time Status** window, shown in Figure 7.2o. Activate the **Real Time Status** window by clicking the **REAL TIME STATUS** icon in NanoScope tool bar.

Figure 7.2o   Real Time Status Window

Real Time Status icon

Detector Schematic

Laser Signal Values
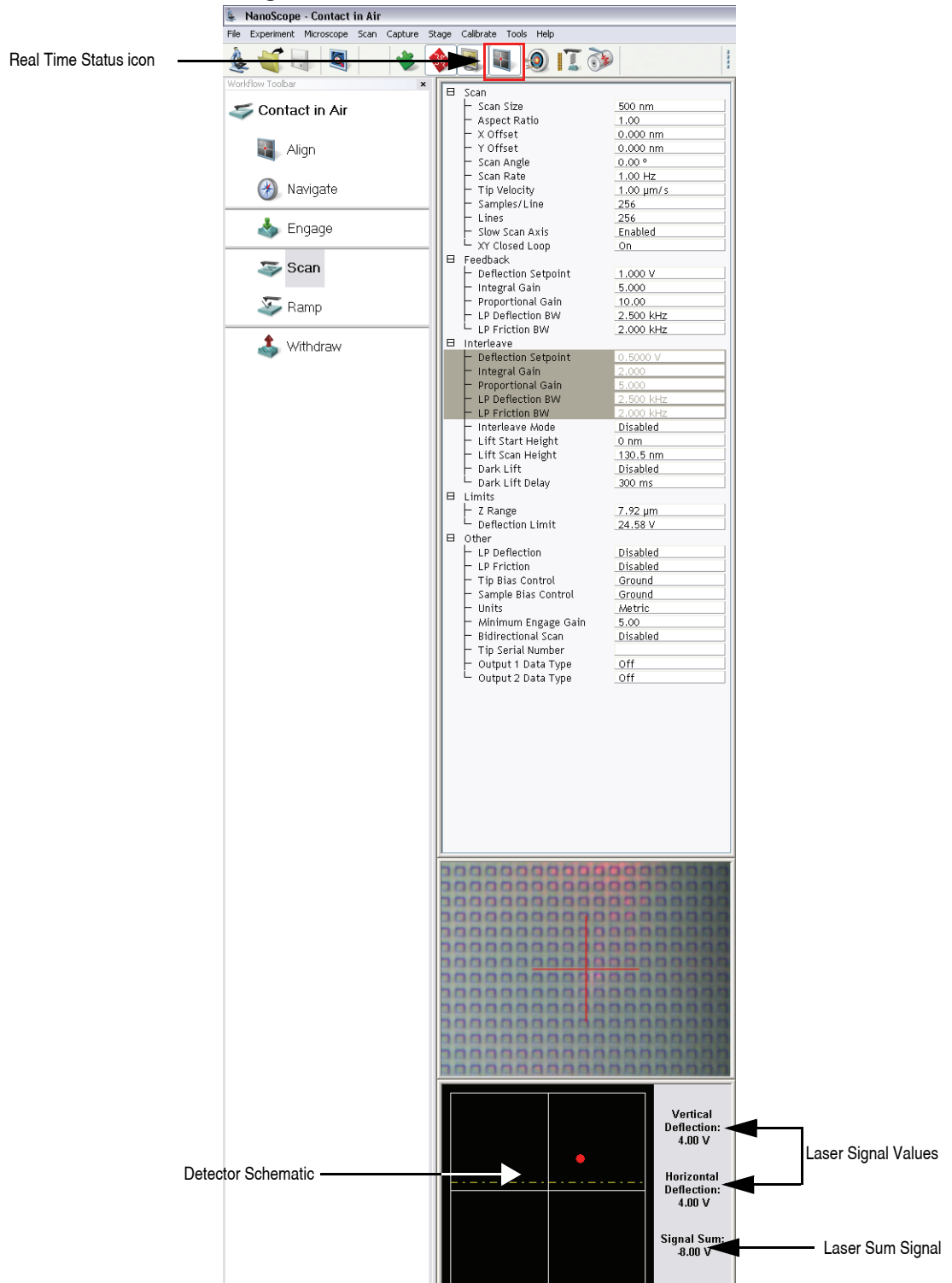
Laser Sum Signal

Note: If the laser sum signal is low, either the laser is not aligned or the photodetector knob needs adjusting (described in Step 3).

Note: Figure 7.2o shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

3. Center the laser detector signal using the photodetector adjustment knobs (see Figure 7.2m) located on the left side of the Dimension head. The **Real Time Status** window displays the laser signal values and a schematic of the detector quadrants. The position of the laser is denoted by a red dot on the detector schematic (see Figure 7.2o).

- The Vertical Deflection signal is the difference between the top and bottom photodetectors. For TappingMode, adjust this signal to 0. For Contact Mode, adjust this signal to -2V (see Figure 7.2m).

Note: In TappingMode, the **RMS Ampl** is an AC signal and does not have any real magnitude until the cantilever tune has been completed.

Note: When the laser is positioned in the center of the detector schematic, the laser is also in the center of the screen on the front of the head. If the laser is severely out of alignment, it may help to first center the laser on the screen on the head using the photodetector adjustment knobs, then use the detector schematic on the image monitor to finish positioning the laser.

7.2.11 Locate Tip

For initial start-up where the Z-stage and optics have been almost fully retracted (such as when you performed **STAGE** > **LOAD NEW SAMPLE**), use the **Alignment Station**. 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. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever.

> **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.

7.2.12 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 1.5cm diameter metal disks used for sample mounting. The Dimension Icon SPM ships with several steel sample disks that you may attach to a magnetic sample holder located in the Dimension Chuck Spare Parts 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 7.2p).

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 7.2q).

   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 Icon is not optimal for imaging soft magnetic materials due its the magnetic hold-down properties. If magnetic imaging is intended, use the vacuum chuck.

   Note: For small samples with flat backs or samples mounted on metal discs, use the vacuum chuck.

Figure 7.2p   Securing Double-sided Tape to the Sample Disk

Figure 7.2q   Securing the Sample
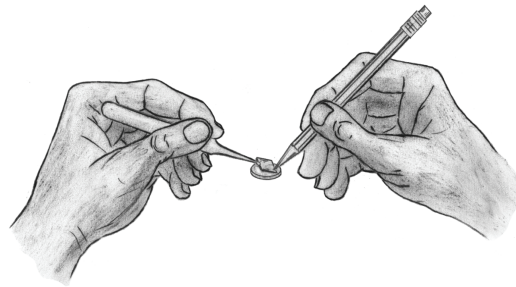
5.   Secure the sample atop the stage.

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 labeled **Vacuum** at the upper right of the Dimension Icon, shown in Figure 1.4b. The stage has five holes available for holding samples: the center hole, three holes with machined rings for holding wafers and discs, and an isolated hole near the edge of the chuck for small samples. Remove the screw from the hole you will use, and block all unused holes with screws.

7.2.13 Focus Surface

1.   Click the NAVIGATE icon in the **Workflow Toolbar**.

2.   Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3.   Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4.   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. be careful when performing this in the downward direction

	CAUTION:	Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

7.2.14 Cantilever Tune (TappingMode only)

1. Select **Cantilever Tune** from the **Microscope** > **Cantilever Tune** pop-down menu, or click on the **Cantilever Tune** icon in the **Workflow Toolbar**.

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 **500mV**.

3. Click **AUTO TUNE**.

4. When the procedure ends, click **EXIT** to exit the function.

7.2.15 Set Initial Scan Parameters

1. In the **Scan Parameters List** window, set the following:

    •   Initial **Scan Size** is set to **1μm** (or desired scan size). Veeco recommends you always initially engage with small scan sizes.

    •   **X** and **Y Offsets** are set to **0**.

    •   **Scan Angle** is set to **0**.

2. In **TappingMode**, under the **Feedback** panel, set the following:

    •   **Integral Gain** is set to **0.5**.

    •   **Proportional Gain** is set to **5**.

    •   **Scan Rate** is set to **1Hz**.

3. In **Contact Mode AFM**, under the **Feedback Controls** panel, set the following:

    •   **Deflection Setpoint** is set to **0.5V**.

    •   **Integral Gain** is set to **2**.

    •   **Proportional Gain** is set to **5**.

    •   **Scan Rate** is set to **1Hz**.

7.2.16 Engage

- Select **MICROSCOPE > ENGAGE**, or click the **ENGAGE** icon in the **Workflow Toolbar**.

- Once engaged, refer to the appropriate chapter in this manual for further instructions for the mode you are using (e.g. **Contact AFM:** Chapter 10, **TappingMode AFM:** Chapter 11, etc.)

7.2.17 Withdraw

When imaging is complete, withdraw the tip by selecting **Microscope** > **Withdraw**, or the click **Withdraw** icon in the **Workflow Toolbar**. This typically raises the tip 1mm above the sample.

Although the 1mm withdraw distance provides enough clearance to loosen the dovetail and remove the AFM head, if further distance is desired perform one of the following:

- Select **MICROSCOPE > WITHDRAW** to raise the Z-stage with the trackball.

- Execute **WITHDRAW** from the **Workflow Toolbar** multiple times.

  Note: It is impossible to move the Dimension head if the laser sum signal is too low.

7.3   Advanced AFM Operation

7.3.1   Stage Parameters

The system ships with the following default stage parameters (see Figure 7.3a). 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 **MICROSCOPE > ENGAGE SETTINGS > GENERAL** to open the **Engage Setings** window, shown in Figure 7.3a.

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

Figure 7.3a   Default SPM Parameters

Definitions of **Engage Stage-Motor Settings** fields are as follows:

Sample Clearance

Defines the height of the probe tip over the sample prior to **Engage** (set up by performing the **Locate Tip** and **Focus Surface** routines). You may change the sample clearance parameter once you are experienced with the system. The default parameter is 999µm. This parameter also sets the distance that the tip is raised above the sample each time **Withdraw** is executed.

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, but it increases the risk of the tip reaching the surface during the fast engage if the **Locate Tip** and **Focus Surface** focal points are not properly set. If this happens, the tip has "crashed" and may need replacing.

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. 6-7µm).

Load/Unload Height

This parameter defines how high the tip moves above the sample when **Tools > Stage** > **Load New Sample** is executed. After the Z-stage is raised, the sample stage moves as far forward in the Y direction (towards you) as possible.

Chapter 8   Adaptive XY Scanning

This chapter discusses Adaptive XY Scanning, a Veeco-proprietary technology that enables faster scanning and less position noise than traditional closed-loop scanning.

Specifically, this chapter discusses the following areas:

- **Introduction:** Section 8.1

    - **Higher bandwidth tracking:** Section 8.1.1

    - **Improved position accuracy:** Section 8.1.2

- **Operation:** Section 8.2

    - **Adaptive XY Controls:** Section 8.2.1

8.1   Introduction

Adaptive XY control improves on conventional closed-loop XY control in two areas:

- Higher bandwidth tracking

- Improved position accuracy

Conventional closed-loop XY control, while greatly improving positioning accuracy and scan linearity compared to open loop operation, has two issues:

1. Increased XY position noise: The noise of the sensor is introduced into the feedback loop and propagates to the controller output (this is more prominent with scan sizes less than 500nm).

2. Feedback instability and limited tracking bandwidth: the nature of feedback requires that the tracking bandwidth is well below scanner's resonant frequency in XY direction for conventional PID closed-loop XY control.

Noise performance and tracking performance are two conflicting goals.

For AFM applications, the scanner is required to track a repetitive raster scan motion while compensating low frequency position errors introduced by creep and thermal drift. Adaptive XY control separates the two requirements with advanced digital signal processing which results in both increased tracking bandwidth and improved position accuracy.

Figure 8.1a shows a block diagram of the Adaptive XY control mechanism.

Figure 8.1a   Block diagram of Adaptive XY control
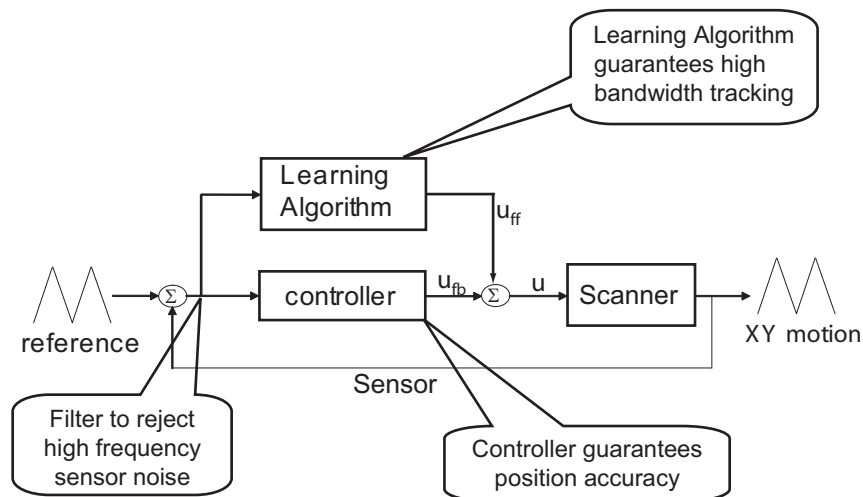
8.1.1   Higher bandwidth tracking

Adaptive XY control takes advantage of a Veeco-proprietary control technology "Model Based Inverted Iterative Control" to achieve a tracking accuracy (e.g., peak error) of less than 1% of the total scan range in a minimum amount of time. This inversion-based iterative control (IIC) is based on the work of Zou et. al.[1]

After a user specifies a new **Scan Size**, **Scan Rate** or **Scan Angle**, the Adaptive XY algorithm automatically generates and optimizes a scan table. The scan table ensures high bandwidth tracking without introducing sensor noise.

For conventional PID closed-loop XY control, the tracking bandwidth is well below scanner's resonant frequency in the X and Y directions.

With Adaptive XY control, the tracking bandwidth can be even higher than the scanner's resonant frequency. Tens of Hertz can be achieved without compromising image quality.

1.Q. Zou et al., "Precision tracking of driving wave forms for inertial reaction devices," Rev. Sci. Instrum. 76, 023701 (2005).

Figure 8.1b shows a scan without Adaptive XY control and Figure 8.1c shows the same scan with Adaptive XY control.The periodic artifact in Figure 8.1b is a product of XY scanner ringing. Adaptive XY control, shown in Figure 8.1c, avoids this ringing.

Figure 8.1b Adaptive XY control disabled
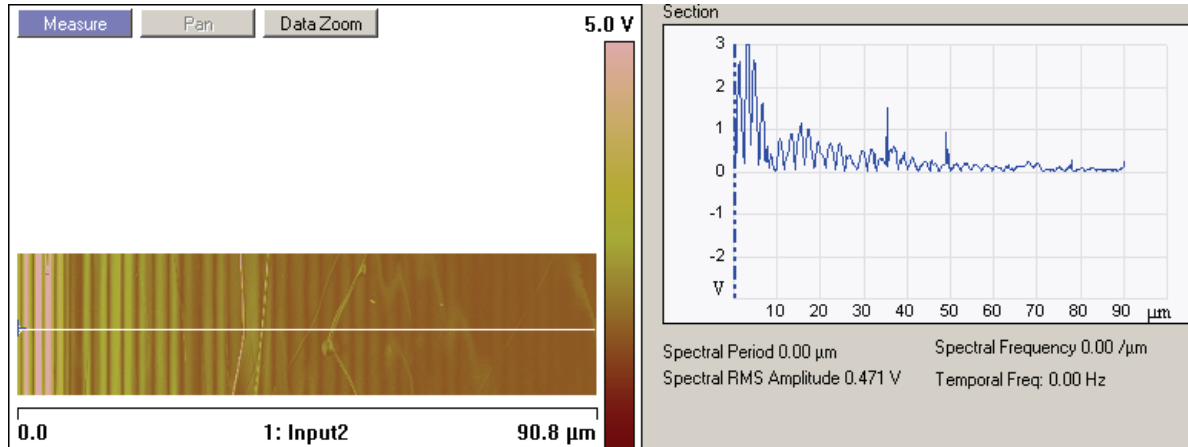
Figure 8.1c Adaptive XY control enabled
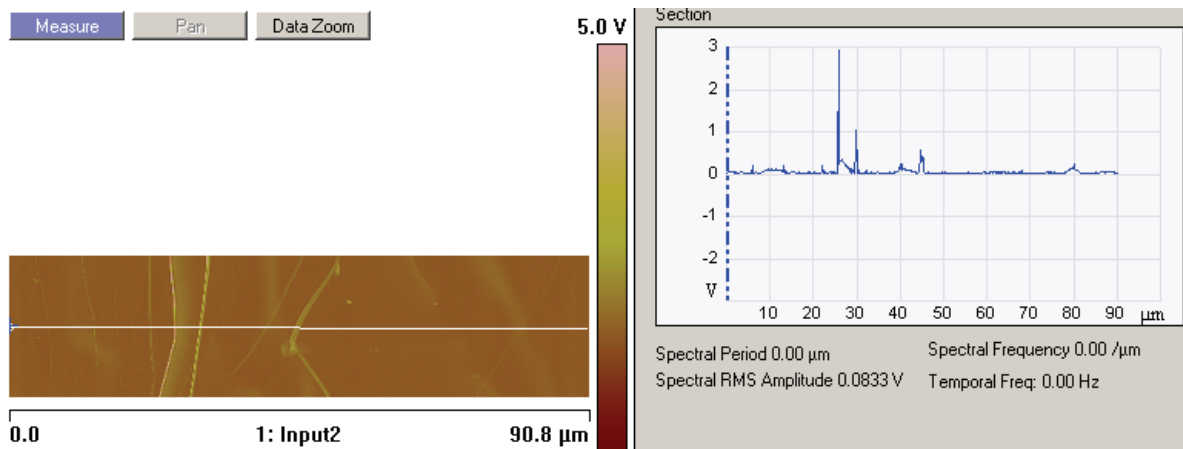
Figure 8.1d shows an Adaptive XY scan (8 nm square) of mica showing atomic resolution.

Figure 8.1d   Adaptive XY scan on mica showing atomic resolution
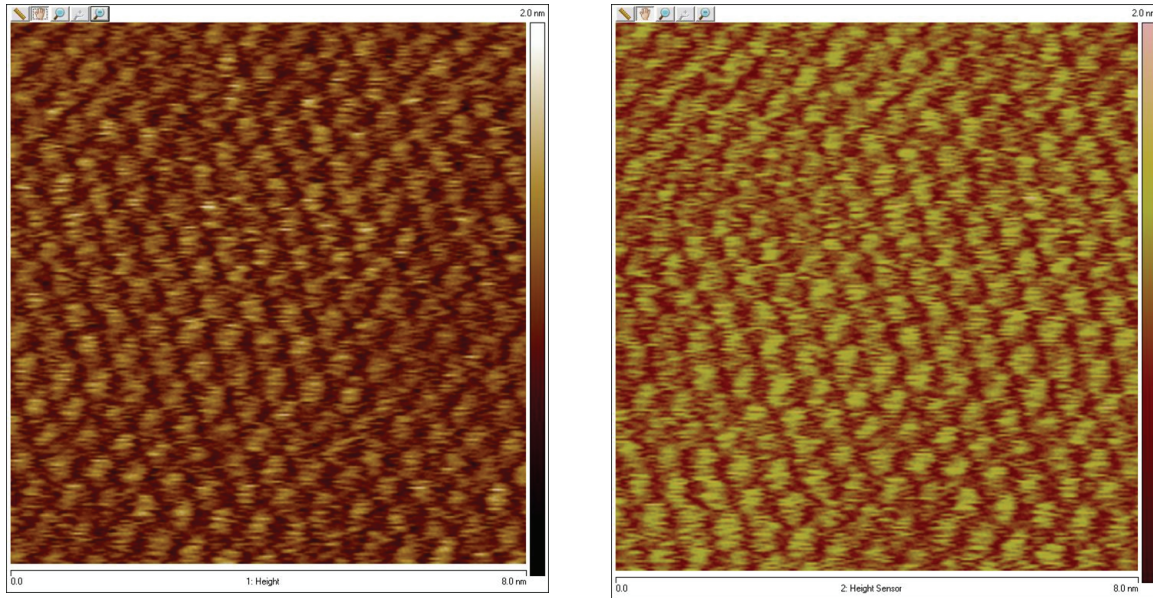
8.1.2 Improved position accuracy

Adaptive XY control uses a low frequency feedback loop to compensate for low frequency position errors introduced by phenomena such as thermal drift and creep.

The sensor signal is filtered to reject the high frequency noise and improve position accuracy.

Figure 8.1e shows a scanned image of calibration grating (10um pitch size, 100um scan size, 10Hz) along with a digital zoom of a 5 µm region of that scan (B). Figure 8.1e (C) shows a new scan (3.2um scan, 10Hz) at the area shown in (B). This shows low noise and precise positioning of the scanner. Creep is not evident.

Figure 8.1e   Scanned image of calibration grating

B: Digital zoom on 5 um area of Fig. A

C: Actual scan of the area indicted in Figs. A and B

A: Original image
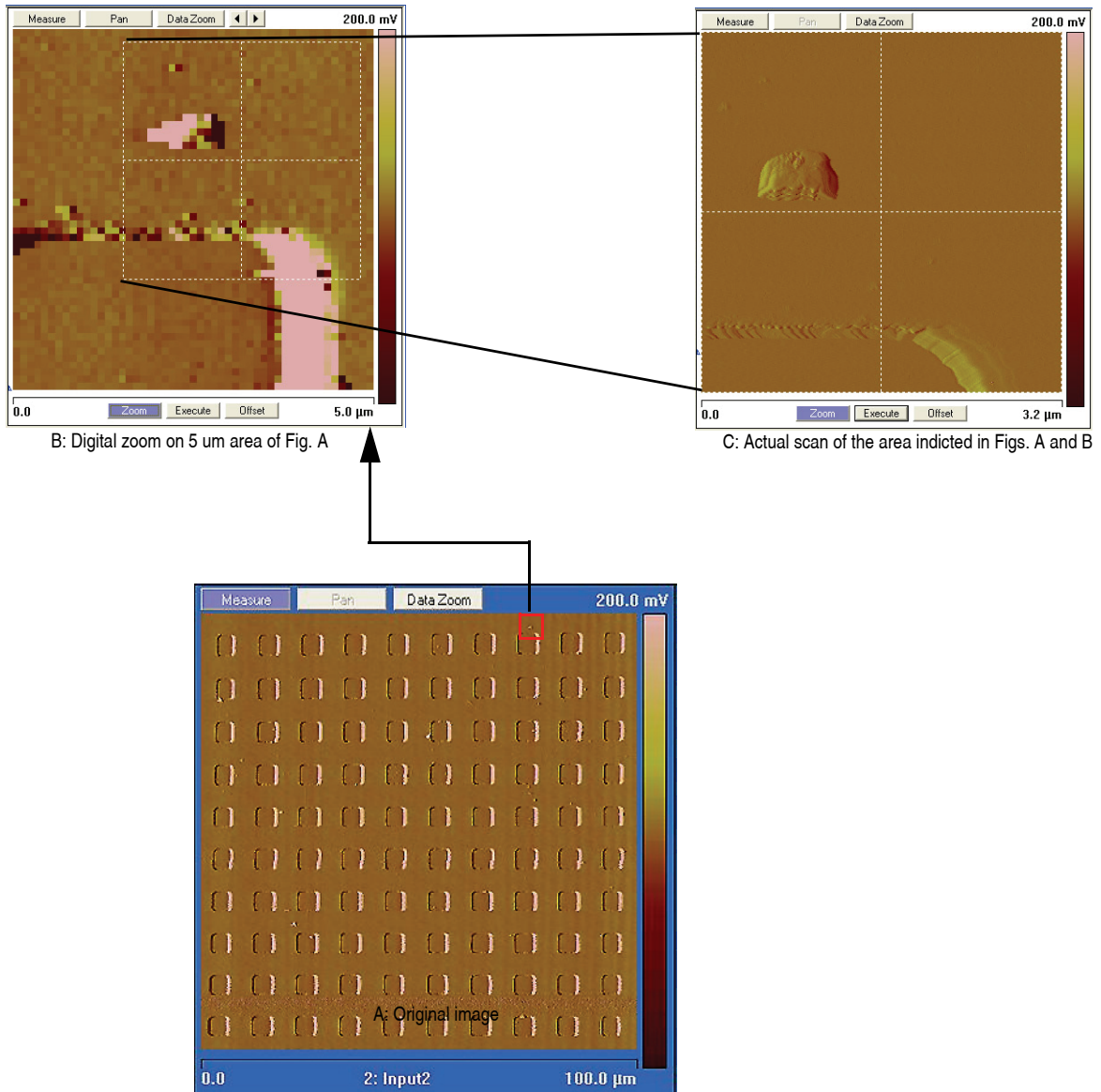
8.2 Operation

1. Click the **EXPANDED MODE** icon, to access the **XY Closed Loop** drop-down box, shown in Figure 8.2a.

2. Select **ADAPTIVE**, shown in Figure 8.2a.

Figure 8.2a Select **ADAPTIVE**
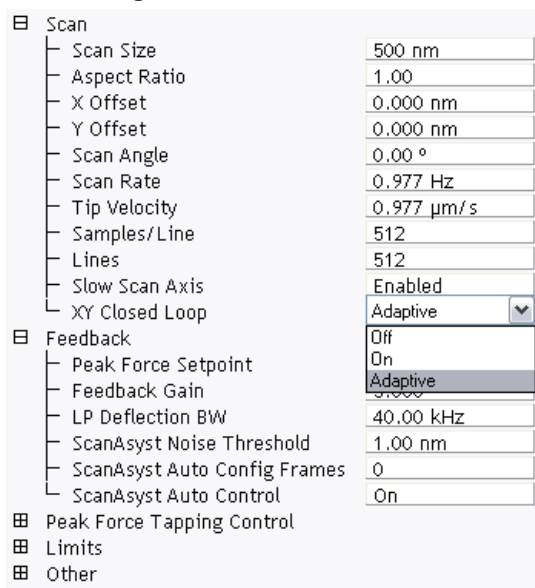
3. Click Calibrate > Scanner > **ADAPTIVE XY FEEDBACK** (see Figure 8.2b) to open the **Adaptive XY Feedback Calibration** window, shown in Figure 8.2c.

Figure 8.2b Select **ADAPTIVE XY FEEDBACK**
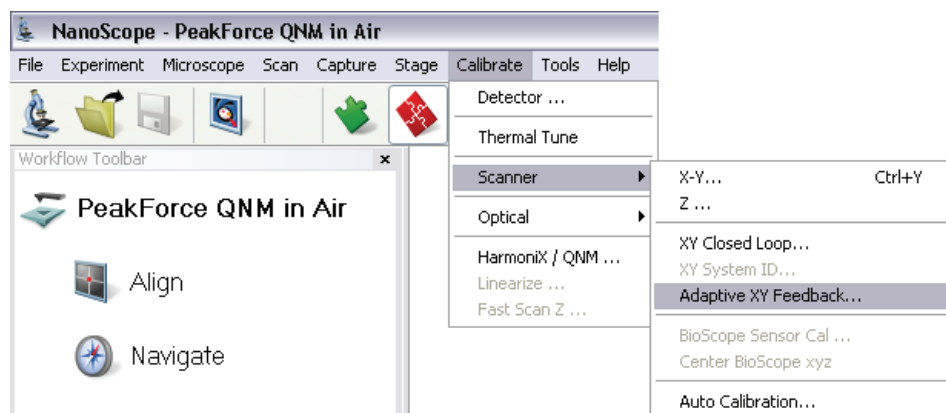
Figure 8.2c   The **Adaptive XY Feedback Calibration** window
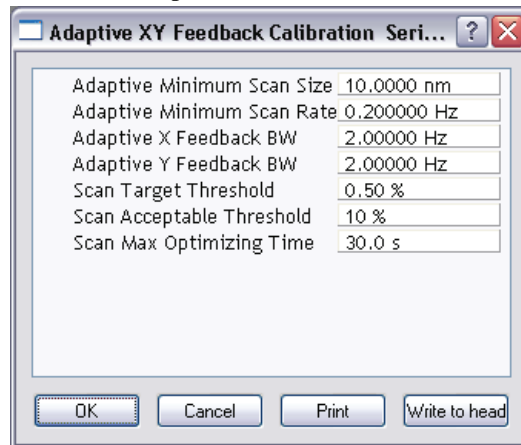
8.2.1   Adaptive XY Controls

The following controls are available for Adaptive XY scanning:

Adaptive XY Minimum Scan Size   To reduce sensor noise, the feed forward portion of the adaptive XY algorithm is turned off if the **Scan Size** is below this value.

Adaptive XY Minimum Scan Rate   The feed forward portion of the adaptive XY algorithm is turned off if the **Scan Rate** is below this value.

Adaptive X Feedback BW   This corresponds to the closed loop bandwidth. Lower values result in cleaner images.

Adaptive Y Feedback BW   This corresponds to the closed loop bandwidth. Lower values result in cleaner images.

Scan Target Threshold   The adaptive XY algorithm will stop converging when the linearity is below this value.

Scan Acceptable Threshold   The adaptive XY algorithm is deemed to have failed if the linearity is greater than this number. This usually indicates that something is wrong with the system.

Scan Max Optimizing Time   The maximum time allowed for the Adaptive XY algorithm to converge.

Chapter 9   ScanAsyst

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

> **Note:**   ScanAsyst is not available on the Dimension Icon-PI microscope.

- **ScanAsyst and Peak Force Tapping Mode Parameters:** Section 9.5

    - **Feedback Parameters:** Section 9.5.1

    - **Peak Force Tapping Control Parameters:** Section 9.5.2

    - **Limits Parameters:** Section 9.5.3

- **Capture buttons:** Section 9.6

- **Optimizing a ScanAsyst image:** Section 9.7

- **Advanced Atomic Force Operation:** Section 9.8

    - **Displaying Parameters:** Section 9.8.1

9.1   Introduction

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

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

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

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

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

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

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

9.2   ScanAsyst Principles of Operation

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

9.2.1   The "Heartbeat"

The **Force vs. Time** display, shown in Figure 9.2a is referred to as the "heartbeat." The initial contact of the probe with the sample (B), peak force (C) and adhesion (D) points are labelled.

Figure 9.2a   The "heartbeat." Blue indicates approach while red indicates retract.
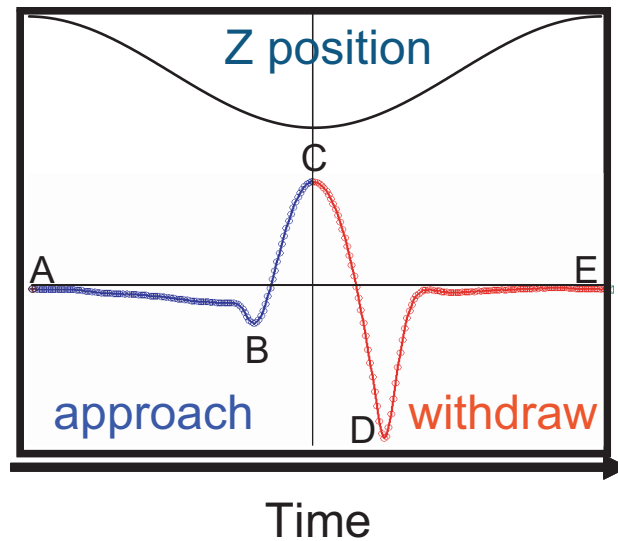
9.2.2 Force curves

Using the Z-position information, the heartbeat is transformed into a force curve, shown in Figure 9.2b. The force curve plot is analyzed, on the fly, to produce the peak interaction force as the control feedback signal and the mechanical properties of the sample (**Adhesion**, **Modulus**, **Deformation**, **Dissipation**).

Figure 9.2b Force curve
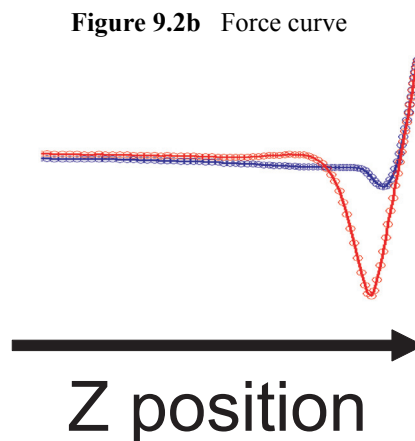
9.3 Probe Selection

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

- ScanAsyst-Air (k ~ 0.4 N/m, tip radius < 10 nm)

- ScanAsyst-Fluid (k ~ 0.7 N/m, tip radius < 10 nm nominal, max. = 15 nm)

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

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

9.4 Basic ScanAsyst AFM Operation

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

9.4.1 Select the Microscope

Follow the **Select the Microscope** procedure described in Section 7.2.1.

9.4.2 Select Mode of Operation

1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 9.4a.

Figure 9.4a The ScanAsyst **Select Experiment** window
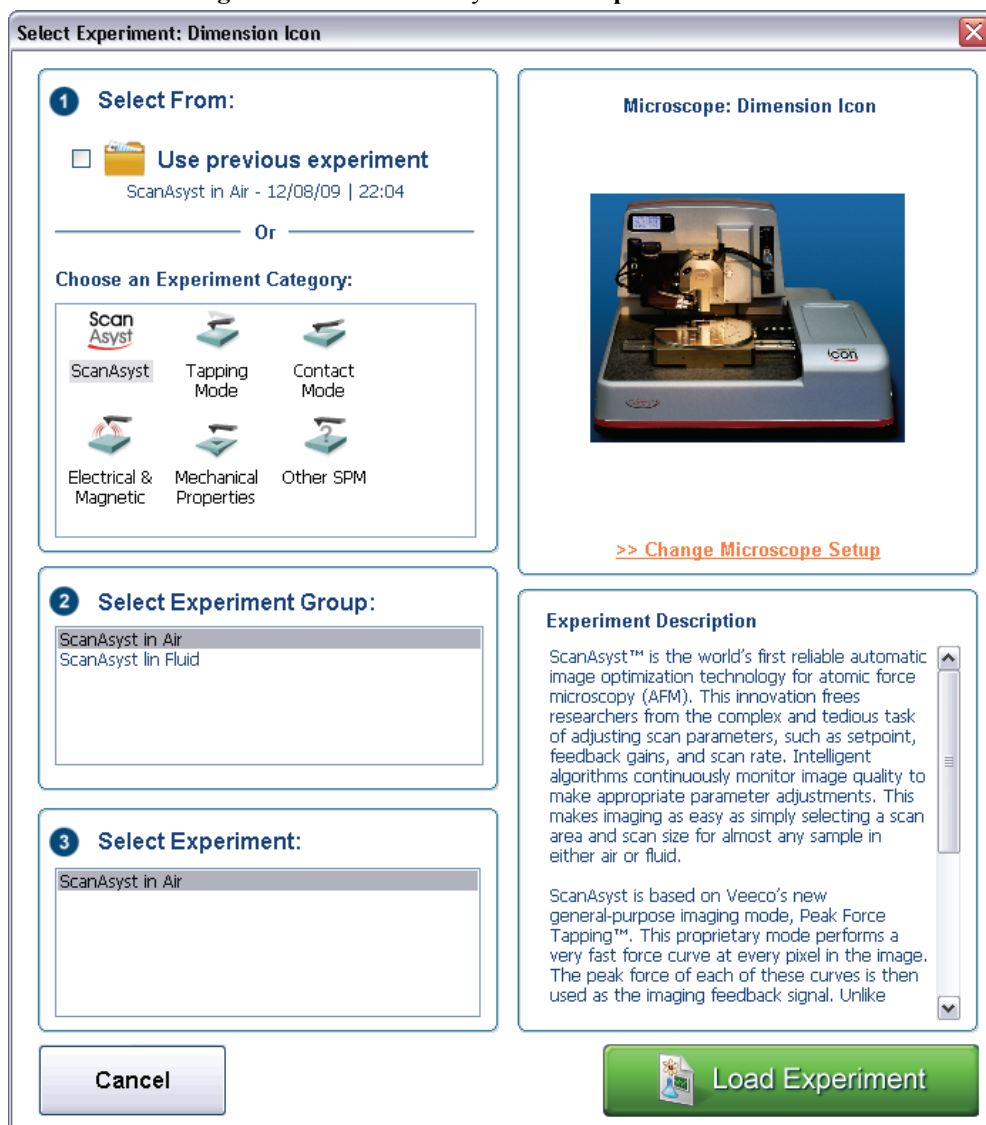
2. Select **SCANASYST** in the **Choose an Experiment Catagory** panel.

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

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

5. This opens the **Workflow Toolbar**, the **Scan One Channel** window, the **Force Monitor** window and the **Scan Parameters List** window, shown in Figure 9.4b.

Figure 9.4b   ScanAsyst Mode in Air (Simple Mode) configuration
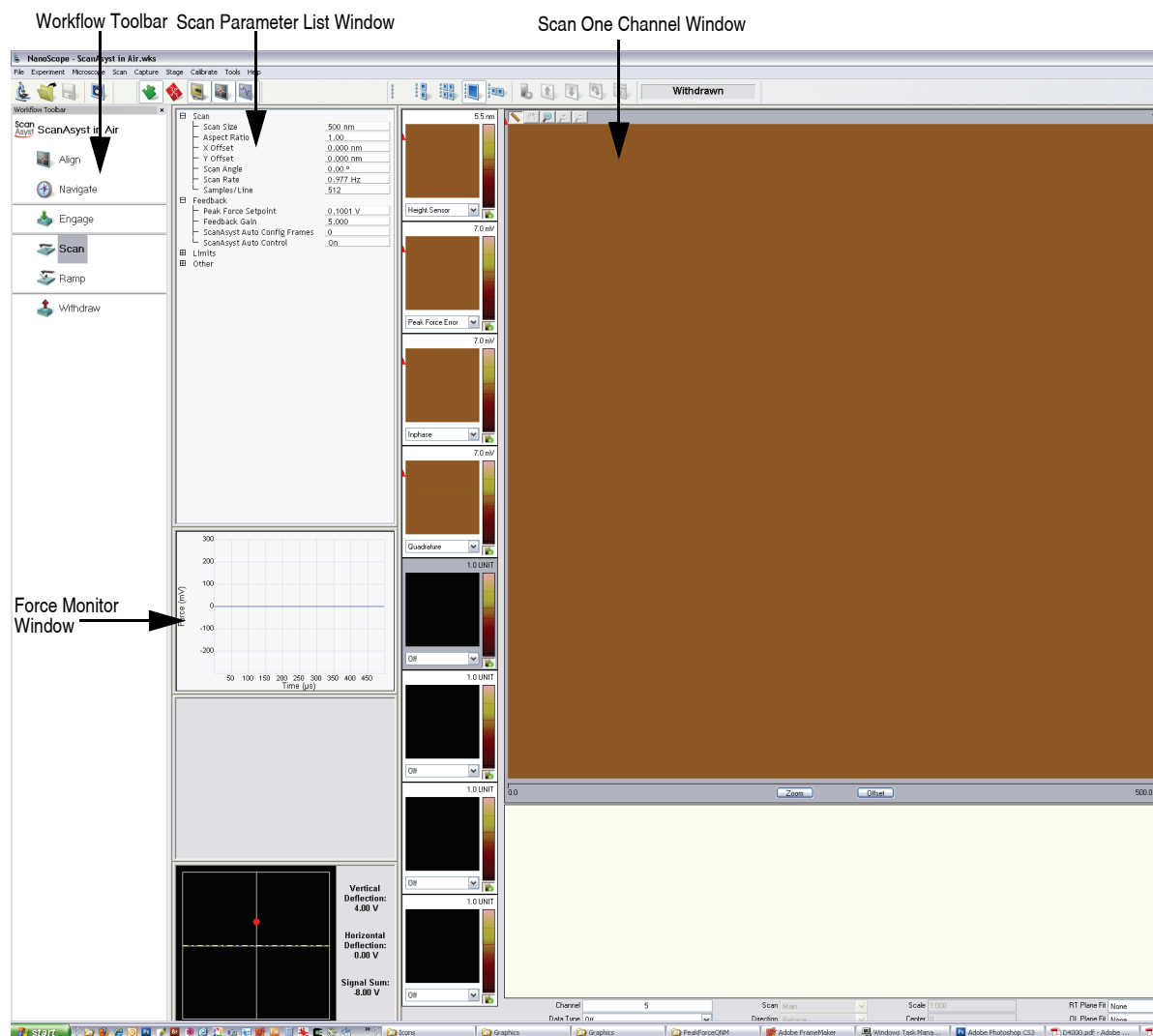
9.4.3   Head, Cantilever and Sample Preparation

1. Install a ScanAsyst probe 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. See **Basic AFM Operation:** Section 7.2.

9.4.4   Align Laser

1. Align the laser using the laser control knobs. See **Align Laser:** Section 7.2.9.

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 **4-6V**.

9.4.5   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. See **Adjust Photodetector:** Section 7.2.10.

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

3. Set the **Vertical Deflection** to -**2V**.

9.4.6   Locate Tip

To locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

9.4.7   Focus Surface

1. Click the Nᴀᴠɪɢᴀᴛᴇ icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

CAUTION:	Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

9.4.8 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 9.4c).

1. Set the **Scan Size**.

2. Set the **Scan Angle**.

Feedback Panel

1. Set **ScanAsyst Auto Control** to **ON** (see Figure 9.4c).

Figure 9.4c   ScanAsyst in Air (Simple Mode) Parameters Panel

Channels 1, 2, 3 and 4

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

1. **Channel 1 Data Type** is **HEIGHT SENSOR** (see Figure 9.4d).

2. **Channel 2 Data Type** is **PEAK FORCE ERROR**.

3. **Channel 3 Data Type** is **INPHASE**.

4. **Channel 4 Data Type** is **QUADRATURE**.

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

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

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

Figure 9.4d Default Channel Settings

9.4.9   Engage

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

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

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

9.4.10 Image the sample

1. If needed, right-click in the **Force Monitor** window and click **UNDOCK**. See Figure 9.4e.
    You may **DOCK** the undocked **Force Monitor** window by right-clicking in it and clicking
    DOCK.

Figure 9.4e   Undock the Force Monitor window
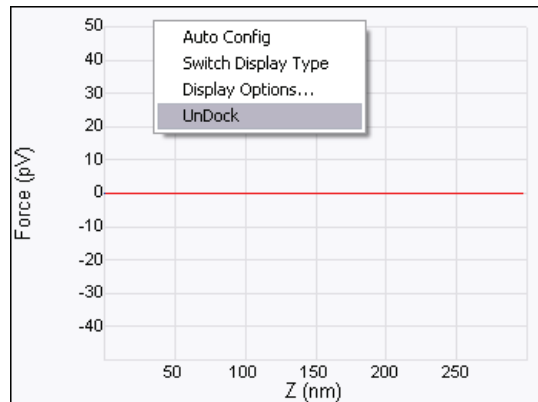
2. Select one plot to be FORCE VS. TIME and the other to be FORCE VS. Z.

3. Once scanning, the **Force Monitor** window, shown in Figure 9.4f, should display a **Force vs. Z** plot and a "heartbeat" (**Force vs. Time**) plot.

Figure 9.4f The **Force Monitor** window
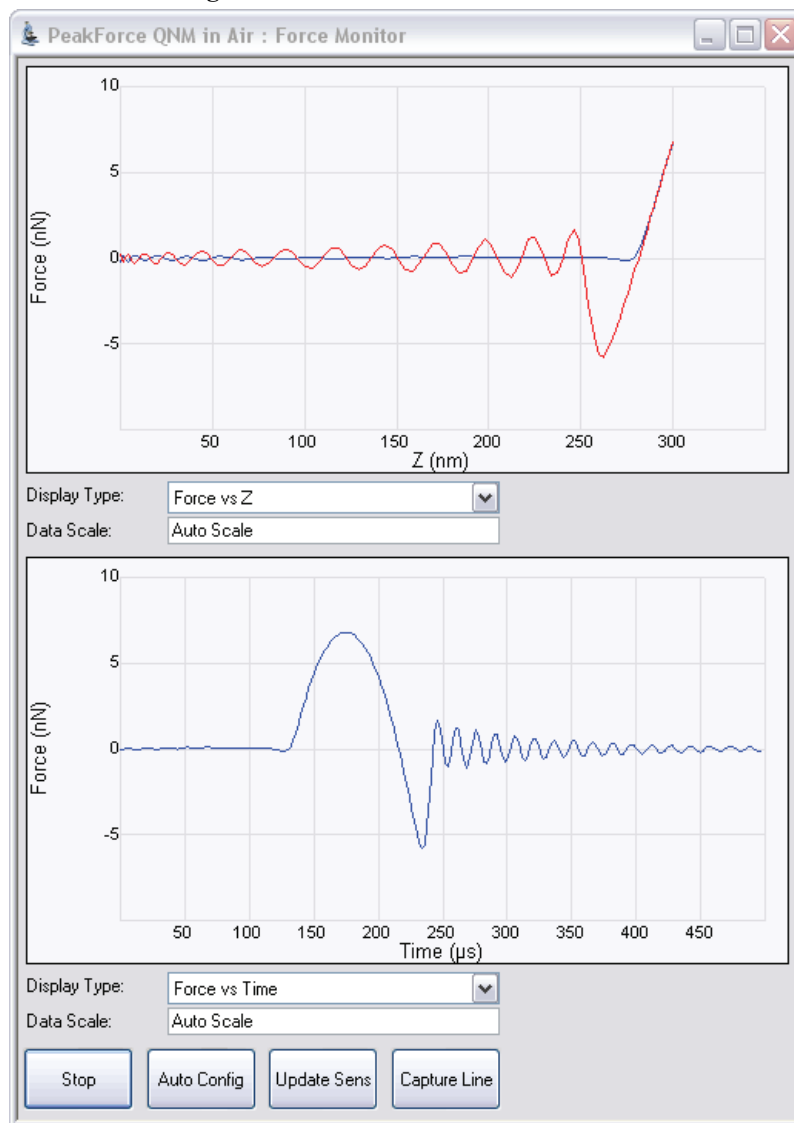
4. The **HEIGHT** channel in the **Scan** window, shown in Figure 9.4g, will display a topographical image of your sample.

Figure 9.4g Height Image of a PS + LPDE blend.
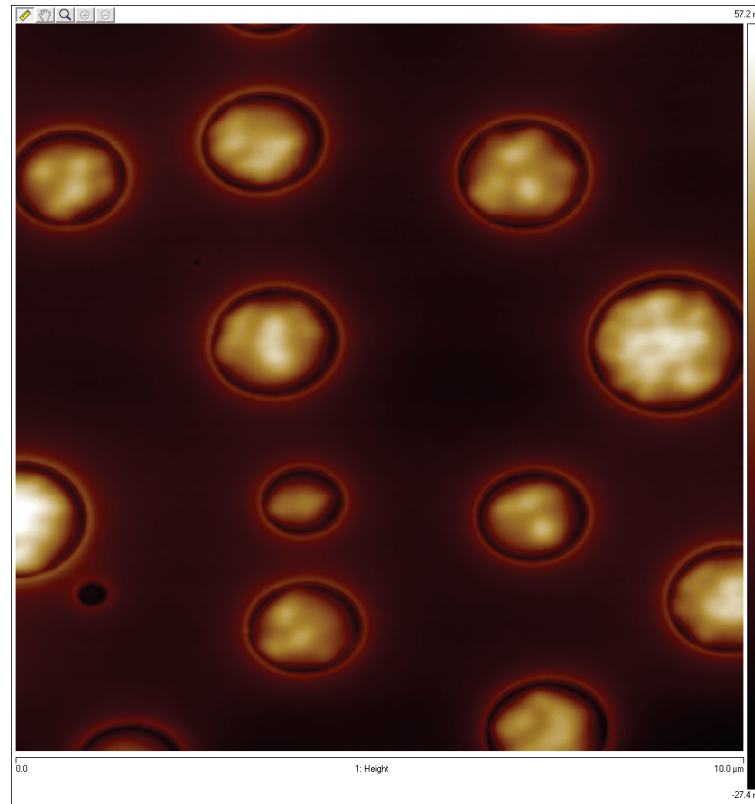
9.5   ScanAsyst and Peak Force Tapping Mode Parameters

9.5.1   Feedback Parameters

Peak Force Setpoint

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

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

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

Feedback Gain

The gain of the Peak Force Tapping feedback control loop.

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

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

Low Pass Deflection Bandwidth

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

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

ScanAsyst Setup

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

ALLOW: Allows ScanAsyst Auto Control.

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

ScanAsyst Noise Threshold

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

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

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

ScanAsyst Auto Config Frames

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

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

ScanAsyst Auto Control

Range and Settings: OFF: Turns ScanAsyst Auto Control OFF.

ON: Turns ScanAsyst Auto Control ON.

INDIVIDUAL: Allows individual control of **ScanAsyst Auto Gain**, **ScanAsyst Auto Setpoint**, **ScanAsyst Auto Scan Rate** and **ScanAsyst Auto Z Limit**.

ScanAsyst Auto Gain

ScanAsyst Auto Gain allows NanoScope to dynamically control **Feedback Gain**.

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

ON: Turns **ScanAsyst Auto Gain** ON.

ScanAsyst Auto Setpoint

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

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

ON: Turns **ScanAsyst Auto Setpoint ON**.

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

ScanAsyst Scan Auto Scan Rate

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

Range and Settings: **OFF**: Turns **ScanAsyst Scan Auto Scan Rate OFF**.

ON: Turns **ScanAsyst Scan Auto Scan Rate ON**.

ScanAsyst Auto Z Limit

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

Range and Settings: **OFF**: Turns **ScanAsyst Auto Z Limit OFF**.

ON: Turns **ScanAsyst Auto Z Limit ON**.

9.5.2   Peak Force Tapping Control Parameters

Peak Force Amplitude

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

Lift Height

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

> **Note:** Changing the **Lift Height** will automatically start the **AUTO CONFIG** function (see **Optimizing a ScanAsyst image:** Page 192) and retract the Z piezo to the specified **Lift Height**. Clicking **AUTO CONFIG** will automatically calculate the **Lift Height** and perform an **AUTO CONFIG** operation.

9.5.3   Limits Parameters

Z Limit

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

Range or Settings: 8.33 V (~0.241 μm) to 309.3 V (~9 μm).

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

Z Range

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

Range or Settings: 0.2 nm to ~7.42 microns.

Deflection Limit

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

Range or Settings: 4.096V - 24.58V.

9.5.4 Parameter Visibility

The visibility of various parameters depends on the selected mode. Table 9.5a shows parameter visibility as a function of microscope mode.

Table 9.5a   Parameter Visibility

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

9.6   Capture buttons

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

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

2.   When you are in a region of interest, click the **CAPTURE LINE** button, shown in Figure 9.6a, to capture a scan line.

Figure 9.6a   CAPTURE LINE button
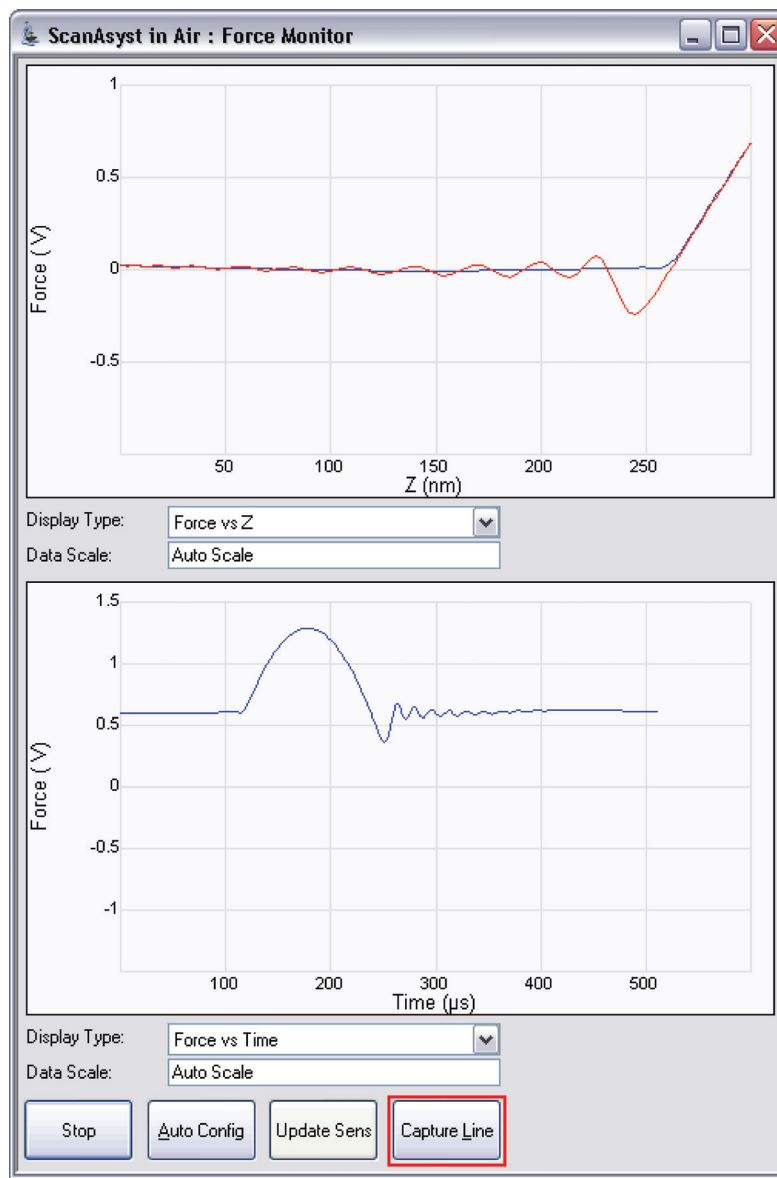
3. The **High Speed Data Capture** window, shown in Figure 9.6b, will open and the **Status** will change when the data has been captured. UPLOAD DATA to the PC when the capture is complete. When CAPTURE LINE is used this way, the off-line NanoScope Analysis software will correctly associate the capture line of the high speed data capture with the line in the image.

Figure 9.6b High speed data capture is complete. However, the data is not immediately transferred to the PC.
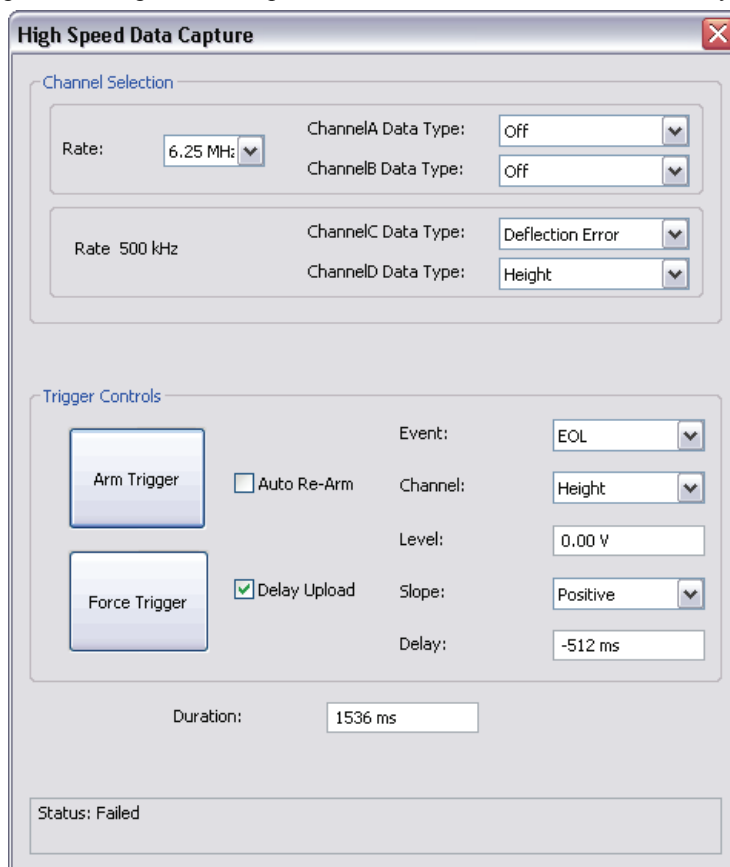
4. Click the UPLOAD DATA button to transfer the captured data to the computer. While the data transfer process takes place, the scan data will look corrupted because the DSP time is shared between PeakForce QNM properties computation and data transfer.

9.7   Optimizing a ScanAsyst image

If your force curves show background noise or otherwise need improvement, click **AUTO CONFIG** to invoke the real-time pattern analysis algorithm that removes parasitic deflection.

Note: Clicking **AUTO CONFIG** will automatically calculate the **Lift Height** and perform an **AUTO CONFIG** operation.

Figure 9.7a   The **AUTOCONFIG** button
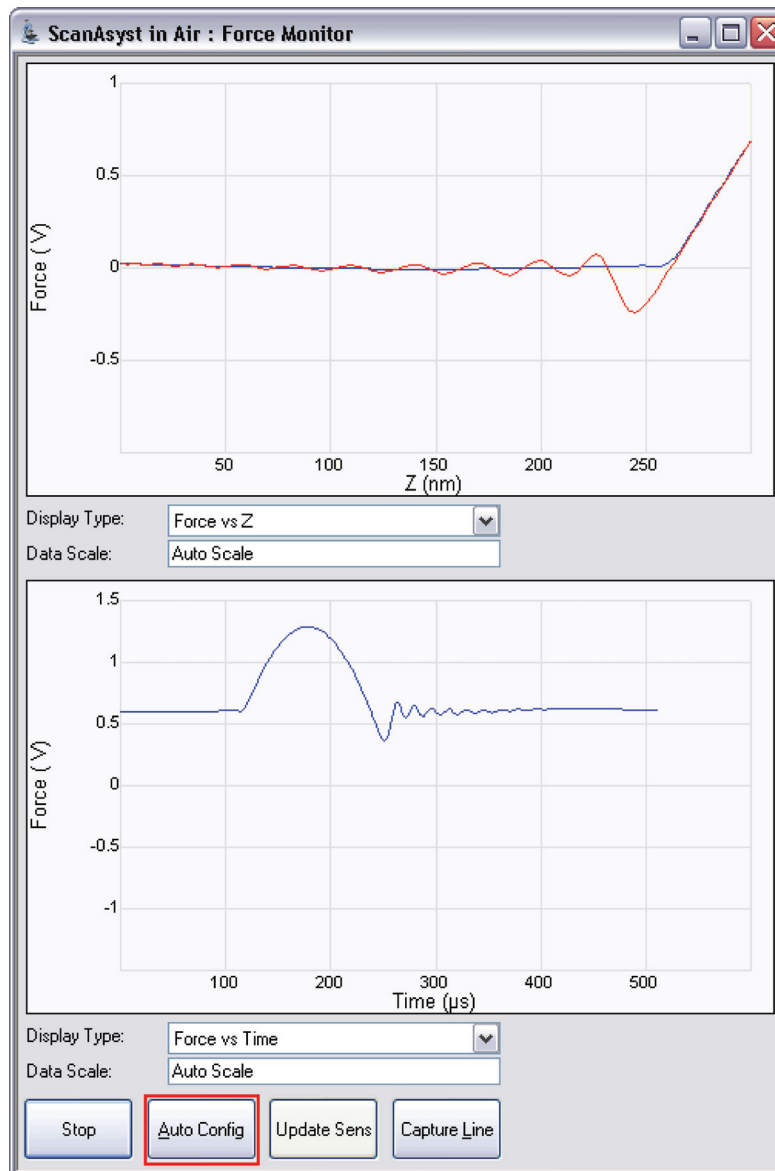
9.8   Advanced Atomic Force Operation

9.8.1   Displaying Parameters

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

Simple Mode

1. The default SIMPLE MODE, intended for novice users and shown in Figure 9.8a, displays the minimum number of parameters needed to make an image.

Figure 9.8a   The SIMPLE MODE view of the **Scan Parameter List** for **ScanAsyst in Air**

Expanded Mode

1. The EXPANDED MODE view, shown in Figure 9.8b, increases the number of displayed parameters enabling expert users to fine tune an image.

Figure 9.8b   The EXPANDED MODE view of the **Scan Parameter List** for **ScanAsyst in Air**

Show All

1. From the Menu bar, click **EXPERIMENT** > **CONFIGURE EXPERIMENT**. This opens an information window, shown in Figure 9.8c.

Figure 9.8c The Configure Experiment information window
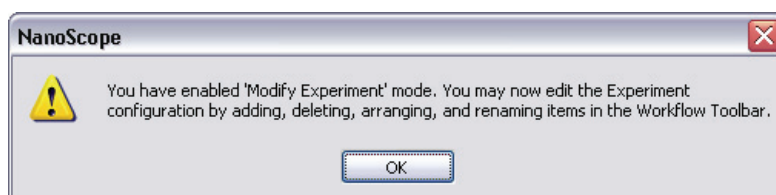
2. Click **OK** to open the **Configure Experiment** window, shown in Figure 9.8d.

Figure 9.8d The **Configure Experiment** Window
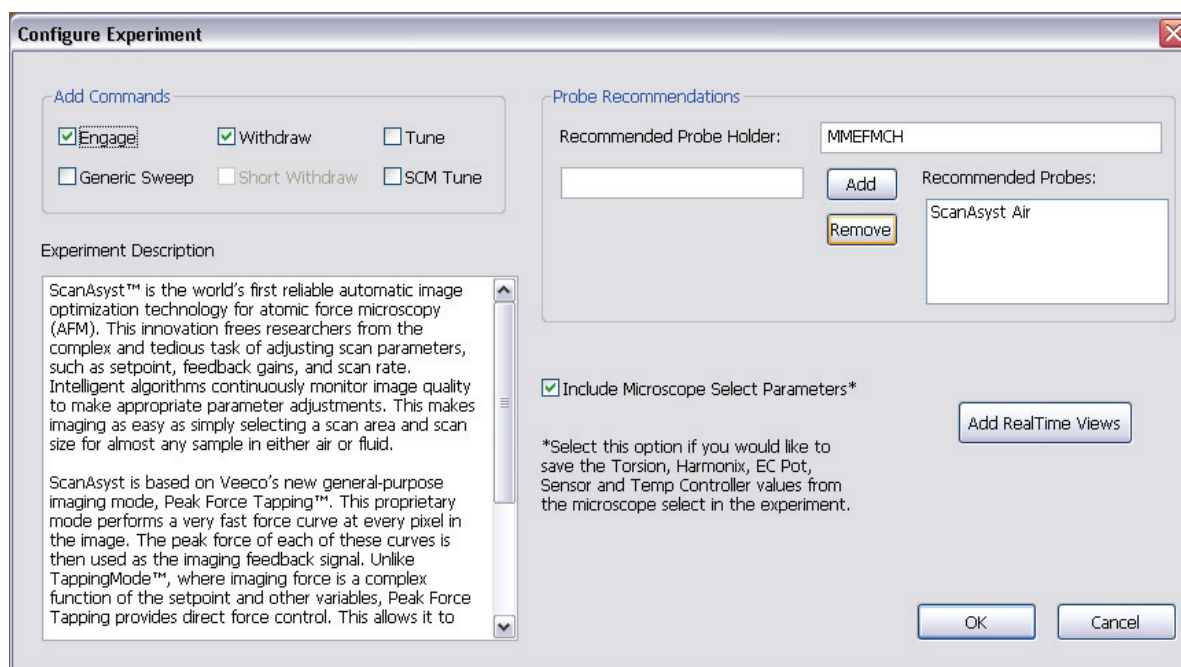
3. Click **CANCEL** which will close the **Configure Experiment** window.

4. Right-click in the **Scan Parameter List** panel and select SHOW ALL, shown in Figure 9.8e.

Figure 9.8e Select SHOW ALL items
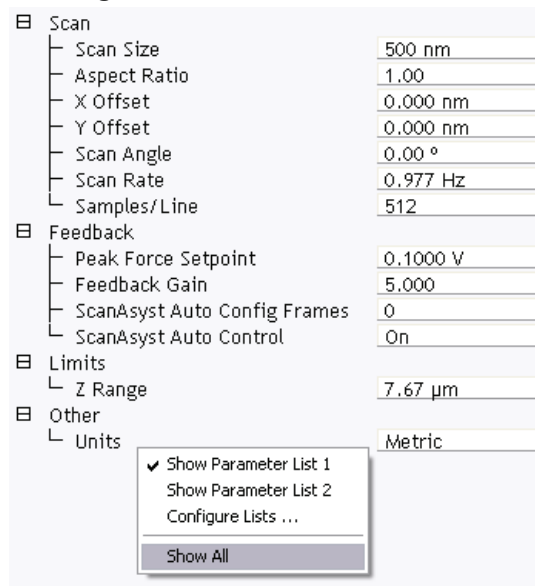
This makes all **Scan Parameters** visible along with two check boxes, the left, green, check box for the Sᴍᴘʟᴇ Mᴏᴅᴇ and the right, red, check box for the Exᴘᴀɴᴅᴇᴅ Mᴏᴅᴇ. See Figure 9.8f.

Figure 9.8f Enable Parameters

With "☑"
Parameter will
display

Without "☑"
Parameter will
not display

The checked ☑ parameters display in normal Real-time mode while those parameters without a ☑ will not display in normal Real-time mode.

Check the parameters that you want displayed and right-click in the **Scan Parameter List** and select **SHOW ALL** items to hide the unchecked parameters. The panel will once again appear in normal Real-time mode.

Chapter 10   Contact AFM

This chapter covers procedures for operating the Dimension Icon 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.

- **Probe Selection:** Section 10.1

    - **Cantilever Selection:** Section 10.1.1

- **Basic Contact Mode AFM Operation:** Section 10.2

    - **Select the Microscope:** Section 10.2.1

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

    - **Head, Cantilever and Sample Preparation:** Section 10.2.3

    - **Align Laser:** Section 10.2.4

    - **Adjust Photodetector:** Section 10.2.5

    - **Locate Tip:** Section 10.2.6

    - **Focus Surface:** Section 10.2.7

    - **Displaying Parameters:** Section 10.3.1

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

    - **Engage:** Section 10.2.9

- **Advanced Atomic Force Operation:** Section 10.3

    - **Displaying Parameters:** Section 10.3.1

    - **Optimization of Scanning Parameters:** Section 10.3.2

- **Force Calibration Mode:** Section 10.4

10.1   Probe Selection

Cantilever selection is critical and becomes more important as tip and cantilever technology continues to develop. A clear understanding of the parameters in the Scan Parameters List allows you 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.

10.1.1   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 Veeco website (www.veeco.com) for more information.

Silicon Nitride Cantilevers

Silicon nitride cantilevers for the Dimension Icon 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 www.veeco.com.

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 10.1a. 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 10.1a   Cantilever Spring Constants

Cantilever Type	k (N/m) Narrow Legs	k (N/m) Wide Legs
115μm (triangular)	0.38	0.58
196μm (triangular)	0.06	0.12

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. For more force curve information, see Chapter 15.

⚠	**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: 450µm) sous peine de destruction de celles-ci.
	VORSICHT:	Wir empfehlen, im Kontakt-Modus mit den geätzten Siliziumspitzen (nur mit den 450µm 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 pressure between the tip and sample. Therefore, etched silicon tips are not suggested for soft samples.

10.2   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.

10.2.1   Select the Microscope

Follow the **Select the Microscope** procedure described in Section 7.2.1.

10.2.2 Select Mode of Operation

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

Figure 10.2a The Contact Mode **Select Experiment** window
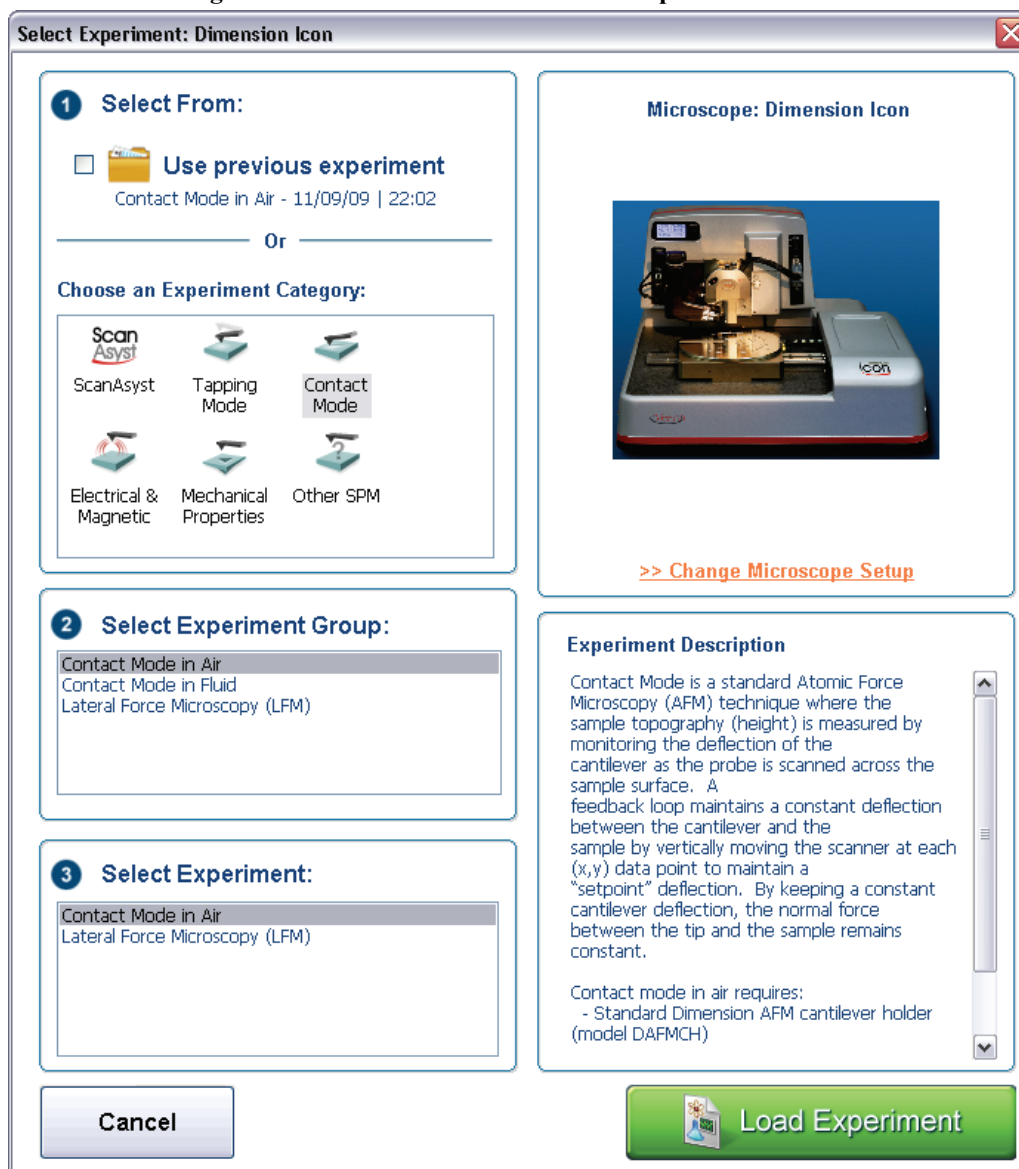
2. Select CONTACT MODE in the **Choose an Experiment Catagory** panel.

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

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

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** (Icon) or the **Scan 8 Channels** (Icon-PI) windows and the **Scan Parameters List** window, shown in Figure 10.2b.

Figure 10.2b Contact Mode in Air (Simple Mode) configuration
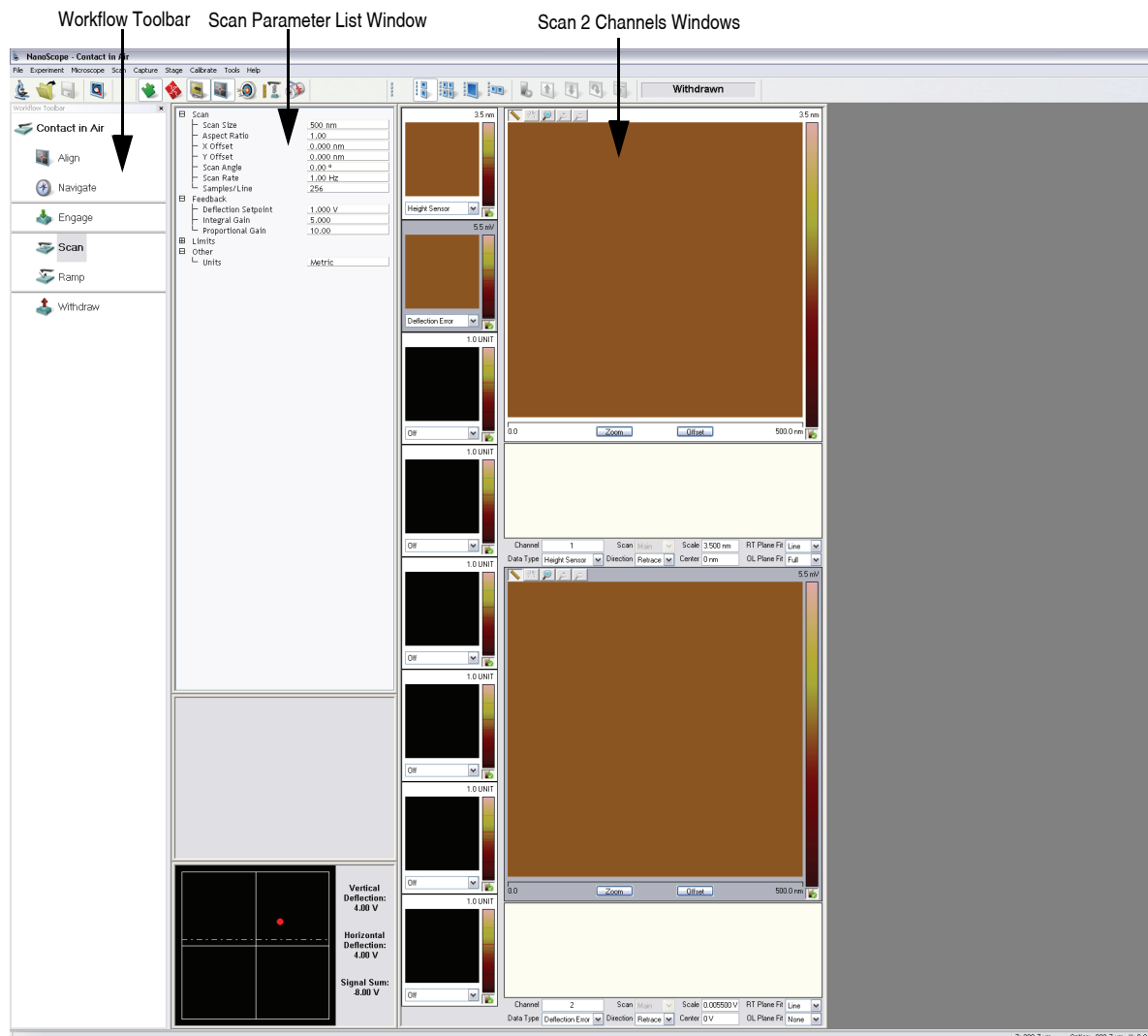
Note: Figure 10.2b shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

10.2.3 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. See **Basic AFM Operation:** Section 7.2.

10.2.4 Align Laser

1. Align the laser using the laser control knobs. See **Align Laser:** Section 7.2.9.

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 **4-6V**.

10.2.5 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. See **Adjust Photodetector:** Section 7.2.10.

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

3. Set the **Vertical Deflection** to **-2V**.

10.2.6 Locate Tip

To locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

10.2.7 Focus Surface

1. Click the NAVIGATE icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

> **CAUTION:** Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

10.2.8 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 10.2c).

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

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

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

Feedback Panel

1. Set the **Integral gain** to **2** and the **Proportional gain** to **5** (see Figure 10.2c).

2.   Set the **Deflection Setpoint** to **0.5V**.

Other Panel

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

Figure 10.2c   Suggested Contact Settings

Channels 1 and 2

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

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

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

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

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

5.   Set the **Channel 3** through **Channel 8 Data Type** to OFF.

Figure 10.2d   Suggested Channel Settings

10.2.9 Engage

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

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

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

10.3   Advanced Atomic Force Operation

10.3.1   Displaying Parameters

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

Simple Mode

1. The default **SIMPLE MODE**, intended for novice users and shown in Figure 10.3a, displays the minimum number of parameters needed to make an image.

Figure 10.3a   The **SIMPLE MODE** view of the **Scan Parameter List** in **Contact Mode**

Expanded Mode

1. The **EXPANDED MODE** view, shown in Figure 10.3b, increases the number of displayed parameters enabling expert users to fine tune an image.

Figure 10.3b The **EXPANDED MODE** view of the **Scan Parameter List** in **Contact Mode**

10.3.2 Optimization of Scanning Parameters

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

Data Type

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

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

Collect **Deflection Error** data with low feedback gains to ensure the piezo remains at a constant position relative to the sample. In this case, the tip and cantilever are deflected by the features on the sample surface. The output fluctuations in the cantilever deflection voltage from the top and bottom photodiode segments are recorded as a measure of the variation in the sample surface. **Deflection Error** data is not automatically calibrated in units of distance. You must measure the sensitivity using the procedures discussed in Chapter 15.

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

Gain Settings

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

In general, set the gain settings as follows:

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

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

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

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

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

Scan Size and Scan Rate

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

Deflection Setpoint

The **Deflection Setpoint** parameter defines the desired voltage (and, therefore, the desired deflection or force of the cantilever) for the feedback loop. The setpoint voltage constantly compares to the present vertical deflection signal of the photodiode to calculate the desired change in the piezo position. When the gain values are high, and when the **Data Type** is set to **Height Sensor**, the Z piezo position changes to keep the photodiode output signal close to the **Deflection Setpoint**; therefore, the cantilever deflection remains nearly constant.

Adjust the **Deflection Setpoint** to increase or decrease the cantilever deflection and, therefore, the contact force of the tip on the sample. The **Ramp Mode** menu allows you to adjust the setpoint while viewing a graph of the tip position versus the deflection voltage. Using this procedure, described in detail in Chapter 15, minimizes the contact force of the tip on the sample. This is especially important on soft materials such as biological samples.

Lowpass Filter (LP Deflection and LP Friction)

LP Deflection (**Other > LP Deflection**, see Figure 10.3c) invokes a user programmable low pass filter to remove high frequency noise from the real-time data. The filter operates on the collected data regardless of scan direction. The cutoff frequency, shown in Figure 10.3c, can be set from 2-20kHz. The filter affects feedback in Contact mode. For faster scanning, set the **LP Deflection BW** to higher values.

LP Friction (**Other > LP Friction**, see Figure 10.3c) invokes a user programmable low pass filter to remove high frequency noise from the friction data channel. The LP Friction filter does not affect feedback or scanning speed. The cutoff frequency, shown in Figure 10.3c, can be set from 2-20kHz.

Figure 10.3c LP Deflection and LP Friction parameters

10.4 Force Calibration Mode

The **Ramp Mode** allows you to check the interaction between the cantilever and the sample surface. For detailed information regarding force ramps, see Chapter 15.

Chapter 11   TappingMode AFM

This chapter details procedures for operating the Dimension Icon SPM in TappingMode in air. For information regarding TappingMode in fluids, see Chapter 12. For information regarding loading a TappingMode 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:

- **Principles of TappingMode:** Section 11.1

- **Basic TappingMode AFM Operation:** Section 11.2

  - **Select the Microscope:** Section 11.2.1

  - **Select Mode of Operation:** Section 11.2.2

  - **Head, Cantilever and Sample Preparation:** Section 11.2.3

  - **Align Laser:** Section 11.2.4

  - **Adjust Photodetector:** Section 11.2.5

  - **Locate Tip:** Section 11.2.6

  - **Focus Surface:** Section 11.2.7

  - **Cantilever Tune:** Section 11.2.8

  - **Set Initial Scan Parameters:** Section 11.2.10

  - **Engage:** Section 11.2.11

  - **Optimize Scan Parameters:** Section 11.2.12

  - **Withdraw the Tip:** Section 11.2.13

- **Advanced TappingMode AFM Operation:** Section 11.3

  - **Resonating Techniques:** Section 11.3.1

  - **Cantilever Oscillation:** Section 11.3.2

- **Decreasing the Cantilever Drive Frequency:** Section 11.3.3

- **Optimization of Scanning Parameters:** Section 11.3.5

- **Data Type:** Section 11.3.6

- **Gain Settings:** Section 11.3.7

- **Scan Size, Scan Rate, and Setpoint:** Section 11.3.8

- **Surface Tune:** Section 11.3.9

- **Troubleshooting:** Section 11.4

- **Frequency Response Plot:** Section 11.4.1

- **Engaging the Sample:** Section 11.4.2

- **Cantilever Will Not Tune:** Section 11.4.3

11.1 Principles of TappingMode

Figure 11.1a depicts a cantilever oscillating in free air at its resonant frequency. A piezo stack excites the cantilever substrate vertically, causing the cantilever to move up and down. As the cantilever moves vertically, the reflected laser beam, or "return signal," deflects in a regular pattern over a photodiode array, generating a sinusoidal, electronic signal.

Figure 11.1a   Tapping Cantilever in Free Air
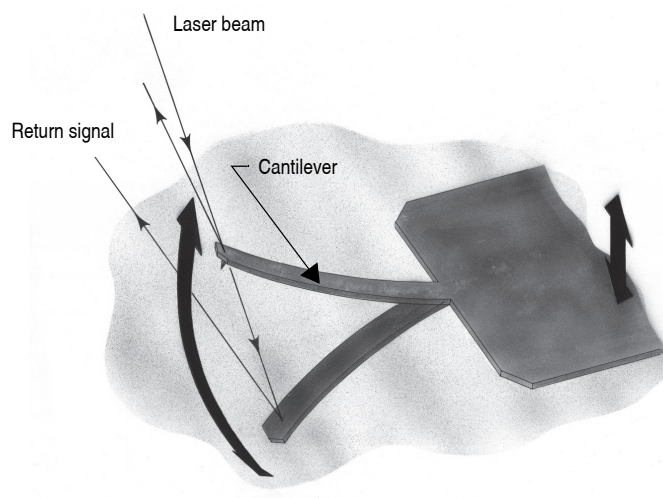
Figure 11.1b represents the same cantilever at the sample surface. Although the piezo stack continues to excite the cantilever substrate with the same energy, the tip deflects in its encounter with the surface. The reflected laser beam reveals information about the vertical height of the sample surface and characteristics of the sample material itself. These material characteristics include elasticity, magnetism, and presence of electrical forces.

Figure 11.1b   Tapping Cantilever on Sample Surface
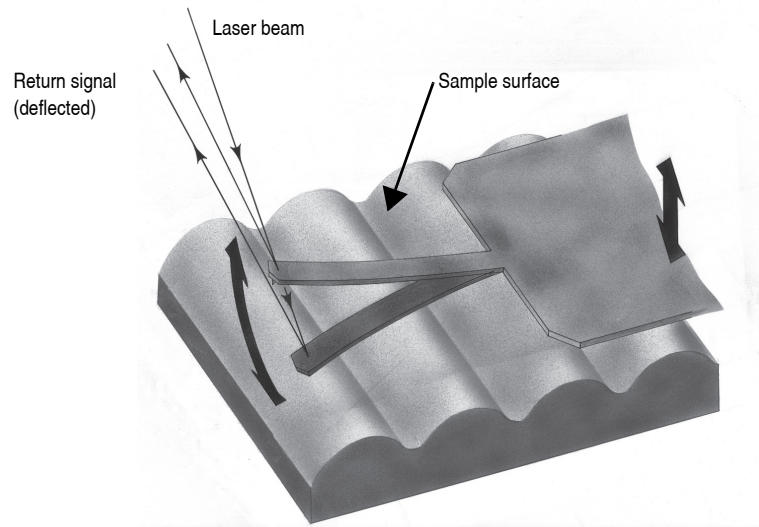
> **Note:**   Deflection of the cantilever and return signal are exaggerated in the figure for illustrative purposes.

11.2   Basic TappingMode AFM Operation

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

11.2.1   Select the Microscope

Follow the procedure described in **Select the Microscope:** Section 7.2.1.

11.2.2 Select Mode of Operation

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

Figure 11.2a   The **Select Experiment** window
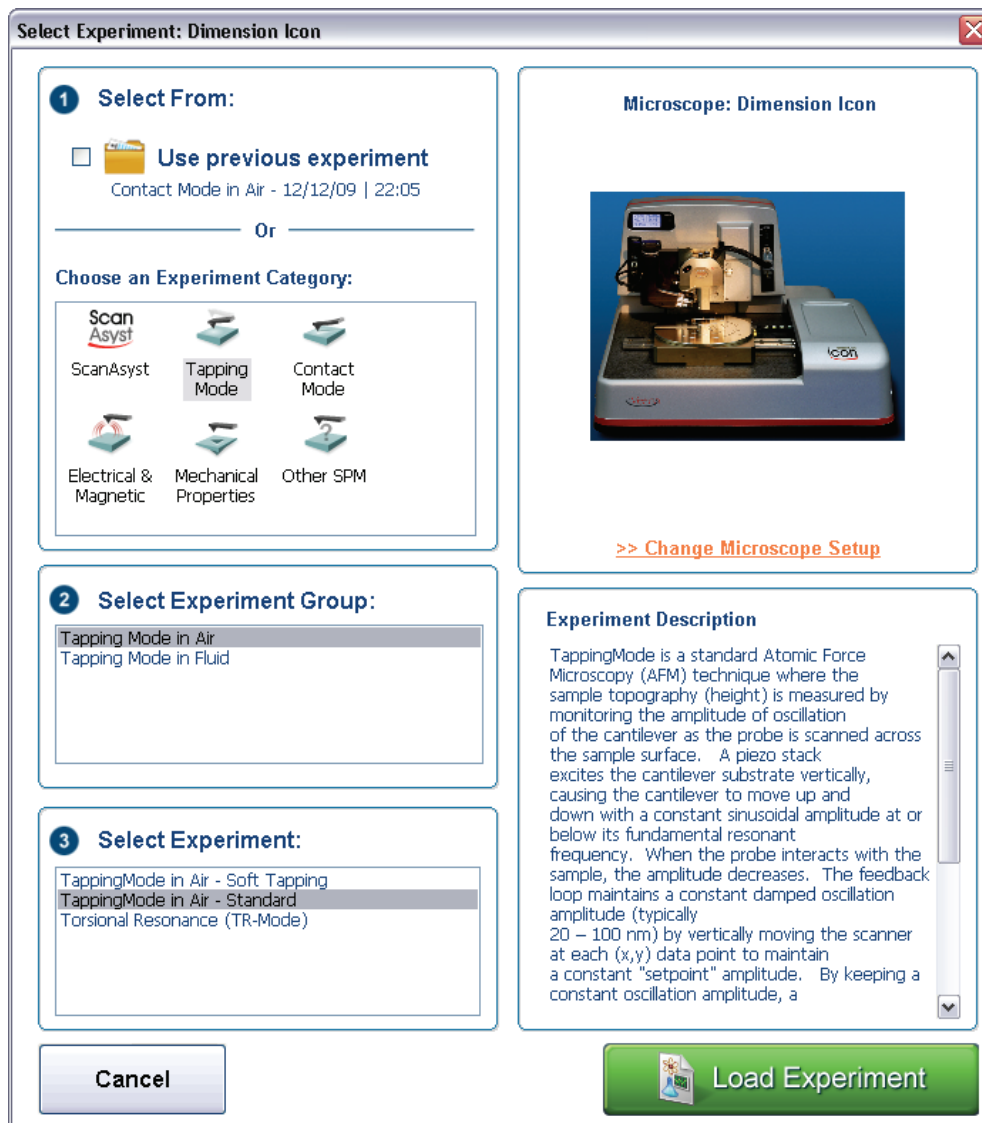
2. Select TAPPING MODE in the **Choose an Experiment Catagory** panel.

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

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

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** (Icon) or the **Scan 8 Channels** (Icon-PI) windows and the **Scan Parameters List** window, shown in Figure 11.2b.

Figure 11.2b Tapping Mode (Simple Mode) configuration
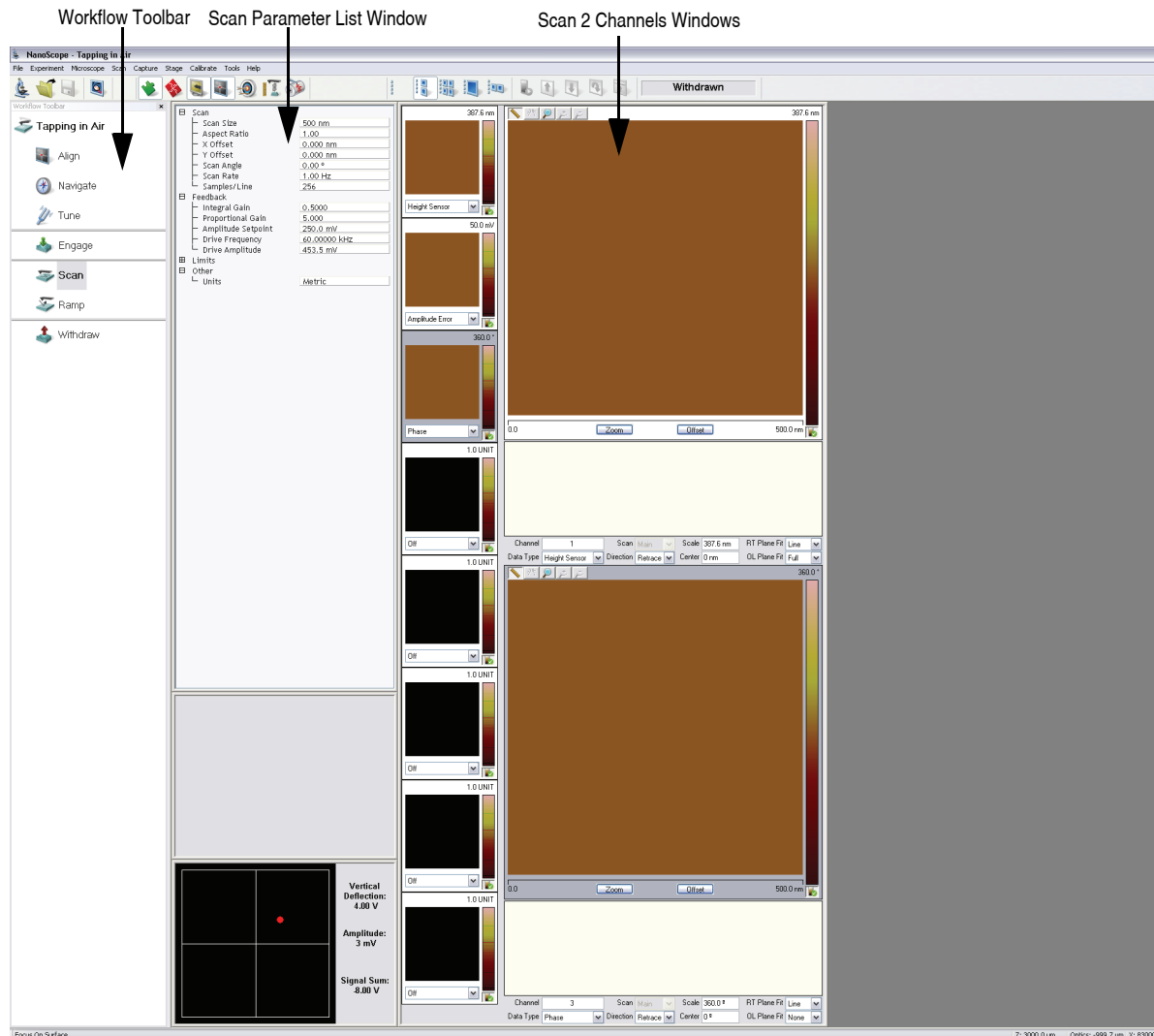
Note: Figure 11.2b shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

11.2.3 Head, Cantilever and Sample Preparation

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

- Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head. See **Basic AFM Operation:** Section 7.2.

11.2.4 Align Laser

1. Align the laser using the laser control knobs (See **Align Laser:** Section 7.2.9. for more detailed instructions).

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 at least **1V**.

11.2.5 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 left side of the Dimension head. See **Adjust Photodetector:** Section 7.2.10.

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

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

11.2.6 Locate Tip

locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **LOCATE TIP** command in the **Align** menu by clicking on **LOCATE TIP**.

4. Zoom out as far as possible using the trackball or **ZOOM OUT** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up

or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

11.2.7 Focus Surface

1. Click the NAVIGATE icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the FOCUS SURFACE icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

CAUTION:	Because the command FOCUS SURFACE moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

11.2.8 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 resonant frequency). In most instances, the resonant 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 **Microscope** > **Cantilever Tune** or click the Tune icon on the Workflow Toolbar.

2. The initial **Cantilever Tune** panel, shown in Figure 11.2c, appears with the **Frequency Sweep** (a plot of cantilever response as a function of applied oscillation frequency).

3. Choose either the automatic or manual tuning method (see **Automatic Tuning:** Section 11.2.9 and **Manual Cantilever Tuning:** Section 11.3.4).

11.2.9 Automatic Tuning

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

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

- Click **Auto Tune**. The computer and controller begin automatic tuning. See Figure 11.2c.

Figure 11.2c Auto Tune Control Panel
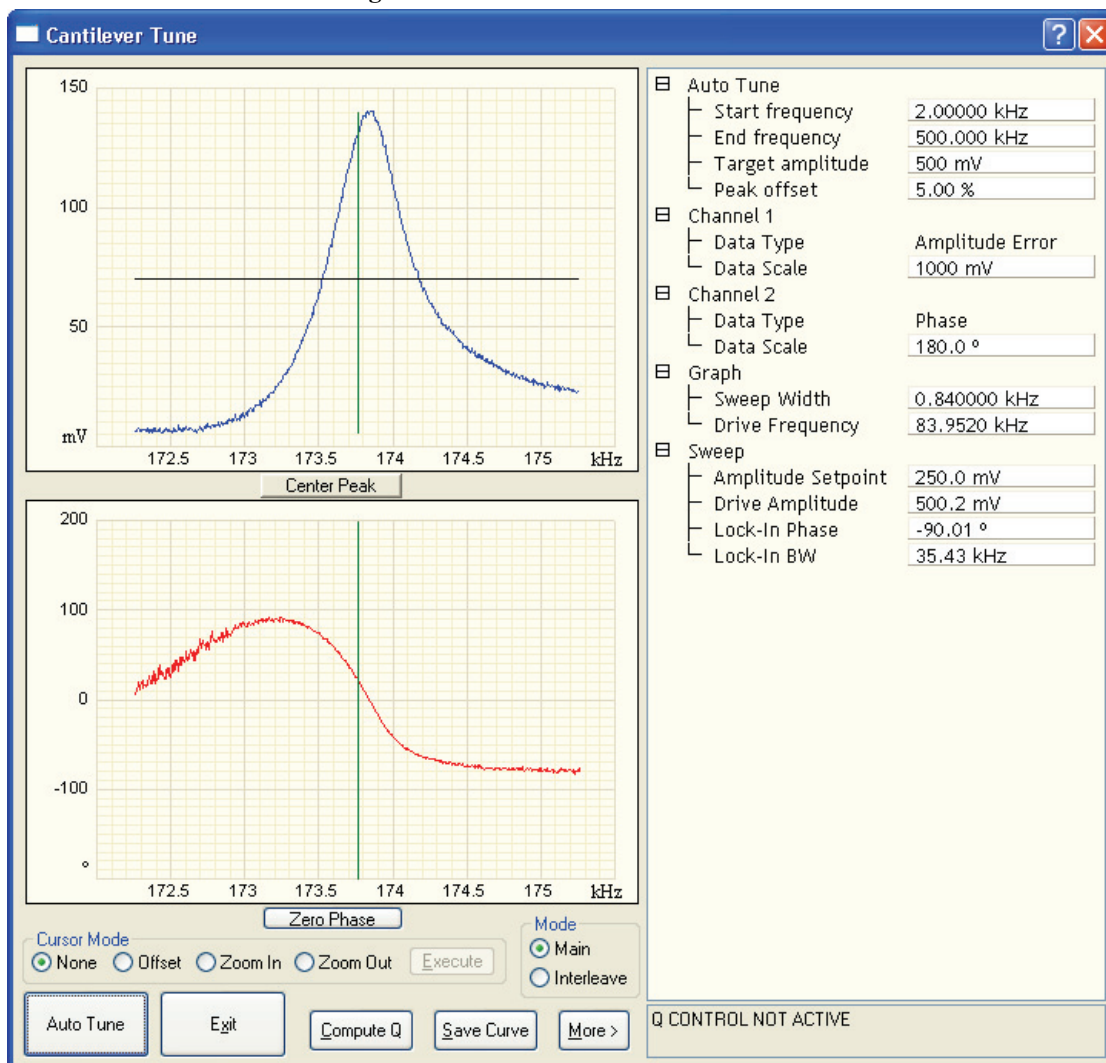
11.2.10 Set Initial Scan Parameters

Scan Panel

In the **Scan Controls** panel, set the following initial scan parameters (see Figure 11.2d).

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

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

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

Figure 11.2d   Suggested Tapping Mode in Air Scan Settings

Feedback Panel

1. Set the **Integral gain** to **0.5** and the **Proportional gain** to **5** (see Figure 11.2d).

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

Other Panel

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

Channels

1. Set the **Channel 1 Data Type** to HEIGHT SENSOR (see Figure 11.2e).

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

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

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

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

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

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

Figure 11.2e   Suggested Channel Settings

11.2.11 Engage

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

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

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

    Note: After the tip engages, adjust the control panel values to provide the desired scan parameters. Refer to Section 11.3.5 for scan parameter optimization.

4. After engaging, it may be necessary to recheck the Cantilever Tune parameters. Select **MICROSCOPE > CANTILEVER TUNE** or click the **TUNE** icon in the **Workflow Toolbar**. In the **Tip offset** box, shown in Figure 11.2f, enter **200nm** and click **OK**. Adjust the **Drive frequency** if necessary.

Figure 11.2f Start Sweep On Surface
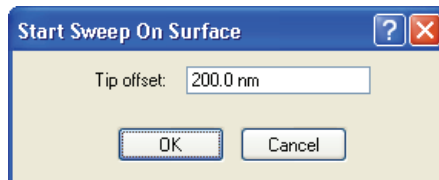
11.2.12 Optimize Scan Parameters

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

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

2. Observe the agreement between the Trace and Retrace lines. Adjust the **Scan Rate, Gains,** and **Amplitude Setpoint** to bring these two lines in to coincidence. A more detailed discussion of these parameters can be found in Section 11.3.

11.2.13 Withdraw the Tip

1. Select **RealTime** > **Withdraw** or click the **WITHDRAW** icon in the **Workflow Toolbar**. The SPM stops scanning, and ascends to the sample clearance height defined in the **SPM Parameters** menu.

2. Replace or move the sample.

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

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

11.3   Advanced TappingMode AFM Operation

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

11.3.1 Resonating Techniques

Without a thorough understanding of principles associated with cantilever resonating techniques, you may generate distorted data. Understanding the **Cantilever Tune** process and the effects of real-time scan parameters is critical for effective operation of the microscope. It is also important to understand similarities and differences between **Ramp Mode** in **Contact AFM** and **Ramp Mode** in **TappingMode** (see Chapter 15).

11.3.2 Cantilever Oscillation

The response of the cantilever to inputs plays an important role in the operation of the Dimension Icon SPM while in TappingMode. There is an important trade-off between the response time of the cantilever and the force applied to the sample. The cantilever does not respond instantly to perturbations in oscillation amplitude. The cantilever drive system pumps energy gradually into the cantilever oscillation. Figure 11.3a illustrates a typical response curve of the cantilever amplitude as a function of time. To demonstrate the conflicting requirements, the performance of the system is analyzed at two operating points.

Figure 11.3a   Cantilever Response Curve
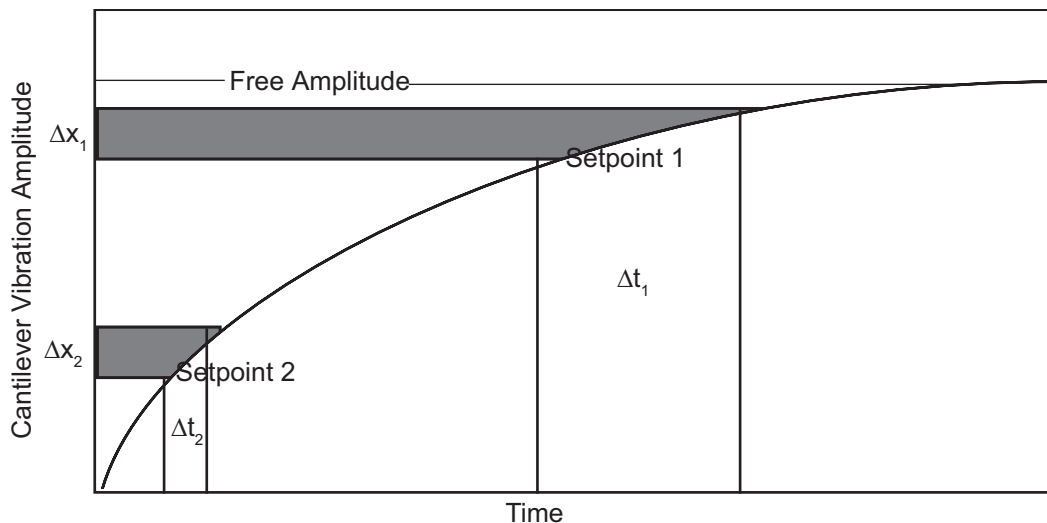
At Setpoint 1 the operating point is only slightly lower than the free oscillation amplitude. This has the advantage of dissipating very little energy to the sample surface. The disadvantage is that the system takes longer to recover from a given perturbation in the amplitude. Consider the situation where the tip travels off a step with a height of Δx. At Setpoint 1 it takes longer for the amplitude of

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

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

Inadequate response time typically occurs when the tip encounters a low point in the sample. The amplitude of the cantilever oscillation decreases very quickly when taller portions of the sample are encountered. As a result, the system response is markedly different depending on whether the tip is climbing or descending a feature in the sample. For this reason, viewing the **Scope** trace (below the scan) is very useful when setting scan parameters. As the tip descends, features are evaluated by comparing the **Trace** and **Retrace**. Figure 11.3b illustrates the effects of poorly selected scan parameters on a calibration standard that includes a series of sharp-walled pits. Regardless of the scan direction, the tip does not track the wall of the pit when the tip encounters a pit. However, it does track the surface closely when moving out of the pit.

Figure 11.3b   Scope Trace with High Scan Rate
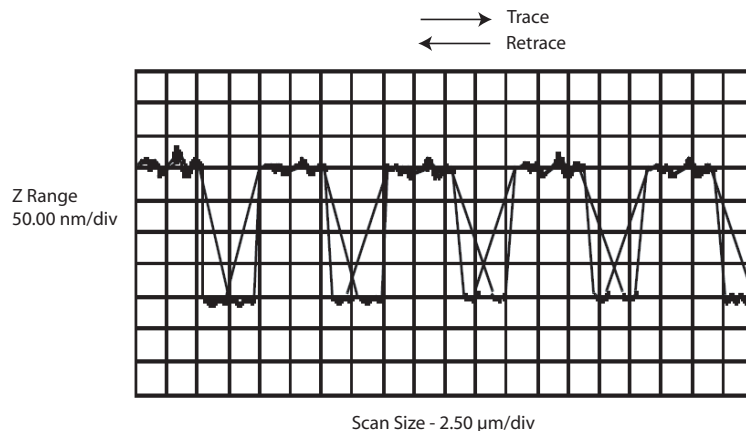
Figure 11.3c depicts the same sample with a slight increase in the **Integral Gain** and a twofold decrease in the **Scan Rate**. The tip now tracks the surface when it descends into the pit as well as when it exits. The **Trace** and **Retrace** lines now coincide closely.

Figure 11.3c Scope Trace with Correct Scan Rate
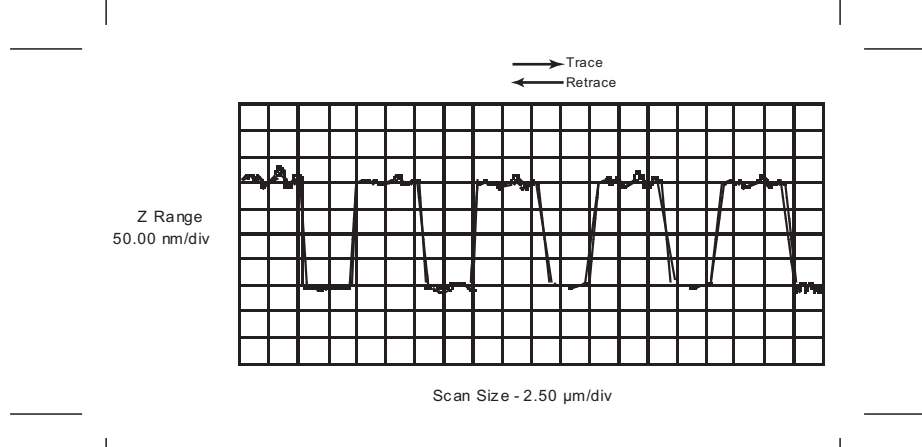
Note: The Ease of Use Interface, shipped with NanoScope V controllers, automatically sets gains, scan rates... to optimize image quality. Details may be found in the *NanoScope V Controller Manual*.

11.3.3 Decreasing the Cantilever Drive Frequency

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

The microscope produces better data in TappingMode when the **Drive Frequency** is set lower than the resonant peak of the cantilever. The **Drive Frequency** is set such that it coincides with a 1-10 percent decrease in the oscillation amplitude by setting **Peak Offset** in the **Auto Tune** controls to the desired percent. Figure 11.3d shows a suggested operating region. This is a suggestion based on our observations; users are encouraged to experiment with the microscope and decide what produces the best results.

Figure 11.3d Suggested Range of Drive Frequencies
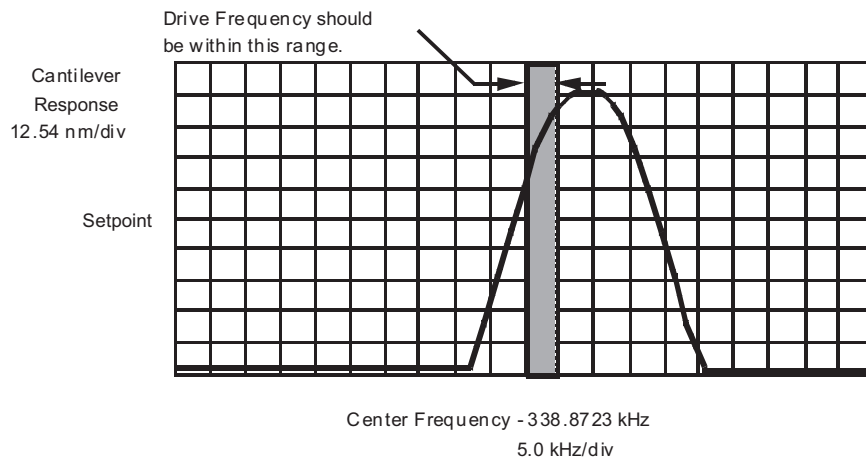
11.3.4 Manual Cantilever Tuning

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

2. Set the **Drive Amplitude** to **200 mV**.

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

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

5. Zero the **Setpoint**.

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

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

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

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

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

> **Note:** The system works well in TappingMode if the center frequency is at, or below, the peak in the resonance plot. The center frequency can decrease to the point where the oscillation amplitude reaches 90 percent of the maximum value. Operate at a frequency lower than the resonant frequency to avoid shifting the resonant frequency upon approach of the tip to the surface (see Figure 11.3f).

Figure 11.3e Cantilever Tune Control Panels for Main Controls
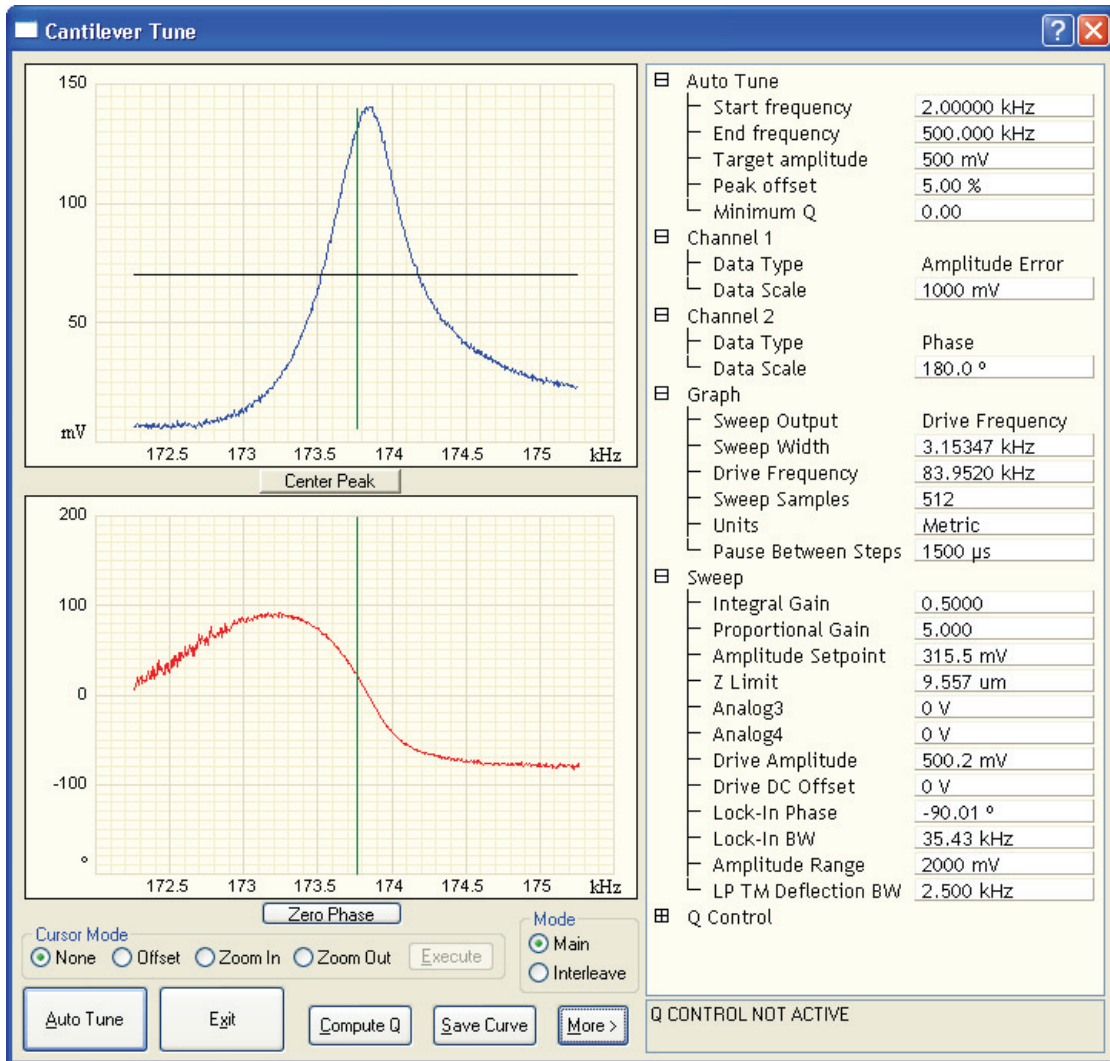
10. Specify the **RMS amplitude** after tuning the cantilever to its resonant frequency. The desired operating amplitude depends on the sample and other scanning conditions.

Figure 11.3f Cantilever Tune Frequency Sweep

Frequency Sweep
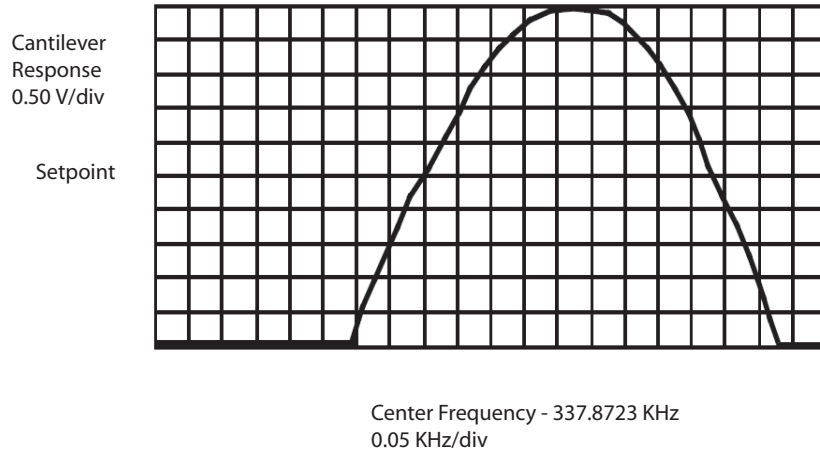
Cantilever
Response
0.50 V/div

Setpoint

Center Frequency - 337.8723 KHz
0.05 KHz/div

11. Click **AUTO TUNE**. The parameters set in the **Cantilever Tune** control panel appear in the **Realtime** control panel.

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

11.3.5 Optimization of Scanning Parameters

The user is encouraged to review Section 10.3.2 which discusses parameter optimization for Contact AFM.

11.3.6 Data Type

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

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

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

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

11.3.7 Gain Settings

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

11.3.8 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.

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

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

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

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

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

11.3.9 Surface Tune

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

A warning, shown in Figure 11.2f, appears indicating that cantilever tuning while engaged can result in damage to the tip or sample. This warning prompts you to enter a **Tip offset**. This is the distance above the sample that the tip will be raised before displaying the tuning spectrum. Use a minimum **Tip offset** of **100-200nm**.

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

11.4   Troubleshooting

11.4.1 Frequency Response Plot

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

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

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

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

11.4.2 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.

11.4.3 Cantilever Will Not Tune

If the Cantilever Tune fails, check the following:

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

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

Chapter 12   Fluid Imaging

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. Contact Veeco for more information.

This chapter details basic operation of the Dimension Icon SPM in fluid, using both Contact Mode and TappingMode. Methods of preparing the sample for fluid operation are also included. Specifically, this chapter addresses the following:

- **Select Mode of Operation:** Section 12.5.3

- **Load the Probe:** Section 12.5.4

- **Install the Fluid Probe Holder:** Section 12.5.5

- **Install the Protective Skirt:** Section 12.5.6

- **Align Laser:** Section 12.5.7

- **"False" Reflections:** Section 12.5.8

- **Load Sample:** Section 12.5.9

- **Lower Probe into Fluid:** Section 12.5.10

- **Readjust Laser Alignment:** Section 12.5.11

- **Adjust Photodetector:** Section 12.5.12

- **Locate Tip:** Section 12.5.13

- **Focus Surface:** Section 12.5.14

- **Cantilever Tune (TappingMode Only):** Section 12.5.15

- **Set Initial Scan Parameters:** Section 12.5.16

- **Engage:** Section 12.5.17

- **Adjust Scan Parameters:** Section 12.5.18

- **Clean Cell and Protective Skirt:** Section 12.5.20

- **Troubleshooting:** Section 12.6

  - **Cantilever Tune Plot Looks Poor:** Section 12.6.1

  - **Laser Sum Signal Absent or Weak:** Section 12.6.2

  - **Poor Image Quality:** Section 12.6.3

  - **Unable to Locate Particulate Samples:** Section 12.6.4

12.1 Basic Principles

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 similar to that for observation 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 Icon AFM in air. If you are not familiar with air operation of the AFM in Contact Mode, please follow the procedures outlined in Chapter 10 before attempting to operate the AFM under fluid. If you are not familiar with air operation of TappingMode, refer to Chapter 11.

12.1.1 Acknowledgments

Veeco appreciates the following individuals' assistance with this chapter: Monika Fritz, Manfred Radmacher, Magdalena Benzanilla, Helen G. Hansma, Jan H. Ho, Craig B. Prater.

12.2 Fluid Operation Hardware

12.2.1 Fluid Probe Holder

The Dimension Icon can be equipped with an optional probe holder, the Dimension Direct Drive Fluid Cantilever Holder (Veeco p/n DTFML-DD) that permits operation in fluids (see Figure 12.2a). The fluid probe holder interchanges with the standard probe holder quickly and easily.

The fluid probe holder consists of a small Kel-F assembly with a wire clip for holding an AFM cantilever substrate and a glass window for the AFM laser. The glass provides a flat, beveled interface so that the AFM laser beam may pass into the fluid without being distorted by an unstable fluid surface. The DTFML-DD includes a small piezo to directly drive the cantilever for improved ease of use in TappingMode. The four sockets located on the top of the fluid probe holder are used to attach the probe holder to the four pins at the end of the scanner tube. More information about the DTFML-DD may be found in Veeco Support Note 410, *Direct Drive Fluid Cantilever Holder*.

Use the fluid probe 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). A method for mounting samples for fluid operation is illustrated in Figure 12.3a in Section 12.3.

Figure 12.2a Direct Drive Fluid Cantilever Holder - bottom view

Spring loaded
probe clip

Glass Window

AFM cantilever
mounting groove
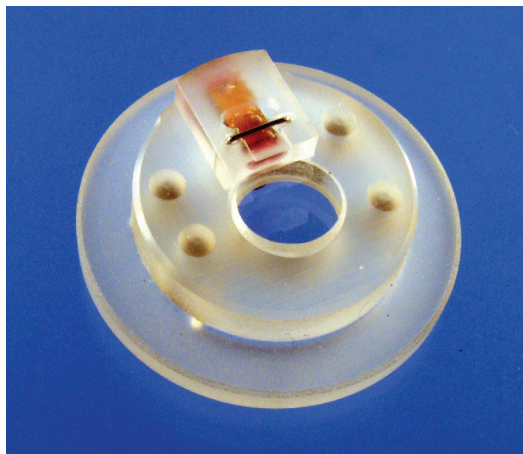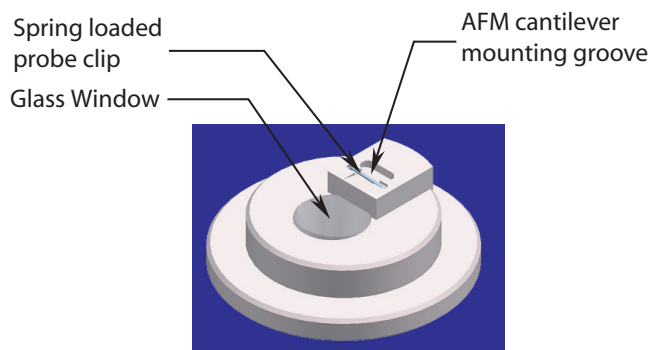
Figure 12.2b Direct Drive Fluid Cantilever Holder - top view

Mounting Sockets-4

Clip release button
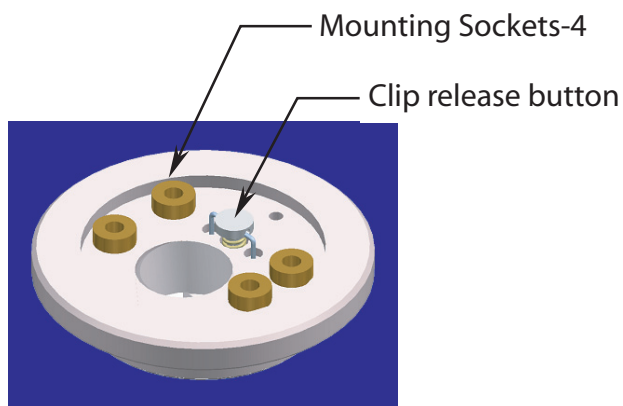
12.2.2   Probe Suggestions

Soft cantilevers, particularly oxide sharpened tips, produce the best results for biological applications. These cantilevers are typically 100μm long, narrow-legged with oxide sharpened silicon-nitride tips. Models DNP-S is recommended. Users should experiment to find which cantilevers work best for their sample. The application note "*Choosing AFM Probes for Biological Applications*" discusses the appropriate probes for different fluid imaging applications. Contact Veeco for a copy.

> **Note:**   For additional information on selecting a cantilever, please refer to the Veeco website (www.veeco.com).

Removing Organic Contamination

⚠️	**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 Icon system, especially onto the SPM head.

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.

> **Note:** Washing probes in 1-5% SDS (sodium dodecyl sulfate) is also effective.

12.2.3   Rubber Protective Skirt

A rubber protective skirt (see Figure 12.2c) is also provided with the fluid probe 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. Skirts wear over time and may need to be replaced.

Figure 12.2c   Rubber Protective Skirt

12.3   Sample Mounting

12.3.1   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 Veeco.

	CAUTION:	Do not attempt to operate the standard air probe holder in a fluid environment. The standard probe 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.
	VORSICHT:	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.

12.3.2   Larger Samples

For larger samples, it may be desirable to image a small region under a drop of fluid (see Figure 12.3a).

1. Put the drop of fluid onto the sample.

2. Lower the AFM fluid probe holder into the drop.

Figure 12.3a   Imaging a Sample Covered by a Drop of Fluid

Fluid Probe Holder

Fluid Meniscus
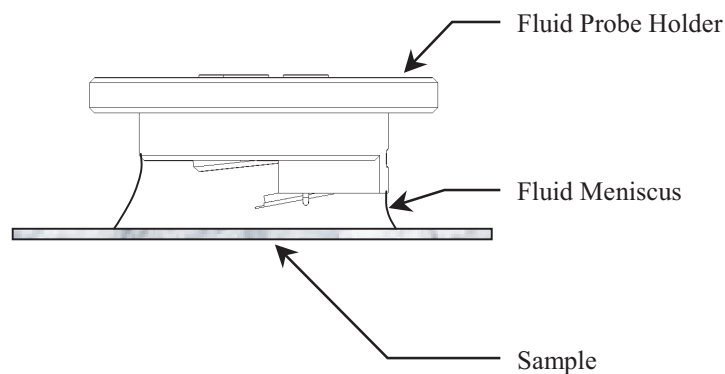
Sample

12.3.3   Smaller Samples

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

1. Place the sample in a plastic petri dish.

    Note: If you wish to construct a custom sample holder, please contact 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.

12.4 Precautions

12.4.1 Spillage Precautions

Throughout all procedures outlined in the following sections, you will work with fluids on and around the Dimension Icon 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 3mm 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.
	VORSICHT:	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.

	CAUTION:	Do not turn the head upside down when the fluid cell is wet. Any moisture present on the end of the scanner must be dried **immediately** to prevent shorting the piezo.
	ATTENTION:	Ne tournez pas la tête du capteur a l'envers quand le réservoir à liquide est humide. Veuillez sécher l'extrémité du numériseur immédiatement en cas d'humidité pour eviter un court-circuit dans le piezo.
	VORSICHT:	Der Scannerkopf darf nicht umgedreht werden, solange die Flüssigkeitszelle nass ist. Sollte sich Feuchtigkeit jeglicher Art am Scannerkopf befinden, muss diese umgehend beseitigt werden, um Kurzschlüsse am Piezo zu verhindern.

	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.
	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.
	VORSICHT:	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.

<table>
<tr><td rowspan="3"></td><td>**CAUTION:**</td><td>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.</td></tr>
<tr><td>**ATTENTION:**</td><td>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.</td></tr>
<tr><td>**VORSICHT:**</td><td>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.</td></tr>
</table>

12.5   Operating Principles

12.5.1   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 probe 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 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.

12.5.2 Select the Microscope

Follow the procedure described in **Select the Microscope:** Section 7.2.1.

12.5.3 Select Mode of Operation

1. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 12.5a.

Figure 12.5a The Contact Mode in Fluid **Select Experiment** window
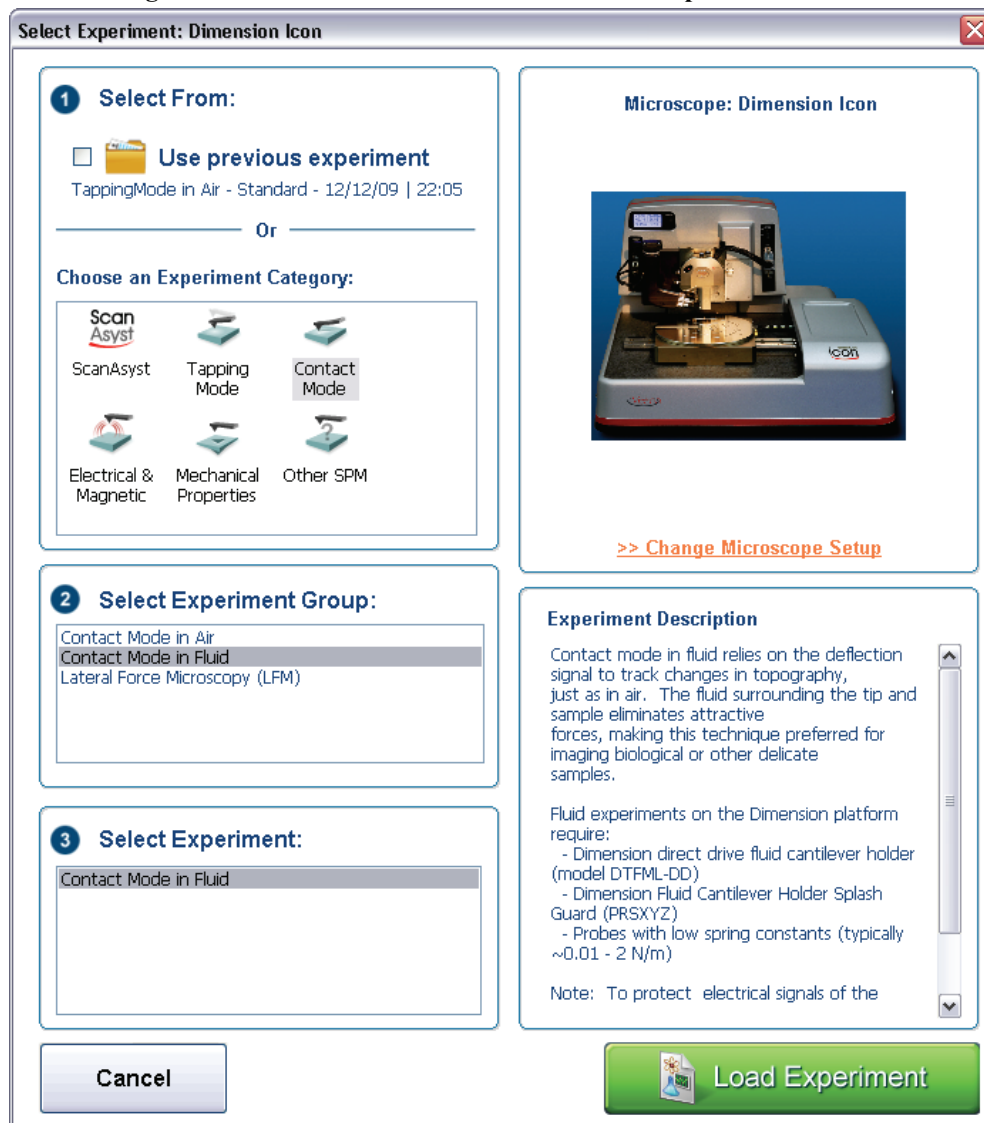
2. Select CONTACT MODE in the **Choose an Experiment Catagory** panel.

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

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

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** (Icon) or the **Scan 8 Channels** (Icon-PI) windows and the **Scan Parameters List** window, shown in Figure 12.5b.

Figure 12.5b   Contact Mode in Fluid (Simple) configuration

Workflow Toolbar   Scan Parameter List Window   Scan 2 Channels Windows
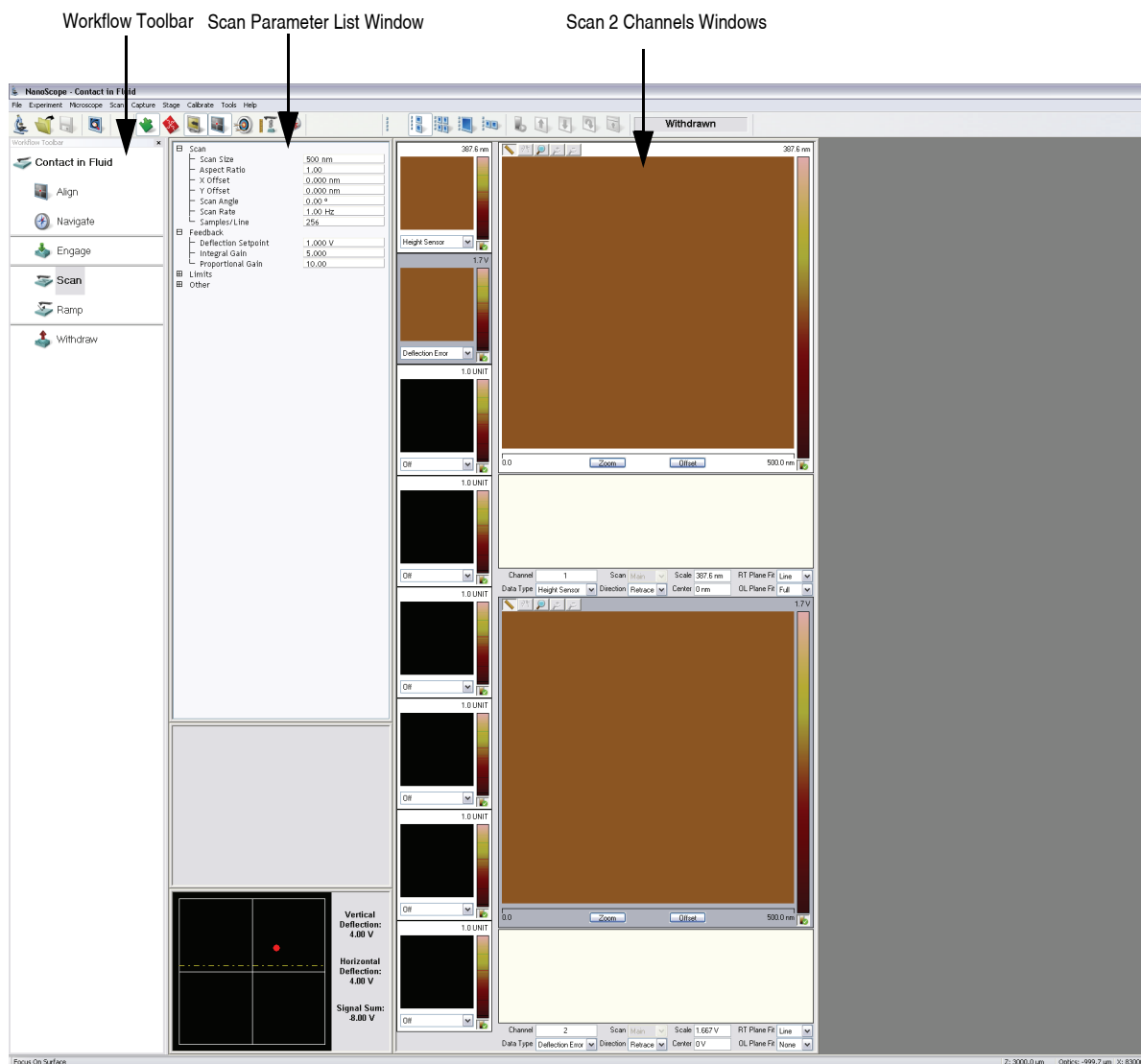
Note: Figure 12.5b shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

12.5.4   Load the Probe

The probe is held in a small pocket on the bottom side of the probe holder by a gold-plated stainless steel wire. The cantilever installation fixture has three docking stations where different probe 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 probe holder.

1. To mount a probe 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 probe 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 probe in the pocket.

3. With the gold wire raised, use tweezers to slide a probe under the wire and into the pocket.

4. Check that the cantilever substrate is set squarely against one side of the pocket and flush against the back.

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

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

    Note: When removing the probe holder from the docking station, the leaf spring should pull the gold wire tight against the cantilever substrate.

6. Check that the probe is held firmly by the wire.

12.5.5   Install the Fluid Probe Holder

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

    Note: The cantilever probe 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: **Navigate** and raise the probe with the **Z motor** control, or the trackball, **MICROSCOPE > STEP MOTOR > TIP UP** 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 probe points to the left of the microscope.

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

12.5.6 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 probe holder and onto the shoulder on the SPM tube. Ensure that the seal is tight on both the probe holder and the SPM tube. See Figure 12.5c below for an illustration of the SPM head and the fluid probe holder with the protective skirt installed.

Figure 12.5c Probe Holder Installed With Protective Skirt
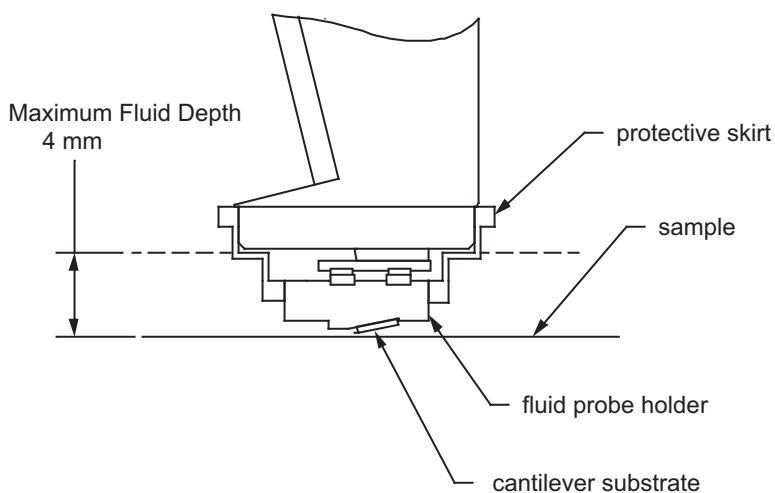
12.5.7 Align Laser

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

12.5.8　"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 12.5d). 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.

Figure 12.5d　False Reflections from Fluid Probe Holder
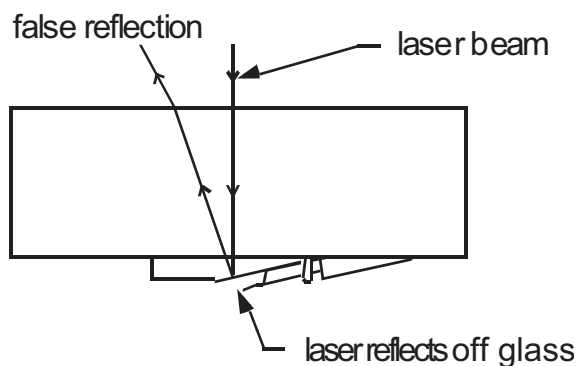
Once the fluid probe holder is plugged into the bottom of the AFM scanner, note the faint laser reflection visible on the glass in the probe holder (in the laser viewing window).

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

12.5.9　Load Sample

Prepare the Sample for Imaging

Set the sample on the microscope stage. For TappingMode 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.

Remount 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.
	VORSICHT:	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.

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 **MICROSCOPE > 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 Section 12.5.11 for more information regarding aiming the laser through a fluid layer.

At this point, the fluid cell should be prepared for TappingMode 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.

Verify that the Microscope is Dry

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

12.5.10 Lower Probe into Fluid

Lower the head, allowing the fluid probe holder to enter the liquid. The fluid should form a meniscus around the fluid probe holder (see Figure 12.3a).

> **Note:** The laser sum signal must be greater than 0.5V 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.

> **Note:** To prevent air bubbles, you may first place a drop of fluid on the probe.

12.5.11 Readjust Laser Alignment

Lowering the probe holder into fluid causes the laser spot to move towards the fixed end of the cantilever by roughly 25µm. 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 probe holder into fluid. In this case, do the following:

3. Remove the SPM head from the dovetail.

4. Lightly rinse the probe holder with liquid.

5. Replace the SPM head.

12.5.12 Adjust Photodetector

Adjust Detector Offsets

Turn the detector mirror adjustment screws to center the laser spot on the laser detector as described in Chapter 7.

Set several parameters:

- For **Contact Mode**: **Vertical Deflection** signal = roughly **-1V**, and **Setpoint** = **0V**. (These are typical starting parameters).

- For **TappingMode**: **Vertical Deflection** signal = **0V**.

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.

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

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.

12.5.13 Locate Tip

To locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window if you are not using a Direct Drive Fluid Cantilever Holder.

> **CAUTION:** Do not use the Alignment Station when using the Direct Drive Fluid Cantilever Holder (see **Fluid Probe Holder:** Section 12.2.1) as it will hit the Alignment Station and knock it off its magnetically secured base. If you have selected a fluid experiment, the Alignment station button will be grayed out.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

12.5.14 Focus Surface

1. Click the **NAVIGATE** icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

> **CAUTION:** Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

5. Add fluid

6. Readjust laser alignment

7. Adjust laser alignment in photodetector

Fluid changes the optical path length as the angle of refraction changes, therefore, you must offset the focus position in **Focus Surface** by 1.3 times 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 -1000µm, you must move the Z motor position down until it is at -1300µm.

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)

<div align="center">**OR**</div>

    d.   Focus surface (beyond surface by 1.3x)

    e.   Realign laser

    f.   Realign laser in photodetector

	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 probe 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.
	VORSICHT:	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.

12.5.15 Cantilever Tune (TappingMode Only)

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.

Manual **Cantilever Tune** is the counterpart to the step used in standard (air) TappingMode to find the resonant frequency of the cantilever. In liquid, however, the cantilever resonance is highly

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.

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

A typical **Cantilever Tune** screen is shown in Figure 12.5e. 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. Figure 12.5e shows a typical operating frequency that produces good fluid tapping images.

Figure 12.5e Typical Cantilever Tune Curve in Fluid
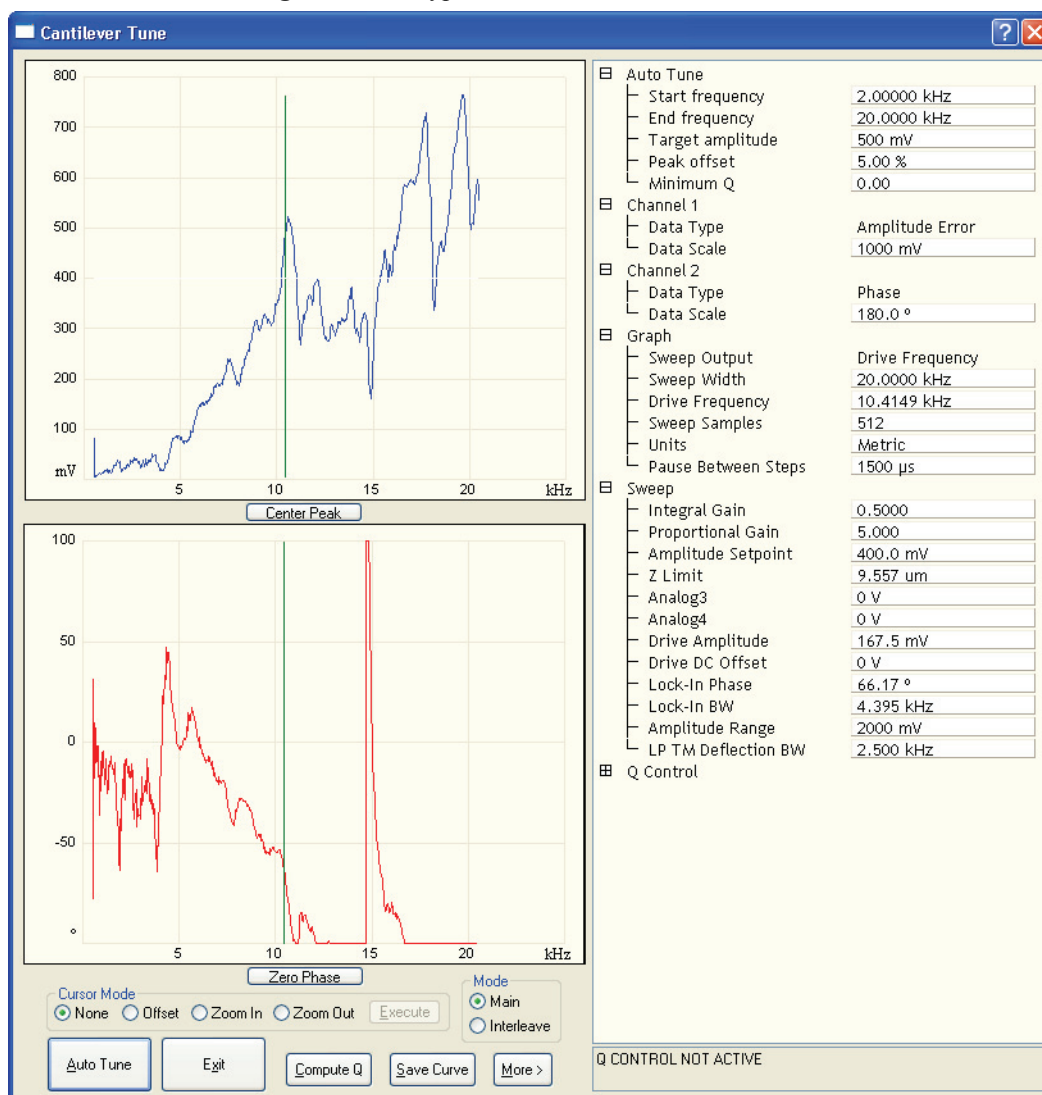
1. Set **Z Modulation** to **DISABLED**.

    Note: When **Z Modulation** is **DISABLED**, the piezo embedded in the fluid cell is
             used to oscillate the cantilever (direct drive). If **Z Modulation** is **ENABLED,**
             the scanner z-tube is used to oscillate the cantilever. Use this setting if you
             do not have a Direct Drive Fluid Cantilever Holder.

2. Select **MICROSCOPE > CANTILEVER TUNE** or click the **TUNE** icon on the NanoScope toolbar
    The initial **Cantilever Tune** panel appears with the **Frequency Sweep** (a plot of cantilever
    response as a function of applied oscillation frequency).

3. In the **Cantilever Tune** panel, set the **Drive frequency** parameter to **10kHz** and the **Sweep
    width** parameter to **20kHz**.

4. Set the **Drive amplitude** to **60mV**.

5. Zero the **Amplitude setpoint**.

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

7. Center the peak on the frequency sweep plot using the **ZOOM IN** and **OFFSET** commands after identifying the maximum amplitude peak with the lowest frequency in the frequency response plot. This peak is usually in the 8-12kHz range.

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

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

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

   Note: The system works well in TappingMode if the center frequency is at, or below, the peak in the resonance plot. The center frequency can decrease to the point where the oscillation amplitude reaches 90 percent of the maximum value. Operate at a frequency lower than the resonant frequency to avoid shifting the resonant frequency upon approach of the tip to the surface (see Figure 12.5f).

10. Adjust the **Drive amplitude** until obtaining an **RMS Amplitude** of **100mV - 200mV**. This value has produced good results for protein samples like RNA polymerase and lysozyme.

   Note: In general, larger RMS amplitudes (approximately 560mV) work better for taller samples such as cells.

Figure 12.5f   Cantilever Tune Frequency Sweep
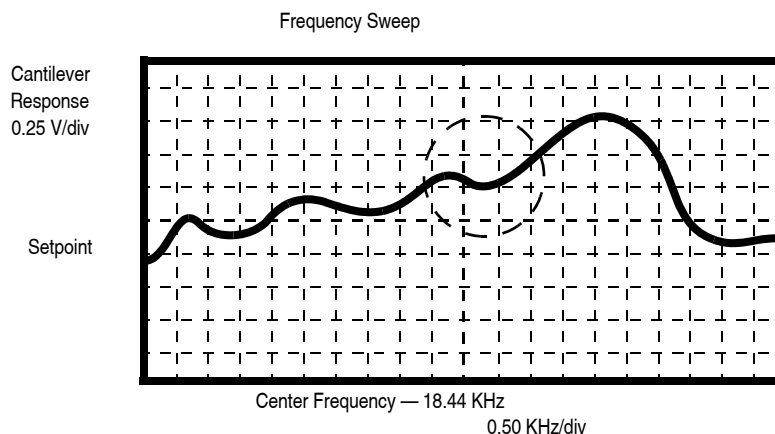
11. Click **AUTO TUNE**. The parameters set in the **Cantilever Tune** control panel appear in the **Realtime** control panel.

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

12.5.16   Set Initial Scan Parameters

Scan Panel

In the **Scan** panel, set the following initial scan parameters (see Figure 12.5g)

1. Set the **Scan Rate** to **0.5Hz**.

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

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

Figure 12.5g   Suggested Settings for Contact Mode in Fluid

Feedback Panel

1. For Contact Mode, in the **Feedback** panel set the **Deflection Setpoint** to **0V**. It is not necessary to enter a value for Amplitude Setpoint for TappingMode because this value is automatically determined during the engage process.

2. For TappingMode, set the **Integral Gain** to **0.5** and the **Proportional Gain** to **5**. For Contact Mode, set the **Integral Gain** to **2** and the **Proportional Gain** to **5**.

Channels 1 and 2

1. Set the **Channel 1 Data Type** to **HEIGHT** (see Figure 12.5h).

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

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

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

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

5. Set the **Channel 3** through **Channel 8 Data Type** to **OFF**.

Figure 12.5h Suggested Channel Settings

Channel		1	Scan	Main	Scale	387.6 nm	RT Plane Fit	Line
Data Type		Height Sensor	Direction	Retrace	Center	0 nm	OL Plane Fit	Full
Channel		2	Scan	Main	Scale	100.0 nm	RT Plane Fit	None
Data Type		Deflection Error	Direction	Retrace	Center	0 nm	OL Plane Fit	None

12.5.17   Engage

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

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

12.5.18   Adjust Scan Parameters

Once engaged, the scan parameters should be adjusted to obtain the best image.

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

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

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

   • Scan rates tend to be slower in fluid.

Adjusting Setpoint

Contact Mode

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 -220V).

2. Increase the **Setpoint** slightly until the cantilever begins to touch the surface again and an image appears.

   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.

TappingMode

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:

- **Optimize the Image Quality**: The **Setpoint** can also be adjusted by simply monitoring the image quality.

  - Select a **Scan size** of **500nm**.

  - Increase the **Setpoint** in small increments until the cantilever pulls off the surface and the **Z center voltage** is at -**220V**.

  - 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.

When operating in TappingMode, it may be necessary to check the cantilever tuning after the system is engaged. To check the cantilever tuning:

1. Select **Microscope** > **Cantilever Tune**.

2. Set the **Tip Offset** to **200nm** and click **OK**.

3. Once in the cantilever tuning window, adjust drive frequency and drive amplitude if necessary.

12.5.19 Withdraw the Tip

1. Select **RealTime** > **Withdraw** or click the **Withdraw** icon in the **Workflow Toolbar**. The SPM stops scanning, and ascends to the sample clearance height defined in the **SPM Parameters** menu.

2. Replace or move the sample.

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

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

12.5.20 Clean Cell and Protective Skirt

When you are finished scanning, remove and clean the fluid probe holder and protective skirt, following the procedure described in Section 12.5.1.

12.6   Troubleshooting

12.6.1   Cantilever Tune Plot Looks Poor

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. Ensure there is good physical coupling between the probe and the fluid cell, and that the laser is positioned correctly at the free end of the cantilever.

12.6.2   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.

12.6.3   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 Icon in Contact Mode by switching the **Experiment Category** parameter in the **Select Experiment** window 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 **TappingMode** and image the sample.

12.6.4   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 cantilever. You should find more individual samples there.

Chapter 13   Scanning Tunneling Microscopy (STM)

This chapter includes the following sections:

13.1   Introduction

STM relies on tunneling current between the probe and the sample to sense the topography of the sample. The STM probe, a sharp metal tip (in the best case, atomically sharp), is positioned a few atomic diameters above a conducting sample which is electrically biased with respect to the tip. At a distance under 1 nanometer ($0.001\mu m$), a tunneling current will flow from sample to tip. In operation, the bias voltages typically range from 10 to 1000mV while the tunneling currents typically vary from 0.2 to 10nA. The tunneling current changes exponentially with the tip-sample separation, typically decreasing by a factor of 2 as the separation is increased by 0.2nm. The exponential relationship between the tip separation and the tunneling current makes the tunneling current an excellent parameter for sensing the tip-to-sample separation. In essence, a reproduction of the sample surface is produced by scanning the tip over the sample surface and sensing the tunneling current.

13.1.1   Overview of STM

The STM option is generally employed under the following conditions:

- When atomic/molecular resolution is desired.

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

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

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

STM relies on a precise scanning technique to produce very high-resolution, three-dimensional images of sample surfaces.   The STM scans the tip over the sample surface in a raster pattern while sensing and outputting the tunneling current to the NanoScope control station. The digital signal processor (DSP) in the workstation controls the Z position of the piezo based on the tunneling current error signal. The STM operates in both the "constant height" and "constant current" data modes, depending on a parameter selection in the **Feedback** panel. The DSP always adjusts the height of the tip based on the tunneling current error signal, but if the feedback gains are low, the piezo remains at a nearly "constant height" and tunneling current data is collected. With the gains high, the piezo height changes to keep the tunneling current nearly constant, and the change in piezo height is collected by the system. The exponential relationship between the tip separation and the tunneling current allows the tip height to be controlled very well. For example, if the tunneling current stays within 20 percent of the setpoint value (the current to be maintained by the feedback system), the variation in the tip-sample separation is less than 0.02nm.

The STM tip is held using a dedicated STM tip holder which plugs into the end of the Dimension head's tube (see Figure 13.1a).

Figure 13.1a Dimension STM Tip Holder and Head Connection

SIDE VIEW

(tip installed)

Op Amp

STM Metal Tip

Tip Holding Tube

BOTTOM VIEW

Electrical Mounting
Sockets (4)
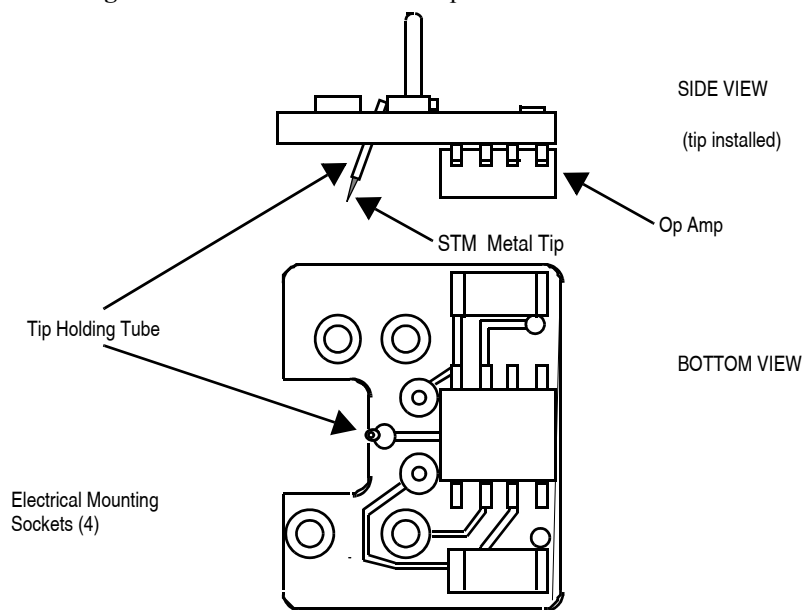
13.2   Basic STM Operation

This section explains how to use the NanoScope to image the 10µm, silicon standard supplied with the system.

13.2.1 Select the Microscope

Follow the **Select the Microscope:** Section 7.2.1 procedure described in Section 7.2.1.

13.2.2 Select Mode of Operation

1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 13.2a.

Figure 13.2a   The **Select Experiment** window
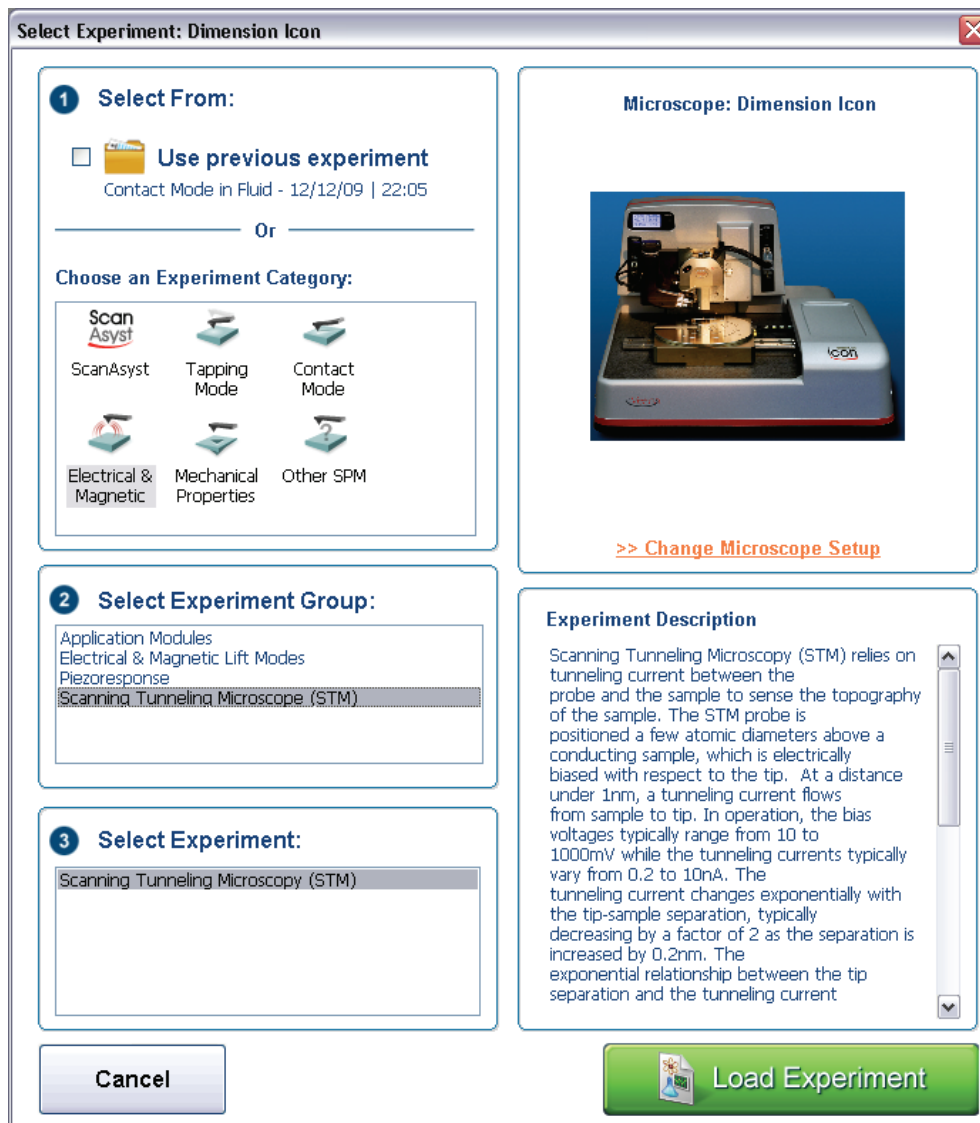
2. Select **ELECTRICAL & MAGNETIC** in the **Choose an Experiment Catagory** panel.

3. Select **SCANNING TUNNELING MICROSCOPE (STM)** in the **Select Experiment Group** panel.

4. Select **SCANNING TUNNELING MICROSCOPE (STM)** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. This opens the **Workflow Toolbar**, the **Scan 2 Channels** (Icon) or the **Scan 8 Channels** (Icon-PI) windows and the **Scan Parameters List** window, shown in Figure 13.2b.

Figure 13.2b STM in Air (Simple Mode) configuration
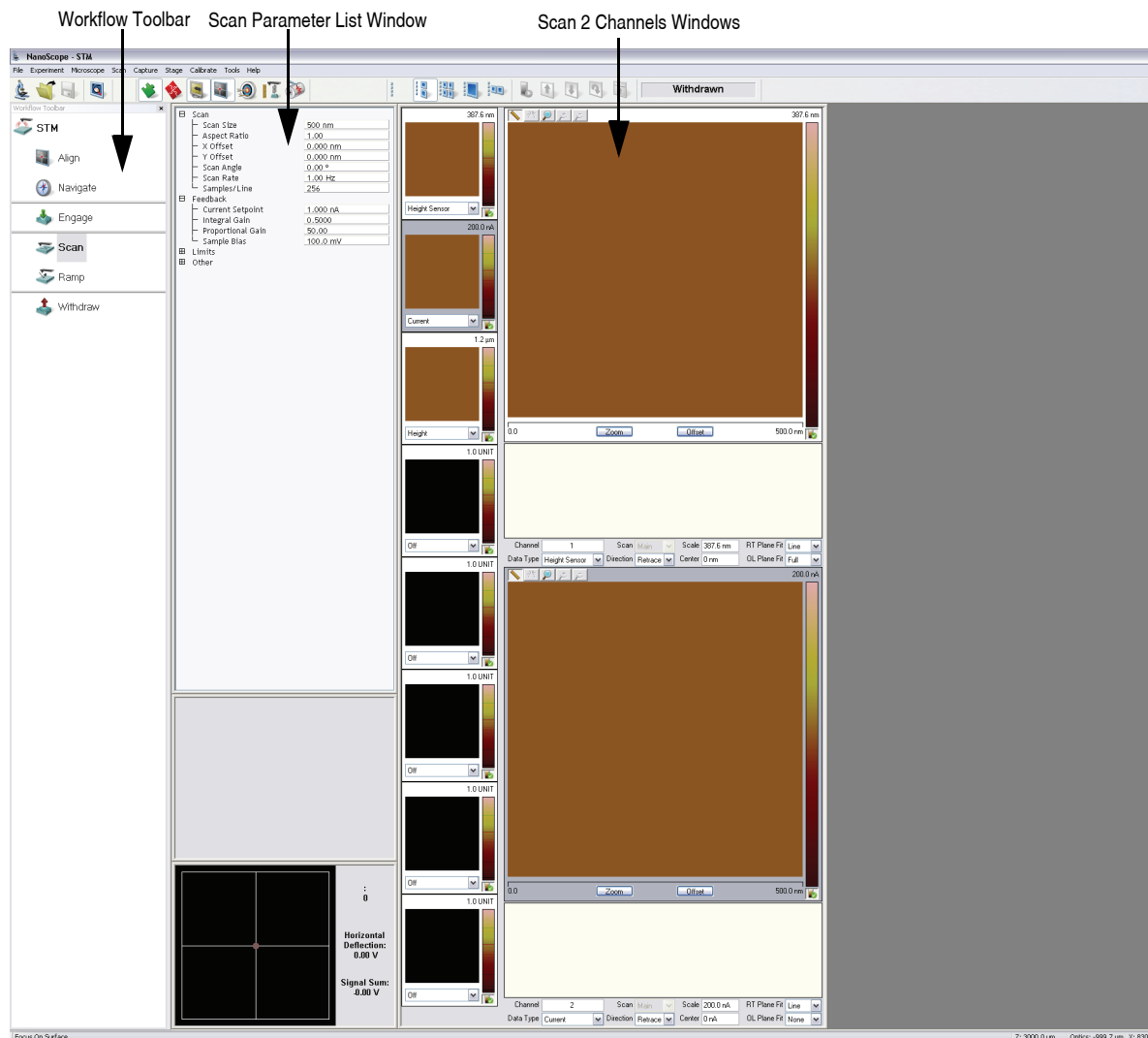
Note: Figure 13.2b shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

13.2.3 Head, Probe and Sample Preparation

1. Insert a new tip in the tip-holding tube mounted on the STM tip holder. The tips come in a small, plastic snap-box filled with black foam. Grip the tip with tweezers near the sharp end, then insert the blunt end of the tip into the tip-holder. For the tip to be held in the tube, it is necessary to put a small bend in the tip before it is completely inserted. The tip should be inserted so that it protrudes about 2mm beyond the end of the head and should be fairly tight in the tip-holder. The business end of the tip must project beyond the bottom profile of the tip holder's preamp chip; otherwise, the chip will bottom out on the sample surface before the tip engages.

2. Load the calibration standard (sample) onto the stage using a magnetic puck. Ensure that the standard is in electrical contact with the stage surface. This usually necessitates using an electrically conductive epoxy (silver-based) to affix the sample to the surface of the puck (e.g., Dynaloy 325).

3. Plug the loaded tip holder into the end of the Dimension head, then mount the head into the dovetail slot.

13.2.4 Locate Tip

To locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

13.2.5 Focus Surface

1. Click the NAVIGATE icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

	CAUTION:	Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

13.2.6 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 13.2c).

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

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

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

Figure 13.2c Suggested STM Settings

Feedback Panel

1. Set the **Integral gain** to **1** and the **Proportional gain** to **2** (see Figure 13.2c).

2. Set the **Sample Bias** to **100mV**.

3. Set the **Current Setpoint** to **5nA**.

Other Panel

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

Channels 1 and 2

1. Set the **Channel 1 Data Type** to **HEIGHT SENSOR** (see Figure 13.2d).

2. Set the **Channel 2 Data Type** to **CURRENT**.

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

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

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

5. Set the **Channel 3** through **Channel 8 Data Type** to **OFF**.

Figure 13.2d   Suggested Channel Settings

Channel	1	Scan	Main	Scale	387.6 nm	RT Plane Fit	Line
Data Type	Height Sensor	Direction	Retrace	Center	0 nm	OL Plane Fit	Full

Channel	2	Scan	Main	Scale	200.0 nA	RT Plane Fit	Line
Data Type	Current	Direction	Retrace	Center	0 nA	OL Plane Fit	None

13.2.7 Engage

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

13.2.8   Adjust Scan Parameters

Once engaged, the scan parameters should be adjusted to obtain the best image. Observe the agreement between the Trace and Retrace lines, and adjust the **Setpoint**, **Gains** and **Scan Rate** to bring the Trace and Retrace lines into coincidence.

    Note: Generally, the **Data Type** field is set to **HEIGHT SENSOR OR HEIGHT** whenever the Dimension is used in STM mode to image sample surfaces with large fields of view.

    Note: A poor tip, either because it has touched the sample, has contamination on the end, or because of its manufacturing, will render a poor image. In this case, there is little likelihood that the tip will recover, and the best procedure is to replace the tip. Click **Withdraw** to stop scanning, and retract the probe from the surface. After the tip is withdrawn, the WITHDRAWN message will appear in the status bar. To simplify tip holder removal, position the stage so that it is no longer beneath the head; the tip holder may be pulled straight off without the need to remove the entire head. Be certain there is sufficient clearance between

tip and sample after installing the new tip—a new tip will extend a different distance out of the tip holder than the old tip.

The nominal Z position of the piezo provides useful diagnostic information. The real time status bar graph reflects the Z position of the piezo during the scan. Under most circumstances, the "Z center position" should be in the center of the graph, indicating that the Z voltage for the piezo is fluctuating around zero volts. If the sample is not level, or if there is drift in the mechanical system after the tip has engaged, the "Z center position" will fluctuate during the scan or drift off-scale.

Improving Image Quality

- Vary the **Scan Size** and the **X Offset** and **Y Offset**. Move to a new location on the sample by changing the Offset of the scan. The X and Y offset parameters define the center of the scan. Often, a better image can be obtained on a different portion of the sample. The X and Y offsets can be changed by altering the values in the control panel, or by using the **Offset** button below the image.

- Oscillate the tip by briefly setting the **Integral Gain** up to about 500. The oscillation will show up on the display as a grainy pattern of light and dark. Set the **Integral Gain** back down after about a second and check if the signal is quieter than before. Setting the **Integral Gain** high for a brief period is also useful for cleaning debris from the tip.

- Alternate the commands **Withdraw** and **Engage** a couple of times. This raises and lowers the tip and may eliminate contamination on the end of the tip.

- Use the **Step Motor** command to single-step the tip down a step or two. Be sure to minimize the **SPM step size** before stepping the motor down. Watch the **Z Center Position** scale to verify that the piezo has sufficient room to retract so the tip will not harm the surface of the sample. Stepping the tip down until the **Z Center Position** goes to the retracted end of the scale where the indicator turns red will usually destroy the tip.

- Vary the **Setpoint** current over the range 2 to 10nA. As a last resort, current may be increased up to 100nA for a very brief period of time.

- If none of the above procedures improves the quality of the signal, replace the tip and try again. If that doesn't work, call Veeco for technical assistance.

- Reduce the **LP Current BW**, available in Expanded Mode.

1. After a clean image has been obtained, saved it to disk with the **Capture** commands (or click a **Capture** icon). The **Capture** command saves the current scan image (or the next image if any changes have been made in the STM control panel) into the specified directory. The **Capture Filename** command can be used to define a custom filename or the captured file will be named with the concatenation of a date/time stamp. In either case, a three-digit suffix which incrementally increases after each capture operation is appended to the filename. If the date/time stamp is used, a file captured at 3:32 p.m. on July 31 would be assigned the file name 07311532.001. A second file captured the same minute would be named 07311532.002.

2. Simple filtering operations can improve the look of the captured image. Double-click the image in the browse window.

3. Apply the **LOWPASS** filter by clicking the Lowpass icon on the toolbar and clicking **EXECUTE**. See the *NanoScope Software 8 User Guide*, Veeco p/n 004-994-000, for information about this command.

13.3 STM-Specific Information and Operations

The NanoScope STM is a digitally-controlled instrument capable of producing high resolution, three-dimensional images. The sample is held in position while a piezoelectric crystal in the form of a cylindrical tube scans the sharp metallic probe over the surface in a raster pattern. The digital signal processor (DSP) calculates the desired separation of the tip from the sample by sensing the tunneling current flowing between the sample and the tip. The bias voltage applied to the sample encourages the tunneling current to flow. The DSP completes the digital feedback loop by outputting the desired voltage to the piezoelectric tube. The NanoScope STM can be operated in two modes. In one case the feedback gains are set high, the tunneling tip closely tracks the sample surface, and the variation in the tip height required to maintain constant tunneling current is measured by the change in the voltage applied to the piezo tube. In the other case, the feedback gains are set low, the tip remains at a nearly constant height as it sweeps over the sample surface, and the tunneling current is imaged. This section provides greater insight into the operation of the STM.

13.3.1 STM Hardware

Some individual STM components are described below:

1. Tip holder—To keep the mass on the end of the tube low, a simple tip holder, designed to take 0.010" diameter tips, is used. The holder is a stainless steel tube with a 0.012" inner diameter mounted in ceramic. This design mounts the tip at the center of the piezo tube on a ceramic support element. This design preserves the symmetry of the scan, although it makes it difficult to see the tip-sample interface.

2. Tips— The probes for the NanoScope STM must be less than 0.012" in diameter to fit into the tip holder. The two most commonly used tips are made from either a platinum iridium (PtIr) alloy or tungsten. The PtIr tips are mechanically formed and can be purchased directly from Veeco. At the end of this chapter, instructions are provided on how to etch tungsten tips from tungsten wire with an electrochemical process. In general, PtIr tips provide better atomic resolution than tungsten tips, but tungsten tips are more uniformly shaped. They may perform better on samples with steeply sloped features such as compact or optical disks.

3. Preamp—Mounted on the tip holder is a circuit which contains the preamplifier for the tunneling current and provides interconnections to the tube electrodes.

13.3.2 Fine Points of STM Operation

Getting the best images on NanoScope requires a good interface between sample and tip, reasonable vibration isolation, and proper settings in the Computer Workstation menus. This section provides greater detail on the operation of the STM.

13.3.3 STM Operating Modes

The two operating modes available on the NanoScope STM are selected with the **Data Type** option in the **Channel** panel (see Figure 13.3a). **Height** or **Height Sensor** data reflects the change in tip position required to maintain a constant tunneling current. The DSP senses the tunneling current, calculates the difference from the desired tunneling current, and determines the voltage that must be applied to the piezo tube to keep the tunneling current constant; the change in voltage applied to the piezo tube translates directly to a change in distance. This distance data is recorded throughout the scan and displayed on the screen as the "height" of the sample.

Figure 13.3a   Typical STM Channel 1(above) and Channel 2 (below) Control Panel Parameters

Current data is a measure of the tunneling current at each point tested on the sample. The DSP measures the voltage drop across a resistor in series with the tip and calculates the tunneling current as the tip scans the surface of the sample. The tunneling current at each data point is recorded and displayed on the screen.

The two scan modes (height and current) require subtle changes in the menu parameters to operate effectively. The parameter changes also affect the application of the two modes. To operate effectively in collecting height data, the tip must closely track the sample surface. The gains must be maximized to force the piezo to respond quickly to the variations in the sample surface. The height mode is used for most applications. Conversely, the gains must be very low to keep the piezo from responding while collecting current data. After engaging, the tip scans the surface of the sample with very little variation in the piezo height. This constant height provides a reference from which to measure and record the fluctuations in the tunneling current. The current mode is most useful for imaging atoms with relatively small scan sizes.

13.3.4 STM-Specific Menu Parameters

In addition to the **DATA TYPE** discussed above, the **Feedback** control panel contains three items that are specific to the operation of the scanning tunneling microscope. The **SPM Feedback** (**Expanded Mode** only), **Sample Bias**, and **Current Setpoint** parameters pertain exclusively to the control of the STM.

The **SPM Feedback** parameters in the **Feedback** panel determine the transformation performed on the tunneling current prior to the feedback calculations. The two settings select LINEAR or LOG operations. Remember that tip-to-sample separation is proportional to the log of the tunneling current. The **Linear** selection causes the error signal for the feedback loop to be the difference between the instantaneous tunneling current and the setpoint current. The **Log** selection calculates the error signal as the difference between the log of the instantaneous tunneling current and the log of the setpoint current.

The **Bias** parameter controls the magnitude and sign of the bias voltage applied between the tip and the sample. A bias voltage encourages the tunneling current to flow. Although settings of 20 to 100 mV are typical for conductive samples, the allowable setting ranges from -10 to 10 volts. Positive settings of the bias voltage induce negative tunneling currents (i.e. electrons flowing from the tip to the sample).

Optimization of STM Scanning Parameters

The process of selecting and optimizing the scan parameters can be streamlined. In most cases the scan parameters are dictated by the sample. The **Data Type** is usually the first parameter set and the **Proportional** and **Integral gains** are directly related to the **Data type**. The **Scan Size** depends on the sample and the features of interest. The maximum **Scan Rate** is usually related to the **Scan Size**. The **Bias** voltage and the tunneling **Current Setpoint** depend on the sample. Usually, they are set at a standard value for engagement and fine-tuned along with the gains and filter to enhance the quality of the image.

As discussed above, a **Data Type** of CURRENT is the best for atomic-scale images. This mode is not practical for rough surfaces, because the tip will crash into the surface at low feedback gains. HEIGHT and HEIGHT SENSOR **Data Types** are usually better for all but atomic-scale scans. In general, height data images are best at higher feedback gains and slower scan rates.

Settings for feedback gains depend on many factors, but perhaps the most important is the **Data Type**. If the **Data Type** is set to **Current**, the **Integral** and **Proportional Gains** should be set as close to zero as possible. The gains should be lowered for data captured using the LINEAR SPM **Feedback** type, especially with high **Setpoint** current levels. Large-scale images should be taken at increased gain. Experimentation may yield other values for your samples.

Often the best way to set the gains for the HEIGHT **Data Type** is to view the scope trace with the **Slow scan axis** disabled (Expanded Mode). This allows the feedback to be tuned while looking at a single scan line of data. First, increase the **Integral Gain** until oscillations start to appear, and then, back off a little. Next, adjust the **Proportional Gain**. High frequency noise will appear on the signal when the **Proportional Gain** is set too high. The **Setpoint** current and the **Bias** voltage can also be adjusted using the Scope display.

Bias voltages below 20mV usually provide the best quality images on samples with surface conductivities equal to or better than graphite, but there are exceptions. Resistivity across the surface of a sample can be measured with an ohmmeter. For samples with high resistivity (greater than 1 megohm/cm), bias voltages of 100mV or even higher may work best. For scans larger than 0.5µm, it is sometimes better to increase the bias voltage by 50mV to 100mV over the value for smaller scans. A higher bias keeps the tip further from the surface, giving the feedback loop greater tolerance in tracking the surface at high speeds.

Increasing the **Current Setpoint** can also be helpful for larger scans. This has the effect of raising the gain, but also brings the tip closer to the surface by a small amount. High setpoint currents of 6 nanoamps or more can also be useful in improving the signal-to-noise ratio for atomic images on some materials.

The **SPM Feedback** type can be set to either **LOG** or **LINEAR** input transformations. Because the tip-to-sample separation is proportional to the log of the tunneling current, the transformation performed on the tunneling current prior to the feedback calculation can have dramatic effects on the performance of the feedback loop. **LINEAR** input is more protective of the tip, because the feedback error signal responds exponentially to tip-sample separation. When the tip-to-sample separation decreases, the error signal rises exponentially, quickly driving the tip away. However, the error signal is unsymmetrical. The same sample-to-tip separation change that caused the tip to move away so quickly will generate a small error signal when the tip is higher than it is supposed to be. This unsymmetrical response in the **LINEAR** mode will distort data. For this reason, the **LOG** mode (with ln(I) used in the feedback calculation) are preferable for most samples, because they respond in a more symmetrical fashion to positive and negative sample slopes. The log input has the advantage of having a gain which is insensitive to the value of the **Current Setpoint**.

Large scans cannot be taken at the same scan rate as small scans. When using **Scan Sizes** above a few microns, the **Scan Rate** should be lowered below 10Hz. Best results can be obtained at scan rates of 1 Hz or less, although image-taking is slow. At these scan rates, the 128 x 128 and 256 x 256 data formats are most useful, quadrupling and doubling the frame rate over the 512 x 512 format for a given scan rate. To be sure that there is no image degradation due to too high a scan rate, lower the rate and check for changes in the image. Check the **Scope** view to ensure that the scan is not slew-rate limited in Z, as evidenced by an artificial "sawtooth" appearance in the scope trace.

The **LP Current BW** (Expanded Mode) parameter provides the option of filtering the tunneling current signal. A lowpass filter with a cut-off frequency of 20kHz can be applied to the analog tunneling signal in hardware. Typically, the filter is selected for atomic-scale images; otherwise, no filtering should be selected.

Tunneling Tips

Although the microscope will accept any 0.010" diameter tip, tips made of platinum iridium (PtIr) and tungsten are used most often. PtIr tips are supplied by Veeco while tungsten tips can be electrochemically etched from tungsten wire by following instructions at the end of this chapter. In general, most of the discussion in this manual involving tips and noise reduction applies to both types of tips, but there are some applications which are tip specific.

PtIr tips seem to give better atomic resolution than tungsten in air and liquids, probably due to the lower reactivity of platinum. The PtIr tips are not as uniformly shaped as the tungsten tips, so freshly etched tungsten tips may provide cleaner data when scanning steeply sloped surfaces such as compact or optical disks. For tunneling on surfaces immersed in conductive liquids, coated tips have been used.

The quality of the mechanically formed PtIr tips will vary. Some give beautiful images as soon as tunneling starts; others start poorly but improve over time; while others start noisy and stay noisy. Some tips have very flimsy points which give an image that is stretched out on one side; this

indicates that the tip is unable to turn around quickly at the ends of the X scan. These tips should be replaced.

Sample Surface

Samples to be imaged with a scanning tunneling microscope must conduct electricity to some degree. In many cases nonconductive samples can be coated with a thin layer of a conductive material to facilitate imaging. The sample surface must be conductive enough to allow a few nanoamps of current to flow from the bias voltage source to the area to be scanned. NanoScopes have been used to scan gold, silver, platinum, nickel, copper under oil, chrome plating, doped silicon under oil, conducting polymers, amorphous carbon, blue diamond, diamond-like carbon films, carbon fibers, graphite, iron-oxide compounds, semi-metals, doped semiconductors (molybdenum disulfide), cobalt-chromium compounds, stainless steel, liquid crystals, and other materials. Oxide layers more than a few atoms thick on the sample, tend to affect the scanning and wear down the tip as it is dragged through the oxide. The feedback loop will extend the tip until a tunneling current flows, even if it must push the tip through an oxide layer (if it can). If oxide presents a problem, keep the sample covered with oil, operate the microscope in a glove bag filled with nitrogen or argon or use a Veeco EnviroScope.

On samples which are noisy or tend to oxidize, tunneling under oil or scanning in a glove box filled with inert gas can improve the imaging. Silicon oil or paraffin oil (mineral oil) also work well with some samples. The only problem involved with the use of oil is the increased difficulty in the coarse positioning of the tip. The reflection of the tip comes off the liquid instead of the surface of the sample. It is difficult to tell when the tip is close to the sample surface. The best approach is to lower the tip until it just touches the surface of the oil, falsely engage the tip (click **MICROSCOPE > FALSE ENGAGE** or use **Ctrl-E** quickly after the **Engage** command), then lower the tip the rest of the way with the stepper motor. It may take the motor about a minute or so, but it is better than smashing the tip on the surface of the sample.

Vibration Isolation

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

As a final note, the best way to reduce coupling from vibrations is to eliminate as many sources of vibration as possible. Remember that vibrations can be transmitted to the microscope over the cable. To reduce this phenomenon, prevent tension in the cable and keep it away from fans and other noise sources. Also, keep the microscope away from sources of acoustic noise. Loud conversation can disrupt atomic scale images. Air currents can also disturb atomic images, so it is best to run the microscope with the cover on.

Piezoelectric Sensitivity and Derating

The sensitivity of the piezoelectric tube in nanometers per volt and the derating of the piezo sensitivity as the scan voltage is decreased are calibrated using the **Calibrate** command. Generally, a silicon calibration standard is used.

The sensitivity of the piezoelectric tube decreases as the applied range of the applied scan voltage is reduced. Over a small scan range, the sensitivity of the piezo is constant and a plot of scan size versus scan voltage is linear. However, for large scans, sensitivity variations are an important factor. A plot of the sensitivity of a long-range head as a function of scan voltage looks similar to the graph in Figure 13.3b. As shown, the **Derating** (the slope of the head's sensitivity plot) indicates how much the piezo sensitivity decreases as the scan voltage is reduced.

Figure 13.3b   Scan Derating Graph
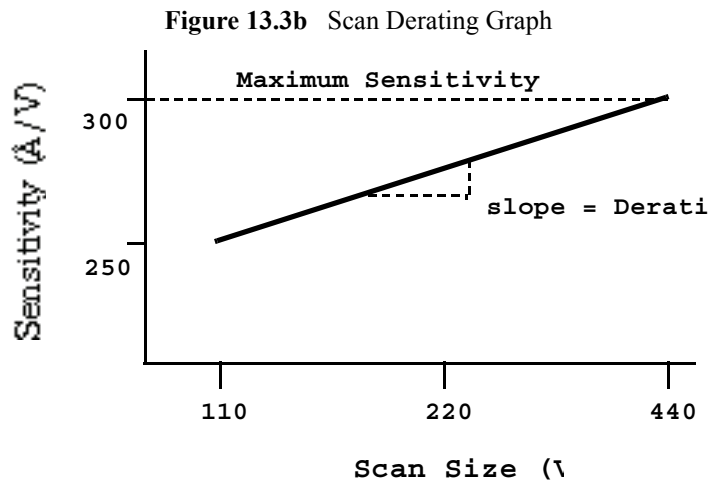
The NanoScope software accounts for the reduction in sensitivity as the scan voltage is decreased through the **Derating** parameters in the **Calibrate > Scanner > X-Y** dialog box.

Head Offset

The value of the head preamplifier offset can be measured with the **Offset** command in the **Tools** menu. The procedure is as follows:

- Withdraw the tip from the sample surface.

- Invoke the **Offset** command to observe the offset current of the preamplifier in nanoamps.

13.4   Spectroscopy with the STM

The NanoScope STM performs limited spectroscopic operations under the two scanning tunneling spectroscopy STS modes of operation. The variation of the tunneling current due to variations of the bias voltage or tip-to-sample separation can be tested and recorded at a single point using NanoScope's **Ramp** mode.

STM Ramps

In the STS ramp operation, the tip is positioned at a point on the surface, and a spectroscopic plot is acquired and displayed in a scope format. Between plots, the feedback is run to establish the tunneling current to the setpoint value. The different types of STS Plots that can be acquired are:

STS i(v) — To do this, select **Ramp** and choose SAMPLE BIAS for the **Ramp Output** in the **Ramp** panel of the **STM Ramp Parameter List**, shown in Figure 13.4a. Select CURRENT as the **Data Type** for **Channel 1** in the **Channel Selection** panel in the **STM Ramp Plots** window, shown in Figure 13.4b. The tunneling current as a function of the bias voltage is displayed. The tip height is held constant while the I-V plot is being acquired. In addition to I vs. V, F(d i,dv), F(d ln(i),dv), or F(d ln(i),d ln(v)) vs. V can be plotted.

Figure 13.4a   STM Ramp Parameter List for STS i(V)

Figure 13.4b   Channel Selection panel for STS i(V) in the **STM Ramp Plots** window. Single monitor view.
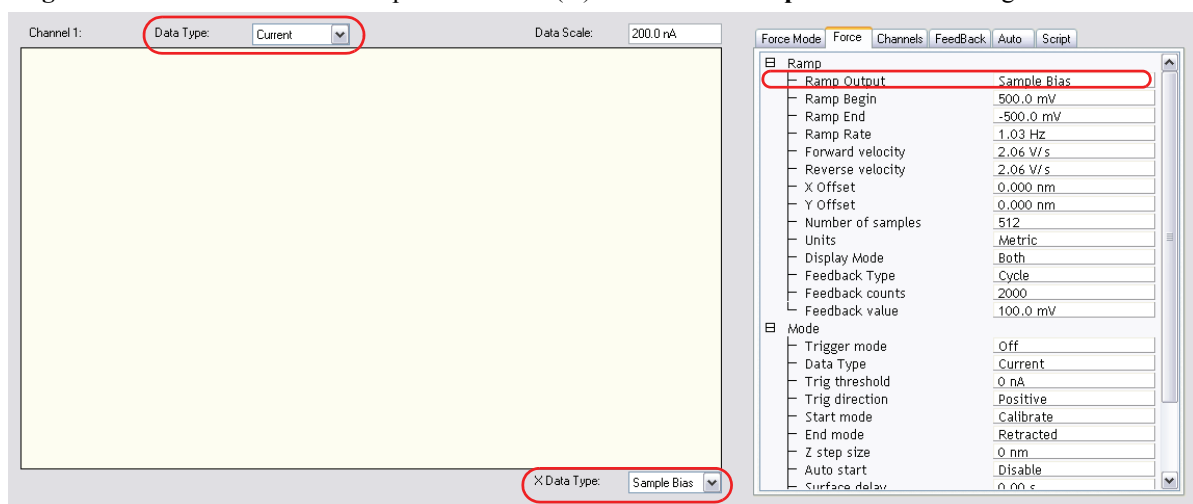
STS i(s) — To do this, select **Ramp** and choose **Z** for the **RAMP OUTPUT** in the **Ramp** panel of the **STM Ramp Parameter List**, shown in Figure 13.4c. Select **CURRENT** as the **DATA TYPE** for **Channel 1** in the **Channel Selection** panel in the **STM Ramp Plots** window, shown in Figure 13.4b. The tunneling current as a function of the tip height is displayed. The bias voltage is held constant while the I-S plot is being acquired. In addition to I vs. S, ln(I) vs. S can be plotted.

Figure 13.4c   STM Ramp Parameter List for STS i(s)
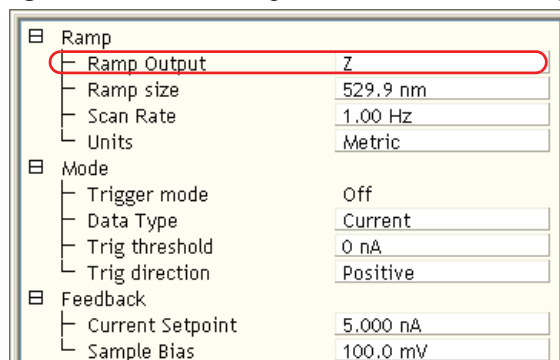
13.4.1 Operation of STS

In the following sections, the operation of the spectroscopic functions of the NanoScope STM will be discussed. Additional information can be obtained from the *Command Reference Manual*.

STS Plot

There are several items that you should be aware of when using the NanoScope to acquire spectroscopic plots with the STS plot commands. The spectroscopic capabilities can provide information that can help distinguish different species although exact species identification is

difficult, especially in air. The spectroscopic plots should aid in comparative studies between samples or between different regions on a sample, but they will not reveal the precise make-up of that sample.

A comparison to STM imaging reveals two somewhat conflicting requirements. As a good starting point, the sample and tip should produce consistent STM images. The images should repeat well from frame to frame and be fairly free of noise or areas on the surface that appear unstable. For current-versus-voltage type plots, the spectroscopic plots may be nice and smooth and repeat well, but switching back to the STM imaging mode reveals images which are quite noisy. This can be attributed to the fact that the quality and uniformity of the tip is probably more critical for imaging than for making the rather simple spectroscopic plots. This is generally not true for the I vs. S plots. Some general recommendations for acquiring spectroscopic plots are:

1. Low settings for the **Integral Gain** are preferred. Because the tip is not tracking topography, lower gains are acceptable and tend to make the plots more stable.

2. The maximum input range of ±100nA on the NanoScope with the standard preamps will restrict the current-versus-voltage plots.

13.5   Troubleshooting Operation of STM

This section addresses errors or malfunctions encountered during the operation of the STM.

13.5.1 Head and Microscope-Related Problems

This section deals with problems related to the scan heads or the microscope. If a problem exists with a scan head, try a second one under the same conditions, if possible. Otherwise, the following list of symptoms and cures may be helpful:

13.5.2 Head Engages Immediately

If the STM engages immediately after initiating the **Microscope** > **Engage** command, then one of the following probably occurred:

1. Tip On Surface—Make sure the tip is not touching the surface of the sample. Withdraw until the tip is far from the surface.

2. Controller Off—Verify that power to the controller is on and that the controller is connected to the computer workstation.

3. Head has Offset—Follow the directions in section 9.3. to determine if your scan head requires its offset to be adjusted.

Head Never Engages

If the scan head never engages, test for the following:

- Disconnected—Make sure the microscope is connected to the Controller.

- Bias Shorted—Measure the bias by using a voltmeter between the head and stage chuck. If this is not in agreement with the settings in the *Bias* voltage item in the STM Parameter control panel and appears to be grounded, then check to see if anything is providing a conduction path between the Base and the Base Support or any other ground.

Tip Crashes

If the tip always crashes into the surface with the Z center either changing erratically or stuck in the fully retracted position, try the following:

- Check Sample Conductivity—There are two problems associated with sample conductivity. First, the bulk conductivity of the sample may make it difficult to image. If the resistance of the sample is greater than 1 Kohm, higher bias voltages should be tried. If the resistance is greater than 1 Megohm, bias voltages of 100 mV or more should be used. Samples with resistances 1 Megohm or greater will be difficult to image even with high bias voltages.

Measuring the bulk conductivity of the sample with probes may not tell the whole story. Probes may easily penetrate oxide or contamination layers on the sample surface yielding reasonable resistance measurements. However, oxide and contamination layers on the sample surface can make imaging very difficult. Higher bias voltages are required for these types of samples.

Problems During Realtime Operation

This section deals with problems that are associated with the Realtime aspects of the NanoScope software although they often point to the scanning head or microscope.

Image goes out of Range

If the Z center position tends to go out of range at the edges, top and bottom or right and left of the scan, then try the following:

- Level Sample to Head—The Z center position is affected by sample tilt and the Dimension head may be less tolerant of sample tilt due to its small vertical range compared to the large, lateral scan range. Try leveling the head relative to the sample by noting the trend in Z center position and adjusting the head tilt accordingly. Setting **Realtime Plane Fit** set to NONE or OFFSET can also be very helpful in eliminating tilt along the scan direction.

- Measure Coupling— A broken wire to one of the electrodes can cause this problem and will usually result in a large, monotonic increase (or decrease) in Z center position from the bottom of the scan to the top.

Z drift

If the Z center voltage tends to drift out of range rather quickly after the head is engaged, it may be a result of the following:

- Thermal Drift— Allow some time for the temperature to stabilize if the microscope and/or scan head has been stored in a cold place overnight. Drift can be minimized by keeping the STM in a thermally stable environment.

- Tip not Tight—Make sure the tip is held tightly in the tip holder. Push and pull on the tip with the tweezers to see that it is not loose. Increase the bend in the tip if the tip seems loose.

- Sample Hold-Down—Verify that the sample is flat against the chuck and cannot move vertically. This may be become problematic if the sample has been affixed using uncured, conductive epoxy, and/or if foam adhesives are used.

Realtime Image Hides Features

If the Realtime image appears to be flat, but captured images reveal detail, then try rotating the sample 90° in the microscope. The Realtime image leveling software tends to hide features that are parallel to the X-scan direction.

Image is Streaky or Wavy

If high-resolution images appear streaky or wavy, it may be a result of the following:

- Insufficient Vibration Isolation—Atomic-scale scans are the most susceptible to vibration in the acoustic and subacoustic frequencies. Verify that the Dimension microscope is acoustically isolated with a functioning isolation table and, if required, an acoustic cover. Make certain that vibration tables are properly pressurized and adjusted.

- Bad Tip—Many strange effects have been pondered and hunted down for hours only to find that changing the tip fixes the problem. Try a different tip to see if the problem persists.

Image is Triangular over Step-like Features

- The performance of the STM over large scans with high vertical features is very dependent on the ability of the feedback loop to force the tip to track the sample surface. The digital feedback used in the NanoScope has been designed to maximize performance on a variety of samples. Try the following to improve the image quality over step-like features:

- Log Feedback—The most dramatic increase in performance of the feedback over large scans is achieved using the **Log** feedback mode. These make the error signal symmetrical for conditions with the tip too far or too close to the surface.

- Gain—The settings of the **Integral Gain** term also tend to be critical for large scans. They should be high enough to produce a little fuzz on the image but not so high as to cause large oscillations.

- Scan Rate—The **Scan Rate** should be lowered for large scans, especially, if the sample surfaces are rough or contain large steps. Moving the tip quickly along the sample surface at high scan rates with large scan sizes will usually lead to a tip crash. Essentially, the **Scan Rate** should be inversely proportional to the **Scan Size**, since the tip must still be maintained roughly 1 nanometer above the surface.

- Setpoint Current—Raising the **Current Setpoint** will effectively raise the gain of the feedback loop which can be quite helpful for large scans. It will also bring the tip closer to the surface but only by a small amount ($i \approx e^{-s}$).

13.6   Etching Tungsten Tips

You can purchase tungsten tips from Veeco or make them yourself. This section describes the process of etching tungsten tips for use with the NanoScope.

Materials Required

- Variac auto transformer

- Optical microscope (20-100X)

- Sodium nitrite ($NaNO_2$)

- Distilled water

- Ethyl alcohol

- WD-40 (anti-oxidant)

- Two 50ml beakers

- Tip holder

- Platinum wire

- Tungsten Wire, 0.010" Diameter

- Miscellaneous Wire/Clips

13.6.1 Procedure

1. Mix a 5 percent (by weight) solution of Sodium Nitrite in water.

2. Pour ∼ 40ml of the Sodium Nitrite solution into a beaker.

3. Pour ∼ 40ml of WD 40 into a beaker.

4. Construct an electrode out of the platinum wire and insert it into the beaker.

5. Adjust the variac for 30V, and with it Off, connect one output to the platinum electrode.

6. Cut 10 to 12 pieces of tungsten wire ∼ 1.25cm long. Before etching, check to make sure that at least one end of the wire has not split by inserting the ends into a tip holder. If an end has split, you will not be able to insert it into the tipholder. You can etch the end that has split, however, preserve the unspoiled end.

7. Place the tungsten tips into a holder. We like to use an IC socket (the low cost, edge-grip square contact type, not the machine-grip round contact type), with all the pins soldered together. The tips will then be held in place while inverting them over the solution. We also find it helpful to solder the IC socket to the back of a proto-board (perf-board). You can then invert the tips over a beaker with the proto-board sitting on the rim of the beaker.

8. Invert the tips over the Sodium Nitrite solution with ∼ 2mm of the tips' surface submerged. More than 2mm will cause excessive foaming of the solution during etching, and less than 2mm will result in tips that are too blunt.

9. Connect the other output of the variac to the common of all the tips.

10. Turn on the variac and etch the tips. While the tips are etching, the solution will foam, and the tips will start to glow. As the tips etch towards the surface, the foaming will be reduced. Continue to etch the tip until it stops.

11. Re-submerge the tips ∼ 1mm into the solution for ∼ 15 seconds at 30V. Turn on the variac and re-etch the tips. There should be only slight bubbling from the tip and it should not glow.

> **Note:** It is possible to vary the tip shape at this point by lowering the voltage on the variac and increasing or decreasing the amount of time the tips are submerged.

> **Note:** Longer time gives blunter tips.

12. Dip the tips into ethyl alcohol to clean them. If you plan to keep the tips around for more than a day, then dip them into the WD 40 after cleaning. Dispose of unwanted tips in an appropriately labelled solvent-contaminated waste container.

13. Examine the tips under the optical microscope. Tips that are too long, too blunt, or split at the end will hardly ever be good tips and can be thrown out at this time. Of course, this is a subjective process. As your experience in etching grows, you will get better at throwing out bad tips.

14. Repeat the etching procedure. Replace the etching solution when a fairly large amount of residue is present. Typically, you can etch 60 to 80 tips in a 40ml solution.

Chapter 14   Lateral Force Mode

This chapter includes the following sections:

- **Introduction:** Section 14.1

- **Basic LFM Operation:** Section 14.2

- **Advanced LFM Operation:** Section 14.3

  - **Scan Direction:** Section 14.3.1

  - **Probe selection:** Section 14.3.2

  - **Understanding the LFM Signal:** Section 14.3.3

  - **Understanding the Color Scale:** Section 14.3.4

  - **Enhancing the LFM Data by Subtracting Two Images:** Section 14.3.5

  - **Height Artifacts in the Signal:** Section 14.3.6

14.1   Introduction

The Dimension Icon SPM is capable of measuring frictional forces on the surfaces of samples using a special measurement known as lateral force microscopy (LFM). The name derives from the fact that cantilevers scanning laterally (perpendicular to their lengths) are torqued more as they transit high-friction sites; low-friction sites tend to torque cantilevers less. The relative measure of lateral forces encountered along a surface yields a map of high- and low-friction sites.

After obtaining a good topographical image in Contact mode, it is relatively easy to use LFM to view and acquire lateral force data. It is important to obtain a good image in Contact mode before measuring LFM data. The NanoScope system will continue to run the feedback based on measuring the vertical deflection signal and feedback gains in the **Feedback** panel while LFM data is acquired and displayed.

14.2   Basic LFM Operation

14.2.1   Select Mode of Operation

1.   Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 14.2a.

Figure 14.2a   The Contact Mode LFM **Select Experiment** window
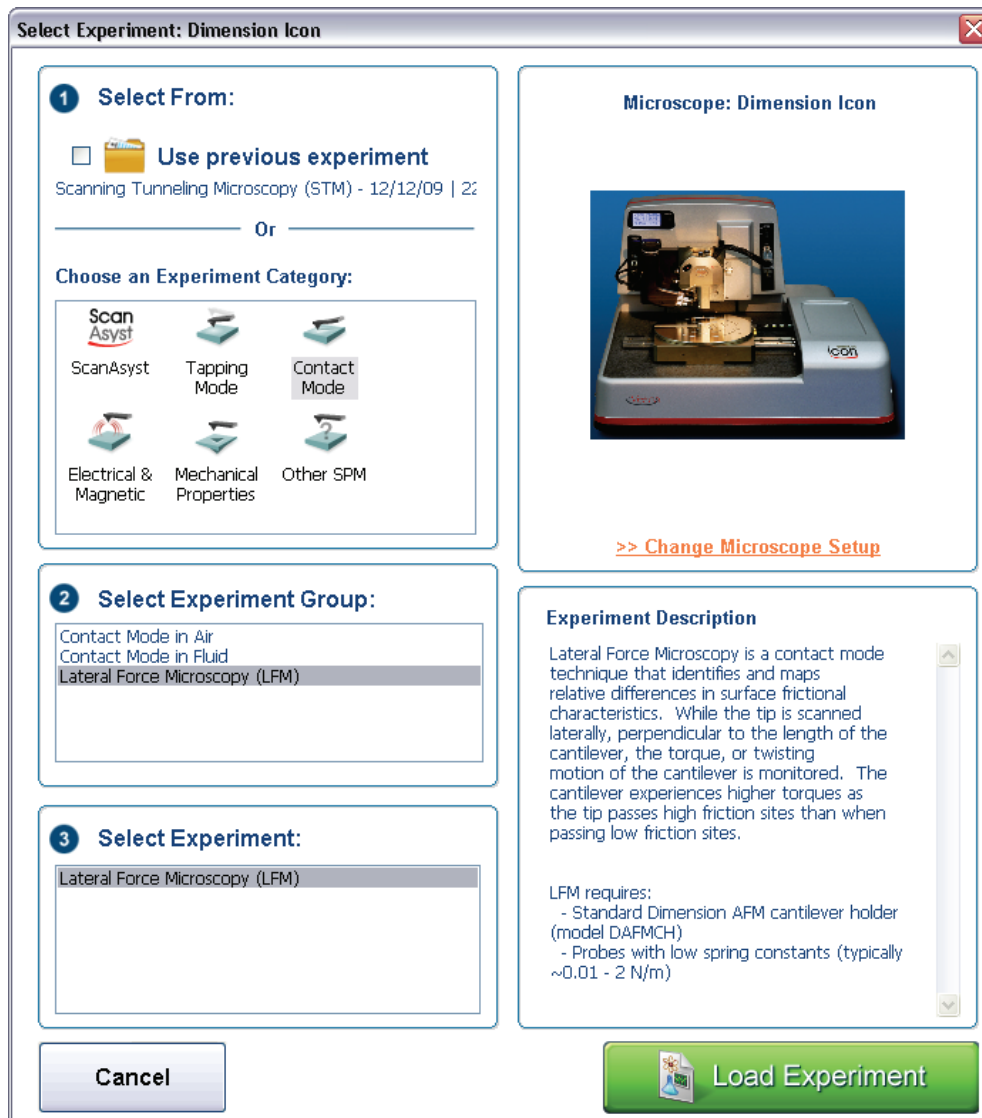
2.   Select **CONTACT MODE** in the **Choose an Experiment Catagory** panel.

3.   Select **LATERAL FORCE MICROSCOPY (LFM)** in the **Select Experiment Group** panel.

4. Select **LATERAL FORCE MICROSCOPY (LFM)** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. This opens the **Workflow Toolbar**, the **Scan 4 Channels** (Icon) or the **Scan 8 Channels** (Icon-PI) windows and the **Scan Parameters List** window, shown in Figure 14.2b.

Figure 14.2b   LFM (Simple Mode) configuration
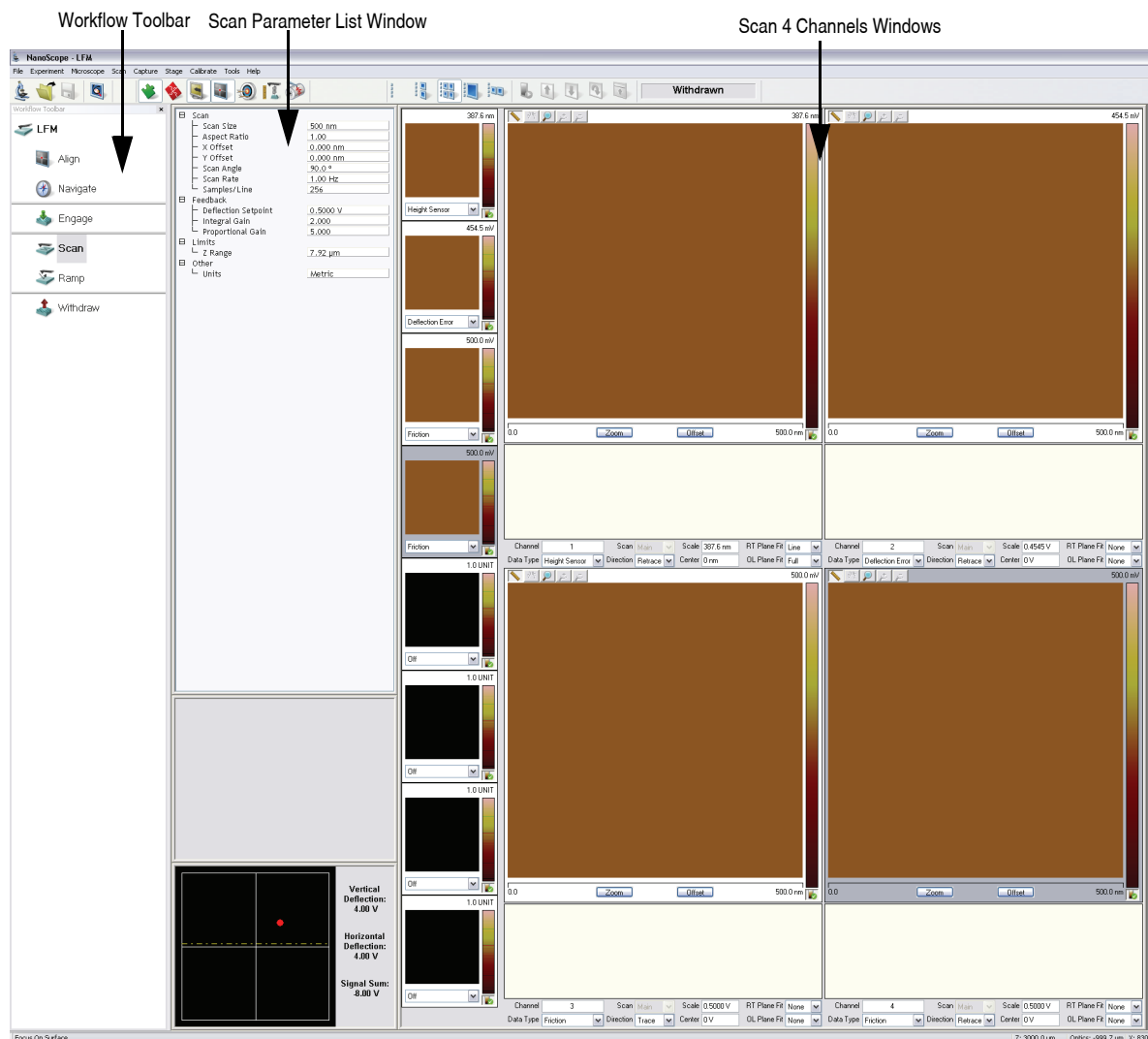
Note: Figure 14.2b shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

14.2.2 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. See **Basic AFM Operation:** Section 7.2.

14.2.3 Align Laser

1. Align the laser using the laser control knobs. See **Align Laser:** Section 7.2.9.

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 **4-6V**.

14.2.4 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. See **Adjust Photodetector:** Section 7.2.10.

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

3. Set the **Vertical Deflection** to **-2V**.

14.2.5 Locate Tip

To locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

14.2.6 Focus Surface

1. Click the NAVIGATE icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

CAUTION:	Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

14.2.7 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 14.2c).

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

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

3. Set the **Scan Angle** to **90.0** degrees.

Figure 14.2c   Suggested Contact Settings

Feedback Panel

1. Set the **Integral gain** to **2** and the **Proportional gain** to **5** (see Figure 14.2c).

2. Set the **Deflection Setpoint** to **0.5V**.

Other Panel

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

Channels 1 and 2

1. Set the **Channel 1 Data Type** to HEIGHT (see Figure 14.2d).

2. Set the **Channel 2 Data Type** to FRICTION.

3. Set the **Channel 3 Data Type** to DEFLECTION ERROR.

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

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

5. Set **Channel 2 Line direction** to TRACE.

    Note: If the **Line Direction** parameter is changed to **Retrace**, the plus-minus sign of the lateral force changes and low lateral force will be displayed on the top of the scope display. Setting the scan angle to 270 degrees will invert the polarity of **Trace** and **Retrace**.

Figure 14.2d   Suggested LFM **Channel 1**, **Channel 2** and **Channel 3** Settings

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

7. Optimize the scan parameters in AFM mode for **Channel 1**.

    Note: LFM is a qualitative measurement, not quantitative. NanoScope software does not provide a way to calibrate the torsional spring constant of a cantilever.

    Note: The trace and retrace LFM signals may not be the same magnitude. The tip will never be exactly perpendicular to the sample. In most cases, internal stress in the material of the cantilever will cause the cantilever to have a natural bias in some direction.

14.3   Advanced LFM Operation

14.3.1   Scan Direction

The cantilever is most susceptible to frictional effects when the scan direction runs perpendicular to the major axis of the cantilever as shown in Figure 14.3a. The **Scan Angle** parameter in the **Scan** panel must be set to **90°** or **270°** to produce this scan direction.

Figure 14.3a   Scan Angle Selection
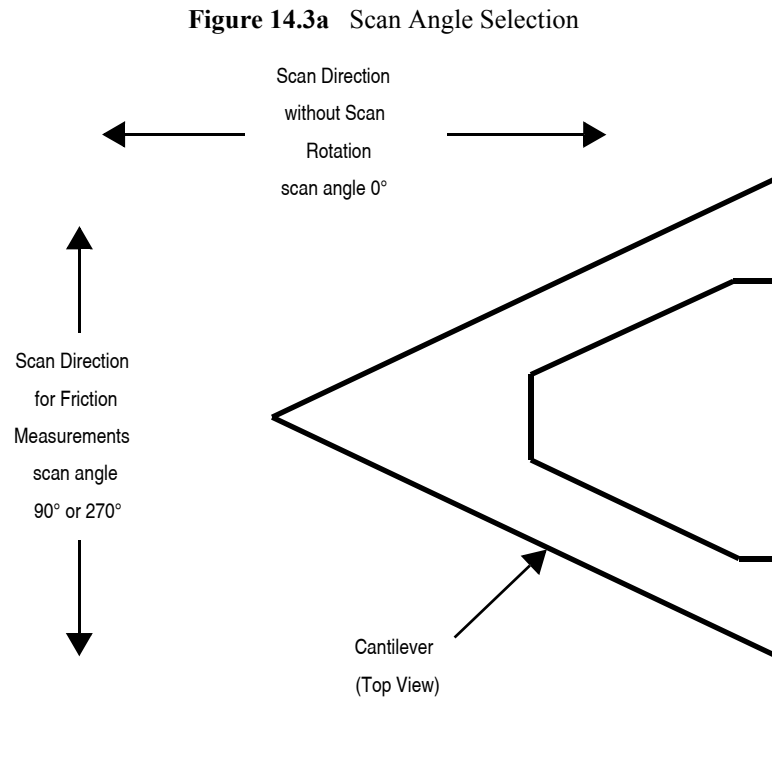
The contact mode setpoint voltage will slightly adjust the gain of the lateral force signal. By increasing the setpoint, the contact force applied will increase, and so will the frictional or torsional forces in an approximately linear fashion. If the frictional effects are too large or too small, change the cantilever, 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.

14.3.2 Probe selection

The analog-to-digital converter on the auxiliary input channel which is used for LFM data has a maximum input range of ±10V. This, and the anticipated interaction between tip and sample define the selection of the cantilever to be used for the measurement.

A 200µm cantilever with wide legs provides a good starting point for frictional measurements. It is flexible enough to provide reasonable signal levels on samples with moderate friction. If the signal exceeds ± 10.00V with a 200µm wide-legged cantilever, one of the stiffer 100µm cantilevers should be used. If the signal level is too small, the narrow-legged 200µm cantilever will provide a larger signal.

14.3.3 Understanding the LFM Signal

To understand LFM, examine a sample's trace and retrace in the scope window. Figure 14.3b, below, illustrates the movement of the tip as it encounters friction during a scan.

Figure 14.3b   Example of Frictional Forces on Tip During Scan (Front View)
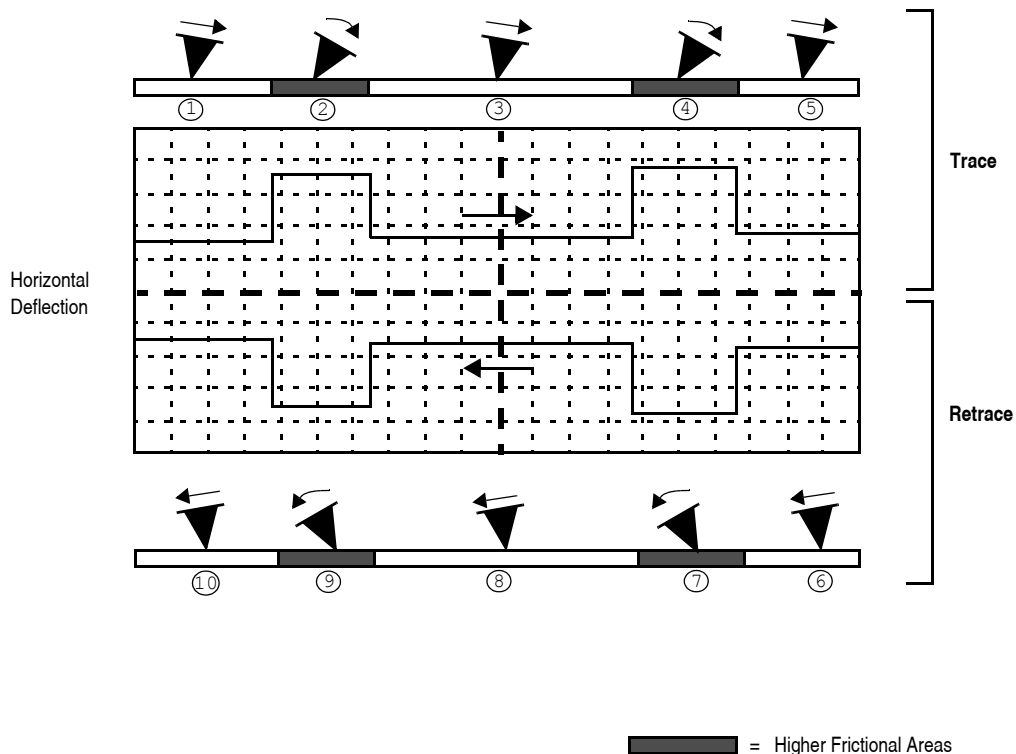
Table 14.3a   Key for LFM Example Above

Number	Trace or Retrace?	Tip Movement	Level of Friction	Photo Diode Voltage
1	trace	slightly right	slight	slightly positive
2	"	severely right	higher	increases positive
3	"	straightens	slight	returns to slightly positive
4	"	severely right	higher	increases positive
5	"	straightens	slight	returns to slightly positive
6	retrace	slightly left	slight	slightly negative
7	"	severely left	higher	increases negative
8	"	straightens	slight	returns to slightly negative
9	"	severely left	higher	increases negative
10	"	straightens	slight	returns to slightly negative

14.3.4 Understanding the Color Scale

LFM data is typically monitored in the trace direction with the scan angle set to 90 degrees. The data then follows a convention where lighter colors mean higher friction. Darker colors mean less friction. The sign of the color is inverted if Retrace is monitored (see Figure 14.3c).

Figure 14.3c   Friction Data
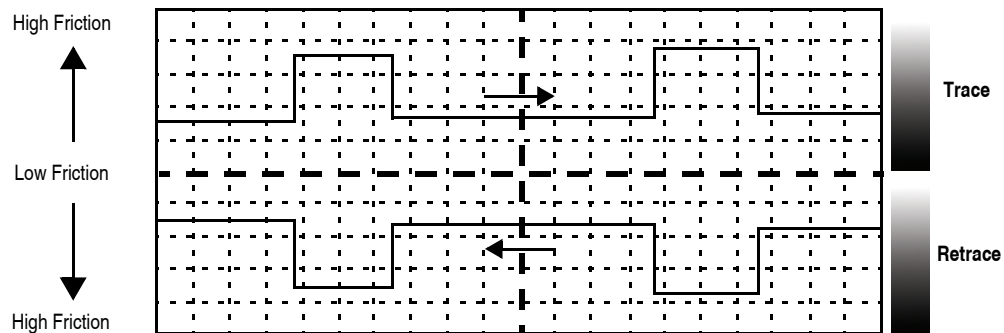
14.3.5 Enhancing the LFM Data by Subtracting Two Images

It is possible to enhance the magnitude of the LFM data by subtracting the trace from the retrace. This is done by collecting three data channels with one scan.

1. Set **Channel 1** to **HEIGHT**.

2. Set **Channel 2 Data Type** to **FRICTION, Line Direction** to **TRACE, Offline Plane Fit** to **NONE.**

3. Set **Channel 3 Data Type** to **FRICTION, Line Direction** to **RETRACE, Offline Plane Fit** to **NONE.**

4. Collect a scan at **90** degree **Scan Angle** and save it.

5. Load the file and use the **SUBTRACT IMAGE** subtract feature to subtract **RETRACE** from **TRACE**. The resulting data will show double the LFM data and half the background noise caused by tracking error. Information about the Subtract Image function can be found the *NanoScope Software 8 User Guide*, Veeco p/n 004-994-000.

14.3.6 Height Artifacts in the Signal

LFM is subject to height artifacts due to coupling with surface topography. Delay in the feedback loop causes the tip to momentarily twist as it climbs up an edge. This will be visible in the friction data if it is severe enough.

To distinguish real friction data from height artifacts, remember that friction causes the cantilever to twist in the opposite direction as it travels. Actual LFM information will always show up as a mirror image in the trace and retrace directions in the scope display. Height artifacts will be the same direction in the scope display (see Figure 14.3d).

Figure 14.3d Height Artifacts in LFM Data
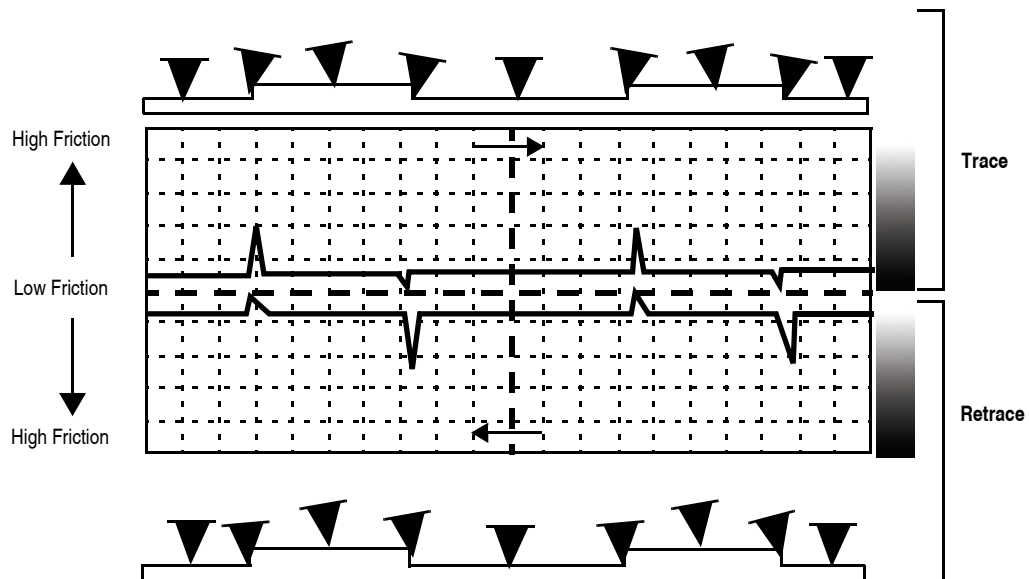
Chapter 15 Force Imaging

Force plots measure tip-sample interactions and determine optimal setpoints. More recently, microscopists have plotted force measurements across entire surfaces to reveal new information about the sample. This area of scanning probe microscopy promises to open new chapters in materials science, biology and other investigative areas.

Specifically, this chapter details the following topics:

- **Force Plots–An Analogy:** Section 15.1

- **Force Calibration Mode:** Section 15.2

  - **Example Force Plot:** Section 15.2.1

- **Ramp Parameter List:** Section 15.3

  - **Ramp Controls:** Section 15.3.1

  - **Channel 1, 2, 3 Panels:** Section 15.3.2

  - **Feedback Panel:** Section 15.3.3

  - **Mode Panel:** Section 15.3.4

  - **Menu Bar Commands:** Section 15.3.5

- **Force Calibration (Contact Mode AFM):** Section 15.4

  - **Obtaining a Good Force Curve:** Section 15.4.1

  - **Helpful Suggestions:** Section 15.4.2

  - **Advanced Techniques:** Section 15.4.3

  - **Interpreting Force Curves:** Section 15.4.4

15.1 Force Plots–An Analogy

A force plot is an observation of tip-sample interactions which yields information regarding the sample and tip. By way of analogy, suppose a materials researcher must determine how powerful two different types of magnets are. One magnet is made of iron, the other is a stronger, rare earth magnet.

A simple way of measuring each magnet's strength would be to measure 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 strength. Similarly, after attachment the researcher could pull each magnet away from the plate and measure the pulling force at regular intervals until the magnet breaks free. The pull-off point of each magnet gives an additional index of its holding strength.[1] A representation of this setup during a lowering cycle of one magnet is illustrated in Figure 15.1a.

Figure 15.1a Comparative Index of Pulling Forces
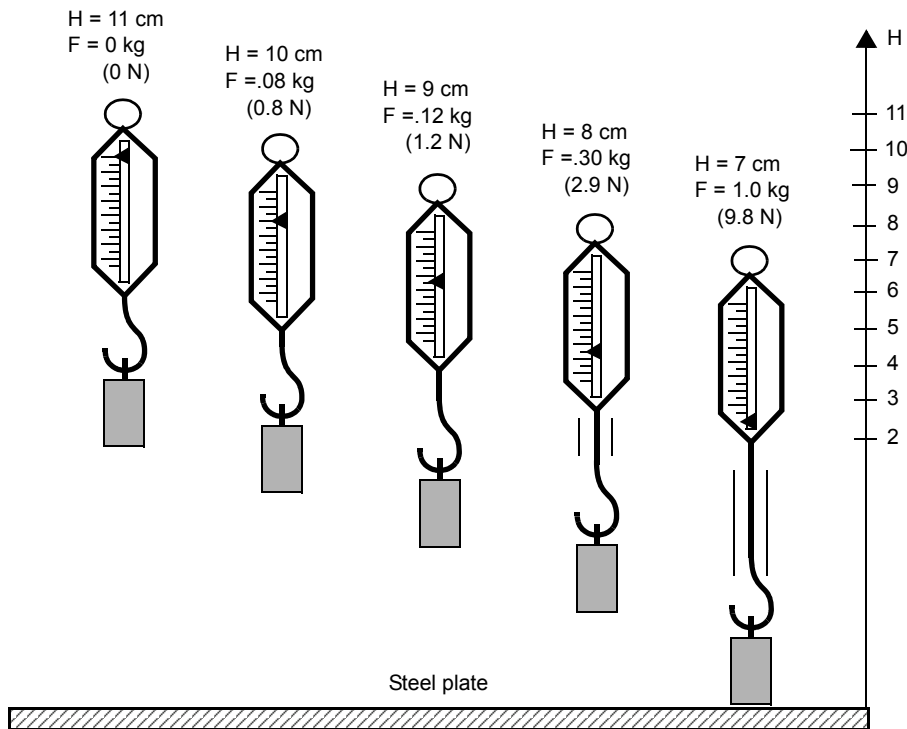
The pulling force is measured at 1 cm height intervals while the scale and magnet lower and lift in a controlled cycle. Figure 15.1b depicts a plot of this experiment using two magnets. The plot depicts each magnet's attraction as it approaches the plate, and its tenacity when pulled off the plate. Assuming both magnets are the same size, this reveals information about each magnet's strength. First, magnet #1 is weaker, attaching to the steel plate with 7N of pulling force at 6cm, and magnet #2 is stronger, attaching at 7cm with 10N.

Figure 15.1b Pulling Forces Graph

Height above steel plate (cm)

---
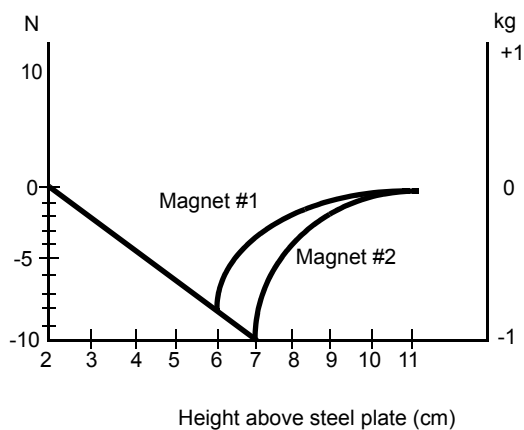
1. For the sake of simplicity, forces are represented here using a common scale in kilogram units; however, force is properly measured in Newtons (1 kg = 9.8 N).

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.

15.2 Force Calibration Mode

The **Continuous ramping** command in the **Ramp** menu allows you to quickly check the interaction between the cantilever and the sample surface. In the **Continuous ramping** mode, the X and Y voltages applied to the piezo tube are held constant and a triangular waveform similar to the one depicted in Figure 15.2a is applied to the Z electrodes of the piezo tube.

Figure 15.2a   Continuous ramping Z Waveform

$$\text{Scan period (sec)} = \frac{1}{\text{Z scan rate (Hz)}}$$
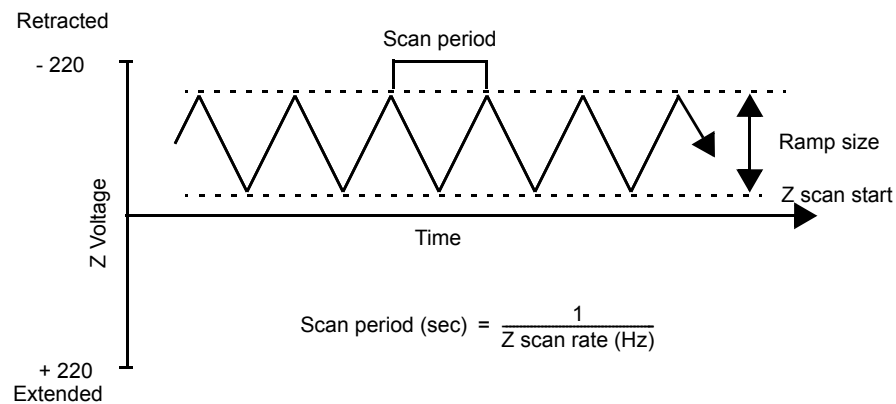
As a result of the applied voltage, the cantilever tip moves up and down relative to the stationary sample as shown in Figure 15.2b. The **Z scan start** parameter, available through the **Ramp Parameter List**, sets the offset of the piezo travel, while the **Ramp size** parameter defines the total travel distance of the piezo. Therefore, you can obtain the maximum travel distance by setting the **Z scan start** to **+220V**, with the **Ramp size** set to **440V**.

Figure 15.2b Piezo Travel in Continuous ramp Mode

A: Z = Z Scan Start - Ramp Size

B: Z = Z Scan Start
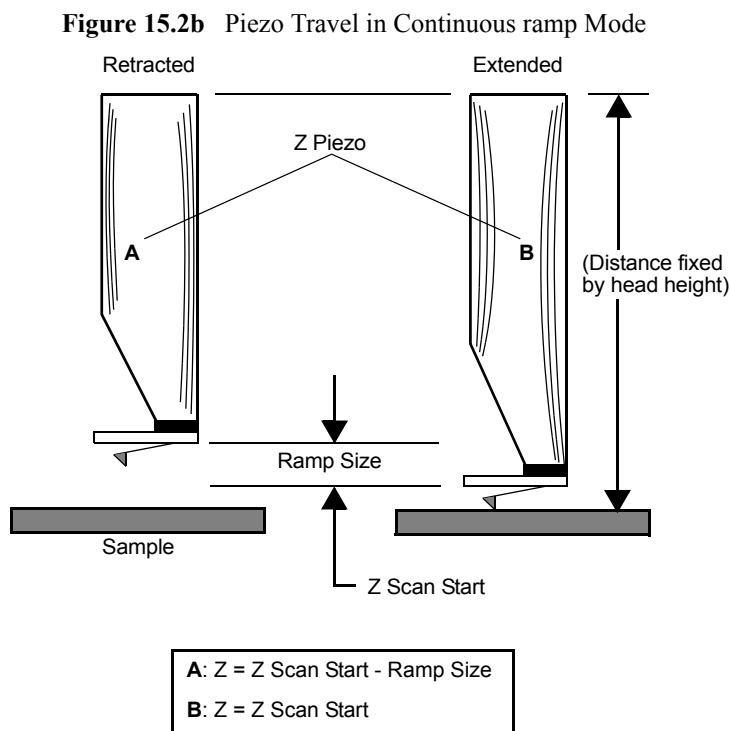
As the piezo moves the tip up and down, the cantilever-deflection signal from the photodiode is monitored. The force curve, a plot of the cantilever deflection signal as a function of the voltage applied to the piezo tube, displays in the **Force Plots** window. The controls detailing parameters for controlling the microscope in **Continuous ramping** mode are available in the **Force Plots** window and the **Ramp Parameters List** window.

Continuous ramp mode is frequently used to adjust and calculate contact forces between the cantilever and the sample. Other uses of **Continuous ramp** mode include characterization of the forces on the cantilever tip, diagnosing AFM performance, and determination of the sensitivity of the cantilever deflection voltage in terms of voltage applied to the piezo. If used correctly, **Continuous ramp** mode provides a variety of useful information.

15.2.1 Example Force Plot

A Contact Mode AFM force plot using a silicon nitride probe is the most simple SPM force plot. Because of the lower spring constant of silicon nitride probes, they are very sensitive to attractive and repulsive forces. A force plot in Contact Mode AFM is shown in Figure 15.2d.

Figure 15.2c compares portions of the force curve shown in Figure 15.2d to relative positions of the tip and sample at seven points. The force curve represents the deflection signal for each complete extension-retraction cycle of the piezo (Figure 15.2a). The **Z scan rate** parameter in the **Ramp Parameter List** defines the rate at which the piezo completes an extension-retraction cycle (and therefore the rate at which new curves are displayed).

Figure 15.2c   Tip-Sample Interaction During a Force Plot
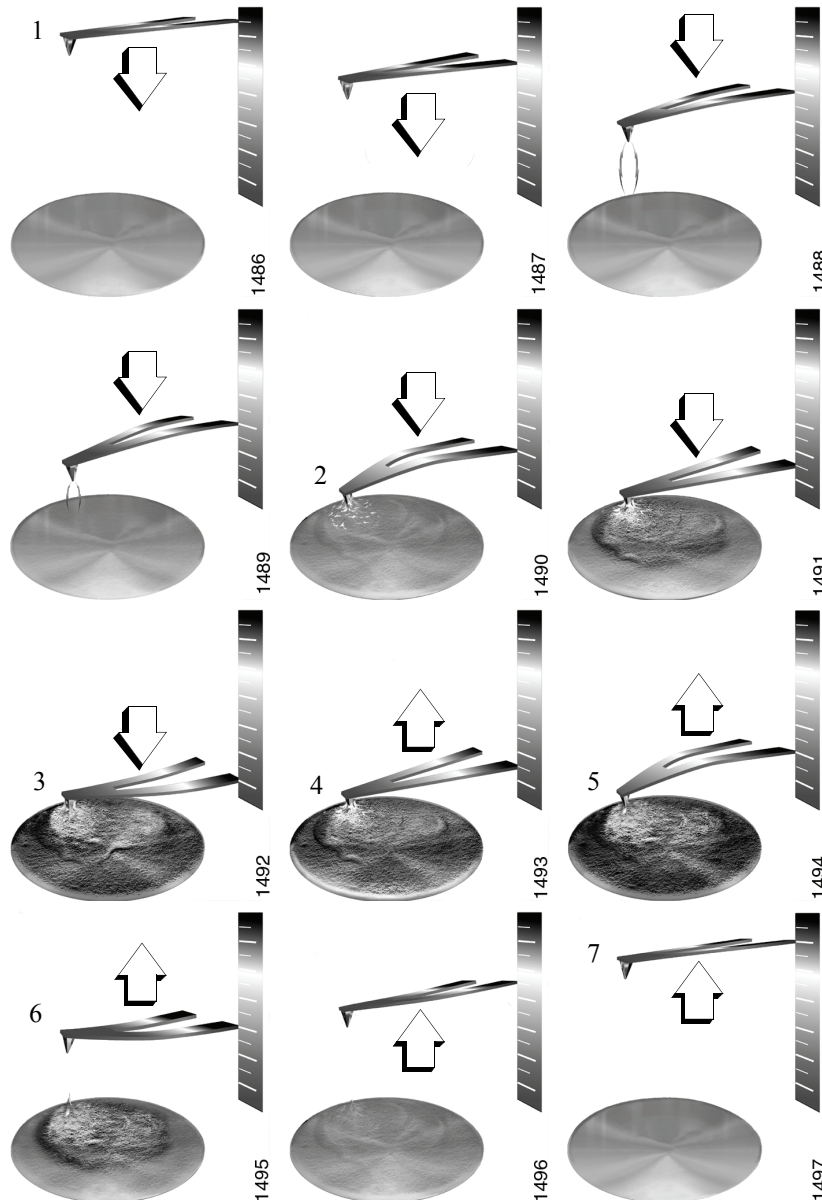
Figure 15.2d   Anatomy of a Force Curve
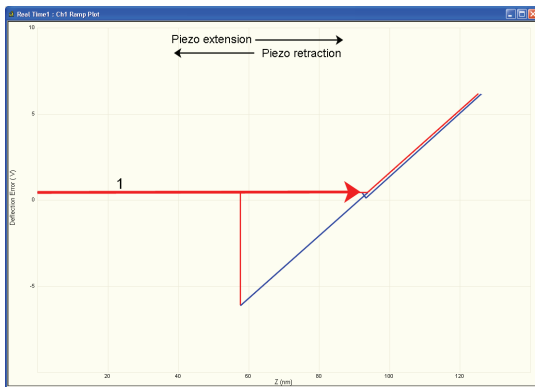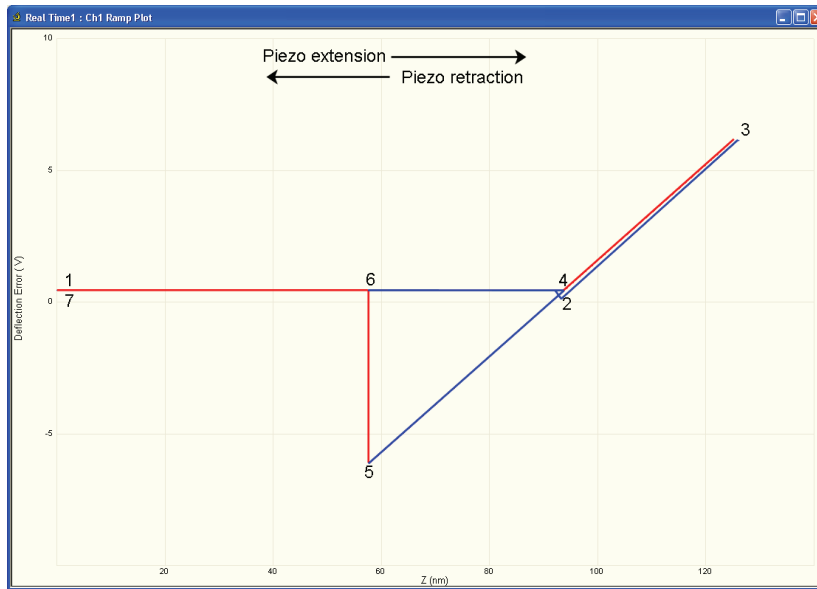
Piezo extends; tip descends.
No contact with surface yet.
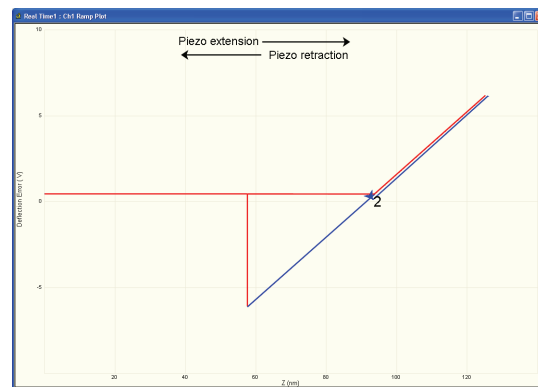
Attractive forces near surface
pull tip down.
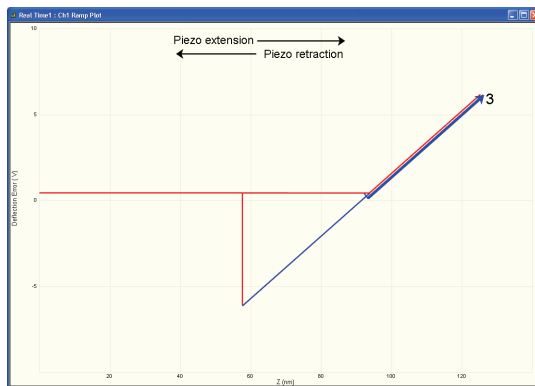
As tip presses into the surface,
cantilever bends upward.
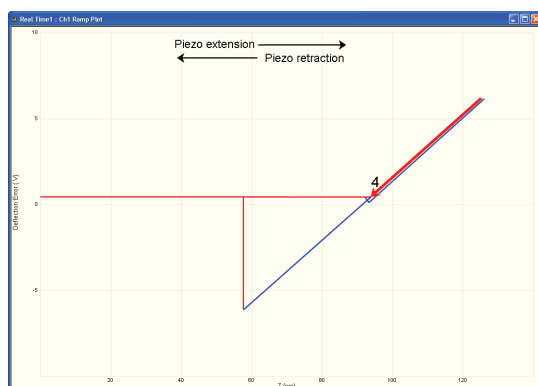
Piezo retracts; tip ascends until forces are in
equilibrium with surface forces.
Cantilever relaxes downward.

Figure 15.2e Anatomy of a Force Curve (continued)
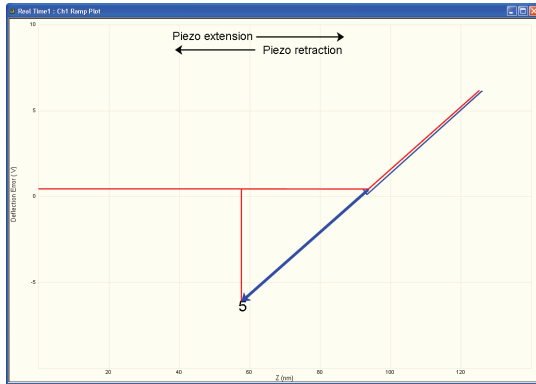
Piezo continues retraction; tip ascends further. Cantilever bends downward as surface attraction holds onto the tip.
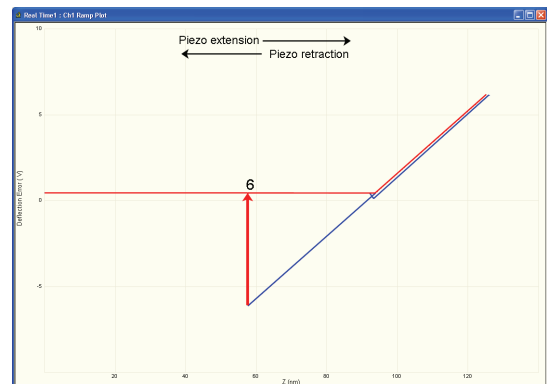
As tip continues its ascent, tip finally breaks free of surface attraction. Cantilever rebounds sharply upward.
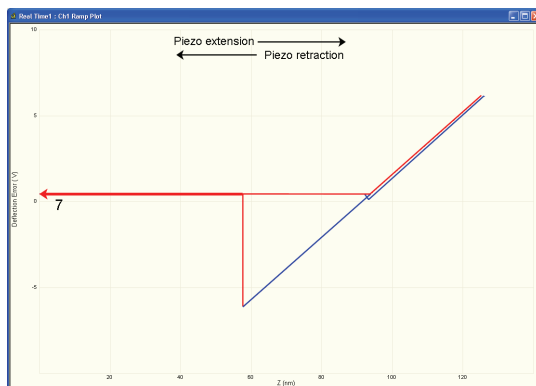
As piezo continues retracting, tip continues its ascent. No further contact with surface this cycle.

The horizontal axis plots the tip movement relative to the sample. By extending the Z-axis piezo crystal, the tip descends toward the sample and the tip-sample distance decreases. The descent plots from left-to-right in red in the NanoScope **Force Plots** window. By retracting the Z-axis piezo crystal, the tip ascends away from the sample and the tip-sample distance increases. The ascent plots from right-to-left in blue on the NanoScope **Force Plots** window.

Cantilever deflection plots on the vertical axis of the graph. When the cantilever deflects downward, it plots on the graph's downward vertical; when the cantilever deflects upward, it plots on the graph's upward vertical.

You can use force plots to adjust the setpoint so that minimal force is applied to the sample. Although attractive forces appear small, the tip is extremely sharp. Because only a few nanometers of the tip actually touch the sample, even minute forces add up quickly when distributed over an exceedingly small area. The tip can easily dent many materials under such conditions.

The graph detailed in Figure 15.2d reveals the following types of information:

Tip-Sample Attraction

As the tip approaches the sample, various attractive forces reach out and grab the tip. This is evident at point 2 (slight dip) in the graph above. The tip plunges toward the sample during its descent. This is also referred to as the "jump-to-contact" point and is usually due to electrostatic attraction or surface tension (capillary) forces.

Attraction is also evident between points 4 and 5 (sloped line) as the cantilever pulls away from the sample. If attractive forces are strong enough, the tip clings to the sample surface as it pulls clear. Eventually, the sample retracts and the tip rebounds sharply upward (white line between points 5 and 6). You can measure attractive forces of tip-sample interactions if you know the spring constant.

Material Elasticity

It is possible to extract information regarding the elasticity of the material by studying force curves. In the graph above, the tip is in constant contact with the sample between points 2 and 4. As the tip presses further into the sample material, the cantilever flexes. The amount of cantilever flexion for a given amount of downward tip movement indicates the material's elasticity.

For example, if the material is extremely hard, pressing the tip downward results in a relatively large amount of cantilever flexion. On the other hand, if the material is soft, the cantilever will flex less during its descent. The shape and slope of the contacted portion of the force curve gives detailed information about surface elasticity. It is possible to obtain quantitative measurements of sample elasticity. (See Radmacher, *et al*. 1994. *Science*, Vol. 265:1577-1579).

Two imaging techniques measure and display elasticity at multiple points on a sample surface: **force modulation** and **force volume imaging**.

15.3 Ramp Parameter List

The **Ramp Parameter List** window (see Figure 15.3a) manipulates the microscope in **Ramp** mode. The parameters control the rate, start position and amplitude of the triangle wave applied to the Z piezo. You can also adjust the **Setpoint** value of the cantilever deflection voltage used in the feedback loop during imaging. The **Capture** button stores the force curve for **Offline** viewing. Some parameters influence the operation of the microscope during imaging; most of the menu parameters affect only **Ramp** mode.

Figure 15.3a   Ramp Parameter List (Contact Mode AFM) Expanded Mode

To view the **Ramp Parameter List** click the RAMP icon in the **Workflow Toolbar**.

Items in the **Ramp Parameter List** window are discussed individually below.

15.3.1 Ramp Controls

Ramp Output

This parameter specifies which output you will ramp. To collect force plots, this parameter should be set to **Z**. For STM IV curves, **Bias** is typically selected.

Ramp Size

As shown in Figure 15.2a, this parameter defines the amplitude of the triangular waveform applied to the Z piezo. The units of this item are volts or nanometers, depending on the setting of the **Units** parameter. Regardless of the size of the scan, the entire scan is shown in the force curve; therefore, increasing the value of this parameter increases the horizontal axis scale on the force plot.

Z scan start

This parameter defines the maximum voltage applied to the Z electrodes of the piezo during force calibration operation. The triangular waveform shown in Figure 15.2a displaces the piezo up and down in relation to the value of **Z scan start**. Increasing the value of the **Z scan start** parameter moves the cantilever closer to the sample by extending the piezo tube. The units of this item are volts or nanometers, depending on the setting of the **Units** parameter. The initial value of **Z scan start** is equal to the average Z-center voltage defined by the feedback just prior to entering the **Ramp Parameter List**. Decreasing the value of this parameter shifts the force curve on the display to the right, while increasing the parameter shifts the curve to the left.

Scan Rate

This parameter defines the rate that force plots are collected (typically ~1Hz). The ramp size, forward velocity and reverse velocity determine the scan rate.

Forward Velocity

This parameter defines the piezo speed to bring together the tip and sample.

Reverse Velocity

This parameter defines the piezo speed to separate the tip and sample.

X Offset, Y Offset

These parameters define the X and Y positions where the force plot will be collected, offset from the image center.

Number of samples

This parameter defines the number of data points captured during each extend and retract operation of the Z piezo during a ramp. This parameter does not affect the number of samples used in **Image Mode**.

Spring Constant

The parameter defines the spring constant of the cantilever. The spring constant will be saved with captured force plots. The spring constant can be evaluated using the Thermal Tune method. Refer to the *NanoScope V Controller Manual* for details.

Plot Units

This item selects the units, either metric lengths or volts on the piezo, that define the parameters and the axes of the graph. Force may be displayed in nN of deflection force. This uses the spring constant. Changing this item in **Ramp** mode also changes it in the **RealTime** imaging mode.

Display Mode

This parameter defines the display as either **EXTEND**, **RETRACT**, or **BOTH**.

X Rotate

X Rotate allows you to move the tip in the X direction during ramping. This is useful because the cantilever is at an angle relative to the surface. **X Rotate** prevents the cantilever from plowing the surface laterally, typically along the X direction, while it indents in the sample surface (in the Z direction). Plowing occurs when the cantilever bends during indentation or with X movement caused by coupling of the Z and X axes of the piezo scanner. **X Rotate** moves the scanner in the opposite direction in which the cantilever points. Without **X Rotate** control, the tip moves in the direction of the cantilever during indentation. Normally, it is set to about **22.0 degrees**. This parameter typically ranges between **12** and **25 degrees**.

15.3.2 Channel 1, 2, 3 Panels

Data Type

This parameter allows you to select the type of data you want to display on the vertical axis. **Data Type**: Channels may be assigned to any input signal available including: **Deflection**, **Amplitude**, **Phase**, **Friction** and so on. Any channel may be switched to Off, however, *at least one channel* is always on.

Data Scale

This parameter sets the vertical scale in the force plot. Increasing this parameter expands the range of the display about the centerline causing more of the force curve to fall on the graph. Initially, set this parameter to its maximum value, then gradually reduce.

Data Center

This parameter shifts the displayed force plot in the vertical axis.

> **Note:** For **Data type** = **Deflection** in Contact Mode or **Data type** = **Amplitude** in TappingMode, the data is also shifted by the setpoint.(i.e., if the **Data type** = **Deflection** and **Data Center** = **0**, then the setpoint is at the center of the vertical scale.

TM Deflection Sensitivity (TappingMode)

TM Deflection Sensitivity relates the vibrational amplitude of the cantilever (in nm) to the amplitude signal (in V) from the controller. To calculate the **TM Deflection Sensitivity**, measure the slope of the **RMS amplitude** versus the **Z piezo position** when the tip is in contact with a very hard, stiff sample. The NanoScope software automatically calculates and enters the value from the graph. You must properly calibrate the **TM Deflection Sensitivity** before amplitude data can be correctly displayed in nm.

15.3.3 Feedback Panel

All of the parameters in the **Feedback** panel also affect **Image** mode.

Deflection Setpoint (Contact Mode)

By changing the deflection setpoint, you can adjust the cantilever deflection voltage maintained by the feedback loop in **Image** mode. This parameter defines the center of the range of Cantilever Deflection Voltages that can be collected. If the **Data Center** = **0**, then the setpoint is at the center line of the force plot. Changing the setpoint shifts the force calibration curve on the graph. For example, if the setpoint is set to -3.0V and the data center is 0, the cantilever deflection axis of the graph centers around -3.0V. Raising the setpoint to -2.0V shifts the force calibration curve down by one volt so the graph centers at -2.0V. Changing the value of this parameter also changes it in **Image** mode.

Amplitude Setpoint (TappingMode)

The RMS value of the cantilever deflection voltage maintained by the feedback loop in **Image** mode can be adjusted by changing **Amplitude Setpoint**. In a TappingMode force plot mode, the setpoint defines the centerline of the vertical, "Amplitude" axis of the amplitude calibration plot (the Data Center must be 0 for that Channel). Changing the **Amplitude Setpoint** shifts the

amplitude curve on the graph. For example, if the setpoint is at 3.0V, the RMS amplitude axis of the graph will be centered around 3.0V; raising the setpoint to 4.0V will shift the amplitude curve down by one volt, so the graph will be centered at 4.0V. Changing the value of this parameter in the **Amplitude Calibration** mode also changes the setpoint parameter in **Image** mode.

Z Limit

This parameter changes the gain applied to the input signal. You can reduce the input signal to see more of the **Force Calibration** plot deflection or amplitude. This parameter also affects **Image** mode and the resolution of the system; therefore, change the value back to the **Image** value before returning to imaging.

Drive Frequency (TappingMode)

This parameter defines the frequency at which the cantilever is vibrated. This frequency should be close to the resonant frequency of the cantilever. Changing the value of this parameter in the **Force Plot** menu also changes the **Drive Frequency** parameter in the **Image** mode.

Drive Amplitude (TappingMode AFM)

This parameter defines the amplitude of the voltage applied to the piezo system which drives the cantilever vibration. Changing the value of this parameter in **Force Plot** mode also changes the **Drive Amplitude** parameter in the **Image** mode.

15.3.4 Mode Panel

Trigger Mode

The **Mode** panel allows you to use various triggers when obtaining **Force Plot** and **Force Volume** plots. The idea of triggering is simple: it limits the total amount of force exerted by the tip upon the sample. Depending upon which trigger you use and how it is set, you may operate the trigger independent of drift (**Relative**) or at some arbitrarily fixed point (**Absolute**).

Figure 15.3b   Absolute and Relative Triggers
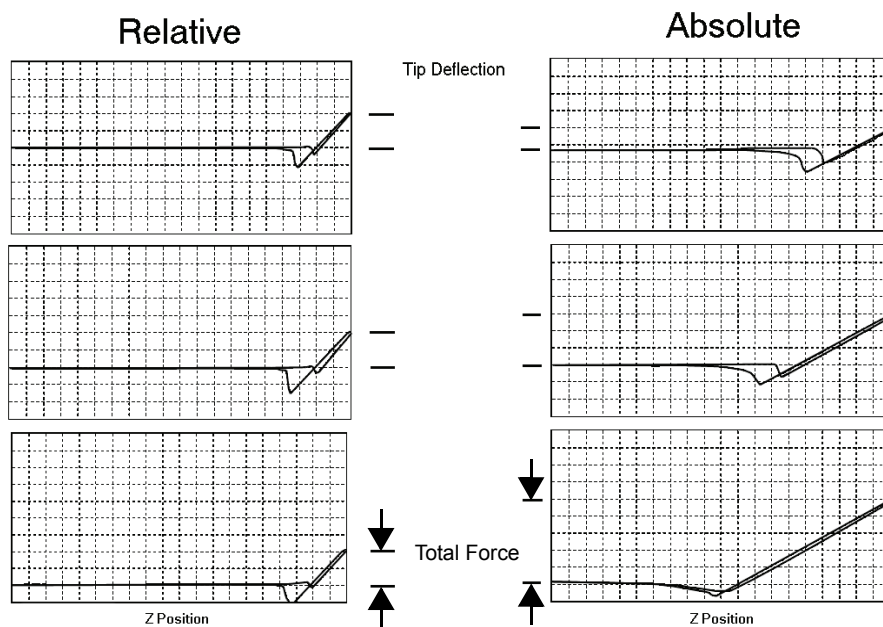
The plots in Figure 15.3b show the effect of drift on each of the two trigger types. The plot series shown on the left side of Figure 15.3b utilizes a **RELATIVE** trigger and maintains force at a constant level defined by the **Trig threshold** parameter. The **RELATIVE** trigger threshold is relative to the data at the right of the plot where the cantilever is typically undeflected. As illustrated, the total force is maintained, even as the system drifts.

The plot series on the right side of Figure 15.3b utilizes an **ABSOLUTE** trigger. Notice that as the system drifts (plot moves slowly downward), total force increases. Drift may be due to mechanical causes, or due to thermal effects on the cantilever. An **ABSOLUTE** trigger permits the total force to be set using **Setpoint** values.

Data Type

This parameter defines the Data Type to watch to determine when to reverse the ramp.

Trigger Threshold

This parameter as well as the **Trigger direction** and **Trigger mode** define the level at which triggering occurs.

Trigger Direction

The **Trigger Direction**s are defined as follows:

- **POSITIVE**: Trigger occurs on positive values only (**Trigger Threshold** must be positive)

- **NEGATIVE**: Trigger occurs on negative values only (**Trigger Threshold** must be negative)

- **ABSOLUTE**: Sign of **Trigger Threshold** is ignored (trigger may occur on positive or negative values).

Start Mode

The Start Mode parameters are defined as follows:

- **CALIBRATE** and **STEP**: Normal ramping (no stepping) unless you use **RAMP** > **RAMP SINGLE** or **RAMP CONTINUOUS**.

- **MOTOR STEP**: Uses the stepper motor to offset ramp instead of the piezo.

End Mode

The **End mode** parameters specify the location of the tip when the mode is switched back to Image mode. **RETRACTED** is the typical setting.

⚠️	**CAUTION:**	Use caution when using the **EXTENDED End mode** parameter; you could damage the tip.

Z Step Size

This parameter defines the size of Z step for **Step** or **Motor Step** modes. For **Step**, it is the amount to move the piezo; for **Motor Step** it is the amount to move the motor.

Auto start

When **Enabled** this parameter starts taking force curves upon entering Force Calibrate Mode.

Surface Delay

This parameter sets the amount of time to wait with the piezo extended before retracting.

Retracted Delay

This parameter sets a delay to occur each time the piezo is retracted while continuously ramping.

15.3.5 Menu Bar Commands

Capture

The **CAPTURE** button stores the force plot for **Offline** viewing. Use other options in the **Capture** menu to change a capture filename and for **CONTINUOUS CAPTURE** of all the force plots collected.

Probe Menu Commands

These commands allow for highly controlled tip movement designed to prevent damage to the tip. Each of these commands has an icon to access directly from the menu bar.

- **RAMP CONTINUOUS**: The tip is continuously lowered and raised by a distance equal to the Z scan size. This is the normal, default motion during **Force Calibrate**.

- **RAMP SINGLE**: Lowers and raises tip *once* by a distance equal to the Z scan size, then halts.

- **STOP**: Halts all tip movement.

Step Motor

The **Microscope > Step Motor** menu allows you to withdraw or manually control the tip position using the stepper motor. Selecting **Step Motor** opens a dialog box containing the following buttons:

- **Tip Up**: This command moves the tip up by the **SPM step size** displayed inside the window.

- **Tip Down**: This command moves the tip down by the **SPM step size** displayed inside the window.

15.4 Force Calibration (Contact Mode AFM)

15.4.1 Obtaining a Good Force Curve

Figure 15.4a   Typical Force Calibration Curve
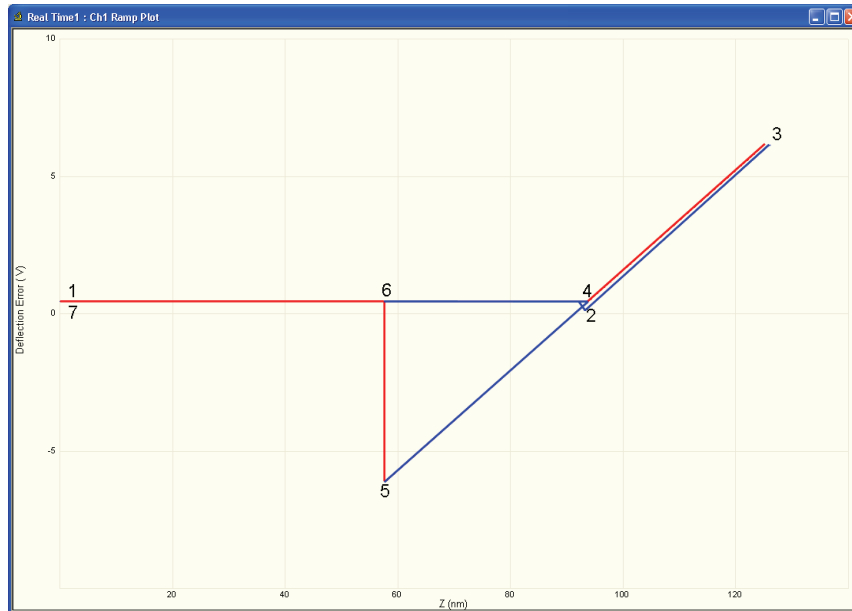
Figure 15.4b   Piezo Positions for Typical Force Curve
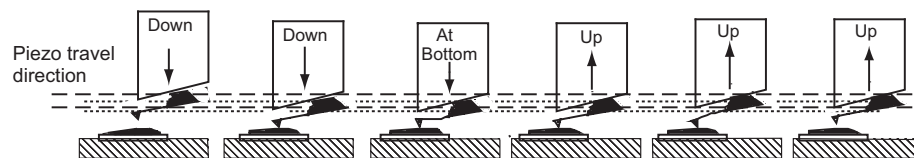
To minimize or calculate the contact force between the tip and sample, obtain a good force curve which shows the typical features displayed in the example curve in Figure 15.4b. However, the force curve rarely looks "typical" immediately after invoking the **Ramp Continuous** mode. This section discusses general approach adjustments to improve force curves obtained after engaging the microscope.

The basic approach to obtaining a good force curve entails adjusting the Z motion of the piezo relative to the sample (with the **Z scan start** and **Ramp size** parameters) and shifting the graph (with the **Setpoint** parameter) so the pull-off point of the tip displays on the graph. In general, use the following steps to obtain a good force curve:

1. **Engage** and verify the tip is tracking the surface. Then switch to **Ramp Continuous** mode.

2. Maximize the **Z Limit** parameter in the **Feedback** panel.

3. Select **DEFLECTION ERROR** as the **Data Type** for **Channel 1**.

4. Maximize the **Data Scale** parameter.

5. Adjust the **Ramp size** parameter to about **1μm**.

6. If the tip does not reach the sample surface (for example, see Figure 15.4a, between points 2 and 3), slowly increase the **Z scan start** value.

7. As the **Z scan start** increases, the traces on the force curve move to the right.

8. Adjust the **Setpoint** parameter.

   Note: Adjusting the **Setpoint** value moves traces up and down on the graph. Decreasing the value moves the curve up while increasing the value moves the curve down. (Remember that the red horizontal centerline of the graph is equal to the **Setpoint** if the **Data Center** = **0**, so that changing the **Setpoint** actually shifts the graph up and down.) Typically, changing the **Setpoint** value to **-2 volts** shows the desired features of the force curve. If it does not, decrease the **Setpoint** value further.

   Note: Sometimes the entire ramp will occur in the contact portion of the curve (see Figure 15.4a, between points 3 and 5). If this occurs, increase the **Ramp size** and/or decrease the **Z scan start**.

15.4.2 Helpful Suggestions

To minimize or calculate the contact force between the tip and sample, it is important to obtain a good force curve; however, there will be situations where you will not obtain a good force curve no matter how much you adjust the **Ramp size**, **Z scan start** and **Setpoint**. The following sections discuss a few such situations. Suggestions on working in **Ramp Continuous** mode are included.

False Engagement

Figure 15.4c illustrates a force curve resulting from a falsely engaged tip. The photodiode receives light reflected off the sample, causing an increase in the deflection signal until the signal equals the setpoint and the system "engages" (even though the tip is not on the surface). Interference in the reflected light causes the hump-shaped waveform.

The easiest way to correct a false engagement is to withdraw the tip, adjust the photodetector positioner to make the top/bottom differential voltage more negative, then re-engage the tip. This compensates for the slight slope in Figure 15.4c).

Figure 15.4c   False Engagement
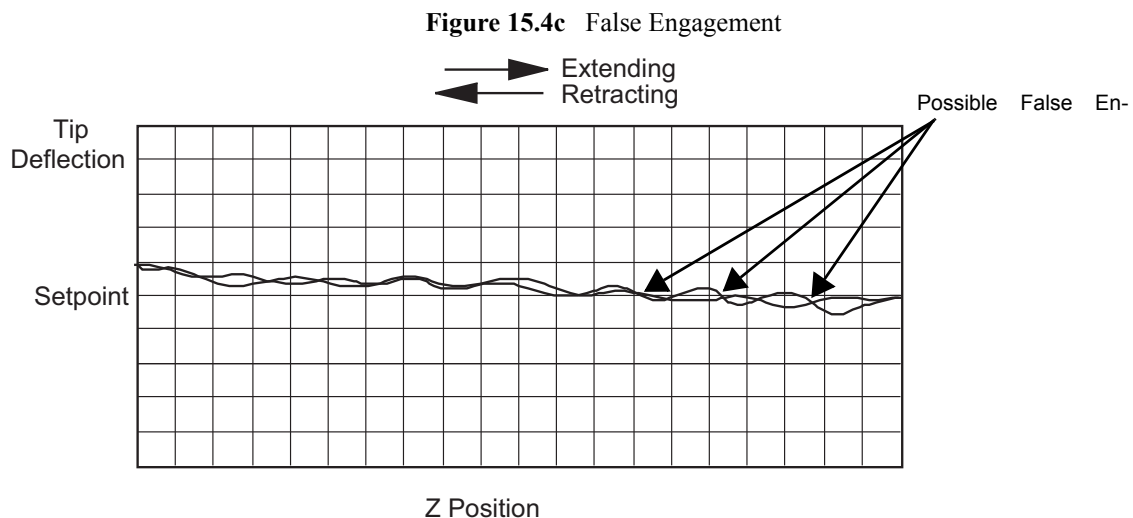
Motor Control

The **Microscope > Step Motor** > **Tip Up** and **Tip Down** buttons provide coarse adjustment of **Z center** voltage. These buttons move the SPM head vertically. The feedback loop causes the Z piezo to adjust to compensate for the head movement. If you use **Tip Up**, the Z piezo extends. If you use **Tip Down**, the Z piezo retracts.

> **Note:**   The **Step Motor** function is generally used only when the scanning range of the Z piezo is exceeded or when it is necessary to position a force measurement in the center of the scanner range. Because the Z-axis leadscrew has some backlash, it may be necessary to rotate the leadscrew several turns by clicking on the **Tip Up** or **Tip Down** buttons before obtaining movement.

Adjust Photodiode

In an analogous manner, you can use the photodiode positioner as a coarse adjustment for **Setpoint** voltage. Changing the laser beam position on the photodiode by rotating the photodiode adjustment knobs shifts the force curve on the graph. Rotating the top photodiode adjustment knob clockwise shifts the curve up, just as decreasing the **Setpoint** parameter shifts the curve up. Conversely, rotating the top photodiode adjustment knob clockwise moves the curve down.

15.4.3 Advanced Techniques

Sensitivity Determination

The **Deflection Sensitivity** allows conversion from the raw photodiode signal (in Volts) to deflection of the cantilever (in nm), and is normally set from the **Ramp Continuous** mode. The sensitivity must be calibrated before accurate deflection data can be obtained. Sensitivity is equal to the inverse of the slope of the force curve while the cantilever is in contact with a hard sample surface. Complete the following steps to calculate the sensitivity:

1. Obtain a good force curve using a hard sample so that the sample does not deform.

2. Drag vertical cursors from the plot edge to contact.

3. Click **RAMP > UPDATE SENSITIVITY** to calculate the cantilever sensitivity (see Figure 15.4d). Click **YES** to accept the computed sensitivity.

Figure 15.4d Set the Sensitivity Parameter
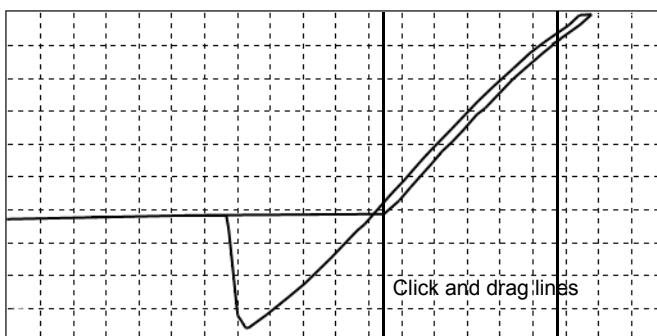
If **Sensitivity** is calibrated on a material much stiffer than the cantilever, it measures the inverse of the value of the AFM's optical lever sensitivity; i.e., how many volts of deflection signal are produced by a given deflection of the cantilever tip. The sensitivity will change for different cantilever lengths and styles (shorter cantilevers give lower **Sensitivities**). **Sensitivity** will also change with the position of the laser on the cantilever and the quality of the laser beam reflection from the cantilever.

Note: It is important to calibrate the **Sensitivity** parameter on a hard substrate as described here BEFORE using the force curve's vertical scale for quantitative measurements.

Note: **Deflection Sensitivity** can be expressed in terms of the photodiode voltage versus the distance traveled by the piezo, or the photodiode voltage versus the voltage applied to the piezo, depending on the setting of the **Units** parameter.

Force Minimization

The **Ramp Continuous** mode allows minimization of the contact force of the cantilever on the sample surface. The force curve clearly illustrates the relationship between the **Setpoint** and the cantilever deflection voltage when the cantilever is off the sample surface. You can adjust the **Setpoint** to set the normal deflection of the cantilever and, therefore, the normal force applied by the cantilever during data collection.

You can run the microscope below the point of zero deflection of the cantilever to minimize the contact force of the cantilever on the sample. It is possible to get a negative deflection whenever the cantilever sticks to the surface. To engage, you must the set **Setpoint** greater than the deflection voltage when the tip is not interacting with the surface. However, you can change the setpoint after entering **Ramp Continuous** mode; you can lower the setpoint to the zero cantilever-deflection point and beyond, while viewing the force curve. You can adjust the setpoint (most often made more negative) so that it lies between the flat segment of the force curve which corresponds to the zero deflection point, and the tip of the retraction scan where the cantilever pulls off the sample surface V_{CSmin} (see Figure 15.4e). The contact force is at its minimum when V_{CSmin} is on the centerline of the deflection-signal axis (**Setpoint** = V_{CSmin}).

In practice, V_{CSmin} must be a little below the centerline because V_{CSmin} is the point where the cantilever pulls off the surface and operation at this deflection is unstable. Changing the **Setpoint** option in the **Feedback** panel changes the setting of the **Setpoint** parameter in its **Image Mode** counterpart when you exit the **Ramp Continuous** mode. After exiting, if the image looks good, you can decrease the force further by lowering the **Setpoint** in small increments until the cantilever pulls off the sample surface. Resetting the **Setpoint** to a value higher than the voltage when the tip is not interacting with the surface, recaptures the cantilever. (Slowly adjust to a more positive value until the tip is back on the surface.) Adjusting the **Setpoint** a few tenths of a volt above the point where the cantilever pulled off provides a low contact force.

If a high initial contact force adversely affects the sample, engage the cantilever with a very small scan size. Then, minimize the force while the tip is confined to a small area of the sample where it experiences the relatively high initial engagement force. Once the force is minimized, increase the **Ramp size** or offset the scan to a different area of the sample. However, keep in mind that if the force is minimized in a smooth area of the sample, the cantilever may pull off when it translates to a rougher part of the sample.

Calculating Contact Force

The force curve clearly shows the relationship between the setpoint and the deflection of the cantilever. Because the setpoint defines the value of the deflection signal maintained by the feedback loop, the force curve can be used to calculate the contact force of the tip on the sample if the spring-constant, k, of the cantilever is known. The contact force is defined by the equation:

$$F = k \times d$$

where "d" is the deflection measured from the setpoint to V_{CSmin} in nanometers. An example of how to compute the contact force from the **Force Plot** graph is shown in Figure 15.4e.

Figure 15.4e Computing Contact Force
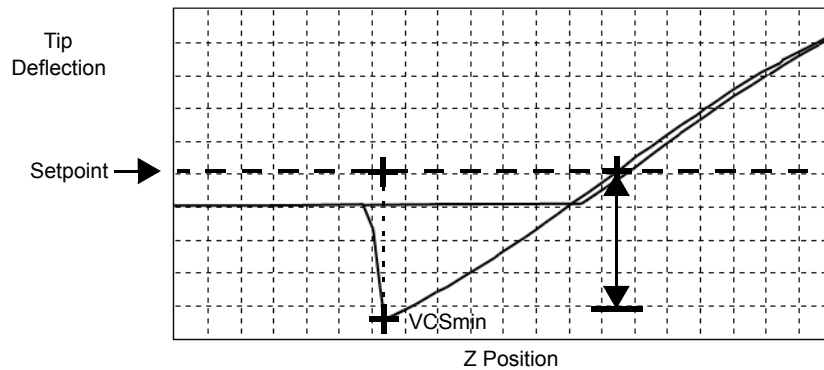
Recalling that contact force $F = kd$, we can calculate the contact force from the sample plot above. Let us assume, for example, that the spring constant of the cantilever is $k = \dfrac{0.6N}{m}$ and that deflection sensitivity $= \dfrac{70nm}{V}$. The plot above may be measured at the points where the retract portion of the curve intersects the setpoint to the pull-off (point). The distance is then multiplied times the deflection sensitivity to obtain d. In this example:

$$d = (4.5 \text{ div}) \, (1V/\text{div}) \, (70nm/v) = 315nm$$

Therefore, the contact force is calculated as:

$$F = (0.6N/m) \, (315nm)$$
$$= 189nN$$

When the **Data Type** is set to **Height** with the feedback gains set high, the tip tracks the sample surface with nearly constant deflection of the cantilever. When the cantilever deflection is constant, the force is constant and the force calculation above determines the contact force between the tip and the surface over the entire scan area.

Force calculations are not as straightforward on images captured with the **Data Type** set to **Deflection Error**. When collecting deflection data, the feedback gains are ideally set low so the sample stays at a nearly constant position relative to the cantilever holder. In this case, the cantilever deflection (and therefore the force applied to the sample) varies as features on the surface are encountered. The contact force can be calculated by adding the deflection data from the image to d before multiplying by the spring constant: $F = k$ (deflection image data + d). Note that the **Sensitivity** parameter must be accurate—that is, previously determined and entered—before the deflection data will be accurate.

A simple alternative to calculating force is to set the units to force.

15.4.4 Interpreting Force Curves

An examination of force curves can prove useful in determining adhesion, hardness, and elastic characteristics of samples. The examples in Figure 15.4f represent some of the general variations in force curves. For more information regarding force imaging, refer to Veeco's application note *Probing Nano-Scale Forces with the Atomic Force Microscope*.

Figure 15.4f Force Curve Examples
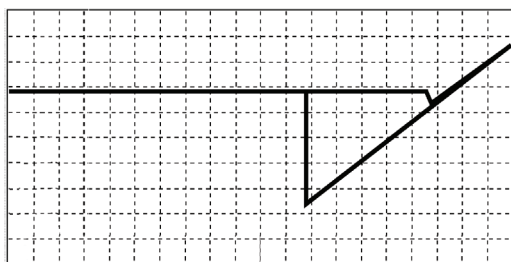
Large adhesion
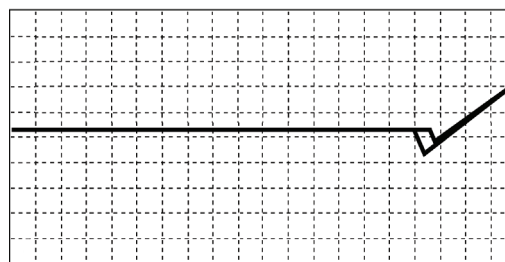
Small adhesion
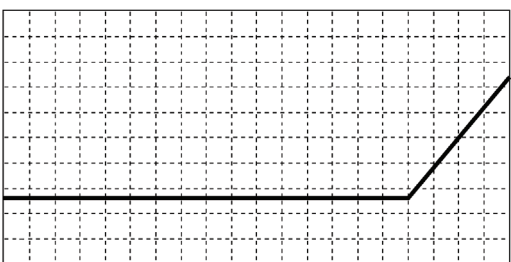
Stiff sample
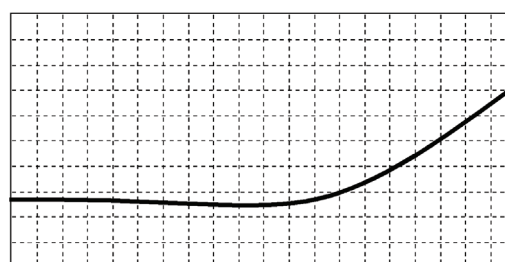
Soft sample
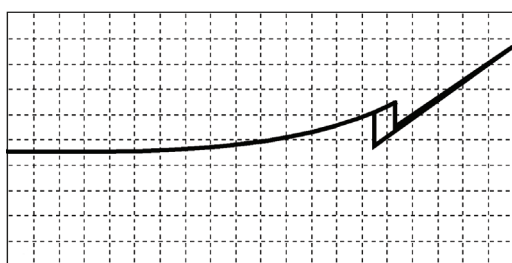
Long-range repulsion
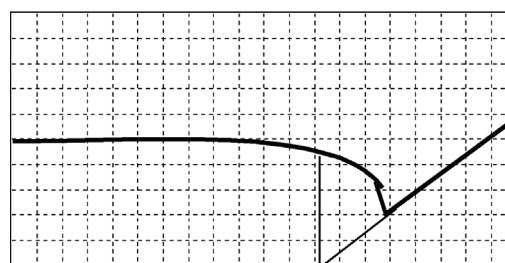
Long-range attraction
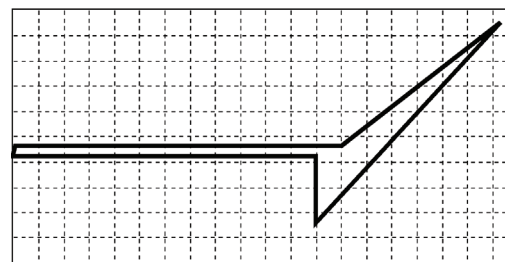
Plastic Deformation

15.5 Force Calibration (TappingMode)

> ⚠️ **CAUTION:** Because TappingMode cantilevers are relatively stiff, **Force Mode** can potentially damage the tip and/or surface. Before using Force Calibration, read and understand the following section.

Force Mode allows the imaging of forces between the tip and surface, including chemical bonds, electrostatic forces, surface tension and magnetic forces. In TappingMode, you can observe forces by measuring changes in tip RMS amplitude, phase, or TM deflection. The user may collect force plots in one of two forms: **Force Plot** and **Force Volume**. The two forms are similar, with **Force Volume** generating a map of many individual force plots. To produce high-quality force plots, it is necessary to precisely control tip position relative to the surface.

15.5.1 Force Plots

When performing **Force Plot** in TappingMode, the piezo moves to the center of the current X-Y scan, then turns off the X-Y scan motion. Next, a triangular waveform is applied to the Z electrodes of the piezo tube resulting in the oscillating tip moving up and down relative to the sample. The same Z-axis piezo motion occurs in Contact Mode AFM force plots. However, in TappingMode, the force plot is a graph of the piezo's extension versus oscillating tip amplitude, phase or TM deflection, instead of deflection or friction.

Uses of **Force Calibration** in TappingMode include characterizing forces on the cantilever tip, diagnosing SPM performance, calibrating the RMS amplitude or TM deflection, and configuring **Lift Mode**. For example, as the oscillating tip is brought closer to the surface, tip motion is dampened, which shows as an immediate drop in amplitude. When plotted, the graph resembles Figure 15.5a.

Figure 15.5a   Piezo Extension Versus RMS Amplitude and Deflection
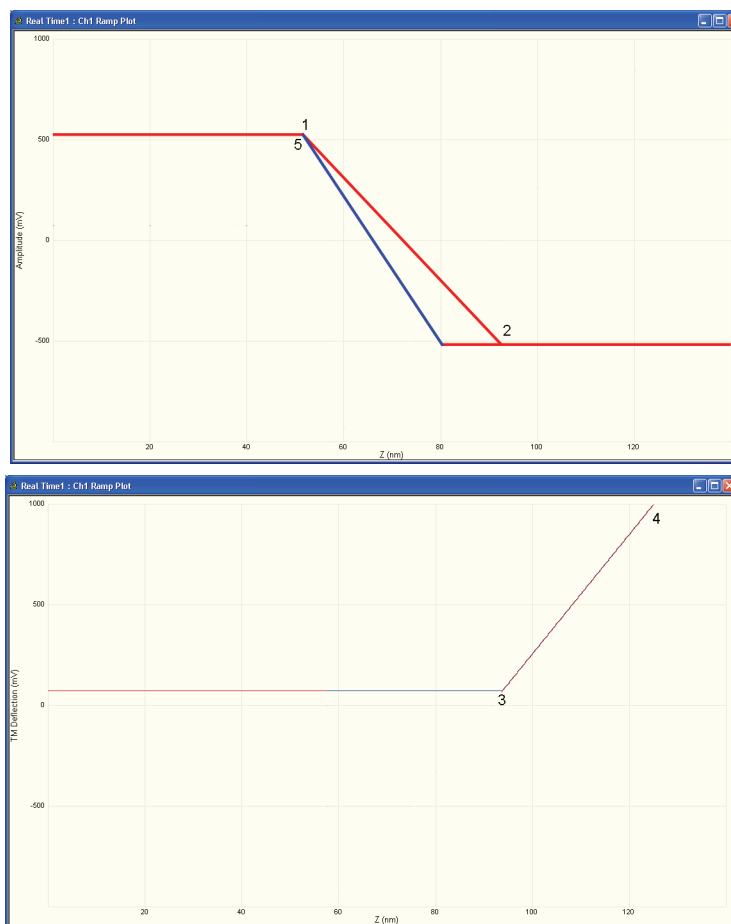
Figure 15.5a illustrates a two-channel TappingMode force plot. The vertical axes of the graphs represent the amplitude (top) and TM deflection signal (bottom) of the cantilever. The position of the Z piezo plots along the horizontal axis. **Channel 1** (top) demonstrates how the cantilever amplitude decreases as the tip moves closer to the sample. The plot represents the amplitude for one complete extension-retraction cycle of the piezo. The **Scan Rate** parameter in the **Ramp** panel defines the rate at which the piezo completes an extension-retraction cycle; therefore, the rate at which new curves display. At point 1, the tip encounters the sample surface. Upon encountering the surface, the tip oscillation amplitude decreases, dropping off as the tip moves closer to the sample surface. Between points 1 and 2, voltage to the Z piezo is increased, bringing the tip about 50 nm closer to the surface. Over the same interval, the cantilever amplitude diminishes about 2 volts due to dampening effects.

Collecting a force plot on a hard sample and dividing the change in amplitude by the change in Z piezo position gives the responsiveness of the electronics that measure the amplitude. The **Amplitude Sensitivity** value in the **Ramp** menu is the inverse of the responsiveness. You can determine this value by using the mouse to drag vertical markers from the edge (between points 1 and 2 where the tip amplitude dampens) and then selecting **RAMP > UPDATE SENSITIVITY**. Tip damping occurs as a result of mechanical-acoustic coupling between the tip and sample. As the z piezo is extended further, oscillation eventually ceases and the amplitude drops to zero (point 2).

For **TappingMode** in air on a hard sample, each nanometer decrease in the cantilever position decreases the peak-to-peak vibration of the cantilever by two nanometers. Once the tip encounters the sample surface, the oscillation amplitude of the cantilever decreases as the piezo is extended. When the piezo turns around and begins to retract, the oscillation amplitude of the cantilever increases until the tip is free of the surface, leveling off at the free-air amplitude (point 5).

Channel 2 (bottom) in Figure 15.5a plots average cantilever deflection (TM deflection) versus piezo extension. The deflection signal is low-pass filtered to eliminate the high-frequency TappingMode oscillation. Even as tip RMS amplitude dampens during its encounters with the sample surface, the average deflection is unchanged. This condition changes once the tip is so close to the sample that all oscillation ceases. Extending the piezo still further causes the average deflection to increase, applying an increasing force to the sample (point 2).

At point 3 in Figure 15.5a, the cantilever begins to deflect. The region between points 3 and 4 may be hazardous to the tip, because the tip is pressed firmly against the sample surface. Most single crystal silicon TappingMode tips rapidly become dull in this region, depending upon the hardness of the sample.

15.5.2 Obtaining a Force Plot (TappingMode)

⚠️	**CAUTION:**	Use **Ramp Continuous** with caution or when it is important to obtain experimental information shown in **Force Calibration.** When using stiff TappingMode cantilevers, it is easy to blunt the tip with excess contact force during **Force Calibration** measurements.

When obtaining force plots in TappingMode, set up scan parameters so that the reduction of amplitude is minimal (approximately 25 percent of the free air amplitude). If the amplitude is reduced to zero, the tip and sample may sustain damage.

To generate a TappingMode force plot of a silicon calibration reference, complete the following steps:

1. Verify the cantilever holder is loaded with a TappingMode tip.

2. Mount the calibration reference on the SPM stage.

3. Click the **SELECT EXPERIMENT** icon.

4. Select **TAPPINGMODE MODE AFM** in the **Experiment Category** panel.

5. Select **TAPPINGMODE IN AIR** in the **Experiment** panel and click **START EXPERIMENT**.

6. Tune the cantilever.

7. Engage. and obtain a TappingMode image.

> **Note:** You are now in **Image mode**.

8. To switch to **Force Mode**, click the RAMP button in the **Workflow Toolbar**. Two panels should be visible:

   • **Force Plots**

   • **Ramp Parameter List**

9. Set the **Ramp Output** to **Z**. This is the default in the **Simple Mode**.

10. Set the **Channel 1 Data Type** to **TM DEFLECTION**.

11. Set the **Channel 1 X Data Type** to **Z**.

12. Adjust the **Z scan start** parameter to obtain a satisfactory force plot using the left-right arrow keys.

15.5.3 High Contact Force

Figure 15.5b shows a curve produced when the tip pushes too far into the sample. The flat portion on the right side of the amplitude curve in Figure 15.5b occurs because the tip is so close to the surface that it no longer vibrates. As the piezo extends the tip further, the amplitude of vibration does not change because the tip is always in contact with the sample surface. The contact force is very high due to the stiffness of the TappingMode cantilevers. The tip may shatter if deflection continues.

Figure 15.5b   Amplitude Force Plot with High Contact Force
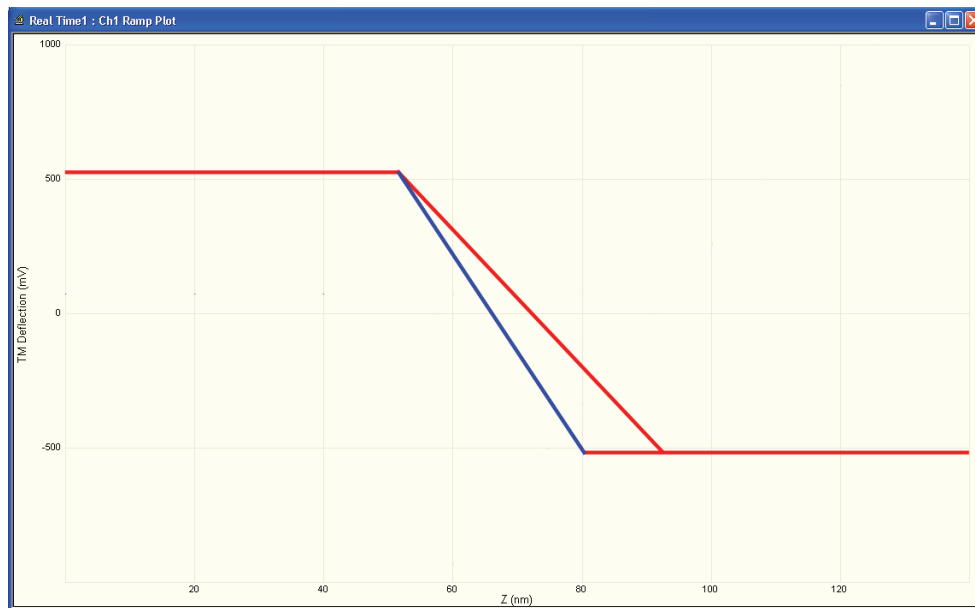
You can avoid this situation by using triggers (see Section 15.3.4) or by reducing the value of **Z scan start** until there is no flat portion on the right side of the curve. Rapidly increasing the value of **Z scan start** is dangerous because the total oscillation amplitude of the cantilever is small relative to the total Z travel of the long-range scanner.

15.5.4 Probe Selection

You may use virtually any TappingMode tip to obtain TappingMode force plots; however, the ultimate choice depends upon the delicacy of the sample and the magnitude of the forces to be gauged. Longer cantilevers have lower spring constants (i.e., they are more pliant) and therefore offer greater sensitivity for most samples. Shorter cantilevers afford better control when gauging strong attractive forces and are less prone to entrapment by surface tension forces. Sharper tips are more likely to plastically deform the sample, and are less sensitive to adhesive forces, than tips that are more blunt. Experiment to determine the tip that best meets your needs.

15.6 Force Modulation

15.6.1 Introduction

This section describes the operation of force modulation mode, which you can use to image local sample stiffness or elasticity. This method is useful for imaging composite materials or soft samples on hard substrates where you can obtain contrast between regions of different elasticity. This section assumes knowledge of operation of Contact Mode AFM in air (see Chapter 10). It is useful, but not essential, to have experience operating in TappingMode (see Chapter 11).

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 15.6a.

Figure 15.6a   Contrast Generation in Force Modulation Mode
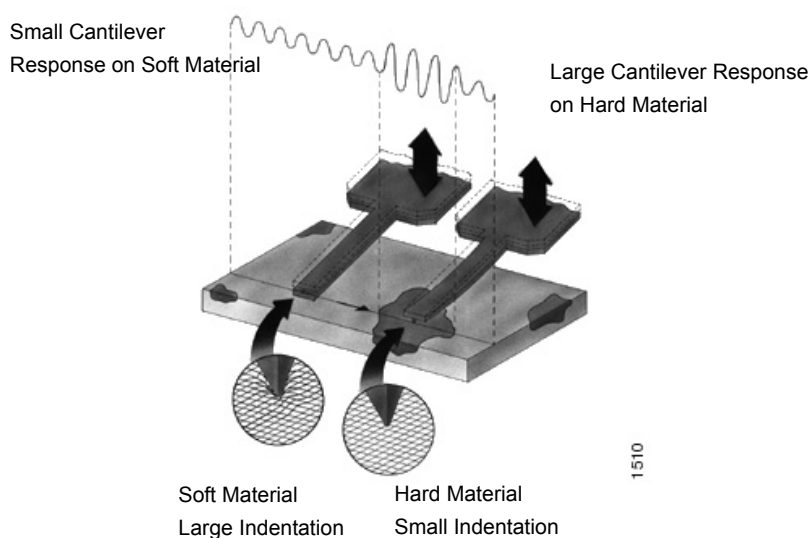
Force modulation requires the use of a special optional cantilever holder, shown in Figure 15.6b. This cantilever holder uses a piezoelectric bimorph to oscillate the cantilever against a sample surface. The force modulation cantilever holder is similar to the standard tapping cantilever holder; however, its piezo stack is much larger, allowing larger driving amplitudes.

Figure 15.6b   Force Modulation Cantilever Holder
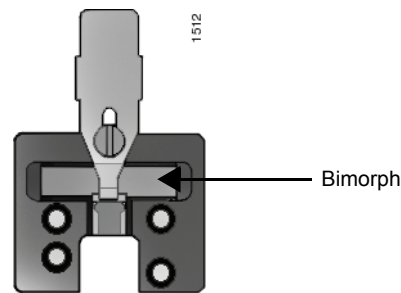
15.6.2 Selecting a Force Modulation Probe

The key consideration when selecting a force modulation cantilever is its spring constant. Ideally, the cantilever must have a spring constant which complements the pliancy of the two contrasting materials (or close to the pliancy of one, but not the other). This way, the tip indents into one material more than the other providing good force modulation image contrast. If the tip is so stiff that it indents equally into both materials, or so soft that it indents neither material, then you will not see contrast in the force modulation image. Instead, the image will consist primarily of edge and frictional artifacts. It may take experimentation to find a cantilever that matches the sample's requirements. For rubber and plastic samples Veeco recommends using 225μm long force modulation (Model # FESP) silicon cantilevers. For more delicate, samples, use 450μm long silicon cantilevers or silicon nitride cantilevers. For hard materials, use stiffer tips like the 125μm single crystal silicon TappingMode tips.

Veeco offers cantilevers with a wide range of spring constants (see Table 15.6a). Choosing a probe depends upon how stiff the sample is For samples of unknown hardness, start with a force modulation cantilever (Model FESP) and determine whether the probe is sufficiently stiff then adjust accordingly.

Table 15.6a Force Modulation Probes

Cantilever	Model No.	Cantilever Length	Spring Constant	
Standard Silicon Nitride	NP, DNP	100—200μm	0.01—0.6 N/m	SOFTER
Oxide-sharpened Silicon Nitride	NP-S, DNP-S	100—200μm	0.01—0.6 N/m	
Contact AFM Etched Silicon	ESP	450μm	0.02—0.1 N/m	
Force Modulation Etched Silicon	FESP	225μm	1—5 N/m	
TappingMode Etched Silicon	LTESP	225μm	20—70 N/m	HARDER
TappingMode Etched Silicon	TESP	125μm	20—100 N/m	

15.6.3 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 is oscillated up and down by a piezoelectric bimorph in the probe holder so that the tip indents slightly into the sample surface as it is scanned across the surface. The NanoScope system records the amplitude of the cantilever motion, which indicates the relative indentation of the tip into the surface. For softer samples the tip penetrates further into the surface resulting in a smaller change in the angle of the cantilever. A small change in angle results in a small measured amplitude which displays as a bright area on the image. For harder samples the tip penetrates less into the surface resulting in a larger change in angle of the cantilever. A large change in angle causes a large measured amplitude which displays as a dark area on the image.

15.6.4 Force Modulation Procedure

This section gives instructions for operating in **Force Modulation** mode.

15.6.5 Select Mode of Operation

1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 15.6c.

Figure 15.6c The **Select Experiment** window
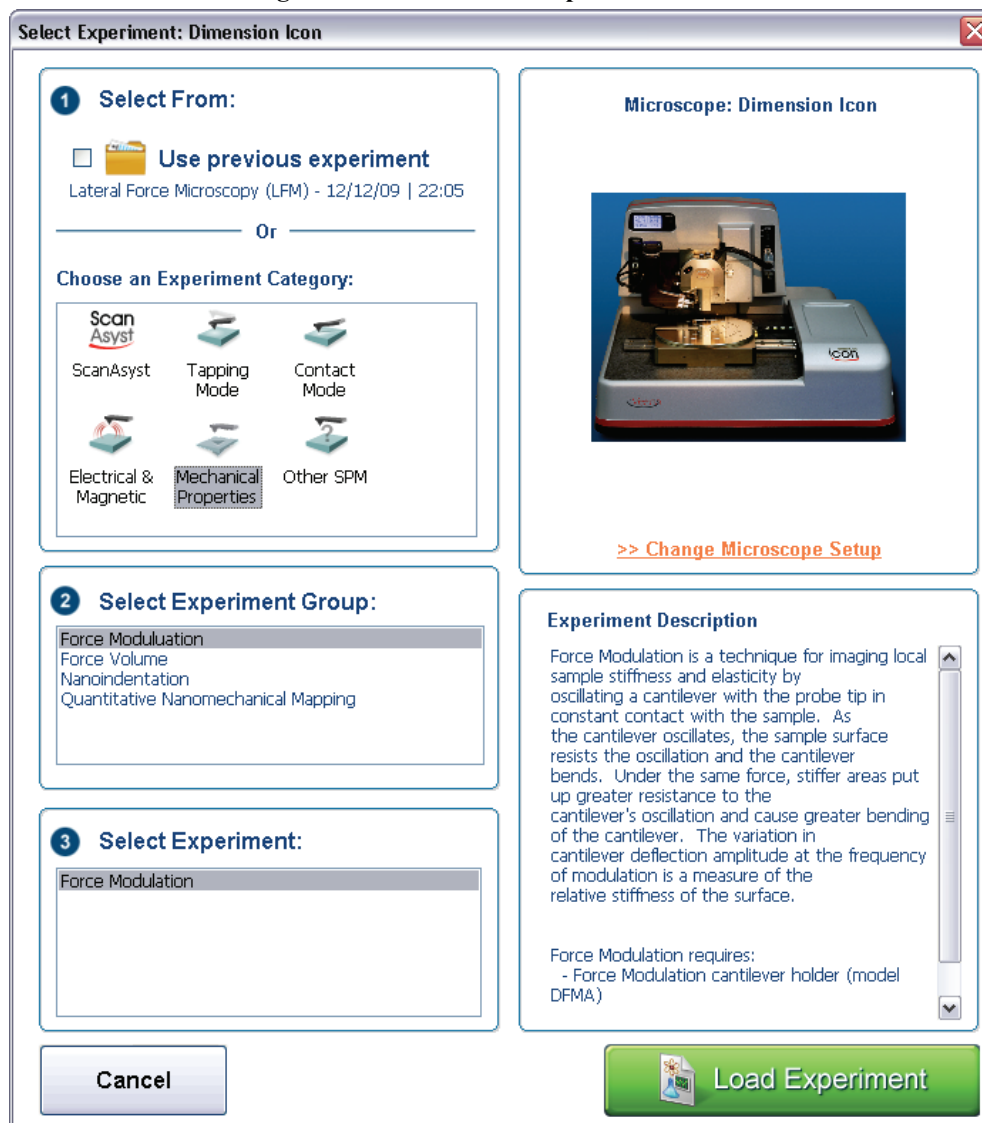
2. Select **MECHANICAL PROPERTIES** in the **Choose an Experiment Catagory** panel.

3. Select FORCE MODULATION in the **Select Experiment Group** panel.

4. Select FORCE MODULATION in the **Select Experiment** panel and click LOAD EXPERIMENT.

5. This opens the **Workflow Toolbar**, the **Scan 4 Channels** (Icon) or the **Scan 8 Channels** (Icon-PI) windows and the **Scan Parameters List** window, shown in Figure 15.6d.

Figure 15.6d Force Modulation (Simple Mode) configuration
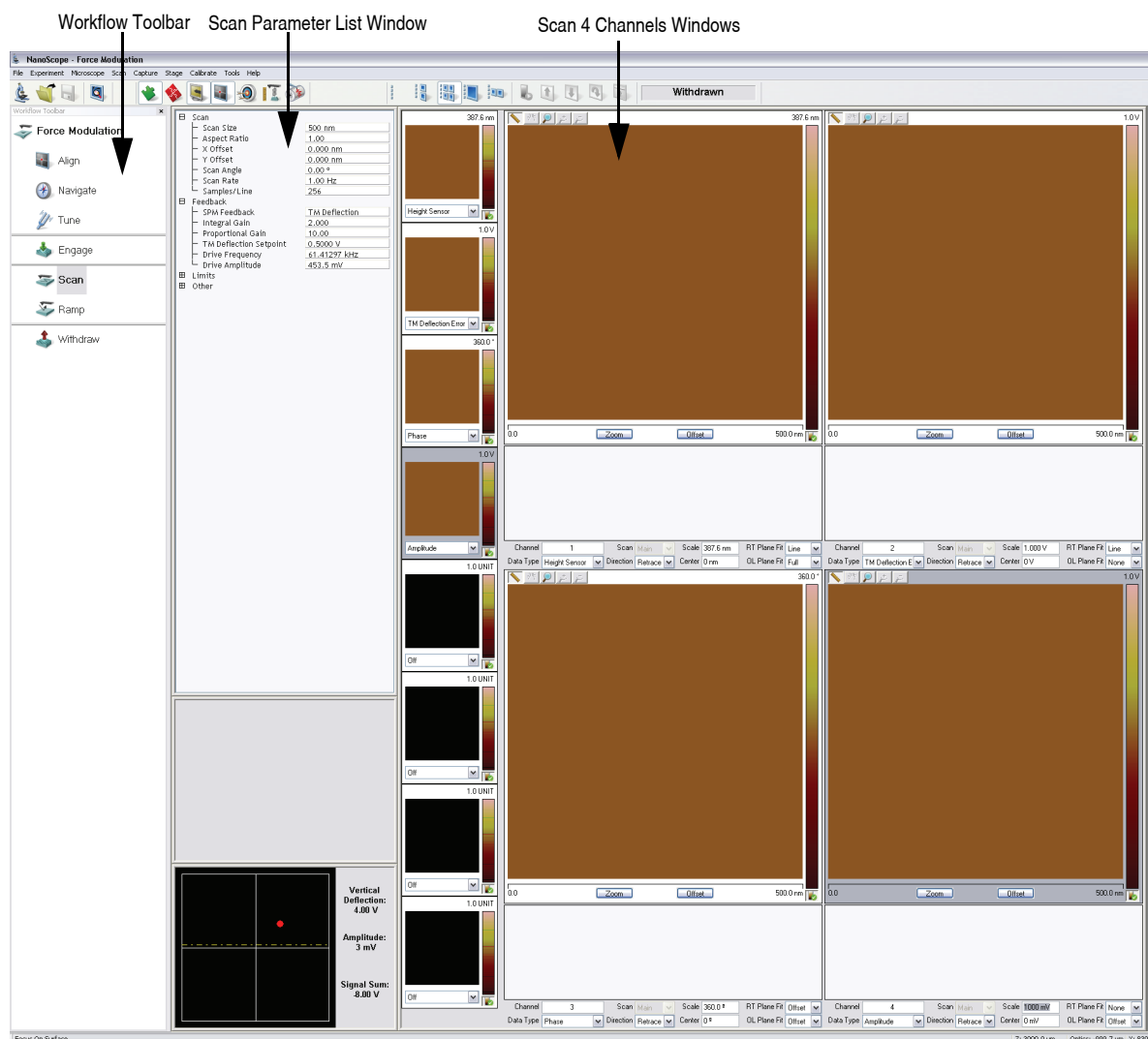
Note: Figure 15.6d shows the single (30 in.) monitor Dimension Icon interface. The dual (19 in.) monitor interface used by the Dimension Icon-PI is slightly different. Refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000 for additional information.

15.6.6 Head, Cantilever and Sample Preparation

- Load the special force modulation cantilever holder with a cantilever (see Chapter 7).

  Note: Refer to the section on Probe Selection earlier in this chapter to help you choose an appropriate cantilever.

- Load the cantilever holder with installed tip onto the scanner tube of the Dimension SPM head. See **Basic AFM Operation:** Section 7.2.

15.6.7 Align Laser

- Align the laser using the laser control knobs (See **Align Laser:** Section 7.2.9. for more detailed instructions).

- 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 at least **1V**.

15.6.8 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 left side of the Dimension head. See **Adjust Photodetector:** Section 7.2.10.

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

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

15.6.9 Locate Tip

To locate the tip, complete the following steps:

1. Click the **ALIGN** icon in the **Workflow Toolbar**.

2. Click the **ALIGNMENT STATION** icon in the **Align** window.

3. Initiate the **Locate Tip** command in the **Align** menu by clicking on **Locate Tip**.

4. Zoom out as far as possible using the trackball or **Zoom Out** button. This helps you locate the cantilever in the optical field of view.

5. Using either the trackball while holding down the bottom-left button or the **FOCUS UP** and **FOCUS DOWN** buttons in the **Focus Optics** panel, shown in Figure 5.2f, move the focus up or down to focus the cantilever. See **Align:** Section 5.2.1 and **Locate Tip:** Section 7.2.11 for detailed instructions for locating the 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.

15.6.10 Focus Surface

1. Click the **NAVIGATE** icon in the **Workflow Toolbar**.

2. Focus on the surface by clicking the **Focus Surface** icon in the **Navigate** window.

3. Focus on the sample surface either with the Focus: Z Motor arrows, shown in Figure 5.2j, or 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.

4. 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. be careful when performing this in the downward direction. Focus on the sample surface using the trackball with the bottom-left button depressed. See **Navigate:** Section 5.2.2 and **Focus Surface:** Section 7.2.13 for details.

> **CAUTION:** Because the command **Focus Surface** moves the scanner vertically, be careful when making this adjustment to ensure that the tip does not hit the sample surface.

15.6.11 Find the Bimorph Resonant Frequency:

The cantilever is oscillated by a small piezoelectric bimorph mounted in the cantilever holder. For Force Modulation, oscillate the bimorph at or near its resonant frequency. The bimorph resonance frequency is usually the largest peak in the 5-30kHz range. This ensures the cantilever moves with sufficient amplitude to produce elasticity contrast.

> **Note:** You need to find the bimorph's resonant frequency only once. (Resonant frequencies are unique to each force modulation cantilever holder.) Once you find the resonant frequency, write it down for future use. This **Drive Frequency** may be used as a starting place each time you perform force modulation imaging; however, recheck the bimorph's resonant frequency in free air using **Cantilever Tune** each time you install a new cantilever.

1. Use a FESP or TESP cantilever. NP probes have confusing low frequency resonances.

2. Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window, shown in Figure 15.6e.

Figure 15.6e   The Cantilever Tune window.
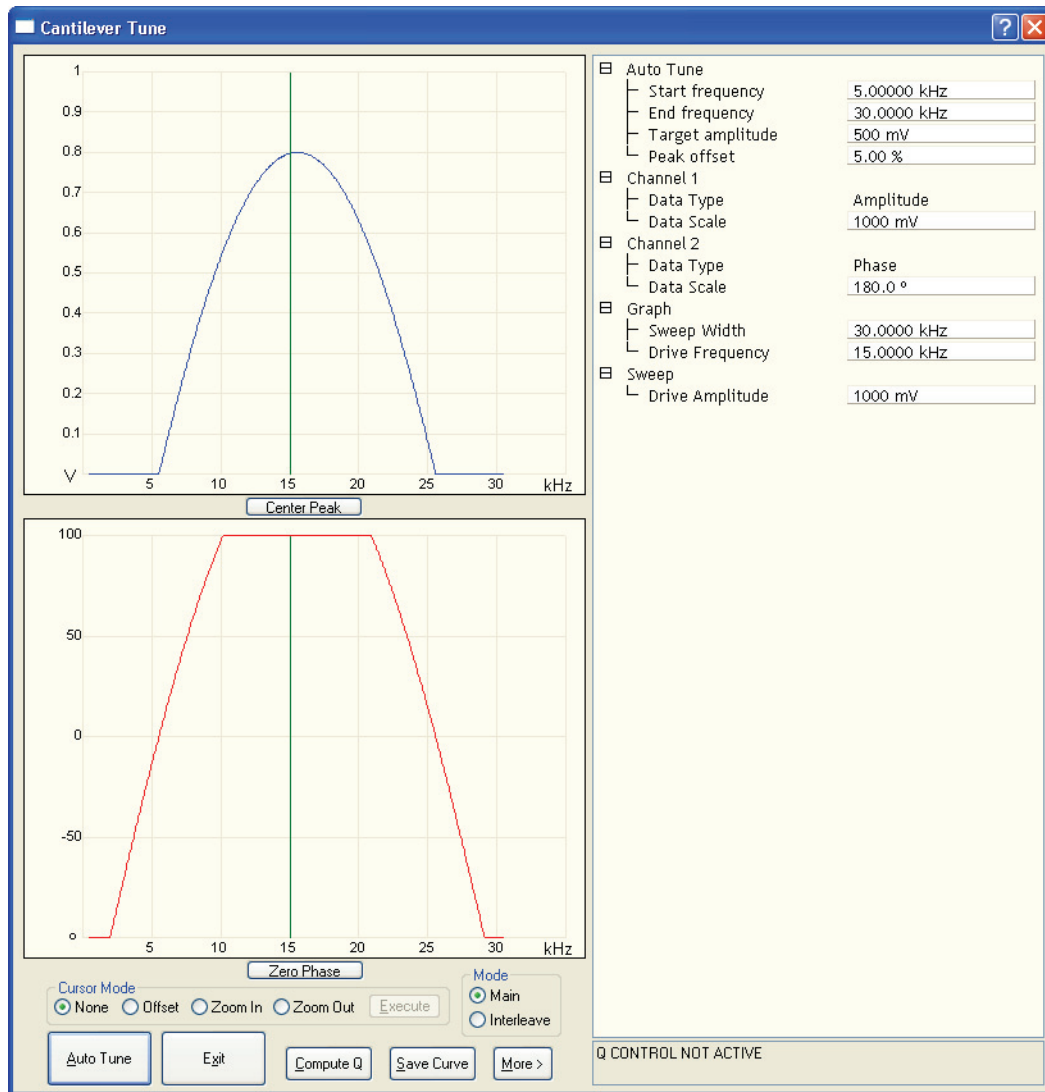
The graphs plot the **Frequency Sweep**, showing cantilever oscillation amplitude and phase versus frequency. To set the parameters controlling the **Frequency Sweep** plot, use the parameters in the **Sweep** panel.

3. Set the **Drive frequency** to **15kHz** and the **Sweep width** to **30kHz**.

4. Set the **Drive amplitude** at **1000mV** to start. You can readjust this value later.

> **Note:** The large drive amplitude is necessary because peaks are smaller than normally seen during TappingMode operation. This is because the cantilever is not at resonance; therefore, its motion is mostly vertical. Vertical motion is not amplified by the beam deflection detection technique which is sensitive primarily to changes in cantilever angle.

5. Adjust the **Sweep Width, Data Scale** and the **Drive Amplitude** until you can clearly see peaks. Bimorph resonances typically occur between **8-20kHz**.

6. Click the **AUTO TUNE** button, shown in Figure 15.6e, to begin optimizing the cantilever tune to the above parameters. The computer automatically changes the **Drive Frequency** so that the peak is centered.

Once you choose the desired frequency, the **Frequency Sweep** plot should be recentered similarly to the plot shown in (see Figure 15.6f).

Figure 15.6f   Correctly Tuned Force Modulation Frequency
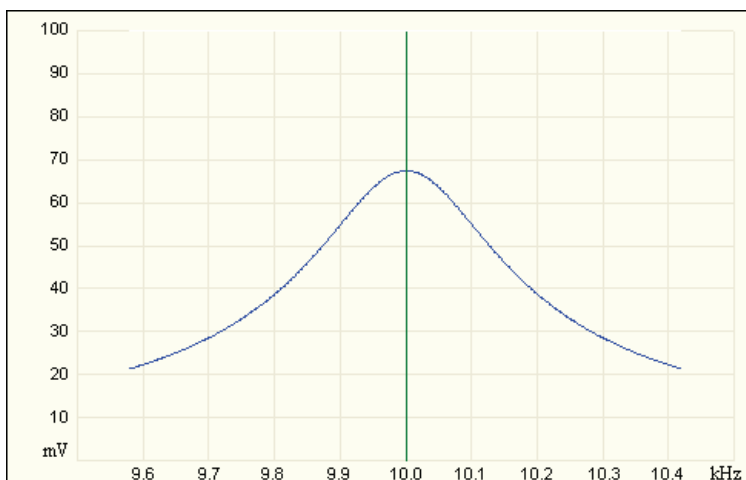
> **Note:** Bimorph resonance should be between **5 kHz** and **30 kHz**. If peaks are found at a much higher frequency, these are cantilever resonances, *not* bimorph resonances.

7. Record the **Drive Frequency**. This is the resonant frequency of your force modulation bimorph.
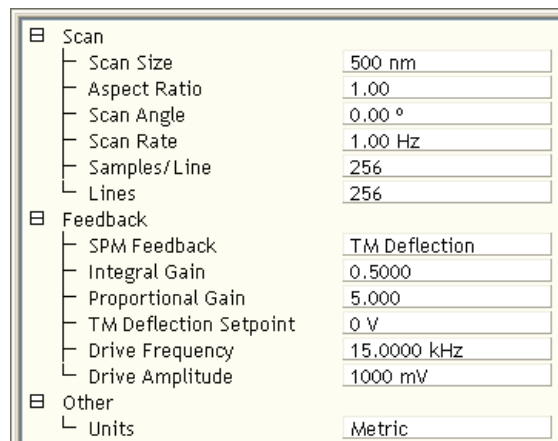
15.6.12 Set Initial Scan Parameters

Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see Figure 15.6g).

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

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

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

Figure 15.6g   Suggested Force Modulation Settings

Feedback Panel

1. Set the **SPM Feedback** to **TM DEFLECTION**.

2. Set the **Integral gain** to **0.5** and the **Proportional gain** to **5** (see Figure 15.6g).

3. Set the **TM Deflection Setpoint** to **0V**. Recall that higher setpoints cause the cantilever to push harder on the surface during scanning.

Other Panel

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

Channels

1. Set the **Channel 1 Data Type** to HEIGHT SENSOR (see Figure 15.6h).

2. Set the **Channel 2 Data Type** to TM DEFLECTION ERROR.

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

4. Set the **Channel 4 Data Type** to AMPLITUDE.

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

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

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

7. Set the **Channel 5** through **Channel 8 Data Type** to OFF.

The multi-image capability views both the topography data and the force modulation (elasticity)
data at the same time.

Figure 15.6h   Suggested Force Modulation Channel Settings

Channel	1	Scan	Main	Scale	14.33 um	RT Plane Fit	Line
Data Type	Height Sensor	Direction	Retrace	Center	0 nm	OL Plane Fit	Full
Channel	2	Scan	Main	Scale	1.475 um	RT Plane Fit	None
Data Type	TM Deflection Error	Direction	Retrace	Center	0 nm	OL Plane Fit	None
Channel	3	Scan	Main	Scale	360.0 º	RT Plane Fit	None
Data Type	Phase	Direction	Retrace	Center	0 º	OL Plane Fit	None
Channel	4	Scan	Main	Scale	1000 mV	RT Plane Fit	Line
Data Type	Amplitude	Direction	Retrace	Center	0 mV	OL Plane Fit	None

15.6.13 Engage

1. Reduce the **Drive Amplitude** to **0** before engaging.

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

15.6.14 Optimize Scan Parameters

1. Adjust the **Integral Gain**, **Proportional Gain**, **Setpoint,** and **Scan Speed** to obtain a good topography (**Height**) image. For force modulation operation, set the **Integral Gain** and **Proportional Gain** to values of **0.5-10** and set the **Setpoint** as low as possible using the cursor keys (or by typing in new **Setpoint** values) until the cantilever pulls off the surface and the Z-center voltage jumps to -**220V**.

2. Record the **Setpoint** value where the cantilever pulls off the surface (the "pull-off value").

3. Increase the **Setpoint** until the cantilever touches the surface, and an image appears. Then decrease the setpoint to a value just above the value where the pull-off occurred.

   Note: If you adjust the **Setpoint** very close to the pull-off value, imaging perform with the smallest and least damaging force. However, in this condition imaging may be more unstable, as the cantilever may pull off the surface unexpectedly. The setpoint value affects the force modulation contrast. Examine the contrast of the force modulation image.

4. Optimize **Drive Amplitude** for Force Modulation imaging. The amount of contrast and the quality of both **Height** and **Amplitude** images depends on the **Drive Amplitude**. In general, increasing the **Drive Amplitude** provides greater contrast in the force modulation image.

   Note: It is possible to set the **Drive Amplitude** too high. If this occurs, the **Drive Amplitude** no longer increases the contrast in the amplitude image. Instead, the overall contrast in the force modulation image remains roughly constant, but you will observe more artifacts in the image. For example, if the drive amplitude is too high, the force modulation image becomes contaminated by "edge effects" or "friction effects." Operate at the lowest drive amplitude that gives sufficient contrast to examine the sample. Low drive amplitudes also help extend the life of the cantilever tip and reduce sample damage.

Procedure for Optimizing Drive Amplitude

1. Start with a small **Drive Amplitude** value of **50mV**.

2. Increase the **Drive Amplitude** with the right arrow keys or type in new values. The contrast of the **Amplitude** (force modulation) image increases.

3. Continue increasing the **Drive Amplitude** until sufficient contrast appears in the **Amplitude** image. If the **Drive Amplitude** increases to the point where contrast is no longer improving, reduce the **Drive Amplitude** slightly.

   Note: Force modulation contrast also depends on the **Setpoint**. In general, if using a larger **Setpoint** (larger tracking force) use a smaller **Drive Amplitude** to obtain good force modulation images without artifacts. If it is difficult to obtain a clear force modulation image that is free of artifacts, try reducing or increasing the **Setpoint** and then optimizing the **Drive Amplitude**.

4. Readjust gains (if necessary).

5. The value of the **Drive Amplitude** may also affect the Contact Mode AFM image, causing the system to go into unwanted oscillations. If the **Drive Amplitude** changes by a large amount, readjust the **Integral Gain** and **Proportional Gain.** Set the gains as high as possible to track the sample topography, but not so high that they cause oscillation due to the bimorph oscillation.

Sometimes an oscillation that appears in the data will be due to "aliasing" as described in the next section. If you cannot adjust gains to eliminate unwanted oscillations without compromising the height image's quality, see **Notes About Artifacts:** Section 15.6.15.

15.6.15 Notes About Artifacts

It is possible to see artifacts in force modulation images that are not due to differences in elasticity. Some artifacts to look for are outlined below:

Aliasing

Under some conditions, unwanted oscillations appear in the data due to aliasing of the **Drive Frequency** with the image pixel rate. This problem can be eliminated by changing the **Drive Frequency** by small increments. Use the arrow keys to change the **Drive Frequency** up or down very slightly until the oscillation disappears. On some materials, shifting the **Drive Frequency** slightly (1-3Hz) below or above a resonant peak value may improve image contrast. Operators are encouraged to experiment.

Edge Effects

Sometimes force modulation images show changes in amplitude at the edge of topographic features like steps or bumps. These artifacts typically look like the derivative of the sample topography.

To see if a feature is an edge effect, try reversing the **Line Direction** from **Trace** to **Retrace,** for example. If the contrast reverses or the amplitude change now appears on the other side of the topographic feature, then the amplitude change is likely due to the topographic edge, not differences in elasticity. To minimize edge effects, reduce **Drive Amplitude, Setpoint** or **Scan Speed.** Set the **Integral Gain** and **Proportional Gain** as high as possible without causing unwanted oscillations.

Frictional Effects

Because the cantilever is held at an angle to the sample surface, the cantilever tip will slide laterally ("skate") as the tip pushes into the sample (see Figure 15.6i). The amplitude of cantilever motion is affected by differences in friction between two different materials. For this reason, force modulation images may contain information about differences in local frictional forces. To reduce the influence of frictional effects, use a smaller **Setpoint** or **Drive Amplitude**. If you are using "Negative LiftMode" (see Section 15.7), use a more positive **Lift Height**.

Figure 15.6i Friction on Force Modulation Images

Effect of friction on force modulation images
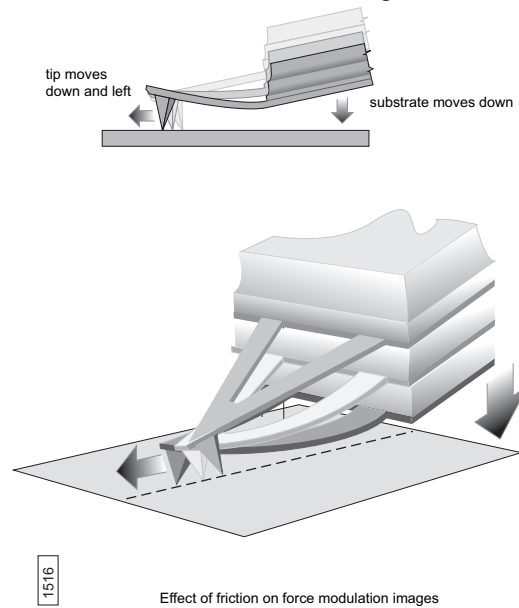
Tip Shape

The amount of indentation into a surface for a given applied force depends on the shape of the cantilever tip. For the same **Drive Amplitude** a sharper tip indents deeper than a dull tip. Because it is possible for the tip to dull during imaging, replace the cantilever if the force modulation contrast deteriorates over time. Also, note that it will be difficult to obtain reproducible, quantitative elasticity measurements between different samples and different cantilevers because the tip shapes may be different. In general, the force modulation technique is a qualitative tool for identifying regions of harder and softer material on a sample, rather than a tool for quantitative analysis.

Not all samples lend themselves to standard force modulation imaging. Even samples with excellent elasticity contrast may not show up in force modulation if their absolute elasticity is out of instrumental range. "Negative LiftMode" may prove useful for imaging otherwise difficult materials (see Section 15.7.3).

15.7 Force Modulation with 'Negative LiftMode'

A new form of force modulation imaging utilizing TappingMode and LiftMode operation known as "Negative LiftMode," allows imaging of certain materials previously not visible with Contact Mode AFM force modulation. This method is especially suited for softer materials, yielding higher resolution. Best results using negative LiftMode are obtained on relatively smooth samples (< 500nm vertical features); however, Veeco encourages experimenting with this technique on rougher surfaces as well. A general procedure for Force Modulation with "Negative LiftMode" is described in Section 15.7.3.

> **Note:** Force modulation contrast is very sensitive to the spring constant of the probe, which varies according to the length and thickness of the cantilever. You may perform TappingMode easily with either force modulation or TappingMode cantilevers, but you may have difficulty with Contact Mode AFM silicon and silicon nitride cantilevers. (see Table 15.6a).

1. Verify the probe is withdrawn from the sample surface.

2. Switch to the **Expanded Mode**.

3. Verify in the **Feedback** panel that **SPM Feedback** = **TM DEFLECTION**.

15.7.1 Set Interleave Controls

In the **Interleave** panel, shown in Figure 15.7a, set the following

1. Click the **Drive Frequency** and **Drive Amplitude** parameters; when enabled (decoupled from the main scan), the buttons appear green.

2. Set the **Drive Amplitude** on the **Interleave** panel to **200-400mV**.

3. Set the **Drive Frequency** on the **Interleave** panel to the bimorph resonant frequency found in Section 15.6.4.

4. Set **Lift Start Height** and **Lift Scan Height** to **0.00nm**.

5. For now, set the **Interleave Mode** parameter to **DISABLED**.

Figure 15.7a Suggested Force Modulation Interleave Parameters

15.7.2 Obtain a TappingMode Image

While negative LiftMode force modulation data is imaged using **Channel 2**, height data is obtained using TappingMode on **Channel 1**. You must obtain a satisfactory TappingMode image to generate good data.

1. Verify that the **Interleave Mode** parameter on the **Interleave** panel is **DISABLED**.

2. Verify that the **Microscope Mode** parameter in the **Other** panel is set to **TAPPING**.

3. Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window, shown in Figure 15.6e.

4. Enter a **Start frequency** value of **5kHz** and **End frequency** value of **100kHz** for FESP tips.

5. Set **Target amplitude** to **0.5V**, and click the **AUTO TUNE** button.

6. Click the **Interleave Controls** button to verify that the bimorph drive parameters are correct.

7. Exit the **Cantilever Tune** dialog box.

8. Verify that all **Scan** and **Feedback** parameters are set for obtaining a TappingMode image.

9. Set the **Channel 1** panel **Data Type** parameter to **HEIGHT**.

10. Set the **Line Direction** to **RETRACE** and enter an appropriate **Data Scale** value for your sample.

11. Engage the tip on the surface and obtain a good TappingMode image.

15.7.3 Obtain a Negative LiftMode Force Modulation Image

1. Set the **Data Type** parameter in the **Channel 2** panel to **AMPLITUDE**. Set the **Line Direction** parameter to **RETRACE**.

2. Switch the **Interleave Mode** parameter in the **Interleave** panel to **LIFT**. Negative LiftMode imaging is now in effect.

3. Using the left arrow key, optimize the negative LiftMode image by slowly decreasing the **Lift Scan Height** parameter in the **Interleave** panel from zero until you reach the surface. In most cases, you should not go below -**60.0nm**.

   Note: When the interleaved scan is enabled, the tip's TappingMode height above the sample reduces by the **Lift Scan Height** amount. This places the oscillating tip in contact with the sample as surface features are profiled. The contrast between light and dark reveals areas of high and low elasticity, with the dark area indicating harder material and the lighter areas indicating softer material.

Note: If the **Lift Scan Height** is too large, the interleave may be tapping, and therefore not in the continuous contact required for force modulation.

4. Adjust the interleaved **Drive Amplitude** and **Lift Scan Height** until the force modulation image is optimized. This may require some experimentation.

Note: If you see a lot of contrast in the amplitude image before reaching the surface, try reducing the **Integral** and **Proportional Gains** in the **Feedback** panel. Adjust gains slightly lower than when performing normal TappingMode imagery. Verify by setting a **Lift Scan Height** of **100nm**, then adjusting the gains (and possibly **Drive Amplitude**) until you see the minimum amount of contrast in the amplitude image. Once you minimize contrast, enter a **Lift Scan Height** of **0.0nm** and approach the surface.

15.8 Force Volume

Force volume imaging with the atomic force microscope (AFM) combines force measurement and topographic imaging capabilities. A force volume data set can be used to map in two or three dimensions the interaction forces between a sample and the AFM tip and correlate the force data with topographic information. Possible applications include elasticity, adhesion, electrostatic, magnetic, and binding studies. Advantages of force volume imaging include the ability to collect distributions of forces at various Z-positions and at thousands of XY positions during a single image scan, correlation of surface topography to interaction force, better quantization of the interaction force, and new methods of analysis. For detailed information regarding force volume imaging, refer to the *NanoScope Software 8 User Guide*, Veeco p/n 004-1025-000.

Chapter 16   Interleave Scanning

This chapter provides general information regarding the **Interleave** procedure and commands, with emphasis on **LiftMode**. Specifically, this chapter details the following topics:

- **Preface: Interleave Scanning & LiftMode:** Section 16.1

- **Interleave Mode Description:** Section 16.2

- **LiftMode Description:** Section 16.3

- **Operation of Interleave Scanning / LiftMode:** Section 16.4

- **Use of LiftMode with TappingMode:** Section 16.5

  - **Main Drive Amplitude and Frequency selection:** Section 16.5.1

  - **Setpoint Selection:** Section 16.5.2

  - **Interleave Drive Amplitude and Frequency Selection:** Section 16.5.3

  - **Amplitude Data Interpretation:** Section 16.5.4

  - **Cantilever Oscillation Amplitude:** Section 16.5.5

16.1   Preface: Interleave Scanning & LiftMode

Interleave is an advanced feature of NanoScope software which allows the simultaneous acquisition of two data types. Enabling **Interleave** alters the scan pattern of the piezo. After each main scan line trace and retrace (in which topography is typically measured), a second (**Interleave**) trace and retrace is made with data acquired to produce an image concurrently with the main scan.

Typical applications of interleave scanning include MFM (magnetic force microscopy) and EFM (electric force microscopy) measurements; Chapter 17 provides detailed instructions for obtaining MFM images of a standard magnetic sample. Enabling **Interleave** with the mode set to **LIFT** enacts **LiftMode**. During the interleave scan, the feedback is turned off and the tip is lifted to a user-selected height above the surface to perform far field measurements such as MFM and EFM. By recording the cantilever deflection or resonance shifts caused by the magnetic or electric forces on the tip, a magnetic force or electric force image is produced; see Chapter 17 and Chapter 18. **LiftMode** was developed to isolate purely MFM and EFM data from topographic data.

INTERLEAVE can also be used in **Interleave Mode**. In this mode, the feedback is kept on while additional topography, phase lateral force, or force modulation data is acquired.

The **Interleave** commands utilize a set of **Interleave** controls which allow several scan controls (Drive amplitude, Setpoint, and various gains) to be set independently of those in the main scan controls.

This chapter provides a general discussion of the **Interleave** action and commands, with emphasis on **LiftMode**. Chapter 17 and Chapter 18 provide step-by-step instructions for using **Interleave** scanning and **LiftMode** for obtaining magnetic and electric force data. For purposes of learning to use **Interleave** scanning, this section may be useful even to those users whose end application is not magnetic force microscopy.

16.2 Interleave Mode Description

Enabling **Interleave** changes the scan pattern of the tip relative to the imaged area. With **Interleave** mode disabled, the tip scans back and forth in the fast scan direction while slowly moving in the orthogonal direction as shown on the left of Figure 16.2a. This is the standard scan pattern of the NanoScope.

Figure 16.2a X-Y Scan Pattern
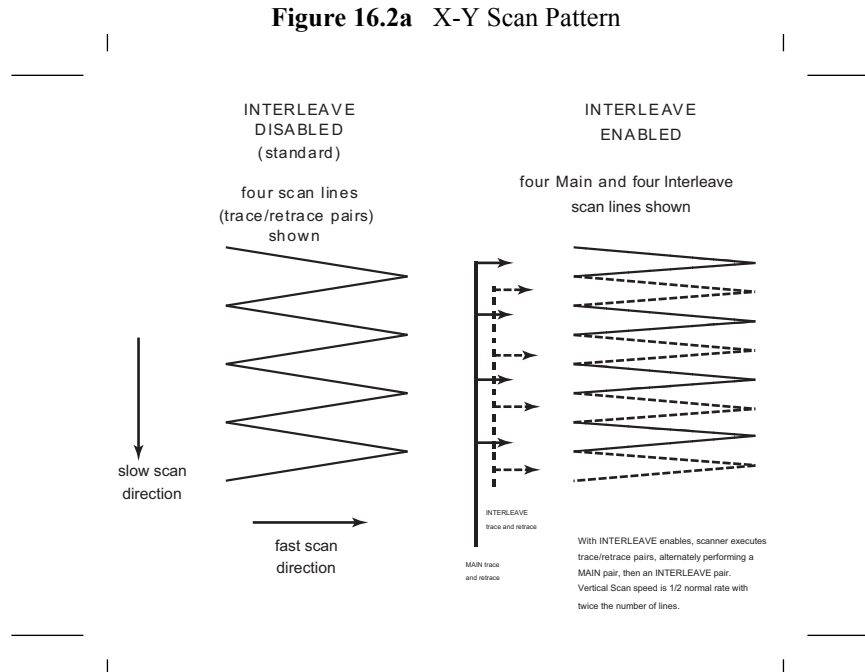
With **Interleave** mode enabled, the system first performs a standard trace and retrace with the main **Feedback** controls in effect. The tip moves at half the normal rate in the slow scan direction. As shown on the right of Figure 16.2a, an additional trace and retrace are then performed with the **Interleave** feedback controls enacted. The frame rate halves because twice as many scan lines are performed for the same scan rate.

Two modes are possible for **Interleave** scan: **INTERLEAVE** and **LIFT**. With **INTERLEAVE** selected, the feedback remains on during the interleave pass with the values under **Interleave** feedback controls (Setpoint, Gains, etc.) in effect. In **LIFT** mode, the feedback is instead turned off, and the tip lifted off the surface and scanned at a user-selected height. Topography data recorded during the main pass is used to keep the tip a constant distance from the surface during the **Interleave** trace and retrace.

16.3   LiftMode Description

With the **Interleave** scan option set to **LIFT**, the motion of the tip during the **Interleave** trace and retrace is as shown in Figure 16.3a.

Figure 16.3a   LiftMode Profiles
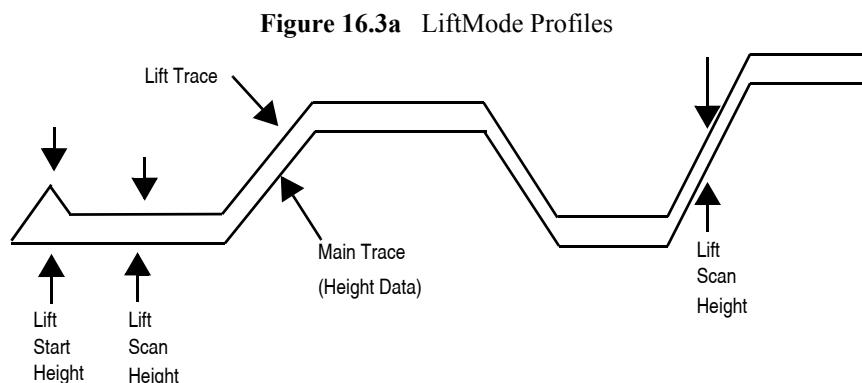
The tip first moves to the **Lift Start Height**, then to the **Lift Scan Height**. A large **Lift Start Height** can be used to pull the tip from the surface and eliminate sticking. The **Lift Scan Height** is the final tip height. This value is added point-by-point to the height data obtained during the Main topography trace and retrace. Values can be positive or negative.

16.4   Operation of Interleave Scanning / LiftMode

These instructions apply to STM, contact AFM, or TappingMode AFM modes. It is assumed the user is familiar with TappingMode or contact AFM to obtain good images of surface topography. Use of interleave scanning requires the steps below; see Chapter 17 for specific examples using MFM.

1. Obtain a topography scan using the appropriate (usually Contact or TappingMode) method: When using **LiftMode**, it is important that the gains and setpoint under **Feedback** controls be adjusted to give a faithful image of the surface. Because the height data is used in the lift pass to trace the topography, a poor measurement of surface height may give inaccurate measurement during the lift pass or cause the tip to strike the surface. Typically, the height data is displayed on Channel 1.

2. Adjust the **Interleave** controls panel to desired settings: Choose the **Interleave Mode** (**INTERLEAVE** or **LIFT**) appropriate for the measurements to be performed. Set the **Interleave** controls as desired. When using TappingMode, the **Drive Amplitude**, **Drive Frequency**, **Gains**, and **Amplitude Setpoint** can be set differently in the **Interleave** panel than in the main **Feedback** panel; see Section 16.5. However, it is often convenient to begin with the main and interleave values set equal; this can be done by toggling the parameters of the appropriate **Interleave** parameters to an "off" (grayed) condition. The values can be changed once engaged. If using **LIFT**, set the **Lift Start Height** and **Lift Scan height**. If using **INTERLEAVE** mode, set the **Gains** and **Amplitude Setpoint**.

Note that certain constraints are imposed: scan sizes, offsets, angles, rates and numbers of samples per scan line are the same for the main and interleave data, and the imaging mode (contact, TappingMode, or force modulation) must also match.

3. Choose the **Interleave Data Type**: Depending on the type of microscope, **Interleave** mode allows the options of amplitude, phase, frequency, potential, input potential, or data types for doing far-field (MFM or EFM) imaging; see Chapter 17 for detailed examples. Auxiliary channels are also available for some applications.

Once the **Interleave Scan Line** is chosen, **Interleave** mode is automatically enabled, triggering interleave scanning. Interleave data typically displays as the second image. Note that the scan rate in the slow direction is halved.

Figure 16.4a Interleave Scan Line

4. Display the interleave data by switching **Scan Line** (in the **Channel** panels) to **Interleave**. See Figure 16.4a.

- **Lift Scan Height**: The lateral and vertical resolutions of the Lɪꜰᴛ 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 Lɪꜰᴛ trace and retrace.

- **Tip Shape**: As shown in Figure 16.3a, the tip separation in the **LiftMode** is defined in terms of the Z direction only. The **Lift Scan Height** is added to the height values taken from previous scan lines point-by-point. However, the tip may be closer to the sample than the Z separation indicates. On features with steep edges, the tip may get very close to the sample even though the Z separation is constant; see Figure 16.3a.

- Scan **Line Direction**: The **Line Direction** should be set to Rᴇᴛʀᴀᴄᴇ for both the main and interleaved scans. If it is set instead to Trace, a band may appear along the left side of the images due to the ramp between the surface and the **Lift Scan Height**.

16.5   Use of LiftMode with TappingMode

Additional considerations when using **LiftMode** with TappingMode are discussed below.

16.5.1   Main Drive Amplitude and Frequency selection

As usual, these parameters are set in Cantilever Tune before engaging. It is helpful to keep in mind the measurements to be done in **LiftMode** when setting these values. For example, if **Amplitude** data will be monitored during the Lift scan for magnetic force imaging, the **Drive Frequency** should be set to the side of the resonance; see Chapter 17. (However, certain parameters can be set independently for the interleave scan; see below.)

16.5.2 Setpoint Selection

When the main and interleave **Drive Amplitudes** and **Drive Frequencies** are equal (parameters in the **Interleave** panel grayed out), the cantilever oscillation amplitude increases to the free oscillation amplitude when the tip is lifted off the surface in **LiftMode**. If a small setpoint value forces the oscillation amplitude low while the feedback is running, the amplitude can grow considerably when the tip is lifted free of the sample surface. The change can also be large if the main **Drive Amplitude** was increased or the main **Drive Frequency** altered after the tip was engaged. (The vibration amplitude remains at the setpoint during the main scan even if these parameters are changed.) This could make the tip hit the surface in the lift scan for small **Lift Scan Heights**.

16.5.3 Interleave Drive Amplitude and Frequency Selection

The cantilever drive amplitude can be set differently in the Lift scan as compared to the main scan by clicking on the parameter of the corresponding parameter in the **Interleave** panel to "on" (green) and adjusting the value. This allows the tuning of a measurement in the Lift scan lines without disturbing the topography data acquired during the Main scan lines. The **Interleave Drive Amplitude** must be set low enough that the tip does not strike the surface during the Lift pass.

WARNING:	Before enabling the **Interleave Drive Amplitude**, check that its value is not much larger than the main Drive Amplitude value to prevent possible damage to the tip.
AVERTISSEMENT:	Lors d'un travail en mode intercalé (**Interleave Mode**), vérifier que la tension appliquée pour osciller le bras de levier lorsqu'il est en positon haute n'est pas beaucoup plus importante que la tension appliquée lorsque ce même bras de levier est en position basse (**Main Drive Amplitude**). Le non respect de cette procédure peut entraîner la destruction de la pointe.
WARNUNG:	Bevor Sie im **Interleave-Mode** die "**Interleave Drive-Amplitude**" einschalten, vergewissern Sie sich bitte, daß der dort eingetragene Wert nicht wesentlich größer ist, als der Wert der "Main Drive-Amplitude", um evtl. Beschädigungen der Spitze vorzubeugen.

The **Interleave Drive Frequency** can also be adjusted, which may be useful if acquiring amplitude data in **LiftMode**; see Chapter 17.

16.5.4 Amplitude Data Interpretation

When monitoring amplitude data in **LiftMode**, brighter regions correspond to smaller amplitude, and darker regions to larger amplitude.

16.5.5 Cantilever Oscillation Amplitude

The selection of the oscillation amplitude in **LiftMode** depends on the quantity to be measured. For force gradients which are small in magnitude but occur over relatively large distances (sometimes hundreds of nm, as with magnetic or electric forces), the oscillation amplitude can be large, which for some applications may be beneficial. The **Lift Scan Height** must be correspondingly large so that the tip does not strike the surface. However, the lateral resolution of far field (MFM or EFM) measurements decreases with distance from the surface. Typically, the resolution is limited to a value (in nm) roughly equal to the Lift scan height; see Chapter 17.

Small amplitudes must be used to sense force gradients, such as Van der Waals forces, which occur over short distances (typically a few nm). As much of the cantilever travel as possible should be within the range of the force gradient.

Chapter 17   Magnetic Force Microscopy

This chapter describes how to perform Magnetic Force Microscopy (MFM) using the Interleave and **LiftMode** procedures discussed in Chapter 16. Please review those sections prior to attempting MFM. These hardware units allows phase detection and frequency modulation for optimal MFM imaging.

Specifically, this chapter discusses the following topics:

- **Magnetic Force Microscopy:** Section 17.1

  - **Force Gradient Detection:** Section 17.1.1

  - **Amplitude Detection Techniques:** Section 17.1.2

- **Basic MFM Operation:** Section 17.2

  - **MFM Using LiftMode:** Section 17.2.1

  - **Magnetic Force Microscopy Procedure:** Section 17.2.2

- **Advanced MFM Operation:** Section 17.3

  - **Lift Scan Height and Magnetic Imaging Resolution:** Section 17.3.1

  - **Fine Tuning Interleave Controls:** Section 17.3.2

  - **Drive Amplitude:** Section 17.3.3

- **Troubleshooting:** Section 17.4

  - **MFM Image Verification:** Section 17.4.1

  - **Saturation in Amplitude Detection:** Section 17.4.2

  - **Optical Interference:** Section 17.4.3

17.1   Magnetic Force Microscopy

In MFM, a tapping cantilever equipped with a special tip first scans over the surface of the sample to obtain topographic information. Using **LiftMode** as shown in Figure 17.1a, the tip then raises just above the sample surface. The surface topography from the initial scan is added to the lift height to maintain constant separation during the lifted scan. The influence of magnetic force is measured using the principle of force gradient detection.

17.1.1   Force Gradient Detection

In the absence of magnetic forces, the cantilever has a resonant frequency f_0. This frequency is shifted by an amount Δf proportional to vertical gradients in the magnetic forces on the tip. The shifts in resonant frequency tend to be very small, typically in the range 1-50 Hz for cantilevers having a resonant frequency $f_0 \sim 100$ kHz. These frequency shifts can be detected three ways: **phase detection** which measures the cantilever's phase of oscillation relative to the piezo drive; **amplitude detection** which tracks variations in oscillation amplitude; and **frequency modulation** which directly tracks shifts in resonant frequency. **Phase detection** and **frequency modulation** produce results that are generally superior to **amplitude detection**.

Figure 17.1a   MFM LiftMode Principles

Magnetic Fields
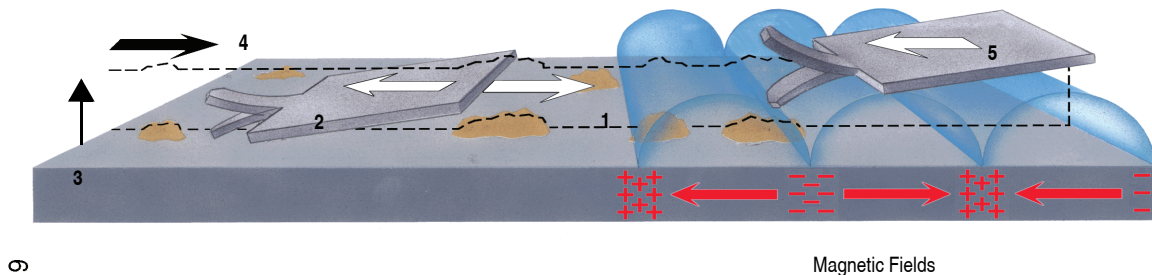
1519

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).

17.1.2   Amplitude Detection Techniques

Amplitude detection has largely been superseded by frequency modulation and phase detection.

17.2   Basic MFM Operation

In the instructions below, steps specific to phase and amplitude imaging are described independently.

17.2.1   MFM Using LiftMode

LiftMode allows imaging of relatively weak but long-range magnetic interactions while minimizing the influence of topography (see Figure 17.1a). Measurements are taken in two passes across each scan line; each pass consists of one trace and one retrace. In the first pass, topographical data is taken in TappingMode. The tip is then raised to the lift scan height and a second trace and retrace performed while maintaining a constant separation between the tip and local surface topography. Magnetic interactions are detected during this second pass. In LiftMode, topographical features are virtually absent from the MFM image (see Figure 17.2f).

This section provides instructions for using the **LiftMode** of Interleave Scanning to obtain MFM images. These guidelines will help you obtain an MFM image of a standard magnetic sample (metal-evaporated video tape). Standard tape samples are provided with the MFM starter kit, and can be obtained from 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).

As mentioned above, NanoScope controllers use force gradient detection for MFM imaging. Within this general technique, there are three possible schemes, known as frequency modulation, phase detection, and amplitude detection. Phase detection and frequency modulation detection are superior methods for magnetic force imaging, offering greater ease of use, better signal-to-noise ratios, and reduced artifact content as compared to amplitude detection.

For MFM procedures, magnetic-coated tips are required. Various kinds of MFM probes are available for specific applications; contact Veeco for more information. For specific information regarding TappingMode and Interleave Scanning please refer to Chapter 11 and Chapter 16 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.

17.2.2 Magnetic Force Microscopy Procedure

1. Magnetize a NanoProbe magnetic probe with a strong permanent magnet before installing the probe holder on the AFM head.

   Note: Tips are usually magnetized with the field aligned along the tip axis (perpendicular to the sample surface). The MFM then senses force gradients from the perpendicular component of the samples's stray field. Tip magnetizers can be purchased from Veeco.

2. Mount a NanoProbe magnetic probe on the scanner or tip holder.

3. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 17.2a.

Figure 17.2a The ELECTRICAL & MAGNETIC AFM **Select Experiment** window
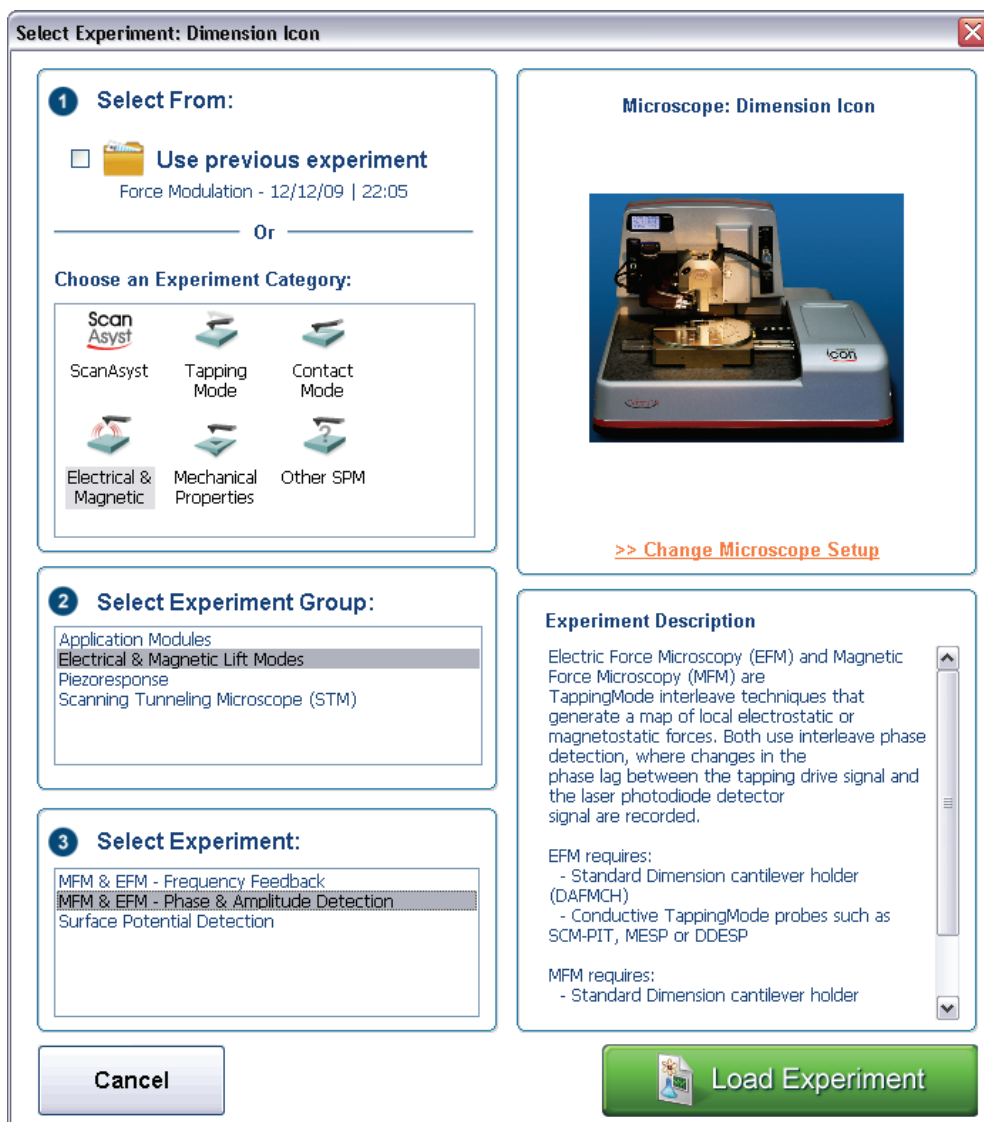
4. Select ELECTRICAL & MAGNETIC in the **Choose an Experiment Catagory** panel.

5. Select ELECTRICAL & MAGNETIC LIFT MODES in the **Select Experiment Group** panel.

6. Select MFM & EFM - PHASE & AMPLITUDE DETECTION in the **Select Experiment** panel and click LOAD EXPERIMENT.

7. Set up the AFM for TappingMode operation (see Chapter 11).

8. Tune the cantilever **Drive Frequency**.

    Note: The procedure to tune the cantilever drive amplitude depends on whether you are using phase detection or amplitude detection. Both cases rely on automatic **Cantilever tune** just as when preparing for TappingMode (see Chapter 11). MFM cantilevers have resonant frequencies between 50 and 100 kHz. If using the **AutoTune** feature, these values can be used as bounds for the frequency sweep.

Setting a Drive Frequency for Phase Detection

The Drive frequency should be set to the center of the cantilever resonance, as shown in Figure 17.2b. This occurs automatically if using **AutoTune.**

Figure 17.2b Cantilever Tune for Phase Detection and Frequency Modulation
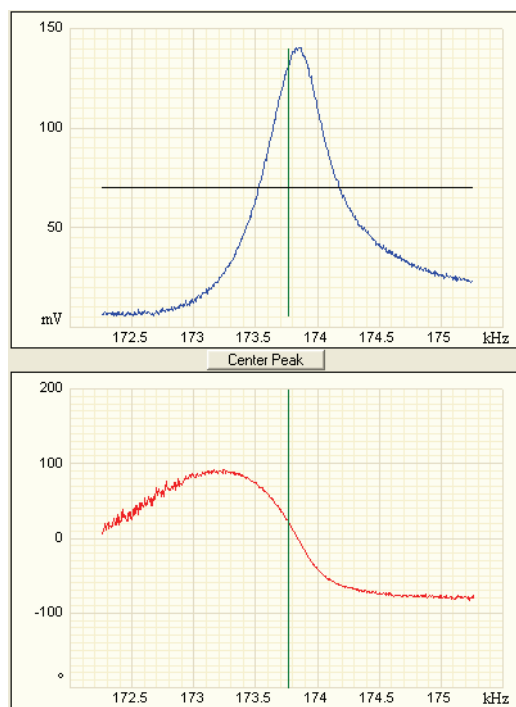
To correctly track the cantilever phase, the **Phase offset** parameter must be adjusted. This automatically occurs in **AutoTune**; alternatively, **Zero Phase** can be selected from the **Channel 2** panel. The phase curve should appear as in Figure 17.2b, decreasing with increasing frequency and crossing the center line (corresponding to a 90° phase lag) at the peak frequency. The phase curve then measures the phase lag between the drive voltage and the cantilever response. Again, vertical gradients in the magnetic force cause a shift Δf_0 in the resonance frequency. In this case, resonance shifts give rise to phase shifts $\Delta\phi$ which then give an image of the magnetic force gradients (see Figure 17.2c**).**

Figure 17.2c   Shift In Phase at Fixed Drive Frequency
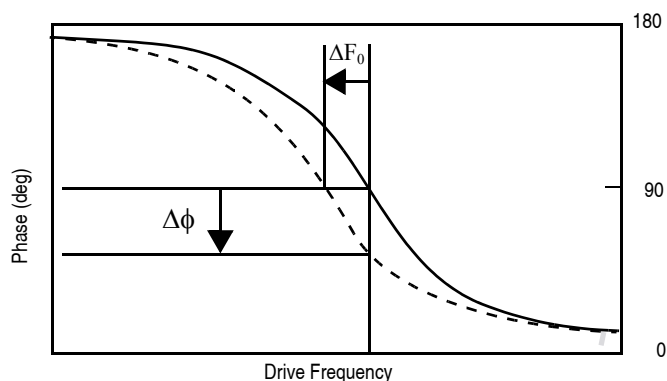
Setting a Drive Frequency for Amplitude Detection

1. Use the **AutoTune** feature to find the resonance peak.

2. Select the **Offset** function in the **Cantilever Tune** window to manually move the drive frequency to the side of the resonance (see Figure 17.2d).

3. Set the **Drive Frequency** to the steepest part of the resonance curve for maximum sensitivity.

   Note: As the tip oscillates above the sample, a gradient in the magnetic force will shift the resonance frequency f_0 (see Figure 17.2e). Tracking the variations in oscillation amplitude while in LiftMode yields an image of the magnetic force gradients.

Figure 17.2d   Cantilever Tune for Amplitude Detection
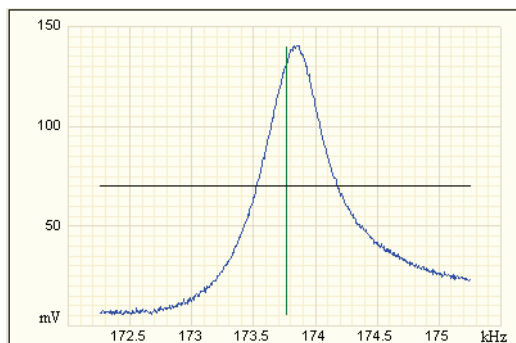
Figure 17.2e Shift in Amplitude at Fixed Drive Frequency
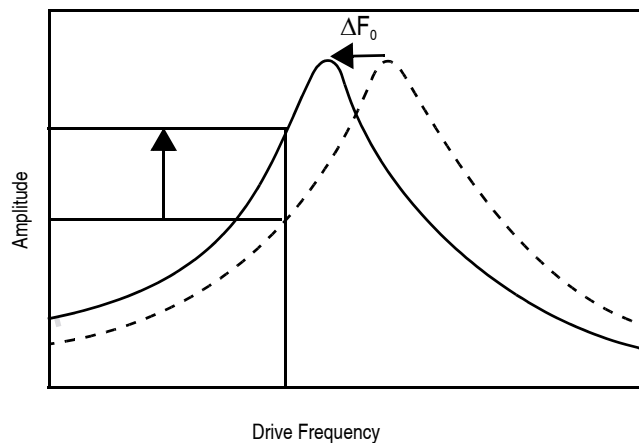
4. Select an appropriate **Target Amplitude** (approximately 560mV) using **Auto Tune** to adjust the **Drive Amplitude** so that the RMS voltage response of the photodetector is approximately 560mV before tuning. Or, exit **Cantilever Tune** and manually adjust the **Drive Amplitude** parameter in the **Feedback** panel.

   Note: Somewhat larger values may be beneficial if using amplitude detection.

5. Exit **Cantilever Tune** and return to **Image Mode**.

6. Under **Interleave** panel set the **Lift Start Height** to **0nm**, and **Lift Scan Height** to **100nm**.

   Note: The lift height can be optimized later.

7. Set the remaining Interleave parameters (**Setpoint**, **Drive Amplitude**, **Drive Frequency**, **Gains**) to the **Main** values by setting the flags left of the **Interleave** column to **Off**.

8. Under **Scan** controls, set the **Scan size** to **5μm** and **Scan rate** to **1–2Hz**.

9. Set the **Channel 1 Data Type** to **HEIGHT**, the **Data Scale** to **75nm** and the **Line Direction** to **RETRACE**.

10. Engage the AFM and make the necessary adjustments to obtain a good topographical image while displaying height data.

⚠️	**CAUTION:** Use the highest possible **Setpoint** to ensure that the tip is contacting the surface only lightly. The image should be similar to the topographic image shown on the left of Figure 17.2f. The surface is fairly flat with lubrication nodules of various sizes. A good image of the nodules indicates that the tip is sharp.

Figure 17.2f Topographic (left) and Magnetic Force Gradient Image (right)
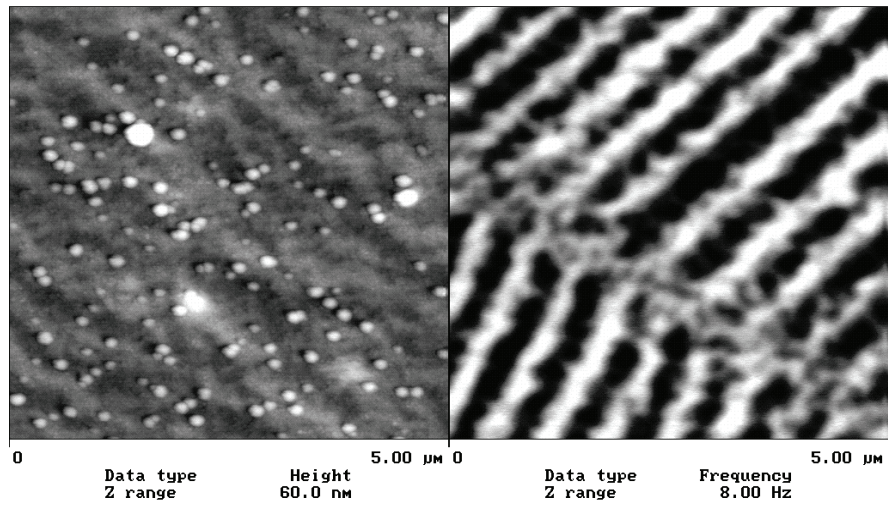
Phase Detection (Recommended)

Set the **Channel 3** image **Data Type** to PHASE, **Scan Line** to INTERLEAVE, **Z range** to **3 degrees**; and **Line Direction** to RETRACE.

> **Note:** It is important that the **Line Direction** be set to RETRACE for both the main and interleave scans. If instead it is set to TRACE, a band may appear along the left side of the images due to the time taken for the tip to move between the surface and the lift scan height.

Amplitude Detection

1. Set the **Channel 3** image **Data type** to AMPLITUDE, **Z range** to **1nm**, and **Line direction** to **Retrace**.

2. Change **Interleave mode** to LIFT to invoke **LiftMode**.

3. Set the **Channel 3 Scan line** to **Interleave** to display the interleaved data.

   > **Note:** This can only be done after **Interleave mode** is enabled. A magnetic force gradient image similar to that shown on the right of Figure 17.2f should appear as the Channel 3 image. The alternating dark and light stripes represent the recorded magnetic information, signifying a varying resonant frequency and magnetic force gradient on the tip.

   > **Note:** Keep the **Setpoint** as large as possible while consistent with a good image. Wider scans (> 25μm) will reveal separate tracks in which the magnetic stripes are at different angles

Frequency Modulation

It may be desirable to use frequency modulation. This activates a feedback loop which modulates the **Drive Frequency** to keep the cantilever phase lag at 90 degrees relative to the drive, corresponding to resonance. The frequency **Data Type** displays the resulting shift in **Drive Frequency** in *Hz*, and gives the most direct, quantitative image of force gradients.

To enable frequency modulation, follow the procedure above for obtaining an MFM image with phase detection with the following modifications:

1. In the **Interleave** panel, set the **Input Feedback** to FREQUENCY.

2. Switch the Channel 2 image **Data type** to **Frequency**. Try a **Data scale** (frequency shift) of approximately **10 Hz**.

3. If necessary, adjust the frequency modulation gains in the **Interleave** panel.

As with topography gains, the scan can be optimized by increasing the gains to maximize feedback response, but not so high that oscillation sets in.

17.3   Advanced MFM Operation

17.3.1   Lift Scan Height and Magnetic Imaging Resolution

The most important parameter affecting imaging resolution is **Lift Scan Height**. The range of 10–200nm is most useful. In general, MFM resolution is roughly equal to the lift height. Smaller **Lift Scan Heights** give better resolution; magnetic features smaller than the **Lift Scan Height** may not be resolved. The tip also experiences stronger fields close to the surface, giving improved signal-to-noise ratios.

For example, the image of metal-evaporated tape in Figure 17.2f has a resolution limited by the 100 nm **Lift Scan Height**. To improve the resolution, try reducing the **Lift Scan Height** to ~ **25nm**. Ensure that the tip does not strike the surface on the low point of its swing in the Lift image. Tip strikes appear as black or white spots, or even noisy, high-contrast streaks crossing the image. If the tip begins to strike the surface, reduce the **Interleave Drive Amplitude**. (In general, MFM tips are not damaged by intermittent tip strikes in **LiftMode**, except in extreme cases of very large amplitude and small lift heights.) An example of an image of the metal-evaporated tape taken with a **Lift Scan Height** of **30nm** is shown in Figure 17.3a. Note the fine magnetic structure that is not visible in Figure 17.2f. When imaging a sample for the first time, begin with moderate **Lift Scan Heights** (**50nm or greater**), then adjust downward. On relatively smooth samples (e.g., hard disks), lift heights down to 0nm can be used, as long as the drive amplitude is adjusted accordingly. (Lift scan heights of 0nm still correspond to a non-zero mean tip-sample distance. See the section on **Setpoint** below.) It is usually not beneficial to use **Lift Scan Heights** much smaller than the surface roughness. Users are encouraged to experiment for the best images on their samples.

The ultimate lateral resolution of MFM is near 20nm. Resolution is affected by properties of the tip, including mechanical sharpness and magnetic structure. When in good condition, magnetically-coated tips routinely give 50nm resolution, and many achieve 30nm or better.

Figure 17.3a   High-resolution Magnetic Force Gradient Image
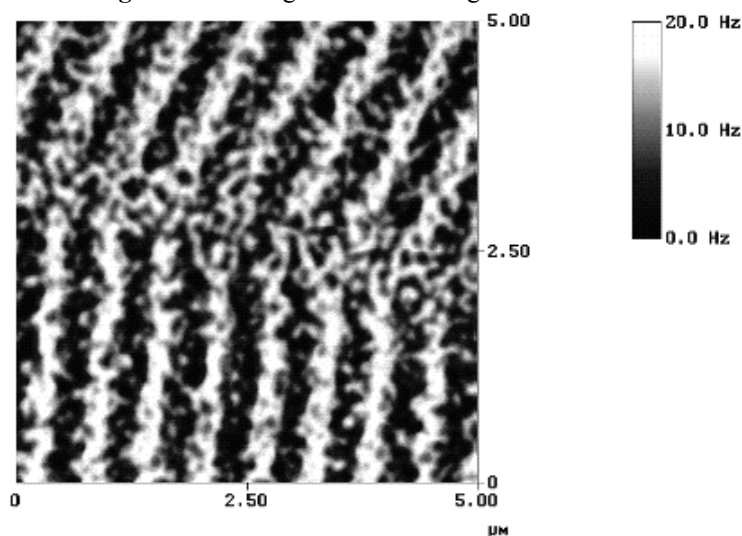
17.3.2 Fine Tuning Interleave Controls

Certain scanning parameters found under **Interleave** can be set to values in the Interleave (Lift) scan that differ from the values in the main scan. These parameters are enabled by clicking on the desired Interleave control, toggling its state from **Off** (gray) to **On** (green). When the flag is set to **Off**, the main scan control parameter setting takes precedence. When the flag is set to **On** the displayed Interleave scan value is active, overriding the main scan value.

17.3.3 Drive Amplitude

For MFM, of particular use is the Interleave **Drive Amplitude**. This parameter can affect a magnetic force image in a variety of ways.

- Increasing the **Drive Amplitude** can improve the signal-to-noise ratio when using phase detection or frequency modulation. This is because intrinsic, low-level noise interferes less when measuring the phase of a larger cantilever oscillation amplitude and hence stronger photodetector output. As an illustration, try setting the Interleave **Drive Amplitude** to **0**; the resulting phase image will be pure noise because one cannot measure the phase of a non-oscillating cantilever.

- In **LiftMode**, the Interleave **Drive Amplitude** can often be set to a value larger than in the main scan, thus giving optimal signal-to-noise. In some cases this is beneficial as long as the **Drive Amplitude** is not increased to the extent that the tip strikes the surface on the low point of its swing. The signatures of tip-sample contact are white and black spots in the image, or, in extreme cases, noisy, high-contrast streaks across the whole image. It is usually safe to increase the **Drive Amplitude** until the first signs of tip strike are noticed, then reduce the amplitude slightly.

- Before enabling Interleave **Drive Amplitude**, check that its value is not much larger than the main **Drive Amplitude** value. This prevents the cantilever oscillation from jumping to a very large amplitude when the parameter is enabled, possibly damaging the tip. The **Drive Amplitude** can be adjusted even when the parameter is disabled (grayed).

⚠	**CAUTION:**	Before enabling the Interleave **Drive Amplitude**, check that its value is not much larger than the main **Drive Amplitude** value to prevent possible damage to the tip.
	ATTENTION:	Lors d'un travail enmode intercalé (Interleave Mode) vérifier que la valeur de tension appliquée à l'oscillateur piézo-électrique est inférieure à celle appliquée à l'oscillateur en mode imagerie (Main Drive Amplitude). Le non respect de cette procédure peut entraîner la destruction de la pointe.
	VORSICHT:	Um mögliche Beschädigung der Meßspitze zu vermeiden, vergewissern Sie sich, daß der Wert der Interleave Drive Amplitude nicht wesentlich größer ist, als der Main Drive Amplitude, ehe Sie den Interleave Mode aktivieren.

- When using **Amplitude Detection**, variations in **Drive Amplitude** affect sensitivity and image contrast as well as signal-to-noise ratio. This is because changes in the oscillation amplitude change the slope of the amplitude vs. frequency curve, and hence the effective sensitivity; see Figure 17.3b. With phase detection and frequency modulation, changes in amplitude produce no change in contrast, and results are thus more reproducible than with amplitude detection.

Setpoint

For the most reproducible results, it is best to use a consistent setpoint. In **LiftMode**, the total tip-sample distance h_{tot} is the sum of the average tip-sample distance in TappingMode h_T, and the lift scan height h_{lift} (see Figure 17.3b). In **TappingMode**, the average tip-sample distance h_T is equal to the oscillation amplitude, which is determined by the setpoint and the amplitude sensitivity of the tip. MESPs typically have an amplitude sensitivity of approximately 25nm/V.

Large variations in setpoint can change the total tip-sample distance in **LiftMode**, sometimes with visible results in the magnetic image. For this reason, reproducible results are most easily obtained by using consistent setpoints. Note that a lift scan height of 0nm still gives a mean tip-sample distance of h_T in **LiftMode**.

Figure 17.3b   Tip Height and Oscillation Amplitudes (TappingMode & LiftMode)
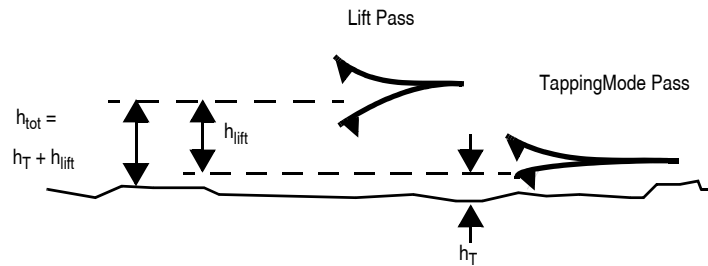
The relationship between setpoint voltage and oscillation amplitude is known as the sensitivity. Its value can be determined with **Force Calibration** (see Chapter 15). For 225μm MFM cantilevers, the sensitivity is typically in the range 20-25nm/V. The exact value depends on the position of the laser spot on the cantilever, the cantilever geometry, and the particular instrument. A 1V **Setpoint** typically corresponds to h_T~14-18nm.

17.4   Troubleshooting

17.4.1   MFM Image Verification

The procedure described above should produce a good magnetic force gradient image of the videotape sample. If there is a problem, check that the **Interleave** mode is set to **LIFT** and that the **Scan Line** is set to **INTERLEAVE**. Check also that the **Interleave** values of **Drive Amplitude** and **Drive Frequency** are initially set equal to the main **Scan** values.

17.4.2 Saturation in Amplitude Detection

If using amplitude detection, the magnetic force image can saturate (appear completely featureless) if the **Interleave Drive Amplitude** is significantly different than the **Drive Amplitude** in the main scan. Adjust the **Interleave Setpoint** to restore the image.

> **Note:** The **Interleave Setpoint** has no physical effect in **LiftMode** since there is no surface feedback during the lift pass.

17.4.3 Optical Interference

Optical interference may sometimes appear in the Lift (magnetic force gradient) image when imaging highly reflective samples. Optical interference appears as evenly spaced, sometimes wavy lines with ~1–2μm spacing superimposed on the lift image. This occurs when ambient laser light (i.e., light passing around or through the cantilever, then reflecting off the sample) interferes with laser light reflecting from the cantilever. Interference can be alleviated by moving the beam spot up the cantilever away from the tip; one-third of the cantilever length works well. The adjustment can be refined by carefully moving the beam spot laterally a small distance on the cantilever while scanning until interference fringes are minimized. Be careful not to move the beam off the cantilever or feedback may be lost.

Chapter 18   Electric Techniques

This chapter describes how to perform two electric techniques: Electric Force Microscopy (EFM) and Surface Potential. EFM is similar to Magnetic Force Microscopy (MFM) and shares many of the same procedural techniques. Both modes utilize the Interleave and LiftMode procedures discussed in previous chapters. Please read those chapters before attempting electric force measurements.

Specifically, this chapter includes:

- **Optimize Tune in Interleave:** Section 18.3.6

- **If Voltage is Needed, Use NanoScope When Possible:** Section 18.3.7

- **Try Uncoated Si Tip:** Section 18.3.8

- **Surface Potential Detection:** Section 18.4

- **Surface Potential Detection—Preparation:** Section 18.5

- **Surface Potential Imaging—Procedure:** Section 18.6

  - **Troubleshooting the Surface Potential Feedback Loop:** Section 18.6.1

18.1   Electric Techniques Overview

There are two types of electric techniques used with Dimension microscopes: **Electric Force Microscopy** (EFM) and **Surface Potential Detection**.

Electric techniques are similar to magnetic force microscopy (MFM) and share many of the same procedural techniques. Electric techniques and MFM both use the **Interleave** and **LiftMode** procedures. The two-pass LiftMode measurement allows the imaging of relatively weak but long-range magnetic and electrostatic interactions while minimizing the influence of topography (see Figure 18.1a). LiftMode records measurements in two passes, each consisting of one trace and one retrace, across each scanline. First, LiftMode records topographical data in TappingMode on one trace and retrace. Then, the tip raises to the **Lift Scan Height**, and performs a second trace and retrace while maintaining a constant separation between the tip and local surface topography.

Figure 18.1a   LiftMode Principles

1.   Cantilever measures surface topography on first (main) scan.
2.   Cantilever ascends to lift scan height.
3.   Cantilever follows stored surface topography at the lift height above sample while responding to electric influences on second (interleave) scan.
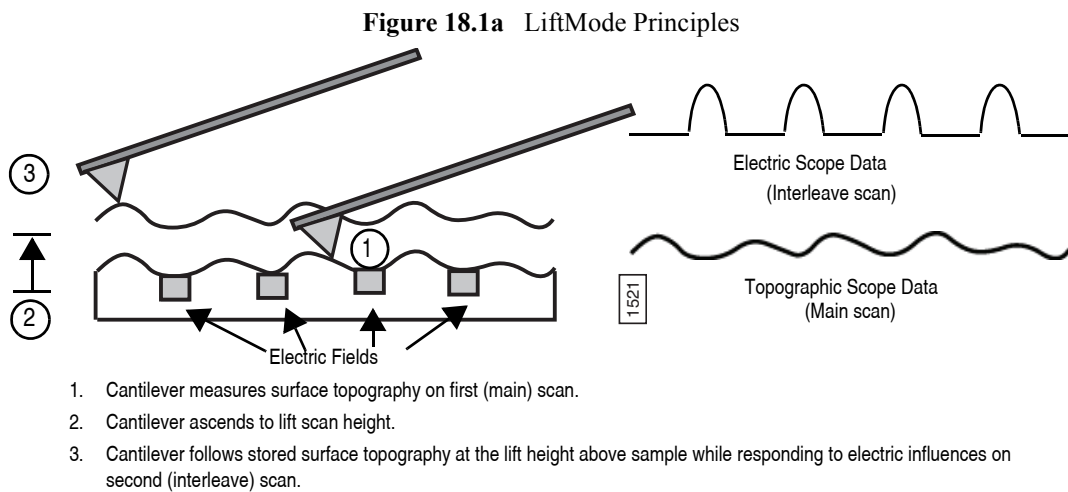
18.1.1 Electric Force Microscopy Overview

Electric Force Microscopy measures variations in the electric field gradient above a sample. The sample may be conducting, nonconducting, or mixed. Because the surface topography (e.g. sharp points on the surface concentrate the field gradient) shapes the electric field gradient, large differences in topography make it difficult to distinguish electric field variations due to topography or due to a true variation in the field source. The best samples for EFM are samples with fairly smooth topography. The field source includes trapped charges, applied voltage, etc. Samples with insulating layers (passivation) on top of conducting regions are also good candidates for EFM.

18.1.2 Surface Potential Imaging Overview

Surface potential imaging measures the effective surface voltage of the sample by adjusting the voltage on the tip so that it feels a minimum electric force from the sample. (In this state, the voltage on the tip and sample is the same.) Samples for surface potential measurements should have an equivalent surface voltage of less than $\pm 10V$, and operation is easiest for voltage ranges of $\pm 5V$. The noise level of this technique is typically 10mV. Samples may consist of conducting and nonconducting regions, but the conducting regions should not be passivated. Samples with regions of different materials will also show contrast due to contact potential differences. Quantitative voltage measurements can be made of the relative voltages within a single image.

18.2   Electric Force Microscopy

18.2.1   Electric Force Microscopy Theory

Electric Force Microscopy is analogous to standard MFM, except that gradients being sensed are due to electrostatic forces. In this method, the cantilever is vibrated by a small piezoelectric element near its resonant frequency. The cantilever's resonant frequency changes in response to any additional force gradient. Attractive forces make the cantilever effectively "softer," reducing the cantilever resonant frequency. Conversely, repulsive forces make the cantilever effectively "stiffer," increasing the resonant frequency. A comparison of these force additives is shown in Figure 18.2a.

Figure 18.2a   Comparison of Attractive and Repulsive Forces

Attractive gradient equivalent to additional spring in tension attached
to tip, reducing the cantilever resonance frequency.

Repulsive gradient equivalent to additional spring in compression attached
to tip, increasing the cantilever resonance frequency.
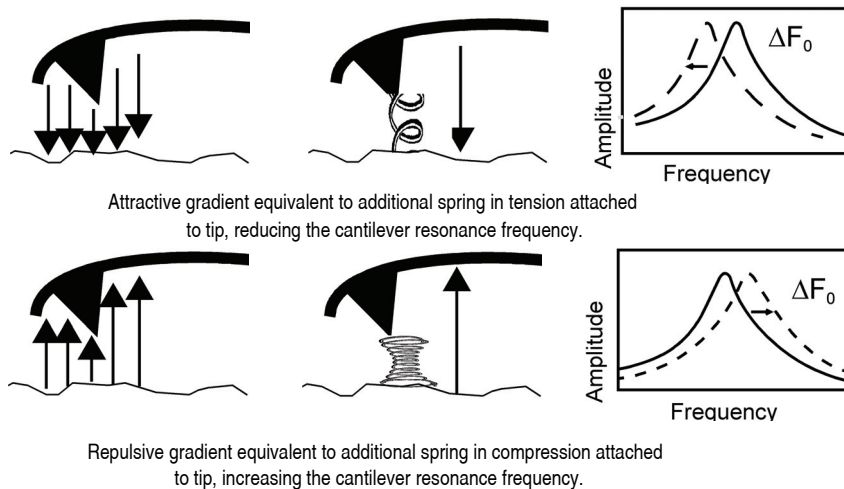
Changes in cantilever resonant frequency are detected in one of the following ways:

- Phase detection

- Frequency modulation

- Amplitude detection (not recommended due to artifacts)

All of the above methods rely on the change in resonant frequency of the cantilever due to vertical force gradients from the sample. Figure 18.2b shows a diagram of how the NanoScope V provides signal enhancement and feedback allowing gradient detection. The best candidates for electric field gradient imaging are samples that have large contrasts in the electric force gradient due to material differences or regions at substantially different potentials. For other samples having rough surface topography or small voltage variations, this technique may be undesirable because topographic features appear in the **LiftMode** image.

Figure 18.2b Block Diagram of NanoScope V for EFM
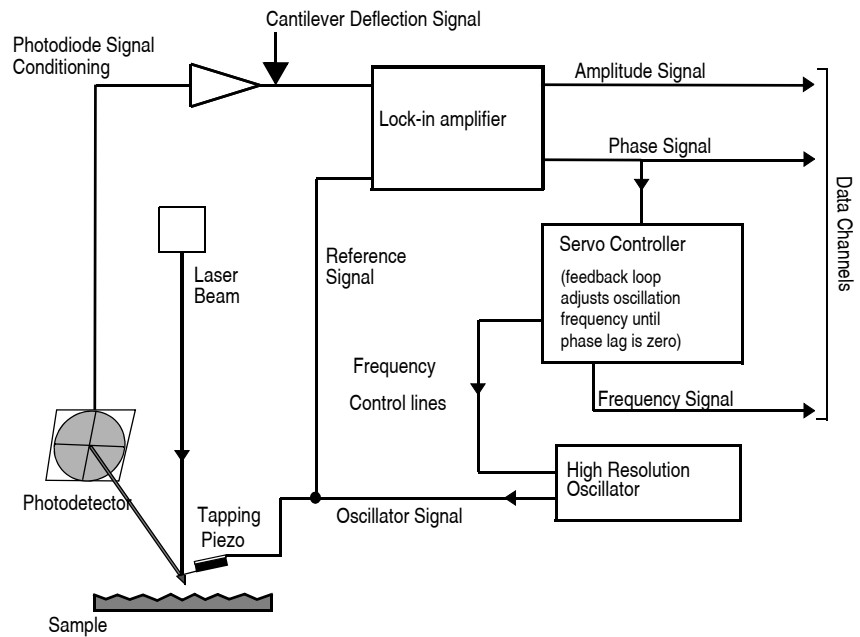
In many cases, you must apply a voltage to the tip or sample to achieve a high-quality image. Various methods for applying voltages to the tip and sample are included in the sections that follow. Samples with permanent electric fields may not require voltage application.

18.2.2 Electric Force Microscopy Preparation

This section explains how to apply a voltage to the tip or sample to generate electric fields. If the sample has a permanent electric field which does not require the external application of voltage, the steps below are not required and you can proceed to Section 18.2.3.

Enabling Tip/Sample in the Software

If you have chosen a configuration where a bias is applied to the tip or sample, you must enable **Tip Bias Control** and/or **Sample Bias Control** in the software. These parameters appear in the **Other** panel, shown in Figure 18.2c. Select **TIP BIAS** and **SAMPLE BIAS** to enable software control of tip and sample voltages.

Figure 18.2c   EFM Scan Parameters

Setting Tip/Sample Bias in the Software

Set the **Tip** and/or **Sample Bias** parameters in the **Feedback** panel, shown in Figure 18.2c, after you have enabled them. The **TIP BIAS** can be set within the range of -12V to +12V. The **SAMPLE BIAS** can be set within the range of -10V to +10V.

18.2.3 Electric Force Microscopy Procedures

1. Verify that **Tip** and **Sample Bias** have been properly set.

2. Electrically connect the sample by mounting it to a standard sample disk or stage using conducting epoxy or silver paint.

3. Verify the connection is good (a poor connection introduces noise).

   Note: If an external power supply connects directly to leads on the sample itself, it is important to electrically isolate the sample from the stage. A piece of Kapton tape covering the bottom of a sample puck works well.

4. Mount a metal-coated NanoProbe cantilever into the cantilever holder.

   Note: MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz, models MESP and SCM-PIT) usually work well. It is also possible to deposit custom coatings on model FESP silicon TappingMode cantilevers. Ensure that any deposited metal you use adheres strongly to the silicon cantilever.

5. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 18.2d.

Figure 18.2d The ELECTRICAL & MAGNETIC AFM **Select Experiment** window
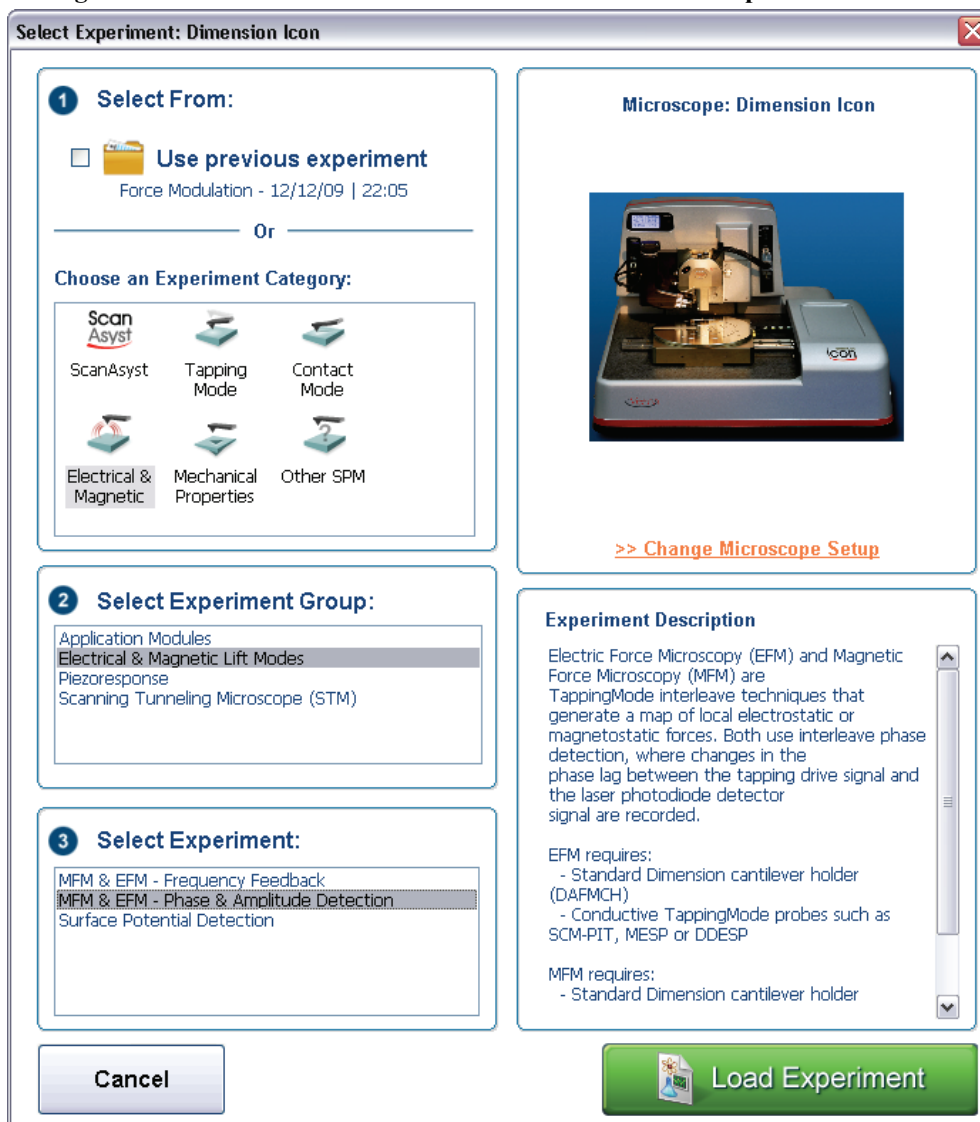
6. Select ELECTRICAL & MAGNETIC in the **Choose an Experiment Catagory** panel.

7. Select ELECTRICAL & MAGNETIC LIFT MODES in the **Select Experiment Group** panel.

8. Select **MFM & EFM - PHASE & AMPLITUDE DETECTION** in the **Select Experiment** panel and click LOAD EXPERIMENT.

9. Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window.

10. Follow the procedure below for the type of electric force imaging desired, **Phase Detection**, **Frequency Modulation** or **Amplitude Detection** (see sections 18.2.4 through 18.2.6).

18.2.4 Phase Detection

1. In the **Auto Tune** panel, set **Start frequency** and **End frequency** to appropriate values for your cantilever (e.g., for 225µm MFM cantilevers, set **Start frequency** to 40kHz and **End frequency** to 100 kHz).

2. Select **Auto Tune**.

   Note: Two curves appear on the Cantilever Tune graph (see Figure 18.2e): the *amplitude* curve on top (the blue line in Figure 18.2e), and the *phase* curve below.

Figure 18.2e Phase Detection Cantilever Tune
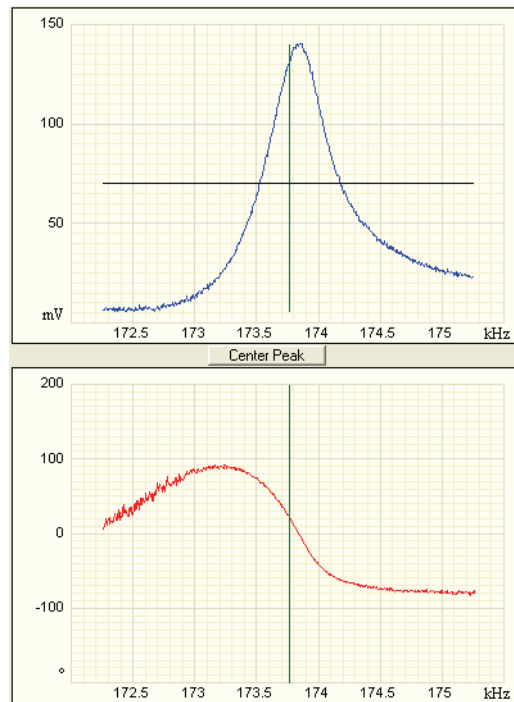
   Note: The phase should decrease with increasing frequency and cross the center line (0° point) at the peak frequency. The phase curve then correctly reflects the phase lag between the drive and the cantilever response. Gradients in the electric force cause a shift ΔF_0 in the resonance frequency. Resonance shifts also give rise to phase shifts $\Delta\phi$ used to generate an image of the electric force gradients; see Figure 18.2f.

Figure 18.2f Shift In Phase at Fixed Drive Frequency
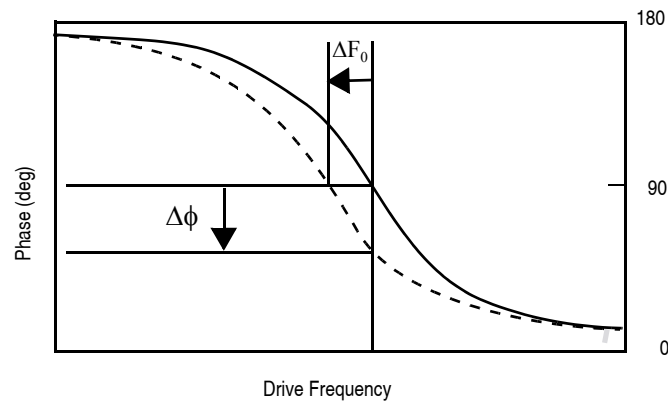
3. **EXIT Auto Tune**.

4. Engage the AFM and make the necessary adjustments to obtain a good topography (**Height**) image on Channel 1.

5. In the **Interleave** panel, shown in Figure 18.2g, set the **Interleave Mode** to **LIFT**.

6. In the **Interleave** panel, set the **Lift Start Height** to 0nm, and **Lift Scan Height** to 100 nm. (The lift height can later be optimized).

Figure 18.2g EFM Interleave Scan Parameters

7. Set the remaining Interleave parameters (**Setpoint**, **Drive Amplitude**, **Drive Frequency**, **Gains**) to the main Feedback values.

> **Note:** This can be done by setting the flags to the left of each parameter to "**off**" (grayed).

8. Set the **Channel 3 Data Type** to **PHASE** and choose **RETRACE** for the scan **Line Direction** on both Channel 1 and 3 images.

9. Set the **Channel 3 Scan Line** to INTERLEAVE to display interleave data. This screen should now display the cantilever phase change due to electrical force gradients from the sample in the right image and topography in the left image.

> **Note:** If a voltage is being applied to the tip or sample, it is recommended to apply it only during the **Interleave line** if feasible. Set TIP BIAS to the desired voltage in the **Interleave** panel. In the **Feedback** panel set TIP BIAS to **0V**.

10. Optimize the **Lift Scan Height**. For high-resolution, make the **Lift Scan Height** as small as possible without crashing the tip into the surface.

18.2.5 Frequency Modulation

For more quantitative results, use frequency modulation (FM). This technique provides a direct measure of change in resonant frequency sensed by the cantilever.

1.   Follow all the steps required to perform phase detection EFM.

2.   In the **Interleave** panel, set the **Input Feedback** to **FREQUENCY**.

3.   Switch the **Data Type** for **Channel 3** to **Frequency**.

   > **Note:**   It may be necessary to optimize the FM gains in the **Interleave** panel to properly track the shifts in resonant frequency (starting values: **Input Igain** = **40** and **Input Pgain** = **60**).

18.2.6 Amplitude Detection

Amplitude detection is inferior to the phase and frequency detection methods described previously and is not the recommended technique.

This section describes the differences in set up for amplitude detection. Changes in the cantilever amplitude provide an *indirect* measure of shifts in the cantilever resonance frequency as shown in Figure 18.2h.

Figure 18.2h   Shift In Amplitude at Fixed Drive Frequency
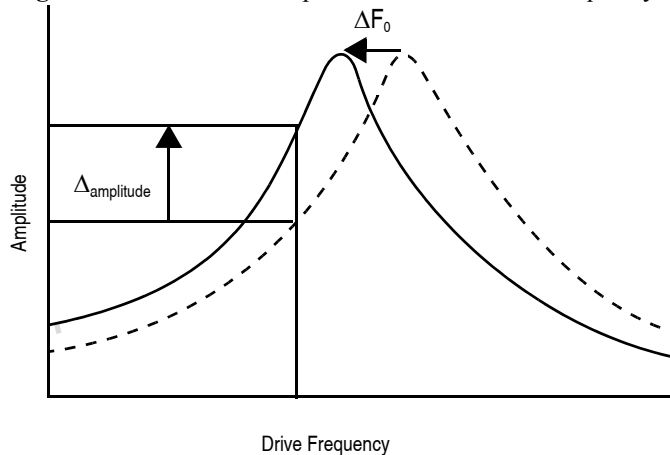
1. Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 18.2i.

Figure 18.2i   Amplitude Detection Cantilever Tune
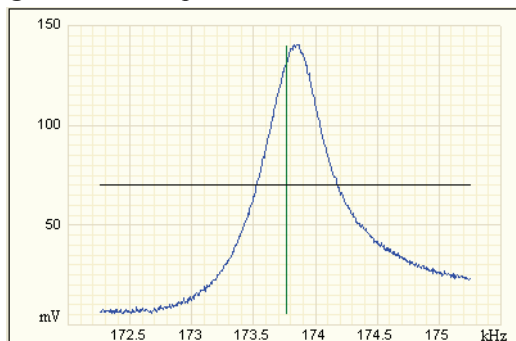
2. For maximum sensitivity, set the **Drive Frequency** to the steepest part of the resonance curve.

   Note: As the tip oscillates above the sample, a gradient in the electric force shifts the resonance frequency F_0 (see Figure 18.2h). Tracking the variations in oscillation amplitude while in LiftMode yields an image of the electric force gradients. You may use either side of the resonance, though we have obtained slightly better results on the low side, as shown in Figure 18.2h.

18.3   EFM Troubleshooting/Pointers

18.3.1 Use Low Setpoint When Tapping in Electric Field

If a voltage is applied during the main TappingMode line (as well as the interleave line) ensure proper operation since it is possible to enter a mode in which the tip is never actually touching the surface, even though the amplitude setpoint is being met. As the feedback moves the tip closer to the sample surface, the resonant frequency of the tip is shifting (see Figure 18.2h). The closer the tip is to the surface, the larger the force gradient and the more the resonant frequency shifts. It is possible for the resonant frequency to shift enough so that the amplitude setpoint is met without the tip touching the surface. The tip can be a significant distance away from the sample. The height image has comparatively poor resolution as does the corresponding electric field gradient image. A significantly lower amplitude setpoint is needed to really tap the surface in the presence of a strong electric field. The probability of this problem can be reduced by applying the **TIP** or **SAMPLE BIAS** only during the interleave scan line.

18.3.2 Verify Electric Field at Surface

If applying a **TIP** or **SAMPLE BIAS** to generate an E-field, set the phase channel **Realtime Plane Fit** to **NONE**. While viewing the scope trace, vary the **Interleave TIP** or **SAMPLE BIAS** value and verify that **PHASE** signal shifts accordingly.

18.3.3 Fine Tune Lift Scan Height

Set the lift height to be as small as possible without hitting the surface, because lateral resolution of EFM improves with decreased tip/sample separation. The minimum lift height depends on the roughness of the sample, the difference between the amplitude setpoint and free air amplitude, and the quality of the height image. Hitting the surface usually produces phase data with extremely high contrast (either black or white pixels).

18.3.4 Fine Tune Interleave Drive Amplitude

Decrease the drive amplitude in the interleave line to further minimize the lift height. Be aware that the S/N ratio also decreases with decreasing drive, so there are diminishing returns after a point.

18.3.5 Optimize Tune in Vicinity of Surface

Click the **Tune** icon in the **Workflow Toolbar** or select **Microscope** > **Cantilever Tune** to open the **Cantilever Tune** window while engaged. A tip offset of 30nm usually works well for smooth surfaces (if the tune has a noisy straight area in the middle instead of a smooth peak, the tip is hitting the surface and a larger tip offset is needed typically for rough surfaces). Adjust the **Drive Frequency** to center of peak or just to the left. If phase data is to be collected during the main line, zero the phase at the drive frequency.

18.3.6 Optimize Tune in Interleave

Find optimal lift height. Choose a **Tip Offset** equal to the **Lift Height**. Make any changes to the **Drive Frequency** and **Drive Phase** to adjust the main tune (tip #3). Activate (turn green by clicking) the **Drive Frequency** and **Phase** in the **Interleave** panel.

18.3.7 If Voltage is Needed, Use NanoScope When Possible

It is often very difficult to get a high quality TappingMode image with an electric field present. The NanoScope controller has an important advantage over an external power supply – it can be used to create an E field only in interleave while set to 0V in the main line. Its range is limited to +/- 12V.

18.3.8 Try Uncoated Si Tip

EFM has been successful with a variety of tips, including standard Si tips with no metal coating (fesp, tesp, ltesp). The Si is highly n-doped and is often conductive enough for EFM measurements. Metal coatings make the tip less sharp, decreasing lateral resolution. The standard tips also seem to give cleaner images over longer periods of time. There is likely a reduction in sensitivity to small E fields with the decreased conductivity.

18.3.9 Minimize Optical Interference

Optical interference may sometimes appear in the lift (electric force) image when imaging highly reflective samples. Optical interference appears as evenly spaced, sometimes wavy lines with about 1–2µm spacing superimposed on the lift image. This occurs when ambient laser light (i.e., light passing around or through the cantilever, then reflecting off the sample) interferes with laser light reflecting from the cantilever. Interference can be alleviated by moving the beam spot up a little along the cantilever away from the tip; about one-third of the cantilever length from the tip usually works well. On the Dimension head, the adjustment can be refined by carefully moving the beam spot laterally on the cantilever while scanning until interference fringes are minimized.

18.4 Surface Potential Detection

### 18.4.1 Surface Potential Detection Overview

Surface potential detection measures the effective surface voltage of the sample by adjusting the voltage on the tip to match that of the surface, thereby minimizing the electric force from the sample. Samples for surface potential measurements must have a surface voltage in the range [-10,+10 volts], and operation is easiest for voltages in the range of [-5,+5V]. The noise level with this technique is typically 10mV. Samples may include both conducting and nonconducting regions. Samples with regions of different metals will also show contrast due to contact potential differences. Quantitative voltage measurements are made of the relative voltages within a single image.

18.4.2 Surface Potential Detection—Theory

Theory Overview

Surface potential detection is a two-pass procedure where the surface topography is obtained by standard TappingMode in the first pass and the surface potential is measured on the second pass (see Figure 18.4a). The two measurements are interleaved: that is, they are each measured one line at a time with both images displayed on the screen simultaneously. A block diagram of the Surface Potential measurement system is shown in Figure 18.4b. On the first pass, in TappingMode, the cantilever is mechanically vibrated near its resonant frequency by a small piezoelectric element. On the second pass, the tapping drive piezo is turned off and an oscillating voltage $V_{AC} \sin \omega t$ is applied directly to the probe tip. If there is a DC voltage difference between the tip and sample, then there will be an oscillating electric force on the cantilever at the frequency ω. This causes the cantilever to vibrate, and an amplitude can be detected.

Figure 18.4a LiftMode Principles Used in Surface Potential Detection

Surface Potential Scope Data
(Interleave scan)

Topographic Scope Data
(Main scan)

Electric Field Sources
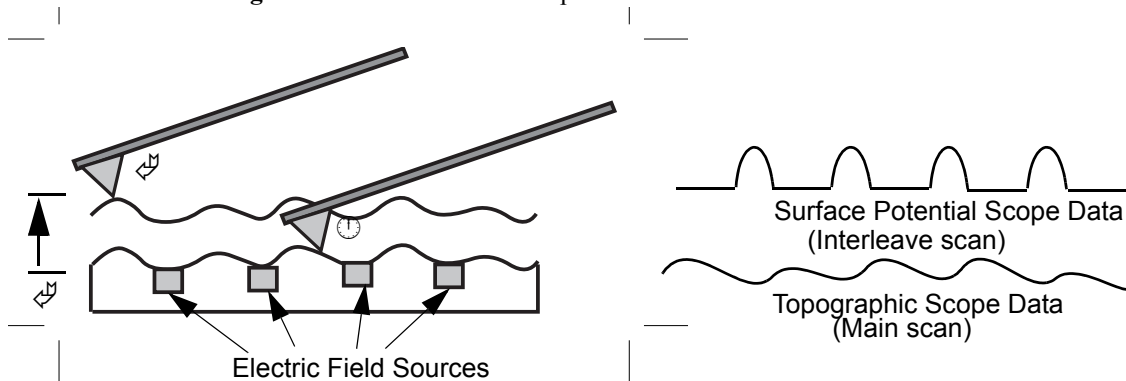
1. Cantilever measures surface topography on the first (main) scan.
2. Cantilever ascends to lift scan height.
3. Cantilever follows stored surface topography at the lift height above sample while responding to electric field influences on the second (interleave) scan.

If the tip and sample are at the same DC voltage, there is no force on the cantilever at ω and the cantilever amplitude will go to zero. Local surface potential is determined by adjusting the DC voltage on the tip, V_{tip}, until the oscillation amplitude becomes zero and the tip voltage is the same as the surface potential. The voltage applied to the probe tip is recorded by the NanoScope Controller to construct a voltage map of the surface.

**Figure 18.4b** Simplified Block Diagram of Surface Potential Detection
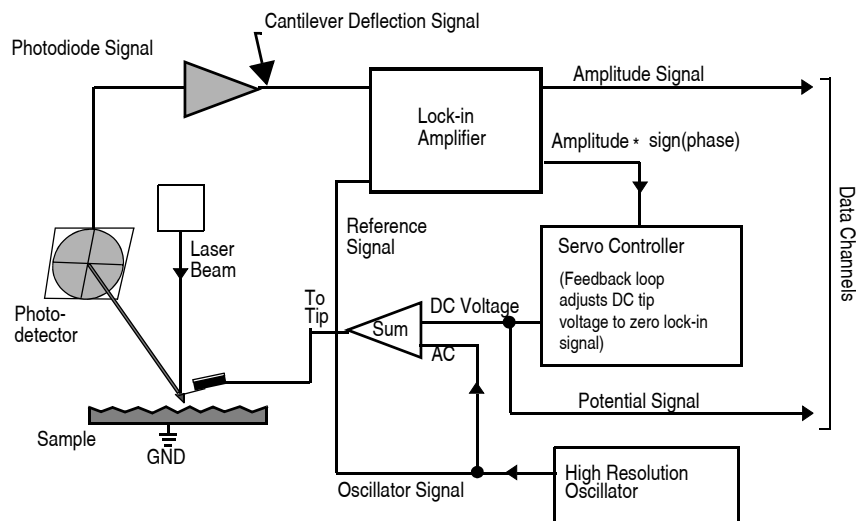
Theory Details

Surface potential microscopy can be modeled as a parallel plate capacitor. When two materials with different work functions are brought together, electrons in the material with the lower work function flow to the material with the higher work function. See Figure 18.4c. If these materials are charged, the system can be thought of as a parallel plate capacitor with equal and opposite surface charges on each side. The voltage developed over this capacitor is called the contact potential. Measuring the contact potential is done by applying an external backing potential to the capacitor until the surface charges disappear. At that point the backing potential will equal the contact potential. In surface potential microscopy (scanning Kelvin probe force microscopy), this "zero-charge" point is determined by adjusting the tip voltage so the electrical force felt by the AFM cantilever is "0".

Figure 18.4c   The electric energy level diagram for two conducting specimens, where ϕ_1 and ϕ_2 are the respective work functions. E_{F1} and E_{F2} are the respective Fermi energies.
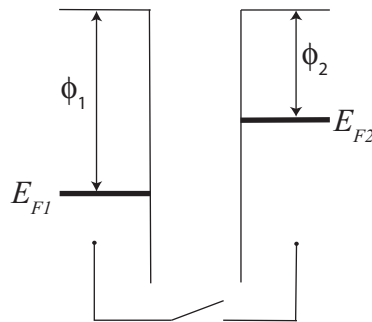

If an external electrical contact is made between the two electrodes, their Fermi levels equalize and the resulting flow of charge (in the direction indicated in Figure 18.4d) produces a potential gradient, termed the contact potential V_c, between the plates. The two surfaces become equally and oppositely charged.

Figure 18.4d   Electrical contact between the two specimens
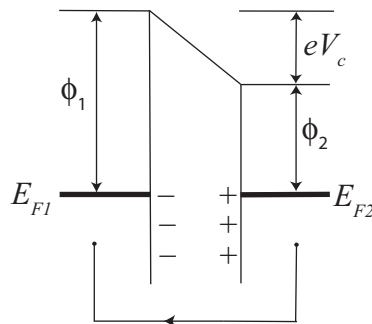
Inclusion of a variable backing potential, V_b, in the external circuit permits biasing of one electrode with respect to the other. When $V_b = V_c = (\phi_1 - \phi_2)/e$ — the average — the electric field between the plates vanishes. See Figure 18.4e.

Figure 18.4e   Variable backing potential, V_b, added

$$V_b = -V_c$$
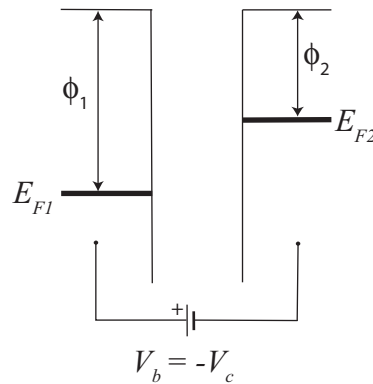
A good way to understand the response of the cantilever during Surface Potential operation is to start with the energy in a parallel plate capacitor, $U = \frac{1}{2}C(\Delta V)^2$, where C is the local capacitance between the AFM tip and the sample and ΔV is the voltage difference between the two. The force on the tip and sample is the rate of change of the energy with separation distance:

$$F = -\frac{dU}{dZ} = -\frac{1}{2}\frac{dC}{dZ}(\Delta V)^2$$

Figure 18.4f   Force as a Function of Voltage
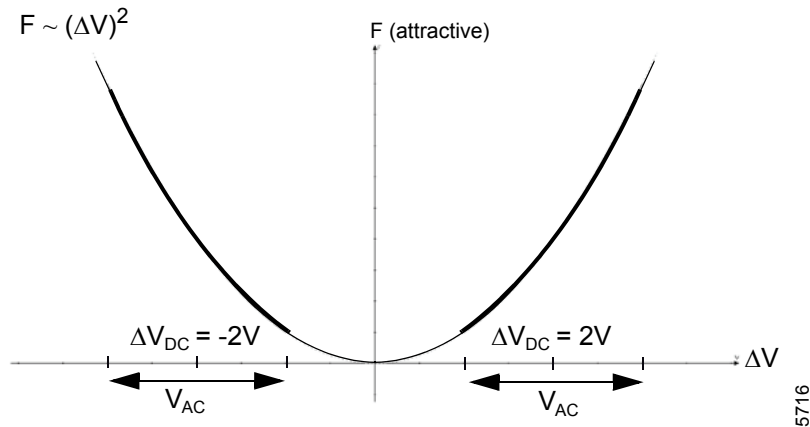
The voltage difference, ΔV, in Surface Potential operation consists of both a DC and an AC component. The AC component is applied from the oscillator, $V_{AC}\sin\omega t$, where ω is the resonant frequency of the cantilever.

$$\Delta V = \Delta V_{DC} + V_{AC}\sin\omega t$$

ΔV_{DC} includes applied DC voltages (from the feedback loop), work function differences, surface charge effects, etc. Squaring ΔV and using the relation $2\sin^2 x = 1 - \cos(2x)$ produces:

$$F = -\frac{1}{2}\frac{dC}{dZ}(\Delta V_{DC}^2 + \frac{1}{2}V_{AC}^2) - \frac{dC}{dZ}\Delta V_{DC} V_{AC} \sin\omega t + \frac{1}{4}\frac{dC}{dZ}V_{AC}^2 \cos(2\omega t)$$

<p style="text-align:center;">DC term ω term 2ω term</p>

The oscillating electric force at ω acts as a sinusoidal driving force that can excite motion in the cantilever. The cantilever responds only to forces at or very near its resonance, so the DC and 2ω terms do not cause any significant oscillation of the cantilever. In regular TappingMode, the cantilever response (RMS amplitude) is directly proportional to the drive amplitude of the tapping piezo. Here the response is directly proportional to the amplitude of the F_ω drive term:

$$\textit{amplitude of } F_\omega = \frac{dC}{dZ}\Delta V_{DC}V_{AC}$$

The goal of the Surface Potential feedback loop is to adjust the voltage on the tip until it equals the voltage of the sample ($\Delta V_{DC}=0$), at which point the cantilever amplitude should be zero ($F_\omega=0$).

The larger the DC voltage difference between the tip and sample, the larger the driving force and resulting amplitude will be. But the F_ω amplitude alone is not enough information to adjust the voltage on the tip. The driving force generated from a 2V difference between the tip and sample is the same as from a -2V difference (see Figure 18.4f).

What differentiates these states is the phase. The phase relationship between the AC voltage and the force it generates is different for positive and negative DC voltages (see Figure 18.4g through Figure 18.4j).

In the case where ΔV_{DC} = 2V, the force is in phase with V_{AC}. When ΔV_{DC} = -2V, the force is out of phase with V_{AC}. Thus, the cantilever oscillation will have a different phase, relative to the reference signal V_{AC}, depending on whether the tip voltage is larger or smaller than the sample voltage. Both the cantilever amplitude and phase are needed for the feedback loop to correctly adjust the tip voltage. The input signal to the Surface Potential feedback loop is the cantilever amplitude multiplied by the sign of its phase (i.e., amplitude for phase ≥0 degrees, -amplitude for phase <0 degrees). This signal can be accessed in the software by selecting **POTENTIAL INPUT** (interleave scan line) in one of the channel panels.

If ΔV_{DC} = 0, the electric drive force is at the frequency 2ω. The component of the force at ω is zero so the cantilever does not oscillate (see Figure 18.4k and Figure 18.4l). The Surface Potential feedback loop adjusts the applied DC potential on the tip, V_{tip}, until the cantilever's response is zero. V_{tip} is the Potential data that is used to generate a voltage map of the surface.
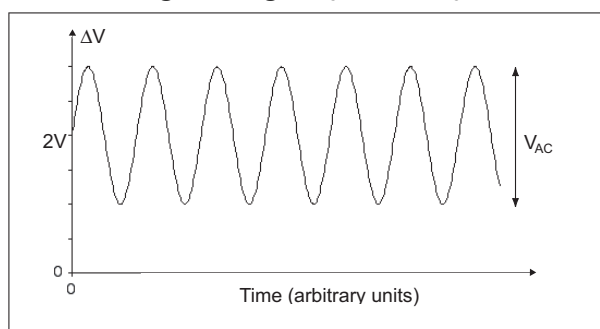
Figure 18.4g V_{AC} at ω, $\Delta V_{DC} = 2V$

Figure 18.4h Major Force Component in Phase with V_{AC} at Frequency ω, $\Delta V_{DC} = 2V$
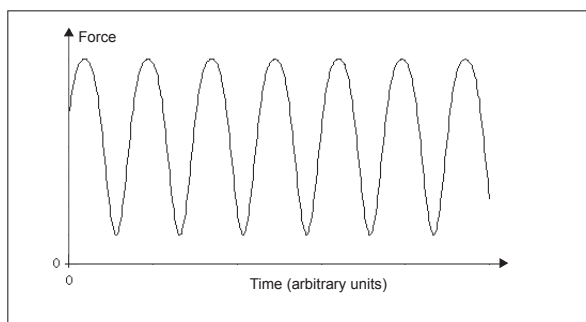

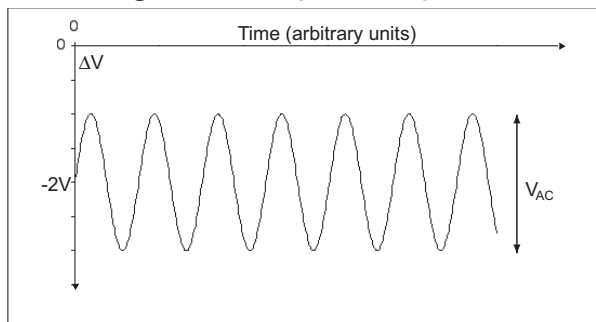**Figure 18.4i** V_{AC} at ω, $\Delta V_{DC} = -2V$

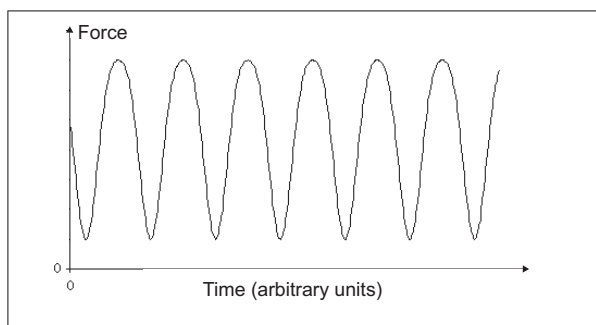**Figure 18.4j** Major Force Component 180° Out of Phase with V_{AC} at Frequency ω, $\Delta V_{DC} = -2V$

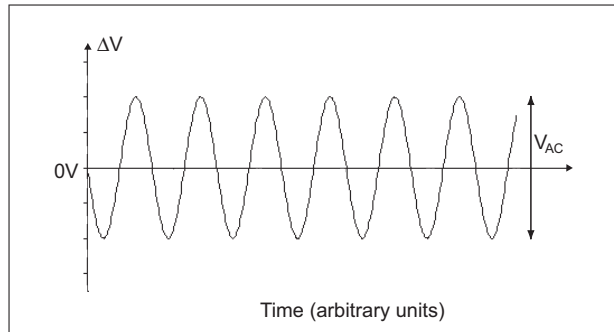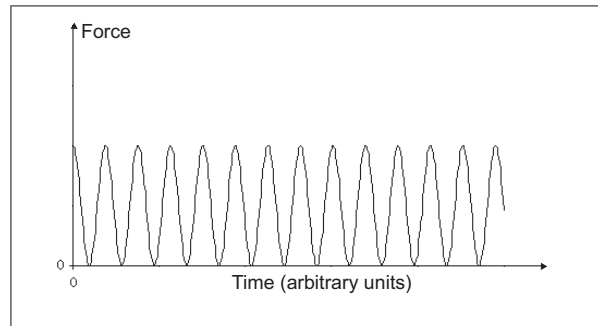**Figure 18.4k** V_{AC} at ω, ΔV_{DC}= 0V
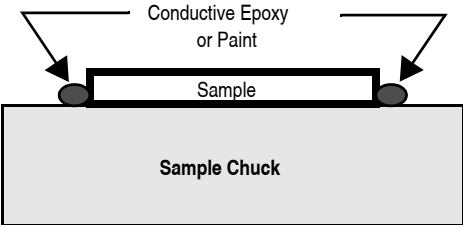

Figure 18.4l   Force at frequency of 2ω, ΔV_{DC} = 0V

18.5   Surface Potential Detection—Preparation

The sample should be electrically connected directly to the chuck, so that it can be held at ground potential (normal operation) or biased through the chuck, or a standard sample puck using conductive epoxy or silver paint as shown below:

18.6 Surface Potential Imaging—Procedure

1. Mount a sample onto the sample holder.

2. Mount a metal-coated cantilever into the standard probe holder. MFM-style cantilevers (MESP, 225µm long, with resonant frequencies around 70kHz) usually work well. It is also possible to deposit custom coatings on model FESP silicon TappingMode cantilevers. Verify that all deposited metal adheres strongly to the silicon cantilever. SCM-PIT and OSCM-PT also work well for surface potential. Uncoated highly doped Si probes such as FESP and LTESP, which provide higher resolution, can also be used.

3. Click the **SELECT EXPERIMENT** icon.

4. Select **ELECTRICAL AND MAGNETIC AFM** in the **Experiment Category** panel.

5. Select **SURFACE POTENTIAL MICROSCOPY (SPoM)** in the **Experiment** panel and click **START EXPERIMENT**.

6. Set up the AFM as usual for TappingMode operation (see Chapter 11).

7. Use **Cantilever Tune**: **AutoTune** to locate the cantilever's resonant peak.

In this case, two curves should appear in the **Cantilever Tune** window: the *amplitude* curve and the *phase* curve. In the event you find more than one resonance, select a resonance that is sharp and clearly defined, but not necessarily the largest. It is also helpful to select a resonant peak where the lock-in phase also changes very sharply across the peak. Multiple peaks can often be eliminated by making sure the cantilever holder is clean and the cantilever is tightly secured.

8. Engage the AFM and make the necessary adjustments for a good TappingMode image while displaying height data.

9. Set the **Interleave** gains:

   a. **Input Igain** = **1**

   b. **Input Pgain** = **5**

10. In the **Interleave** panel, shown in Figure 18.6a, set the **Interleave Mode** to LIFT.

11. In the **Interleave** panel, set the **Lift Start Height** to 0nm, and **Lift Scan Height** to 100 nm. (The lift height can later be optimized).

12. Leave the **Interleave Drive Frequency** at the main **Feedback** value (gray).

13. Set the **Input Feedback** to POTENTIAL.

Figure 18.6a   Surface Potential Interleave Scan Parameters (Expanded Mode)

14. Enter an Interleave **Drive Amplitude**. This is the ac voltage that is applied to the AFM tip. Higher **Drive Amplitude** produces a larger electrostatic force on the cantilever and this makes for more sensitive potential measurements. Conversely, the maximum total voltage (ac + dc) that may be applied to the tip is ±10V. So a large **Drive Amplitude** reduces the range of the DC voltage that can be applied to the cantilever. If the sample surface potentials to be measured are very large, it is necessary to choose a small **Drive Amplitude**, while small surface potentials can be imaged more successfully with large **Drive Amplitudes**. To start choose a **Drive Amplitude** of **500mV**.

15. Set the Channel 4 image **Data Type** to POTENTIAL. Set the scan **Line Direction** for the main and interleave scans to RETRACE. Remember to choose the RETRACE direction because the lift step occurs on the trace scan and can cause artifacts in the data. Set the **Channel 4 Scan Line** to INTERLEAVE.

16. Adjust the Input gains. As with the topography gains, the scan can be optimized by increasing the gains to maximize feedback response, but not so high that oscillation sets in. More information on tuning the feedback loop is given in Section 18.6.1, below.

17. Optimize the lift heights. Set the **Lift Scan Height** at the smallest value possible that does not make the Potential feedback loop unstable or cause the tip to crash into the sample surface. When the tip crashes into the surface during the Potential measurement, dark or light streaks appear in the Potential image. In this case, increase the **Lift Scan Height** until these streaks are minimized.

18. Optimize the drive phase. **Lock-In Phase** adjusts the phase of the reference signal to the lock-in amplifier. As discussed in **Surface Potential Detection—Theory:** Section 18.4.2, the correct phase relationship must exist between the reference and the input signals to the lock-in for the potential feedback loop to perform correctly. **Lock-In Phase** depends on the mechanical properties of the cantilever. For cantilevers with resonant frequencies from 60-80kHz (such as MESP, SCM-PIT, and FESP), use an interleave **Lock-In Phase** of 170 degrees. For cantilevers with higher resonant frequencies, increased electronics phase lag must be compensated. For cantilevers with resonant frequencies around 300kHz (such as TESP, RTESP) an interleave **Lock-In Phase** near 130 degrees often works well. Follow the procedure detailed in **Determination of Lock-in Phase:** Section 18.7 to more accurately determine the **Lock-In Phase**.

19. For large sample voltages or qualitative work, select a **Data Type** = PHASE in addition to POTENTIAL. When the controller has been configured for surface potential measurements, the "phase" signal is actually the cantilever amplitude signal, as measured by a lock-in amplifier. If the feedback loop is not enabled by selecting the **Data Type** = POTENTIAL, the lock-in cantilever amplitude depends on the voltage difference between the tip and sample in a roughly linear fashion. (The lock-in amplifier produces a voltage that is proportional to the cantilever amplitude.) Qualitative surface potential images can be collected using this lock-in signal. Also, if the sample has a surface potential that exceeds ±10V (greater than the range of the "Potential" signal), it is possible to use the lock-in signal to provide qualitative images that reflect the sample surface potential. To view the lock-in signal with the reconfigured controller, select the **Data type** = PHASE.

18.6.1 Troubleshooting the Surface Potential Feedback Loop

The surface potential signal feedback loop can be unstable. This instability can cause the potential signal to oscillate or become stuck at either +10V or -10V. Here are some tips to see if the feedback loop is working properly with no oscillation:

- Look at the scope display for the potential signal. If oscillation noise is evident in the signal, reduce the gains. If oscillations persist even at very low gains, try increasing the **Lift Scan Height** and/or reducing the **Drive Amplitude** until oscillation stops. If the tip crashes into the surface the Lock-in signal becomes unstable and can cause the feedback loop to malfunction. Increasing the **Lift Height** and reducing the **Drive Amplitude** can prevent this problem. Once oscillation stops, the gains may be increased for improved performance.

If the Potential signal is perfectly flat in the **Scope** panel and shows no noise even with a small Z-range, the feedback loop is probably stuck at ±10V. (You can verify this by changing the value of **Realtime Plane Fit** to NONE in the **Channel 1** panel.) Reduce the **Scan Rate** and watch the scope trace which indicates the cantilever amplitude. On the topographic trace, the voltage displayed should be the setpoint selected for the Main scan. On the Potential trace, this voltage drops close to zero if the cantilever oscillation is being successfully reduced. If the value instead goes to a large nonzero value, the feedback loop is probably not working properly. In this case, try reducing the **Drive Amplitude** and increasing the **Lift Scan Height** or optimizing the Lock-In Phase (see Section 18.7). It may also be helpful to momentarily turn the **Interleave Mode** to DISABLED, then back to INTERLEAVE. Also try reducing any external voltage that is being applied to the sample to stabilize the feedback loop, then turn the voltage back up.

18.7 Determination of Lock-in Phase

For surface potential microscopy (also called scanning Kelvin probe force microscopy-SKPFM) measurements, the feedback adjusts the DC voltage applied between the tip and sample to compensate their intrinsic potential difference. The DC voltage map across the surface thus reflects surface potential variations of the sample surface.

When the difference is perfectly compensated, the tapping oscillation amplitude of the metal coated AFM cantilever (excited by an AC bias field) approaches "0", the very reason this technique is called a nulling technique. In doing so, the feedback uses the tapping amplitude and phase to determine whether to increase or decrease the DC voltage. Phase is the one parameter that users may have difficulty selecting. The following sections outline a procedure to address this matter using **Generic Sweep** available in NanoScope software.

While the procedure itself is simple, plots are shown for better understanding of the measurement technique.

Generic Sweeps are taken on **Interleave**. In some cases, it is helpful to switch back and forth between **MAIN** and **INTERLEAVE** for the sweep parameters to take effect. Some inconsistency may occur, for instance, potential feedback may not actually be on when **Input Igain** and **Input Pgain** are non-zero.

18.7.1 Interleave Tuning Curve - Phase Lag

Refer to Figure 18.7a. A phase difference, here $-90°$ (in the bottom plot), is usually seen between tip oscillation signal and the AC bias driving signal. This phase lag varies from tip to tip, with operating frequency, and the potential difference between the tip and the spot on the sample.

**Figure 18.7a** Interleave Tuning Curve - Phase Lag
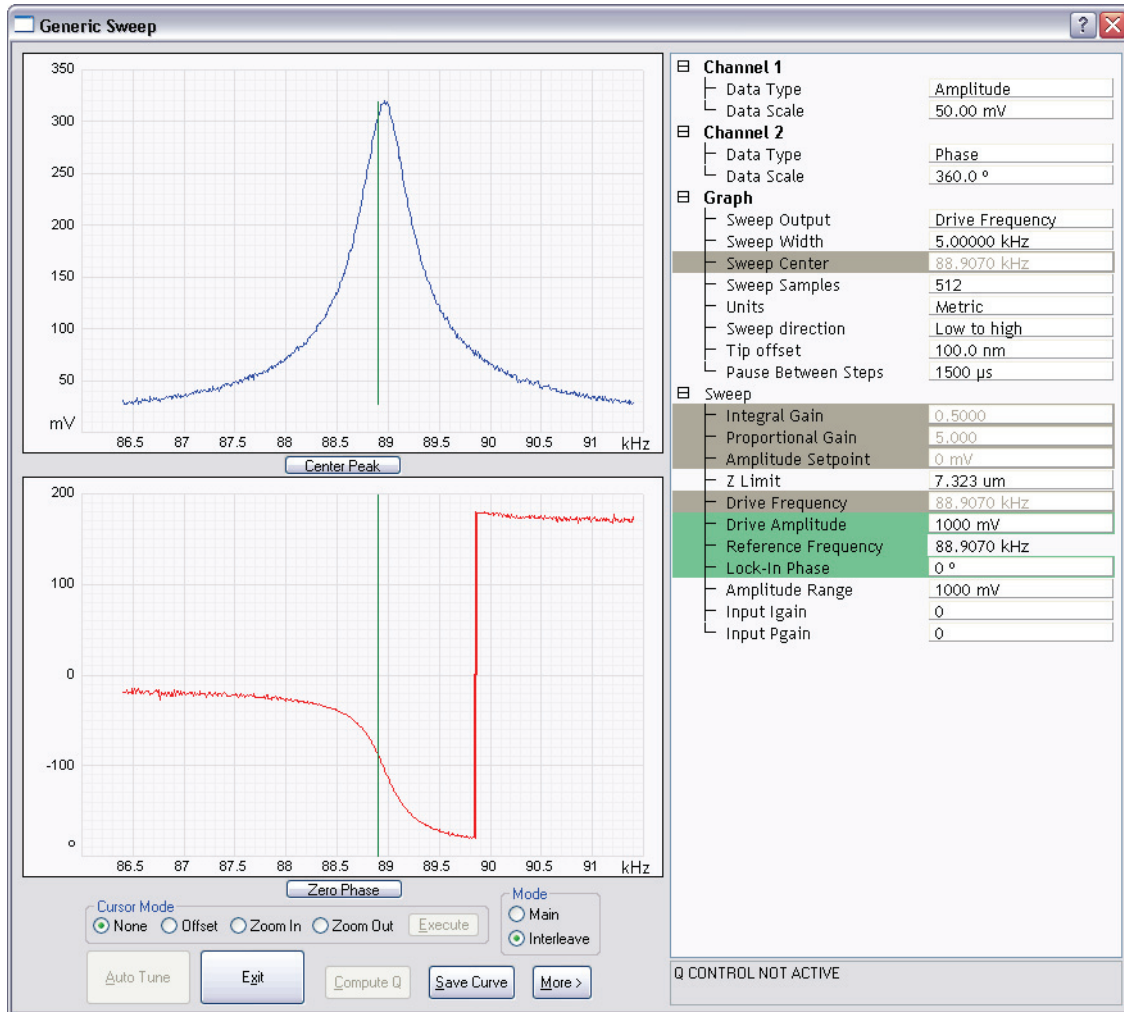
18.7.2 Amplitude and Phase vs. Sample Bias—Feedback Off

Figure 18.7b shows a **Generic Sweep** of SAMPLE BIAS while the potential feedback is turned off. Note that **Input Igain** and **Input Pgain** are 0.

The amplitude, shown in the upper plot, dips to 0 when the sample is biased to match the tip potential; as one moves away from that point, the amplitude increases. Potential feedback seeks that zero point. Because amplitude alone can not determine whether one is on the left side or right side of that zero amplitude point, the feedback relies on the phase to determine which direction to move the DC bias.

Figure 18.7b shows the phase (bottom plot) changing by 180° across "0" amplitude. It is desirable to choose a **Lock-In Phase** to offset the oscillation phase, so that the phase is negative when the tip voltage is positive relative to the sample. Figure 18.7b shows that when the sample bias is -2V (tip is positive), the output **Lock-In Phase** is 90°. The **Interleave Lock-In Phase** then needs to be set to -180° to make it -90°.

Figure 18.7b Amplitude and Phase vs. Sample Bias. Feedback Off
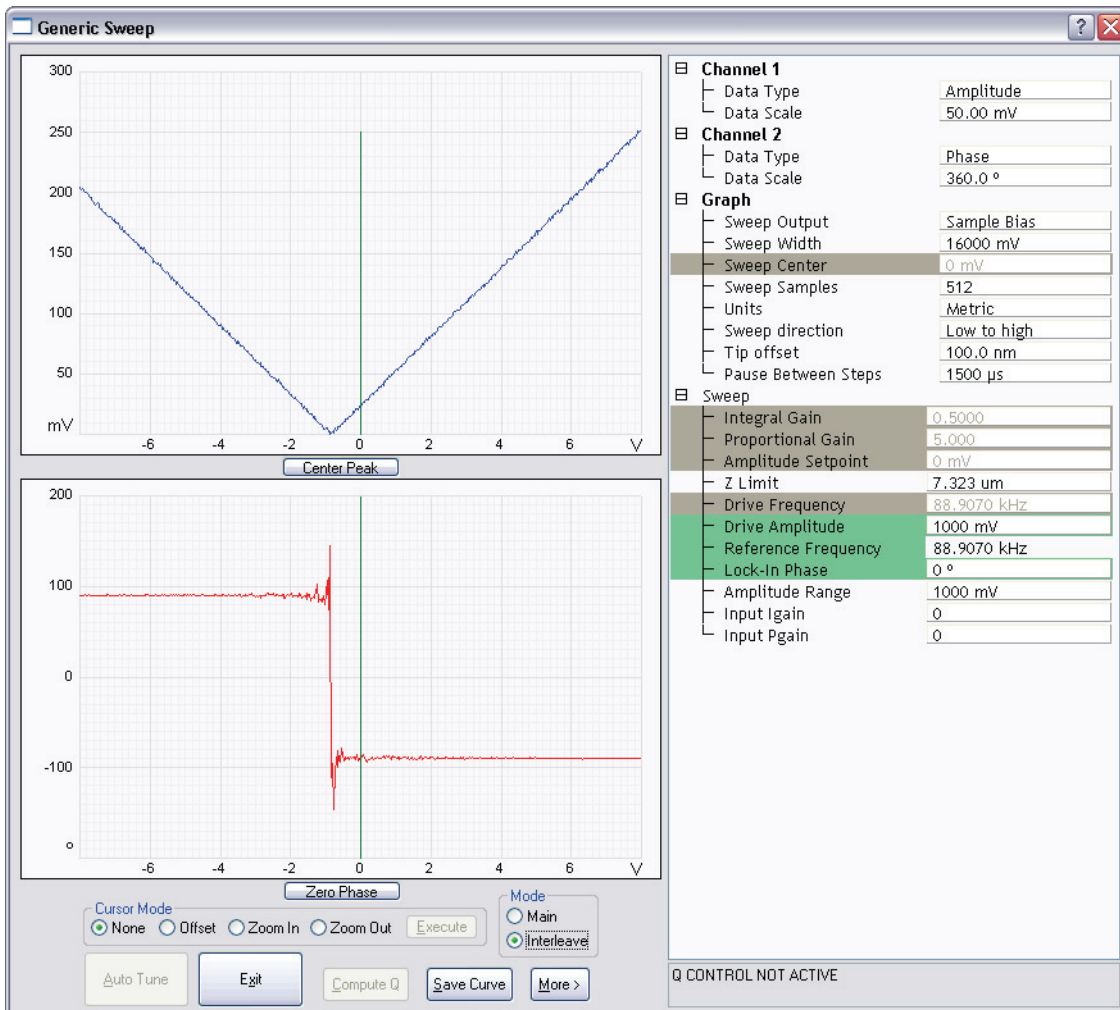
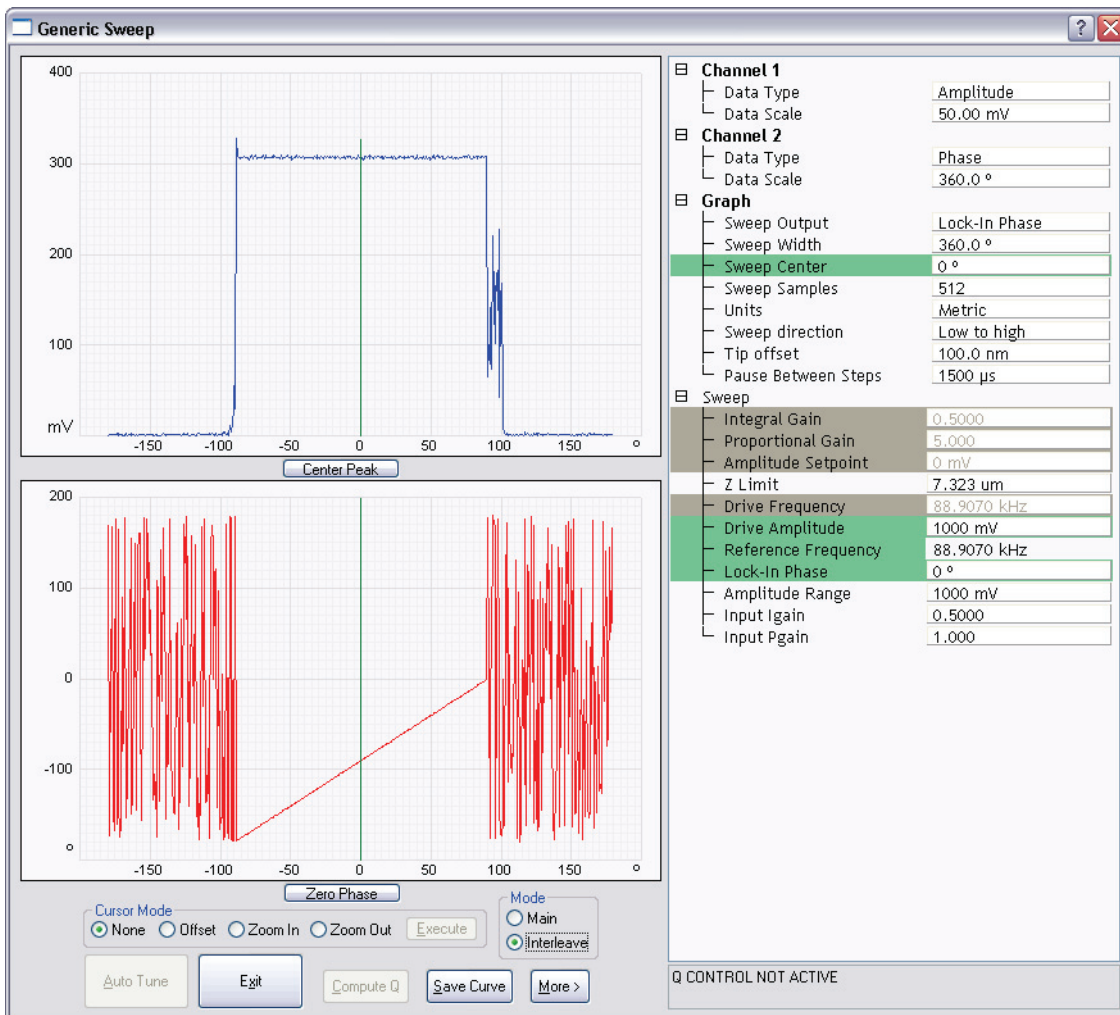### 18.7.3 Phase vs. Lock-In Phase, Feedback On—The Straightforward Approach

Refer to Figure 18.7c. It is logical to sweep the lock-in phase in a full circle to determine the phase range where feedback is working while the feedback is turned on (**Input Igain** and **Input Pgain** are non-zero).

The top plot shows how the amplitude changes while lock-in phase is swept. In the mid-range of the phase sweep (approximately $-100°$ to $+100°$), the amplitude stays non-zero, indicating that the phase is not correct for the feedback to work properly. On both ends, the amplitude remains around "0", implying that feedback is working properly in "nulling" the amplitude. In principle, it is fine to choose any lock-in phase in these two regions $100° \sim 180°$ and $-180° \sim -90°$ (equivalent to $180° \sim 270°$); actually a combined region of $100° \sim 270°$. It makes practical sense, however, to choose a value somewhere in the middle to have a good margin, e.g. $-170°$.

The lower plot shows phase changing linearly in the middle of the sweep range, while jumping around when feedback is working on either end. This is explained in **Amplitude and Phase vs. Sample Bias—Feedback Off:** Section 18.7.2.

Figure 18.7c   Phase vs. Lock-In Phase Feedback On—the Straightforward Approach

18.7.4 Phase vs. Lock-In Phase Feedback Off—An Alternative Approach

Refer to Figure 18.7d, Generic Sweep of Lock-in phase while potential feedback is **OFF** (Input Igain and Input Pgain are "0").

The top plot shows the amplitude remaining unchanged while the **Lock-In Phase** is swept.

The bottom plot shows the phase changing linearly as the lock-in phase is swept. There is one exception where the phase jumps from 180° to -180°, so-called phase wrapping.

In case the plots in the previous section were not obtained (due to software inconsistency), do the following. From the left bottom plot, choose a lock-in phase where oscillation phase falls within -180°~0° (or 0~180°), e.g. 10° (-170°), if this does not work, add/subtract 180°, that is -170° (10°).

Of course, one can simply pick any lock-in phase, if it works, fine. Otherwise, change the phase by 180°. The difference is that with the above outlined approach you know what kind of margin you have.

Figure 18.7d   Phase vs. Lock-In Phase, Feedback Off—an Alternative Approach
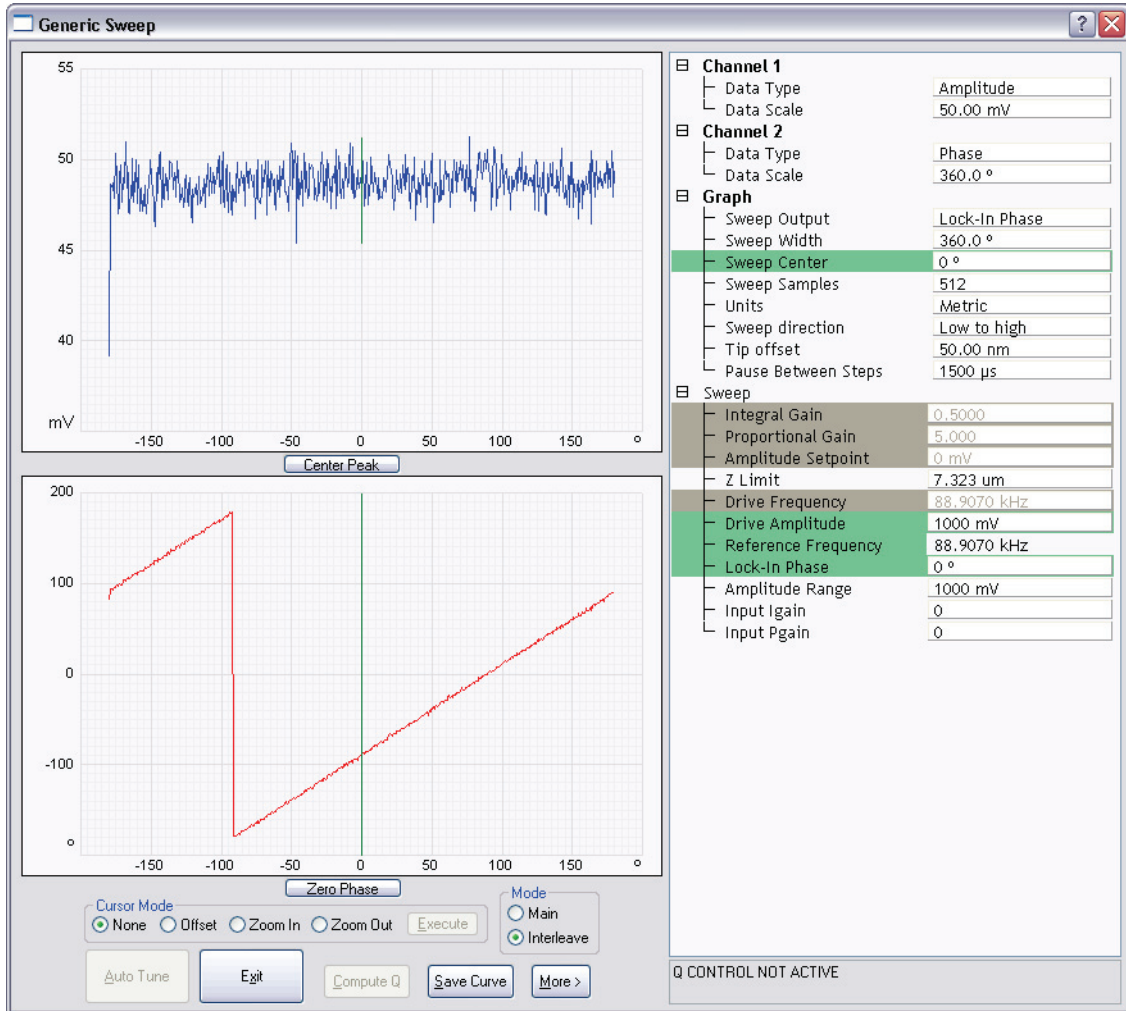
Chapter 19   Calibration

This chapter provides detailed instructions for the fine calibration of Dimension SPMs. Specifically this chapter includes the following topics:

- **Auto Calibration:** Section 19.7

- **Z Sensor Sensitivity:** Section 19.11.6

- **Calculate Retracted and Extended Offset Deratings:** Section 19.11.7

- **Closed Loop Z Calibration:** Section 19.12

19.1 SPM Calibration Overview

Veeco employs a software-guided calibration procedure for all microscopes. The procedural details of how calibration is executed using NanoScope software are beyond the scope of this document and include proprietary methods exclusive to Veeco. A brief overview of the theory is available in Section 19.1.1. The calibration is summarized in four basic steps outlined below.

- **Orthogonality Adjustments**: The orthogonality adjustment compensates for a possible non-square scan pattern.

- **Scanner Linearization**: Adjust parameters for correcting the capacitive nature of the piezo material and force the X-Y motion of the piezo to move at a constant velocity.

- **X, Y Calibration**: Designate the software conversion values using one or both of the following methods.

    - **Capture Calibration and Auto calibration**: A built-in software routine automatically controls the scanner while capturing a series of data files. The data files are then used to "teach" the software the actual distance that the scanner moved. This process creates a customized set of conversion values that will be used to convert the known signals to a metric scale.

        -or-

    - **Fine Tuning**: Manually test and adjust X-Y sensitivity and derating values to within 1% accuracy. The conversion values that scale the scanner motion are confirmed, or are adjusted if necessary to within 1%. These same values should be adjusted by the customer as the scanner ages due to use. A scanner will typically lose 10-20% of its scan range over the first year of typical use. The scanner will then become stable as long as it is not damaged.

- **Calibrate Z**: Calibrate the Z scan tube to within 2%. A separate calibration "look-up" table stores the conversion values for converting the control voltages applied to the Z tube. These values are determined by scanning over a sample with a known depth.

Veeco recommends that you adhere to the Calibration schedule shown in Table 19.1a. After initial installation, perform the **Fine-Tuning X-Y Calibration** (see Section 19.8) 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 19.3). For most applications it is sufficient to perform the Z calibration with the same frequency. For critical height measurements we recommend monthly Z calibration.

Table 19.1a 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

Note: **Small Scan Size Calibration**: If using scan sizes of 5µm or smaller, Veeco recommends calibrating the scanner for small scan sizes. Contact Veeco for further instructions.

19.1.1 Theory Behind Calibration

Scanners typically consist of a hollow tube made of piezoelectric material such as PZT (lead zirconium titanate). Piezo materials contract and elongate when voltage is applied, according to whether the voltage is negative or positive, and depending upon the orientation of the material's polarized grain structure. Scanners are used to precisely manipulate sample-tip movement in order to scan the sample surface. In Dimension SPMs, the sample is stationary while the scanner moves the tip.

Not all scanners react exactly the same to a voltage. Because of slight variations in the orientation and size of the piezoelectric granular structure (polarity), material thickness, etc., each scanner has a unique "personality." This personality is conveniently measured in terms of *sensitivity*, a ratio of piezo movement-to-piezo voltage. Sensitivity is not a linear relationship, however. Because piezo scanners exhibit more sensitivity (i.e., more movement per volt) at higher voltages than they do at lower voltages, the sensitivity curve is just that—curved. This non-linear relationship is determined for each scanner crystal and follows it for the life of the scanner. As the scanner ages, its sensitivity will decrease somewhat, necessitating periodic recalibration.

The diagram below depicts scanner crystal voltage versus photodiode voltage (see Figure 19.1a). In this instance, detector sensitivity is given as volt per volt.

Figure 19.1a Scanner Crystal Voltage and Photodiode Voltage
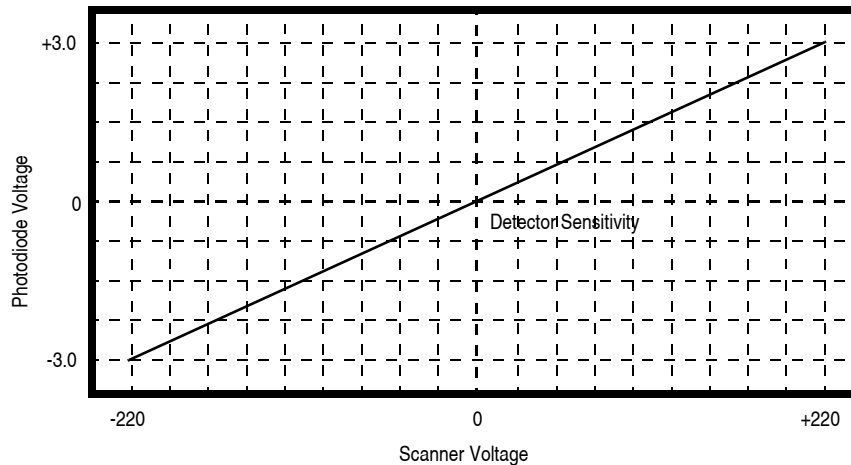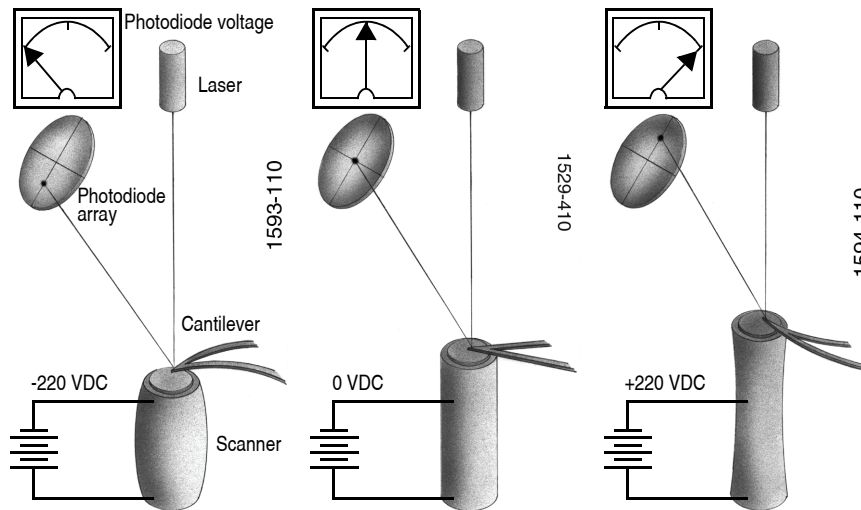
The **Calibrate** > **Scanner > XY...** function displays the **Scanner Calibration** dialog box, allowing users to enter the sensitivity of their scanner's X-Y axes. Sensitivity is measured in terms of lateral displacement for a given voltage (nm/volt). In addition, the NanoScope software employs various derating and coupling parameters to model scanners' nonlinear characteristics. By precisely determining points along the scanner's sensitivity curve, then applying a rigorous mathematical model, full-range measuring capabilities can be achieved with better than 1 percent accuracy. Consider the sensitivity curve represented in Figure 19.1b.

Figure 19.1b Scanner Sensitivity Curve
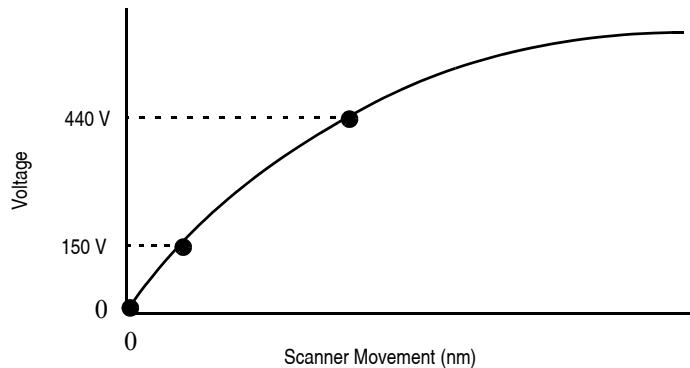
This curve typifies scanner sensitivity across the full range of movement. The vertical axis denotes voltage applied to the scanner. The horizontal axis denotes scanner movement. At higher voltages, the scanner's sensitivity increases (i.e., more movement per voltage applied). At zero volts, the scanner is "motionless." 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. The graph below represents this relationship (see Figure 19.1c).

Figure 19.1c Effect of Nonlinearity and Hysteresis
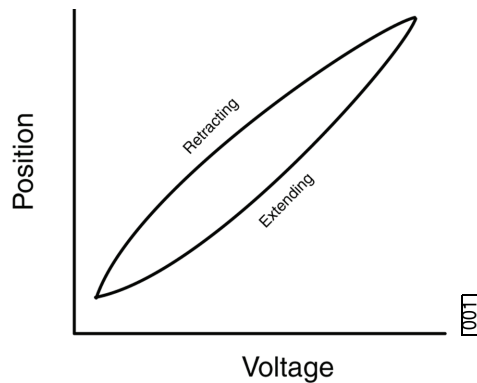
To produce the sharp, linear movements (triangular waveform) required for accurate back-and-forth scanning, it is necessary to shape the applied voltage as shown on the top graph in Figure 19.1d. Moreover, the applied voltage must compensate for scan rate and scan size. As scan rate slows, the applied voltage must compensate for increased memory effects in the piezo material. As scan size is decreased, the piezo exhibits more linearity. These effects are further complicated by X-Y-Z coupling effects (the tendency for one axis to affect movement in other axes).

Figure 19.1d Scanner Voltage and Movement
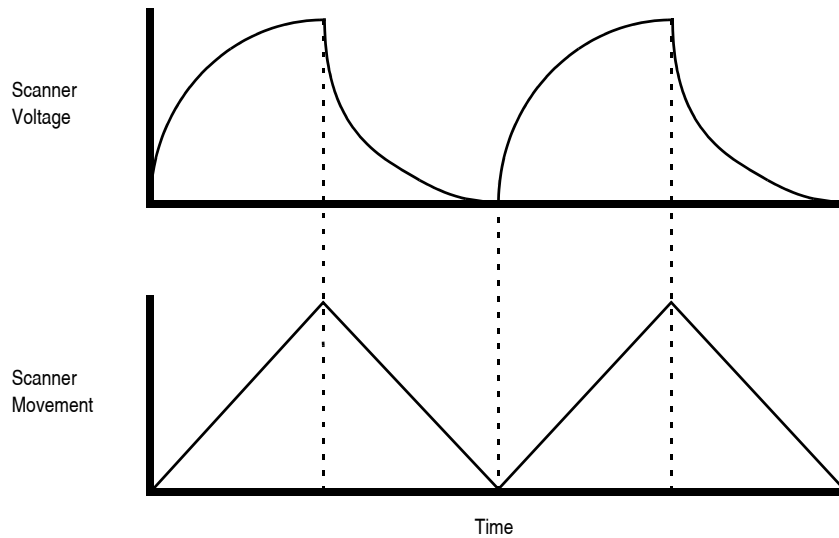
Through rigorous quality control of its scanner piezos, Veeco has achieved excellent modeling of scanner characteristics. Two calibration points are typically used for fine-tuning: 150 and 440 volts. A third point is assumed at 0 volts. These three points yield a second-order sensitivity curve to ensure accurate measurements throughout a broad range of scanner movements.

Because scanner sensitivities vary according to how much voltage is applied to them, the reference must be thoroughly scanned at a variety of sizes and angles. The user dictates, via the software, the distance between known features on the reference's surface and a parameter is recorded to compensate for the scanner's characteristics. The X, Y and Z axes may be calibrated in any sequential order; however, the linearization adjustments must be performed before any calibrations are attempted (see **Linearity Correction:** Section 19.5). Otherwise, calibrations will be undone by the linearity adjustments.

> **Note:** Check the SPM's measuring accuracy periodically to ensure that images are dimensionally represented within acceptable limits of error. If measuring accuracy is critical, or if environmental factors (e.g., humidity, temperature) impact the SPM significantly, this may require a quick check at the start of each imaging session. Establish a three-month service schedule for maintenance and calibration.

19.1.2 Calibration References

As described above, each scanner exhibits its own unique sensitivities; therefore, it is necessary to precisely measure these sensitivities, then establish software parameters for controlling the scanner. This task is accomplished with the use of a calibration reference (see Figure 19.1e).

Figure 19.1e Silicon Calibration Reference
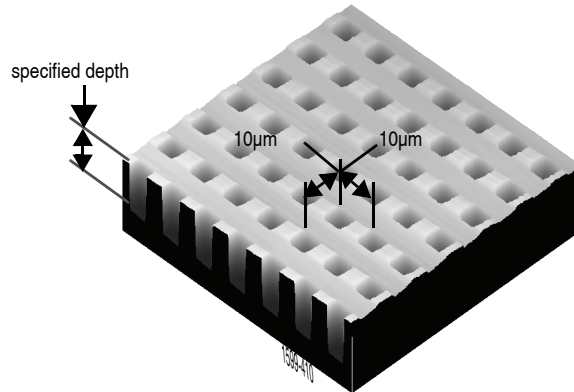
A calibration reference consists of a silicon substrate having a regular series of pits, at a depth specified on the container (typically 180 or 200nm), which is plated with platinum. Pits are spaced apart on 10µm centers. Other similar surfaces are available with different dimensions. Atomic-scale calibrations are generally carried out with mica or graphite, which exhibit very regular atomic lattices. Calibration references serve as the primary tool by which SPMs are calibrated. They serve as measuring sticks with which to gauge scanner displacement for a given voltage.

The SPM should be capable of measuring a calibration reference with an accuracy of 2 percent or better while scanning at the maximum **Scan size** setting. Using fine calibration techniques, it is possible to calibrate the SPM with even greater accuracy.
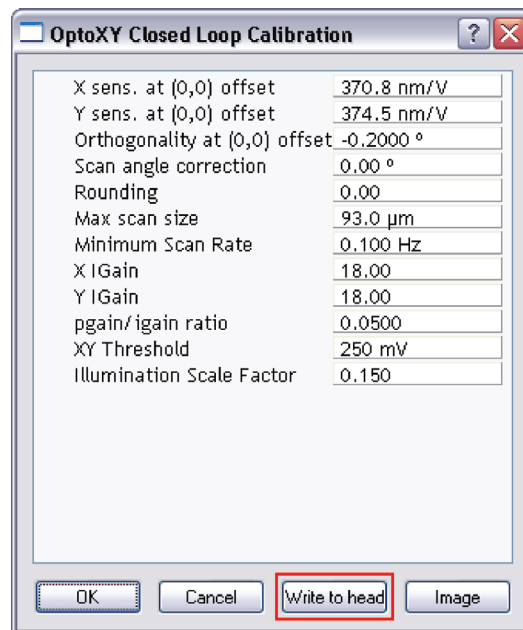
19.2   Calibration Parameters

NanoScope software versions 8.10 and later incorporate the ability to store calibration parameters on flash memory inside the Dimension Icon head. This permits easy swapping of heads and simplifies calibration parameter management.

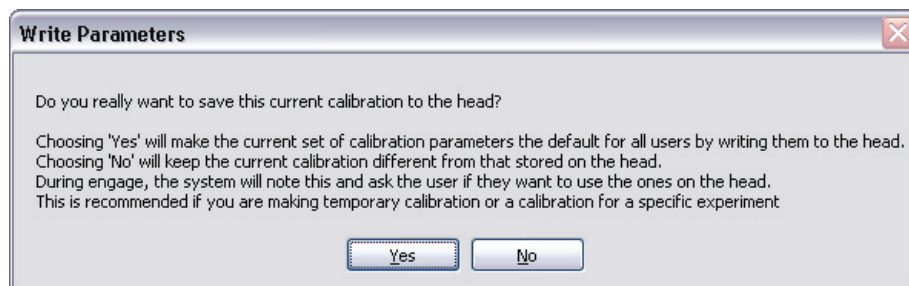### 19.2.1   Writing the head calibration parameters

You may write the calibration parameters to the memory inside the Dimension Icon head at any time by clicking the **WRITE TO HEAD** button, shown in Figure 19.2a.

Figure 19.2a   Click the **WRITE TO HEAD** button to save the calibration parameters to the head.

Clicking the **WRITE TO HEAD** button opens a confirmation window, shown in Figure 19.2b.

Figure 19.2b   The write calibration data to head confirmation window,
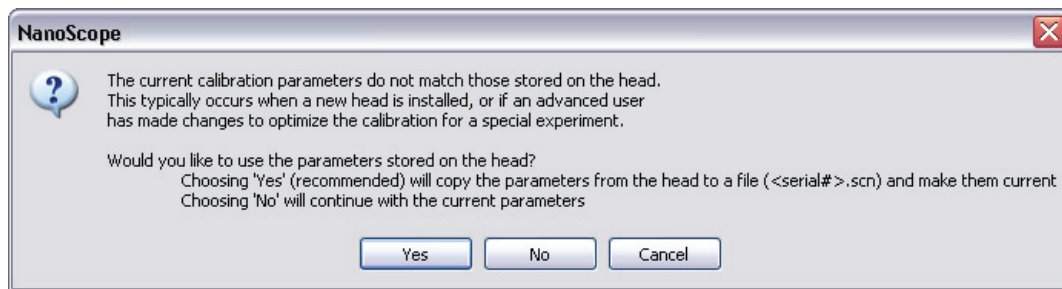
19.2.2 Reading the head calibration parameters

When engaging, the calibration information in the head and the calibration information in the scanner file are compared.

- If there is a difference, the head file should be trusted. Choose **YES** to be safe.

- If you were modifing the calibration file and merely wish to try it out, choose **NO**.

Figure 19.2c

19.3 Calibration Setup

19.3.1 Check Scanner Parameter Values

1. If the system's original scanner parameters are deleted, copy the scanner parameters from the Equip.bak directory located in D:\DI Service. Individually purchased scanners are shipped with a head/scanner disk containing backup files or a hard copy of the scanner parameters.

2. In the event that files are not found, contact Veeco for scanner calibration records.

19.3.2 Align Calibration Reference

1. Load the silicon calibration reference into the SPM.

2. Align the reference with the microscope scanner so that the tip scans parallel to the reference's features with the **Scan angle** set at **0** degrees (see Figure 19.3a).

Figure 19.3a   0 Degrees Scan Angle
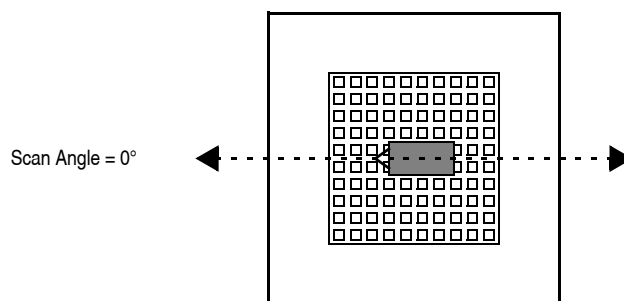
3. Align the reference within approximately 2° of perpendicularity to the scan axes.

19.3.3   Set Realtime Parameters

1. Set parameters in the control panels to the following values:

Panel	Parameter	Setting
Scan Controls	Scan Size	440 V
	X Offset	0.00 nm
	Y Offset	0.00 nm
	Scan Angle	0.00 deg
	Scan Rate	2.44 Hz
	Lines	256
	Slow scan axis	Enabled
	Z limit	330 V
Other Controls	Units	Volts
Channel 1	Data Type	Height
	Data Scale	~ 20 V[a]

a. Adjust the **Data Scale** parameter to obtain the best contrast.

19.3.4 Set Up Contact AFM

1. Set the **Microscope Mode** to **Contact**. Click the SELECT EXPERIMENT icon. This opens the **Select Experiment** window, shown in Figure 19.3b.

   Note: The microscope can be calibrated using STM; however, this example utilizes contact AFM.

Figure 19.3b The Contact Mode **Select Experiment** window
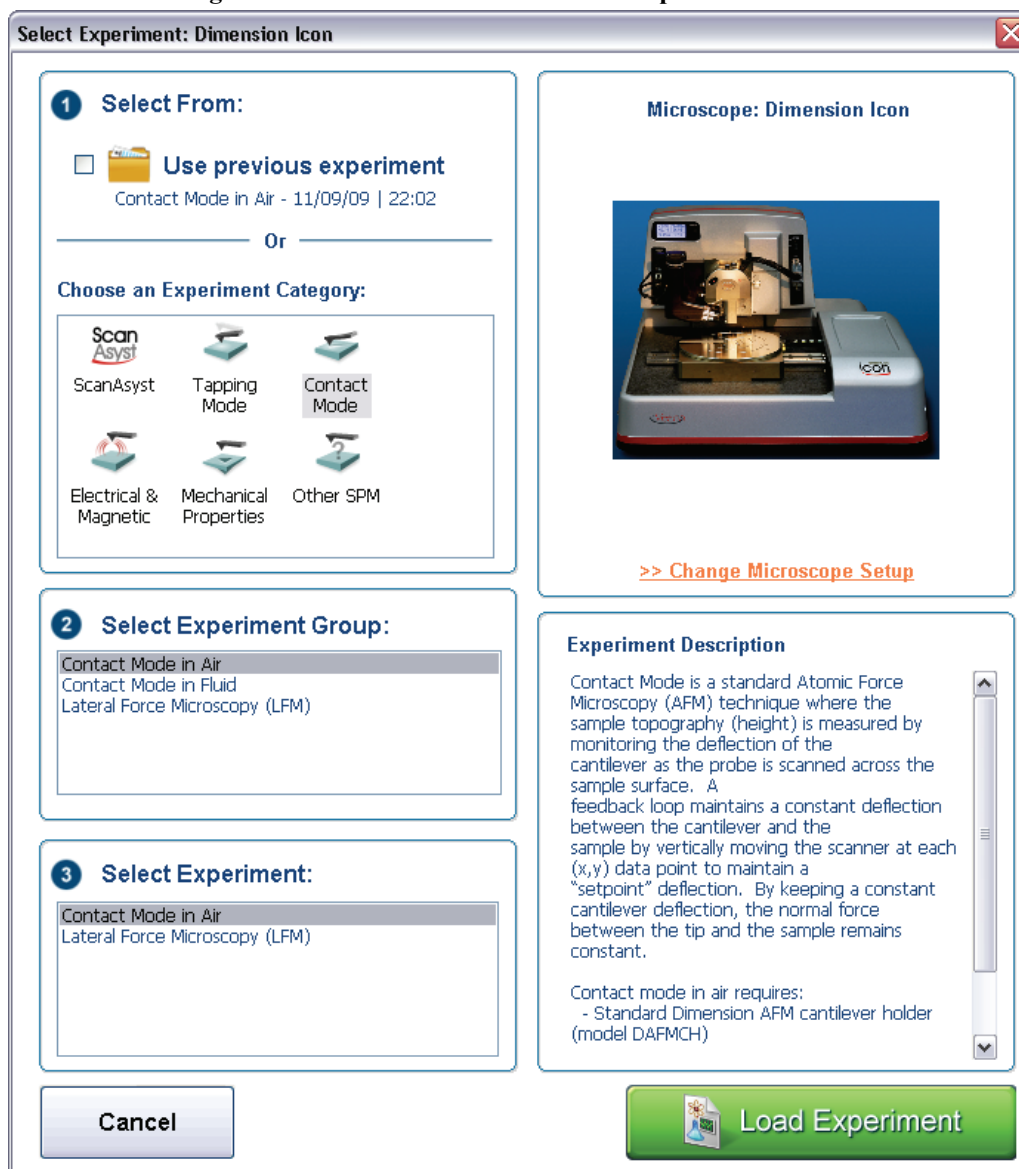
2. Select CONTACT MODE in the **Choose an Experiment Catagory** panel.

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

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

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

6. Adjust scan parameters to obtain a good-quality, maximum **Scan Size** image (**440V**). Set the **Scan Rate** to **2.44 Hz** and the **Lines** parameter to **256**.

7. With the sample engaged, check the scanning line relative to the reference features; the scan should be orthogonal to the pits on the reference.

8. If the reference requires rotation, **WITHDRAW** and rotate the sample to improve orthogonality between sample and scan line.

9. Repeat until features are oriented orthogonally with the scan frame.

19.4   Check Sample Orthogonality

Check the sample scan for orthogonality along both the X- and Y-axes. If the scan is aligned along one axis of the scan but not another, it may be necessary to adjust the microscope's **Orthogonality** parameter in the **Scanner Calibration** panel.

19.4.1 Measure Orthogonality

1. To measure a captured image's orthogonality, draw a horizontal line along an edge near the image center as shown in Figure 19.4a. Note the displayed angle.

Figure 19.4a   Horizontal Reference Line

Draw a horizontal line.

53.83 µm 0.1°
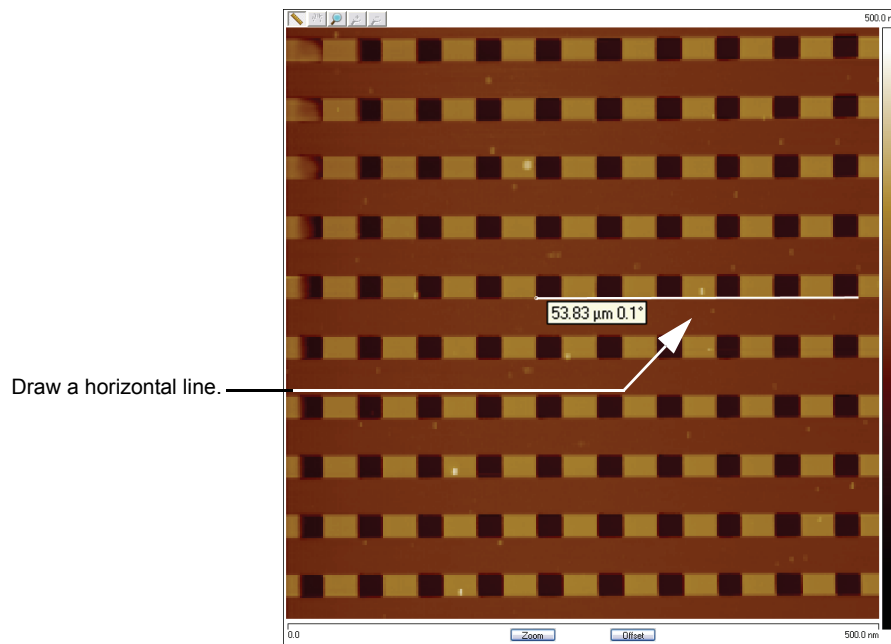
2. Draw a vertical line along an edge near the image center as shown in Figure 19.4b. Note the displayed angle.

Figure 19.4b Vertical Reference Line

Draw a vertical line. ⟶

43.43 µm 90.3°
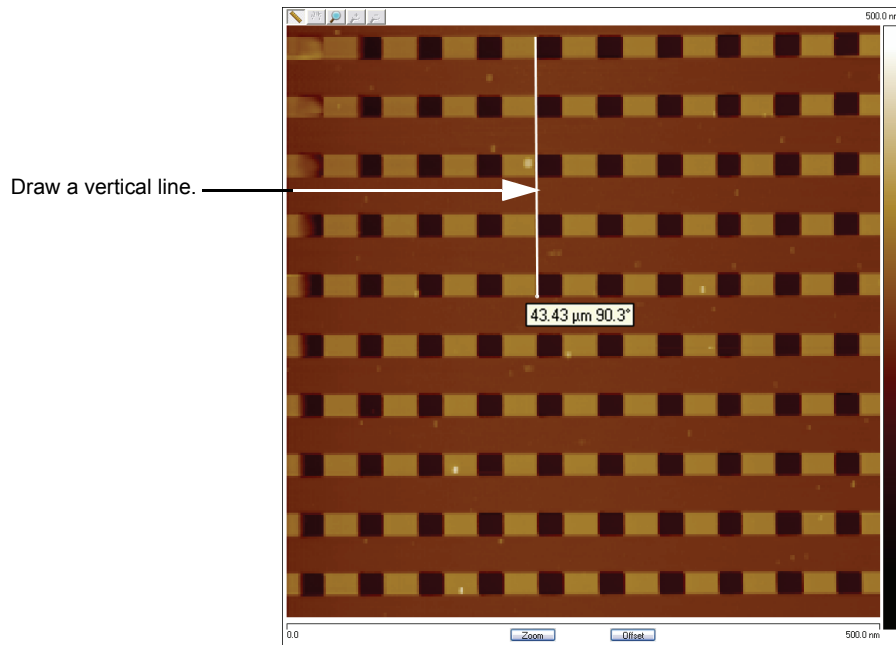
3. Perform the following calculation:

$$
\begin{aligned}
&\text{old } \textbf{ORTHOGONALITY AT (0,0) OFFSET} \\
&+\ 90^{\circ} \\
&-\ \text{(vertical angle - horizontal angle)} \\
\hline
&=\ \text{new } \textbf{ORTHOGONALITY AT (0,0) OFFSET}
\end{aligned}
$$

4. Enter this value into **ORTHOGONALITY** in the **Scanner Calibration** dialog box.

5. Click **OK** to exit the **Scanner Calibration** panel.

6. Repeat correction of **Orthogonality** until the scanned image shows less than 0.5° of error.

   Note: After a major change to the orthogonality parameter, you may need to physically realign the calibration standard to the image frame.

Figure 19.4c Non-Orthogonal and Corrected, Orthogonal Image
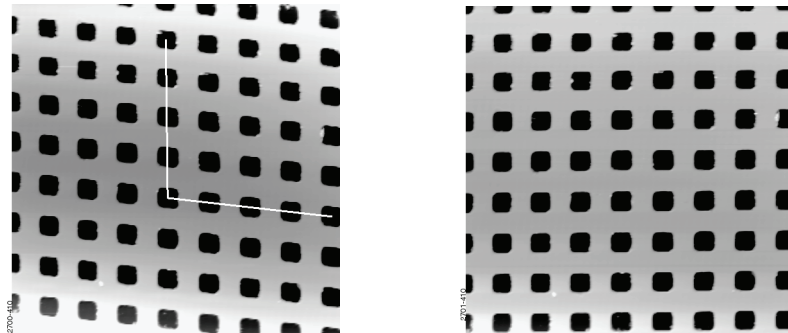
Note: In Figure 19.4c, pits align with the vertical (slow) axis but skew with the horizontal (fast) axis. The angle should be measured with the vertex near the center of the image and the vertices in the upper-right or lower-left quadrant.

Note: Apparent otrthogonality will change slightly between TRACE and RETRACE or UP and DOWN scans due to the software displaying perfectly horizontal lines while the scanner is actually moving at a slight angle during each scan line.

19.5 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.

19.5.1 Adjust Mag0 and Arg

Set the **mag0** and **arg** values while noting the scan beginning and ending.

> **Note:** When **Line direction** is set to TRACE, the beginning of the fast scan is on the left, as indicated by the arrow base. Because the monitor screen itself may be nonlinear, use the Realtime **Zoom** box to check **mag0** and **arg**.

> **Note:** When viewing up or down scans, check the image against itself, not the residual image from the previous scan. Be careful to compare only the part of the scan drawn since the last parameter change.

Adjust Fast Mag0

1. Mount a calibration reference in the SPM and begin imaging. This may consist of a generic (e.g., 10 micron, silicon) reference, or a sample having features of known dimensions (e.g., grating, etc.).

2. Turn the **XY Closed Loop OFF** in the **Scan** panel of the **Scan Parameters List** window (**EXPANDED MODE**). See Figure 19.5a.

**Figure 19.5a** Turn the **XY Closed Loop OFF**

Scan	
Scan Size	3.00 V
Aspect Ratio	1.00
X Offset	0.000 V
Y Offset	0.000 V
Scan Angle	0.00 °
Scan Rate	1.00 Hz
Tip Velocity	0.805 µm/s
Samples/Line	256
Lines	256
Slow Scan Axis	Enabled
XY Closed Loop	Off

3. Verify that the SCAN ANGLE is set to 0.00 degrees.

4. Engage and begin scanning in Contact Mode.

5. Optimize the image quality.

6. Move the mouse cursor to the scan window, click the right mouse button and select **BOX** then draw a box while depressing the left mouse button.

7. Adjust the box until it is about one-third the size of the scan.

8. Click **Calibrate** > **Scanner** > **X-Y...** to open the open loop **Scanner Calibration** window, shown in Figure 19.5b. As parameters values are changed, the effects will be seen on the monitor.

**Figure 19.5b** The Open Loop Scanner Calibration window
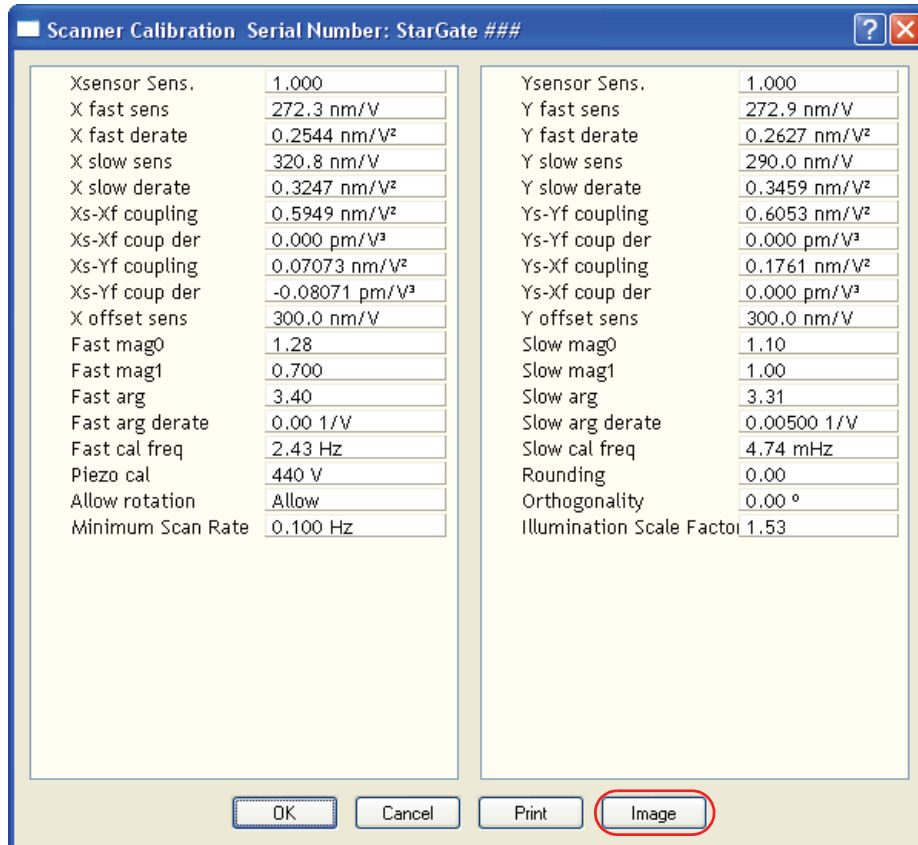
9. Click **IMAGE**, circled in Figure 19.5b, to make a measurement while in the **Scanner Calibration** window.

10. Select **Fast mag0,** the first scanner parameter to modify.

11. Move the zoom box to the start of the fast scan (on the left if the **Line direction** is set to **Trace**).

12. 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.

13. Move the zoom box to the end third of the scan.

14. Align one side of the zoom box with desired features and observe how the other side aligns with the features under it.

15. 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.

16. The entire scan axis is affected each time a parameter changes, so after every change, resize the zoom box at the beginning third of the scan and compare again with the end third.

Figure 19.5c Fast Scan Linearization: Mag0

Mag0 Too Large

Mag0 Too Small
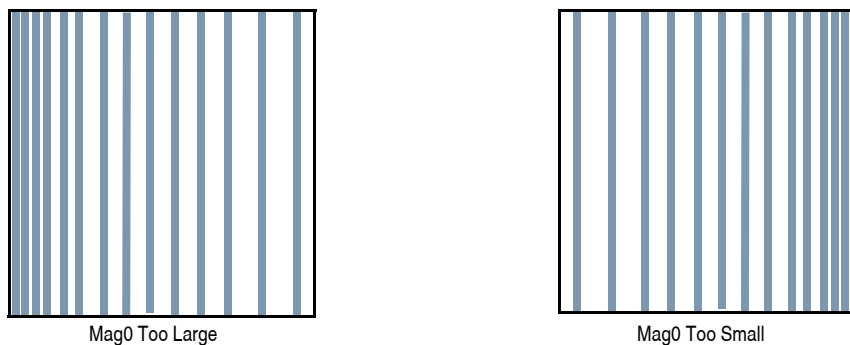
Adjusting Fast arg

1. Once the beginning third of the scan is equal to the end third, check to see if the center needs adjusting.

2. If the center features are too large for the box, decrease the **Fast arg** value. If the center features are too small, increase the **Fast arg** value. Change **arg**s by 0.2 to 0.5 units at a time.

3. Changes affect the entire scan, so continue to resize the zoom box after each change.

4. After an adjustment in **Fast arg** is made, **Fast mag0** may need readjusting. Repeat **Fast mag0** adjustment procedure until the rulings are evenly spaced across the Fast-axis.

Figure 19.5d X Scan Linearization

Center Compressed   Center Expanded

5. After setting **Fast mag0** and **Fast arg**, insert the values for **Slow mag0** and **Slow arg**. These values serve as close starting points before adjusting the slow linearities.

Adjusting Slow Mag0

1. Follow the same instructions as for **Fast mag0**.

2. After a parameter change, wait until a new third of a scan begins before setting new parameters and resizing the zoom box.

    Note: Setting the slow linearities requires a lot of time.

3. Move the resized box to the end of the scan and prepare to measure the end promptly.

    Note: You may type in a new parameter value before the scan starts again.

    Note: Be careful not to confuse scan top and bottom with beginning and ending, as the scan direction alternates.
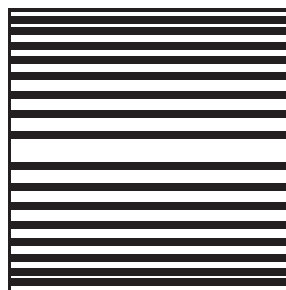
4. Adjust the zoom box to fit the beginning third of the scan and check against the end third.

5. If the features of the end third are too large for the box, decrease the parameter. If the features are too small, increase the parameter.

    Note: Compare only parts of the current scan, not the previous scan.

Figure 19.5e Y Scan Linearization
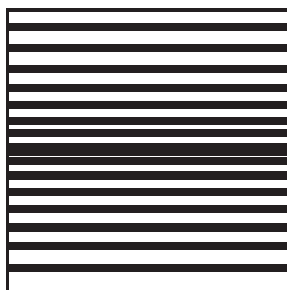
Center Compressed   Center Expanded

Adjusting Slow arg

1. Follow the same instructions for **Fast arg**.

2. Adjust the zoom box to fit the beginning and ending of the scan, then check the center.

3. If the features in the center are too large, reduce the **Slow arg** value. If the features in the center are too small, increase the **Slow arg** value.

4. After adjusting **Slow arg** values, check if **Slow mag0** requires readjusting.

5. Repeat the procedure for adjusting **Slow mag0** until the rulings are evenly spaced along the slow axis.

19.5.2 Adjust Fast mag1

Initial Adjustment

1. For initial adjustment, click **OK** to close the **Scanner Calibration** window.

   Note: Selecting **Cancel** resets parameters to default values when the box is opened.

2. Change the **Scan size** to **150V**.

3. If the two scope traces do not overlap, **Fast mag1** needs adjusting.

4. On the **Scan** panel, select **Slow Scan Axis**. When tall features appear on the scope trace, press the keyboard right or left arrow key to switch the **Slow Scan Axis** to **Disabled**.

5. Select **Tools** > **Calibrate** > **X-Y...** to open the **Scanner Calibration** box.

6. Select **Fast mag1**.

7. Change the value until the two traces align.

   Note: The retrace line will shift to the right as the value is increased.

8. When done, click **OK** to close the **Scanner Calibration** window.

9. Set **Slow scan axi**s back to **Enabled**.

10. Select **Image** mode.

Fine Adjustment

Initial adjustment is usually adequate; however, if more precision is desired, use the following fine adjustment techniques to adjust **Fast mag1**.

1. Use the same procedure for adjusting **FAST MAG0**.

2. As before, set the **Zoom** box for the beginning of the scan and then check the ending. Because the scan is small, use a **Zoom** box up to one-half as large as the scan.

3. If the end of the scan is larger than the beginning, reduce the **Fast mag1** value. If the end is too small, increase the value of **Fast mag1**.

19.5.3 Adjust Slow mag1

1. With the **Scan size** set to **150V**, select **Tools** > **Calibrate** > **X-Y...**

2. Select **Slow mag1** and input the value from **Fast mag1**.

    Note: For medium-sized scanners (C to F), further adjustments should not be necessary. The **Slow mag1** value is usually 100-120 percent of the **Fast mag1** value.

3. If more precision is desired, adjust **Slow mag1** using the same procedure for adjusting **Slow mag0**.

4. Set the **Zoom** box for the beginning of the scan and then check against the ending. Because the scan is small, use a **Zoom** box up to one-half as large as the scan.

5. If the end of the scan is larger than the beginning, decrease the **Slow mag1** value. If the end is too small, increase the value of **Slow mag1**.

6. Wait one complete frame with the new value before readjusting the **Slow mag1** value.

7. Check the final result by capturing an image and checking it with the **Offline** > **Modify** > **Zoom** window. The scanner is now ready for calibration of the X and Y parameters.

19.6   X-Y Calibration using Capture Calibration and Autocalibration

### 19.6.1   Capture Calibration

Scanners are calibrated by setting parameters in the NanoScope software using the **Capture Calibration** command. The basic calibration procedure using a 10µm pitch calibration reference (see Figure 19.1e) is described below.

1. With the **Scan rate** set to **2.44Hz** and **Number of samples** parameter to **256**, a full **Capture Calibration** requires approximately 60 minutes.

    Note: Increasing the **Number of samples** or decreasing the **Scan rate** significantly increases the required time but should be considered if your typical measurements are at higher resolution or slower scan rates.

2. Using the mouse, click on **Capture** > **Capture Calibration**. The **Capture Calibration** dialog box lists twelve parameters used in the calibration procedure (see Figure 19.6a).

Figure 19.6a   Capture Calibration Prompt
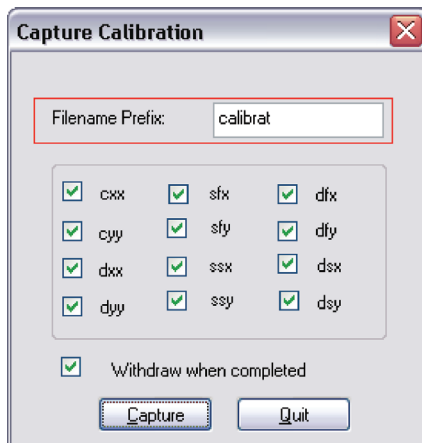
3. If this is a first time calibration, or if the microscope's calibration has not been checked within the last three months, verify that all parameters are selected. Otherwise, select those you need.

4. Change the **FILENAME PREFIX** if desired.

5. Click **CAPTURE** to initiate the automatic calibration routine.

    Note: The microscope begins an automatic series of scans on the reference which require approximately one hour to complete. During each scan, the scanner moves the piezo using carefully calculated movements. Many of these movements are unusual, giving rise to a variety of images which do not resemble the normal reference. For example, pits may resemble trenches and features may be presented at various angles.

6. As each routine is executed, adjust the scan slightly to optimize the calibration image using the **Capture Control** dialog box displayed on the control monitor throughout the calibration routines (see Figure 19.6b).

Figure 19.6b Capture Control Prompt
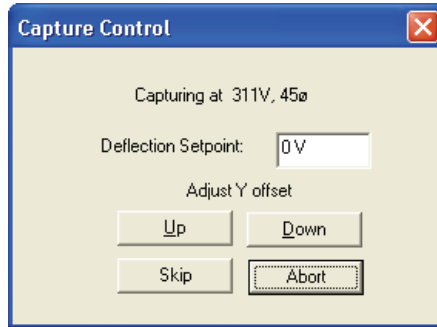
Note: The capture status will begin at skip 2. The program skips the current scan plus one more before capturing an image for later calibration. This allows hysteresis and drift to settle out when the scan changes direction and size between images.

7. For cxx, cyy, dxx, dyy images, if portions of features are missing, or if the image is blank, click repeatedly on **Up, Down**, **Left** or **Right** to adjust the scan until more of the features are imaged. Features should extend across as much of the displayed image as possible (see Figure 19.6c).

Figure 19.6c Improved Calibration Image

Partial Calibration Image   Improved Calibration Image
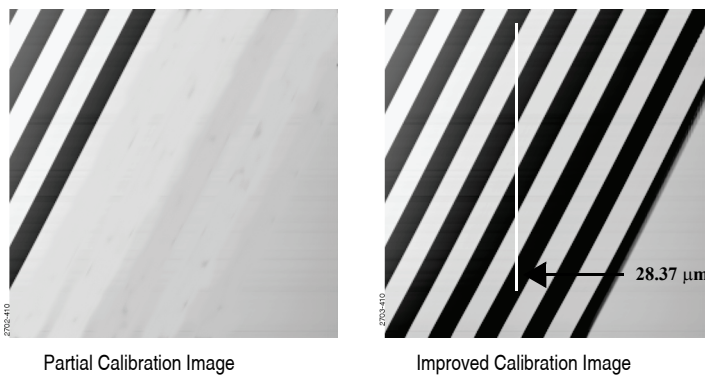
Note: After the first four images with the diagonal stripe pattern are captured, you can leave the system unattended while the program continues to completion. Some of the following images appear stretched in one dimension; however, this is normal.

8. If the scan has not stabilized by the time the capture status changes to **Forced**, click SKIP to increment the capture to skip the current scan.

> **Note:** Do not click on **Abort** unless you want to stop the entire **Capture Calibration** program.

9. Once the image is optimized, allow the software to capture the entire image without disturbing it. The software automatically indexes to the next image. Once all calibration images are obtained, the software prompts the user that it is finished.

10. Go to the directory where all **Capture Calibration** files are saved.

11. Select **View** > **Browse** or click **Browse** to review all **Capture Calibration** files.

12. Verify that all calibration images contain features spanning the full width and height of the image frame (see the right image in Figure 19.6c).

13. Recapture all images unsuitable for calibration. Record the file name extensions for all unusable files (e.g., .cxy, .dyy), then delete the files.

14. Re-engage on the reference surface and select **Capture Calibration**.

15. Verify that the file name prefix is identical to that of the usable files. Remove the "x" from all file name extensions except for the unusable file names recorded in Step 13.

16. Finally, click **Capture** to recapture the selected files.

19.7   Auto Calibration

After the **Capture Calibration** routine is completed, the user measures surface features contained within each image and enters their dimensions into the software. The software compares its estimates with the actual (user-entered) dimensions to make final corrections. This portion of calibration is carried out using the **CALIBRATE** > **SCANNER** > **AUTO CALIBRATION** command.

1. The system will prompt the user to select the calibration image, shown in Figure 19.7a. Click **OK** then do the following:

Figure 19.7a   Select the calibration image
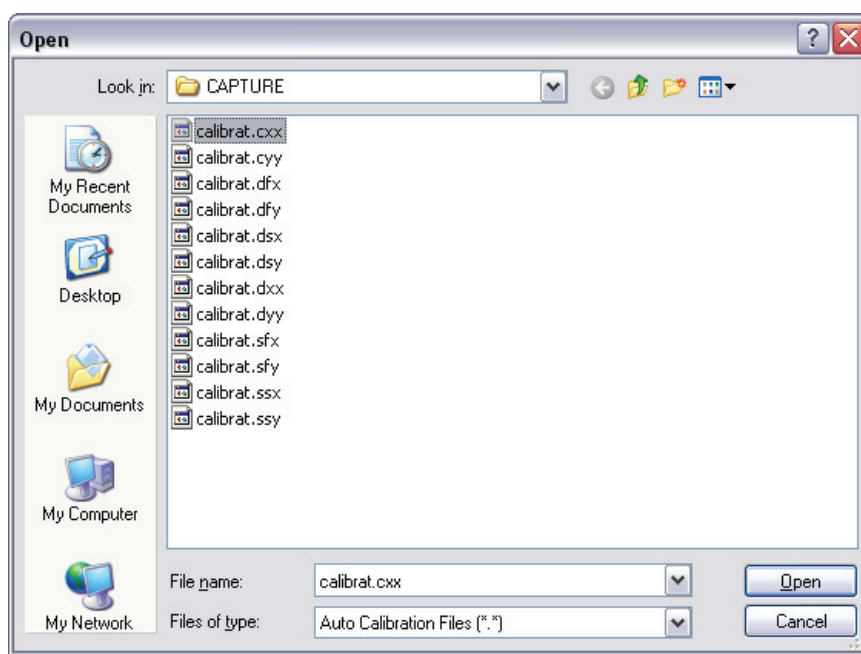
2. Select any of the desired captured calibration images. and click **Open**.

3. The system will open a user interface, shown in Figure 19.7b. In the right upper **Input** panel, select those you wish to calibrate as **YES** and those you wish to skip as **NO**. For normal calibration, verify that all parameters are selected. Click the **CALIBRATE** button to start the calibration.

Figure 19.7b   X-Y Piezo Calibration Selection Prompt
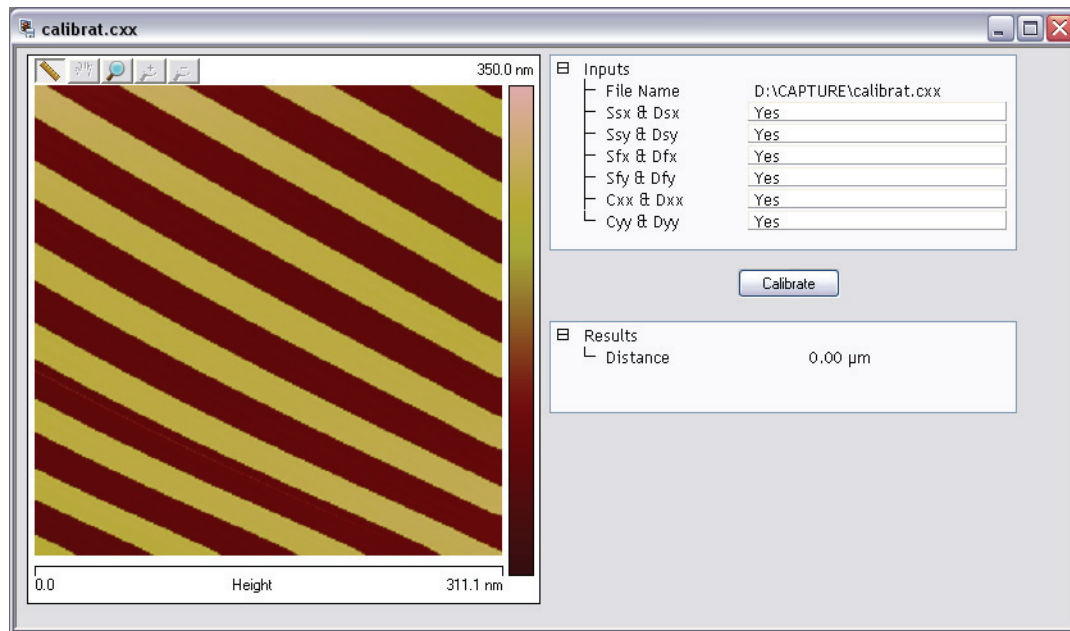
4. Depending on what **Inputs** are selected in Step 3, the software will sequentially present various calibration images. You will be asked to draw either a vertical (shown in Figure 19.7c) or horizontal (shown in Figure 19.7d) line on those images. The software displays the span of that drawn line initially in the **New Distance** field (e.g. 105μm in Figure 19.7c) and in the **Results** / **Distance** /**Value** field. This value is the software's estimate of the length of the line based on the current calibration values. Input the known value (e.g. 100um in Figure 19.7c) in the **New Distance** field to reflect the correct line length.

5. Click the **APPLY** button. The software will finish this image and continue to the next image.

Figure 19.7c Vertical Calibration Line
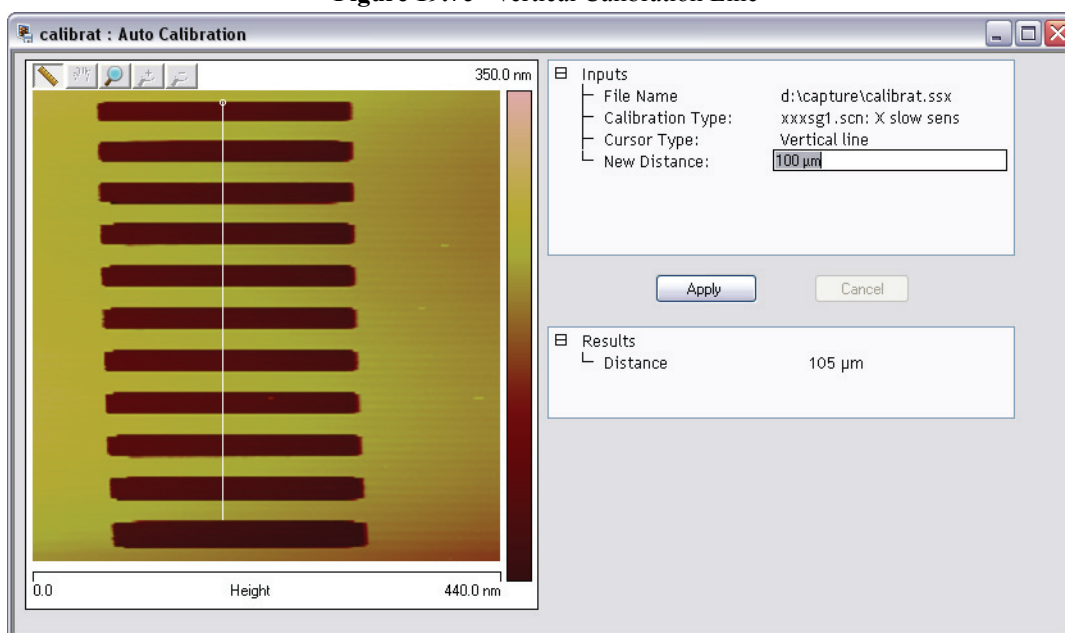
Figure 19.7d Horizontal Calibration Line
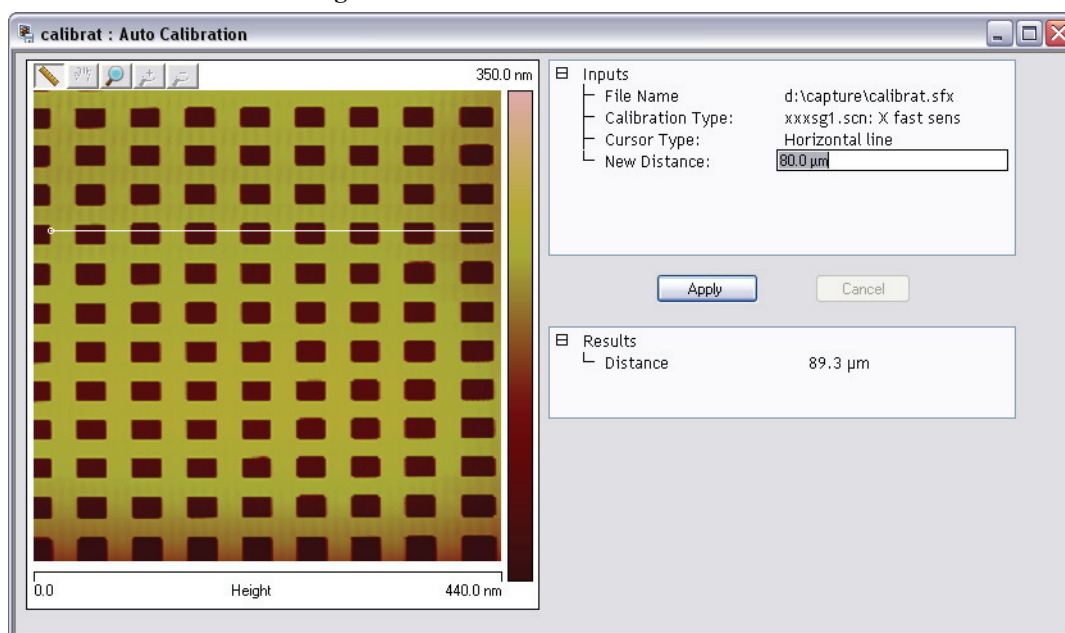
Note: When using the mouse to draw a line on the image, draw it to span as many features as possible, connecting similar edges (see Figure 19.7c and Figure 19.7d). Measure features without regard to how they appear in the calibration images. Features may be represented with stretched, distorted, or angled appearances due to the unusual movements employed during capture calibration scanning. Regardless, features are separated by the same spacing.

6. Continue drawing lines and entering measured distances until all capture calibration images are measured. When the software is finished, it will prompt the user that it is done. Click **OK** to acknowledge this message.

7. The software prompts the user that "The current workspace must be closed. Would you like to save and reload the current workspace." Click either **YES** or **NO**. The software will then close the current workspace and start a new one for the newly calibrated parameters to take effect.

If the **Capture Calibration** and **Auto Calibration** routines are completed correctly, the SPM will be calibrated within 1-2 percent accuracy over most of the scanner's measuring range. To obtain still better accuracy, the SPM can be fine-tuned to obtain maximum measurement accuracy. This is accomplished through the use of calibration parameters discussed in Section 19.8.

19.8   Fine-tuning for X-Y Calibration

Fine-tuning is usually performed at two **Scan size** settings: **150V** and **440V**. Both horizontal and vertical measurements of sample features are made, then compared with actual distances. Based upon this comparison, computer parameters are fine-tuned. To fine-tune your SPM for maximum X-Y measuring accuracy, review the procedure below.

19.8.1   Prepare System for Fine-tuning

1. Set the SCAN SIZE parameter on the **Scan Control**s panel to the maximum value (`440V`).

2. Verify that the SCAN ANGLE is set to `0.00` degrees.

3. Set **Scan** > **XY Closed Loop** to **OFF** for open loop tuning.

4. 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.).

5. Optimize the image quality.

   Note: Your calibration and fine-tuning procedures are no better than the procedures and references used. **Choose both carefully**.

19.8.2   Measure Horizontally at 440V Scan Size

1. Set the **Scan size** parameter on the **Scan** panel to the maximum value (**440V**).

2. Verify that the **Scan angle** is set to **0.00** degrees.

3. **Engage** the surface.

   Note: Contact mode is preferred for this procedure because the scan rate is faster for contact mode than tapping mode.

4. After you have completed a scan pass, click **CALIBRATE** > **SCANNER** > **XY**. The **Scanner Calibration** dialog box displays (see Figure 19.8a).

Figure 19.8a   Scanner Calibration Dialog Box
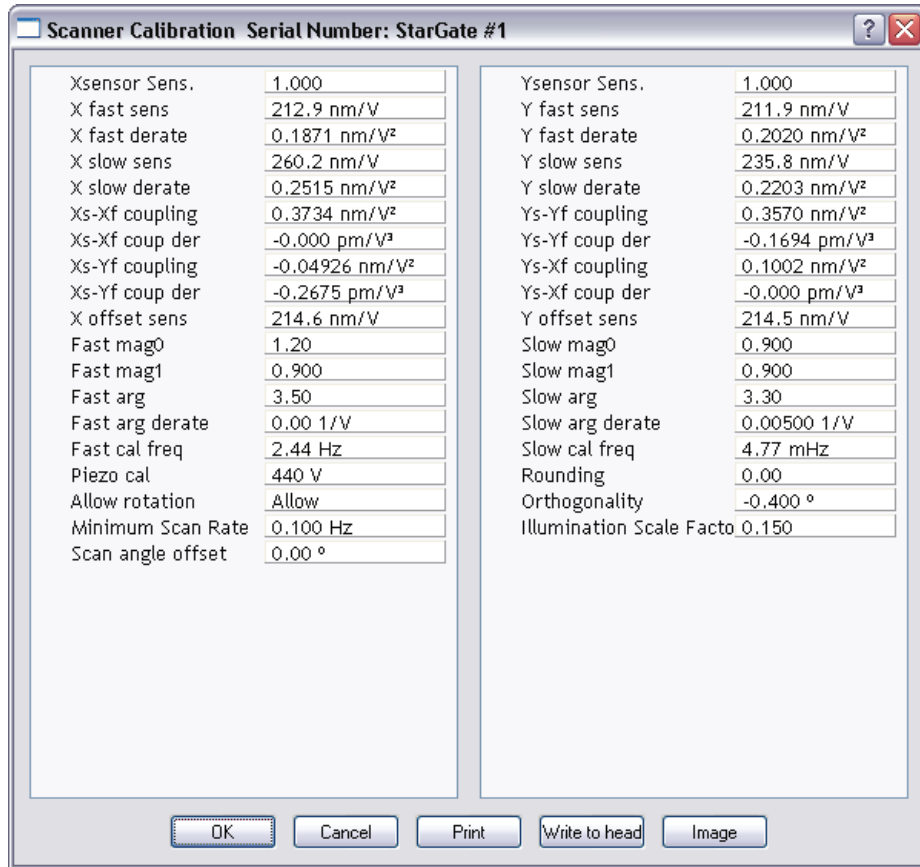
5. Click **IMAGE** to view the image while in the **Scanner Calibration** window, shown in Figure 19.8b.

6. Select two widely-spaced features on the sample image of known separation. Use the mouse to draw a horizontal line between them (see Figure 19.8b). (For example, on a 10µm silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The screen will display the measured distance between pits next to the line.

Figure 19.8b Calibration Horizontal Reference
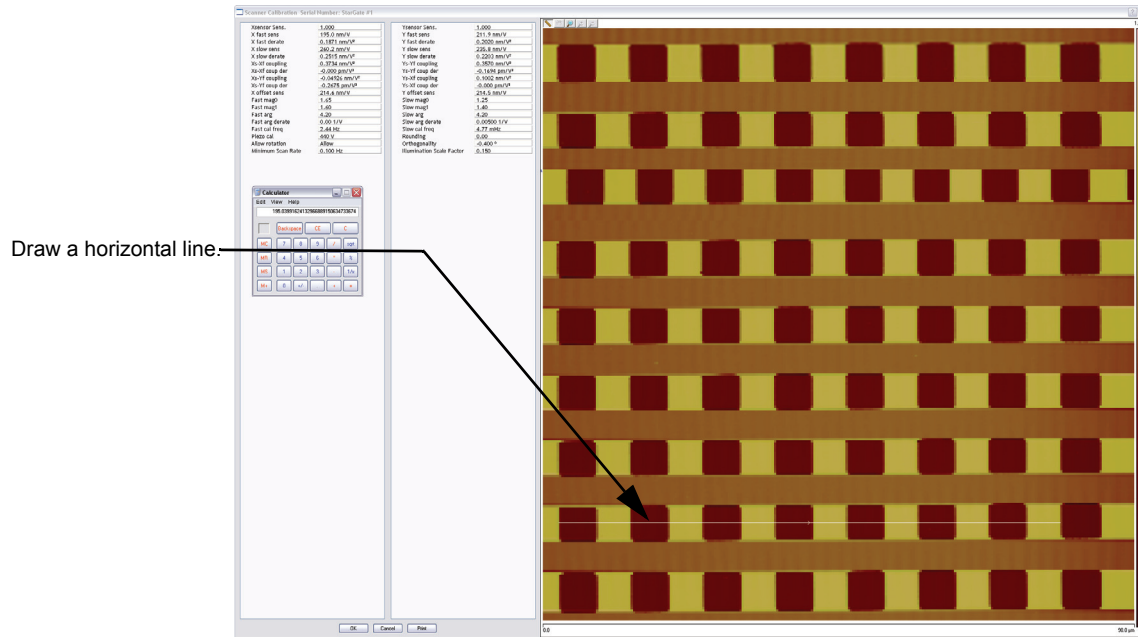
7. Verify that the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to **Measure Vertically at 440V Scan Size:** Section 19.8.3.

8. Based upon the results in the above step, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

9. Multiply the quotient obtained above by the **X fast sens** value shown on the **Scanner Calibration** panel.

10. Enter the new value. The new value adjusts the scanner's fast axis to more closely match calculated distances with actual feature distances. The new sensitivity setting takes effect as soon as it is entered.

11. Repeat Step 6 to check your change.

12. To save new values to the computer's hard disk, click on **OK**. This closes the **Scanner Calibration** panel.

19.8.3 Measure Vertically at 440V Scan Size

1. Return to the image of the calibration reference.

2. Select the **CALIBRATE** > **SCANNER** > **XY** function to display the **Scanner Calibration** dialog box.

3. Click **IMAGE** to view the image while in the **Scanner Calibration** window.

4. Wait for at least three full scans to allow the piezo to stabilize then select two widely spaced features and then draw a vertical line connecting like portions of features (top edge-to-top edge, etc.). See Figure 19.8c.The SPM displays the calculated distance between features.

Figure 19.8c Calibration Vertical Reference
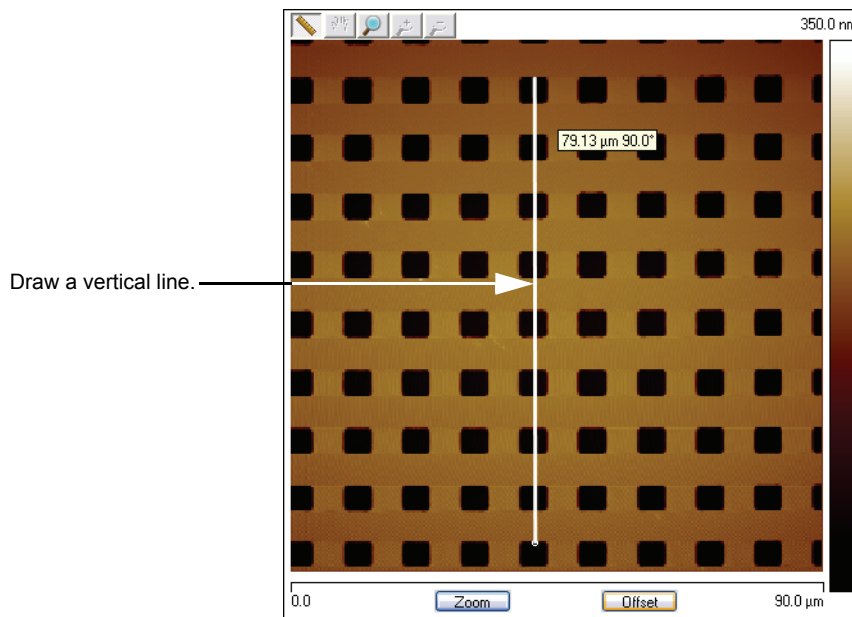
5. Verify that the microscope's calculated distance agrees with the known vertical distance. If there is significant disagreement between the two, fine tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to **Measure Horizontally at 150 V Scan Size:** Section 19.8.4.

6. Using the results from the above step, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

7. Select the **Y slow sens** parameter.

8. Multiply the quotient obtained earlier by the **Y SLOW SENS** value shown on the **Scanner Calibration** dialog box.

9. Enter the new value to adjust the scanner's slow axis to more closely match calculated distances with actual feature distances.

10. To save the new parameter value, click **OK**.

11. Repeat Step 4 to check your change.

19.8.4 Measure Horizontally at 150 V Scan Size

1. Verify that the **Scan angle** is set to **0.00** degrees, and that **Units** (**Other** panel) is set to **Volts**.

2. Set the **Scan size** parameter on the **Scan** panel to one-third the maximum (**150V**).

3. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a horizontal line between them.

   Note: For example, on a 10μm, silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible. The microscope displays the measured distance next to the line.

4. Verify that the microscope's measured distance agrees with the known horizontal distance.

5. If there is significant disagreement between the two, fine-tuning is required; go to the next step. If the displayed distance agrees with the known distance, skip to **Measure Vertically at 150V Scan Size:** Section 19.8.5.

6. Perform fine-tuning adjustments using either trial and error or calculate the precise correction (see Calculation Method).

Trial and Error Method

1. Select the **CALIBRATE > SCANNER > X-Y** function to display the **Scanner Calibration** dialog box.

2. Click **IMAGE** to make a measurement while in the **Scanner Calibration** window.

3. Select the **X fast derate** parameter or **Y slow derate** for Y-axis adjustment.

4. If the measured distance is less than the actual distance, decrease the **X fast derate** parameter slightly or **Y slow derate** for Y-axis adjustment and re-measure image features.

5. Adjust deratings up or down until measurements accord with known feature distances.

Calculation Method

1. Select **CALIBRATE > SCANNER > X-Y** to display the **Scanner Calibration** dialog box.

2. Click **IMAGE** to make a measurement while in the **Scanner Calibration** window.

3. Record the **X fast derate** or **Y slow derate** value.

4. Perform the following calculation:

$$\frac{s - \left(\dfrac{a}{m} \bullet [s - d(440 - v)]\right)}{440 - v}$$

where
s is the **X FAST SENS** or **Y SLOW SENS** value;
a is the actual distance;
d is the **X FAST DERATE** or **Y SLOW DERATE** value you recorded in the above step;
m is the measured distance;
and v is the **SCAN SIZE** in volts.

5. Return to the **Scanner Calibration** dialog box.

6. Enter the new **X FAST DERATE** value calculated in Step 4. This adjusts the scanner's fast axis to more closely match calculated distances with actual feature distances.

7. To set the new parameter value, click **OK**.

8. Measure horizontally again to verify that the measurement is now in agreement with the known distance.

19.8.5 Measure Vertically at 150V Scan Size

1. Select two widely-spaced features on the sample image of known separation.

2. Use the mouse to draw a vertical line between them.

> **Note:** For example, on a 10µm, silicon reference, draw the line from the top edge of one pit to the top edge of another pit as far away as possible. The microscope displays the measured distance next to the line.

3. Verify that the microscope's measured distance agrees with the known vertical distance. If there is significant disagreement between the two, execute the fine tuning procedure; go to the next step. If the displayed distance agrees with the known distance, no further calibration is required.

4. Adjust the **Yslow derate** value by using one of two methods: 1) the Trial and Error Method or 2) the Calculation Method described in **Measure Horizontally at 150 V Scan Size: Section 19.8.4** above.

19.8.6   Change Scan angle and Repeat Calibration Routines

•   Change the **Scan angle** on the **Scan Controls** panel to **90º**.

•   Repeat steps above for the following parameters: **Y FAST SENS, X SLOW SENS, Y FAST DER, AND X SLOW DER** to ensure the scanner is calibrated properly along the X- and Y-axis for scanning at 90º.

19.9   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 Veeco have specified (on the calibration container) vertical features accurate to within ± 3 percent. The calibration reference is referred to throughout the examples provided in this section. If you require greater accuracy, you must select an appropriate calibration standard, and a closed loop head employed with a Veeco Dimension Series microscope.

> **Note:**   Refer to the label on your calibration reference sample to verify the measurement.

19.9.1 Engage

1. Set up the microscope for **TappingMode** imaging.

2. Select **ENGAGE** under the **Microscope** drop-down menu or click the **ENGAGE** icon in the **Main** panel.

3. Find a square pit and center the pit in the image using a **Scan size** of approximately **10MM**.

4. Verify that the **Z Center Position** value shown next to the image display is close to **0 volts** (**±5 VOLTS**), shown in Figure 19.9a.

Figure 19.9a   Z Piezo Voltage (position)
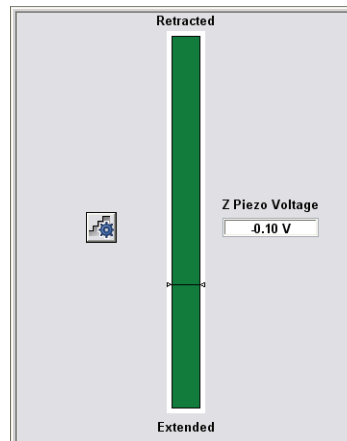
5. If the **Z Center Position** value is not close to zero, use the **Microscope** > **Step Motor** > TIP UP and TIP DOWN buttons to adjust (see Figure 19.9b).

Figure 19.9b   Step Motor
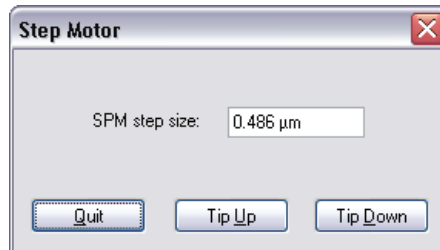
19.9.2 Capture and Correct an Image

1. Set CHANNEL 1 DATA TYPE to HEIGHT and CHANNEL 2 DATA TYPE to HEIGHT SENSOR.

2. Capture an image by selecting **Capture** from the NanoScope tool bar, or click the CAPTURE icon.

3. When the image is captured, load the image by double clicking it in the browser bar.

4. Click the height image (CHANNEL 1) to select it.

5. Remove all tilt and scan line errata from the image by selecting **Modify** > **Flatten** or click the **FLATTEN** icon on the NanoScope tool bar.

   a. Set the **FLATTEN ORDER** parameter in the input box to **1**.

   b. Go to the display window and draw a stopband over the pit as shown in Figure 19.9c.

Figure 19.9c   Draw a Stopband
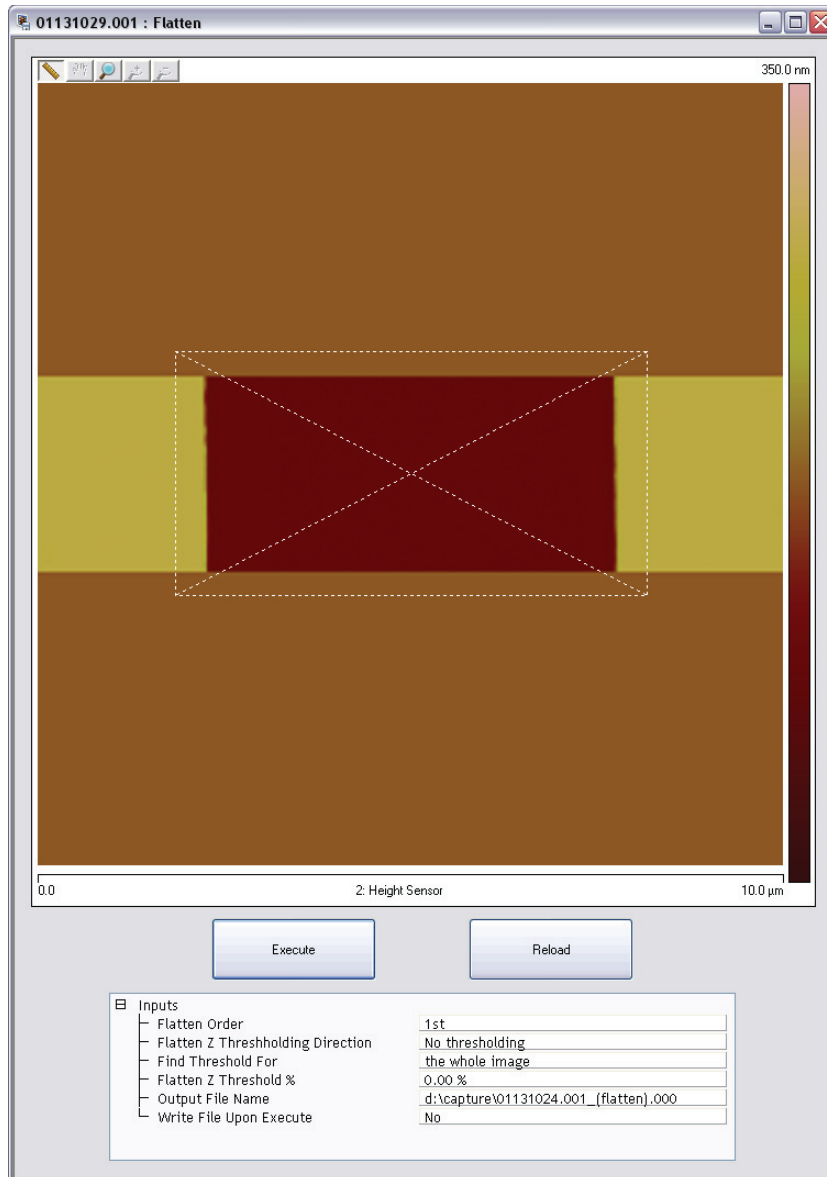
   c. Click **EXECUTE** to complete the flattening procedure.

   d. Right click the image, select **CHANNEL 2** (**HEIGHT SENSOR**) and click **EXECUTE** again to flatten **CHANNEL 2**.

19.9.3 Measure Vertical Features

With the image corrected, its vertical features may now be measured. This is performed using **Depth** analysis to utilize more data points.

1. Select the **Analysis** > **Depth** command or click the **DEPTH** icon on the NanoScope tool bar. This loads the **Depth** analysis screen shown in Figure 19.9d.

2. Right click the image and select **CHANNEL 1**.

Figure 19.9d   Depth Analysis Screen
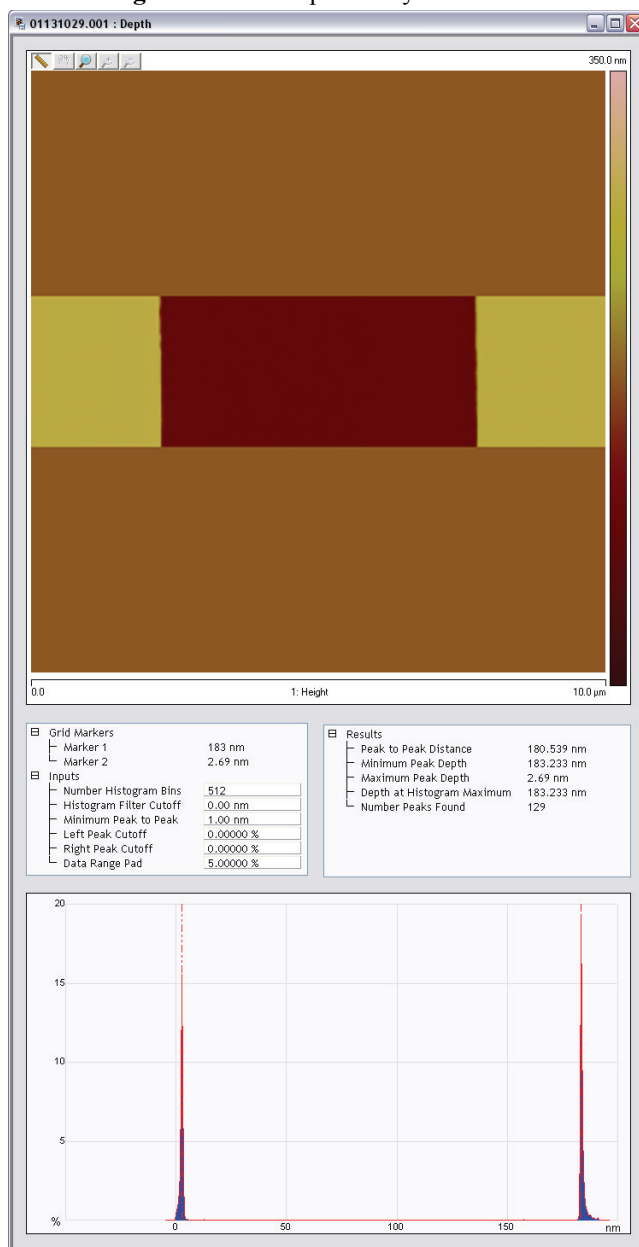
3. If desired, part of the image may be selected for analysis by drawing a cursor box surrounding that region. Otherwise, the whole image will be used (see Figure 19.9d).

> **Note:** A histogram of the height data within the drawn cursor box displays in the window, showing two prominent peaks. These peaks correspond to two elevations on the surface: the bottom of the pit and the top surface. There should be a line cursor on each peak.

4. If the two peaks do not appear in the display, increase the **HISTOGRAM FILTER CUT OFF** in the **Inputs** dialog box to **2–10ɴᴍ**. If the two peaks are too close together, increase the **MIN PEAK TO PEAK** in the **Inputs** dialog box.

5. In the **Results** dialog box in the **Depth** panel, check the **Peak to Peak distance** and record it for future reference.

19.9.4 Correct Z Sensitivity

If the depth of the pit on the 10μm silicon calibration reference deviates significantly from the actual depth, correct the **ZSCAN SENS.** parameter in the **Z Calibration** dialog box.

1. Open the **Z Calibration** dialog box by selecting **Calibrate** > **Scanner** > **Z**.

2. Divide the actual depth of features (200nm for the 10μm calibration reference) by the measured depth (indicated in **Depth** analysis by the **PEAK TO PEAK DISTANCE**):

$$\frac{\text{actual depth}}{\text{Peak to peak distance}}$$

3. Multiply this quotient by the **ZSCAN SENS.** value in the **Z Calibration** dialog box, and replace the former **ZSCAN SENS.** value with the new result.

4. Click **OK** to enter the new **ZSCAN SENS.** value.

19.9.5 Recheck Z-axis Measuring Accuracy

After executing the steps above, recheck the Z-axis measuring accuracy of the SPM by repeating the steps outlined above until you obtain accuracy of 1 to 2%.

19.9.6 Calculate Retracted and Extended Offset Deratings

Piezoelectric materials exhibit greater sensitivity at higher voltages. In the steps outlined above, the Z-axis was calibrated while scanning near the middle of the piezo voltage range (i.e., **Z Center Position** ~ **0V**). In this section, you will calibrate the Z-axis piezo while extended and retracted to determine how much the Z sensitivity varies with **Z Center Position**.

1. If you are not already Engaged, select **ENGAGE** under the **Microscope** drop-down menu or click the **ENGAGE** icon in the **Main** panel. See **Engage:** Section 19.9.1.

2. Use the **Microscope** > **Step Motor** > **TIP UP** button until the **Z Center Position** reads **50V** (±5V).

   > **Note:** By using the motor to move the tip up, the feedback loop forces the Z-axis piezo to extend to continue its tracking of the surface.

3. Refer to the steps above (**Capture and Correct an Image:** Section 19.9.2 through **Measure Vertical Features:** Section 19.9.3) to determine the measured depth of the calibration standard with a **+50V Z Center Position**.

4. Record the measured depth. If the depth measured by the extended piezo is correct within two percent, skip to Step 9.

5. Select **Calibrate** > **Z...** to display the **Z Calibration** pane, shown in Figure 19.9e.

6. Click the **Extended offset der** parameter.

7. Perform the following calculation

$$(1 + \text{current offset der}) \bullet \frac{\text{actual depth}}{\text{meas. depth}} - 1$$

For example, if the current offset equals 4% and the measured depth equals 185nm, then:

$$(1 + 0.04) \bullet \frac{\text{actual depth}}{\text{meas. depth}} - 1$$

8. Enter the new percent value from above (example was 12%) for the **Extended offset der** parameter in the **Z Calibration** panel.

9. The procedure for calculating and setting the **Retracted offset der** is exactly the same as for the **Extended offset der**; however, the piezo must be **retracted** to **−50V**. To calculate the **Retracted offset der**:

   a. Use the **TIP DOWN** button in the **Motor Control** panel to retract the piezo.

   b. Retract the **Z Center Position** to **−50V**.

   c. Repeat Step 3 - Step 8 above.

Figure 19.9e Z Calibration Panel

19.10 Closed Loop XY Calibration

A separate set of parameters is used to control the scanner XY position when **XY Closed Loop** is **ON**. These are called the **OptoXY Closed Loop Calibration** parameters. In addition to the parameter, **XY Closed Loop**, in the **Scan** panel, a new panel, **OptoXY Closed Loop Calibration,** shown in Figure 19.10e, contains scanner calibration parameters associated with the Dimension Icon head.

With **Scan** > **XY Closed Loop** set to **ON**, click **CALIBRATE > SCANNER > X-Y** to open the **OptoXY Closed Loop Calibration** panel.

> **Note:** When the Icon head is in open loop mode, a different panel, **Scanner Calibration**, opens (see Figure 19.8a). It shares two of the same parameters as the **OptoXY Closed Loop Calibration** panel (**ROUNDING** and **MINIMUM SCAN RATE**), but includes considerably more calibration parameters to compensate for piezo nonlinearity

Closed Loop XY Calibration has four components:

1. **Measure Orthogonality at (0,0) offset**: Page 455

2. **Measure Horizontally at 90mm Scan Size**: Page 457

3. **Measure Vertically at 90mm Scan Size**: Page 459

4. **Scan angle correction**: Page 461.

19.10.1 Measure Orthogonality at (0,0) offset

ORTHOGONALITY AT (0,0) OFFSET is the deviation from a 90° angle between the scanned X-axis and Y-axis, positive for an acute angle and negative for an obtuse angle.

1. Capture a 90um scan of the calibration reference.

2. Enlarge the widow for increased resolution.

3. Draw a horizontal line along an edge near the image center as shown in Figure 19.10a. Note the displayed angle.

Figure 19.10a Horizontal Reference Line

Draw a horizontal line. ————

53.83 µm 0.1°
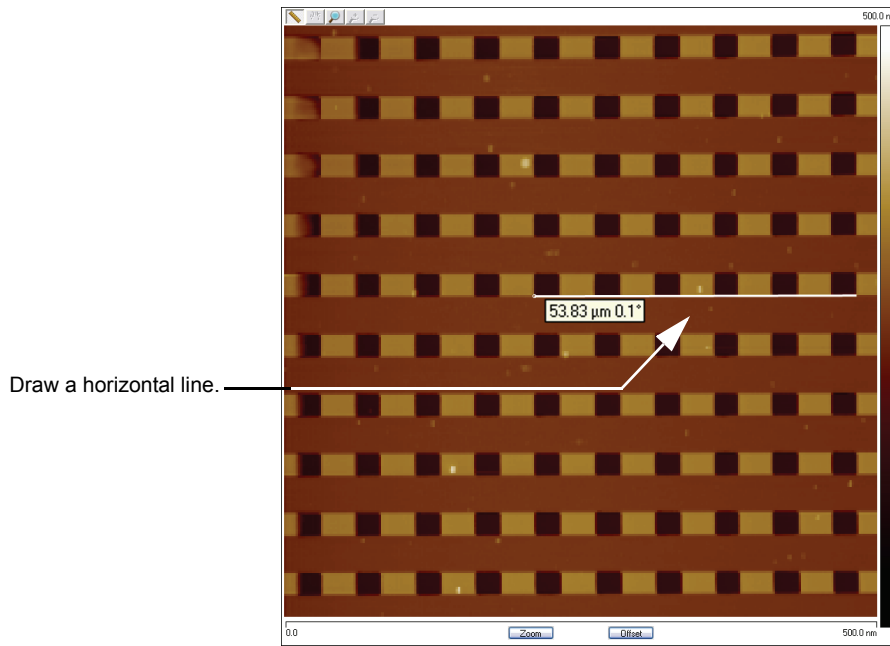
4. Draw a vertical line along an edge near the image center as shown in Figure 19.10b. Note the displayed angle.

Figure 19.10b Vertical Reference Line
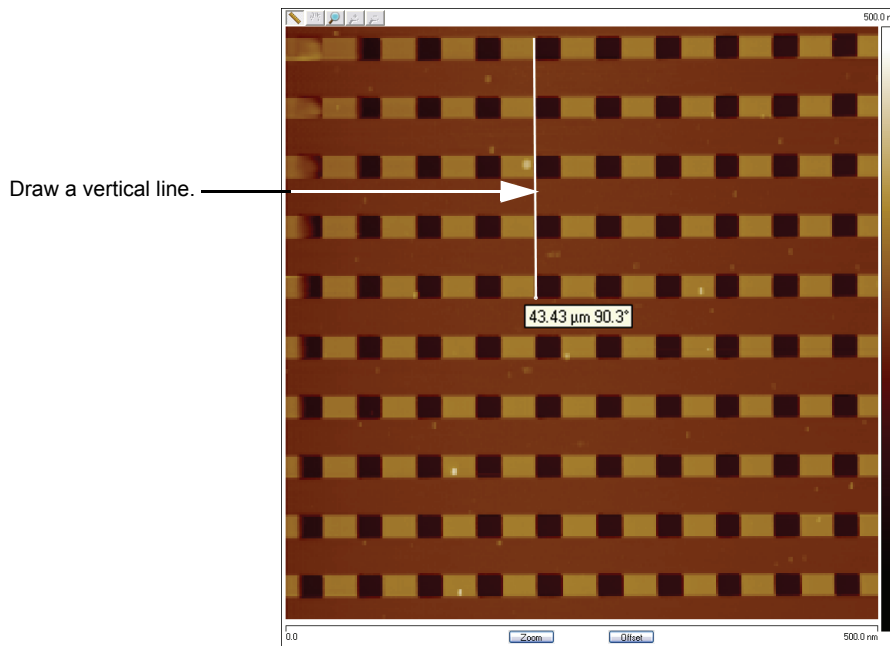
Draw a vertical line. ————

43.43 µm 90.3°

5. Perform the following calculation:

old **ORTHOGONALITY AT (0,0) OFFSET**
+ 90º
- (vertical angle - horizontal angle)

= new **ORTHOGONALITY AT (0,0) OFFSET**

6. Enter this value into **ORTHOGONALITY AT (0,0) OFFSET** in the **OptoXY Closed Loop Calibration** dialog box, shown in Figure 19.10c.

Figure 19.10c ORTHOGONALITY AT (0,0) OFFSET Correction
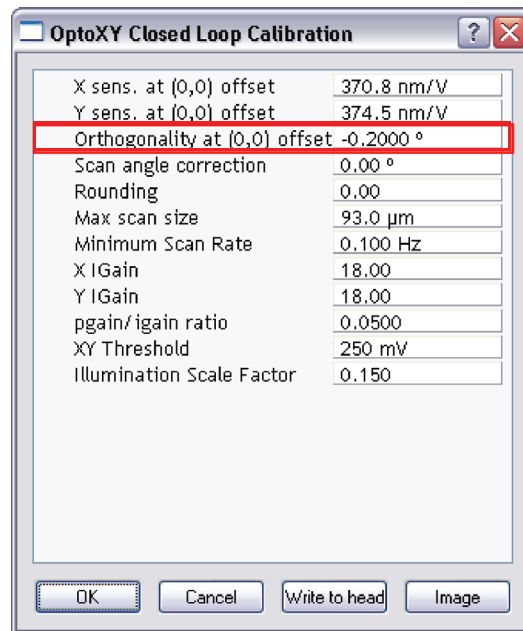
19.10.2 Measure Horizontally at 90µm Scan Size

1. Set **X OFFSET** and **Y OFFSET** to **0**.

2. Verify that the **Scan angle** is set to **0.00** degrees.

3. **ENGAGE** the surface.

> **Note:** Contact mode is preferred for this procedure because the scan rate is faster for contact mode than tapping mode.

4. Select two widely-spaced features on the sample image of known separation. Use the mouse to draw a horizontal line between them (see Figure 19.10d). (For example, on a 10µm silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The screen will display the measured distance between pits next to the line if the cursor is near the line.

Figure 19.10d Calibration Horizontal Reference
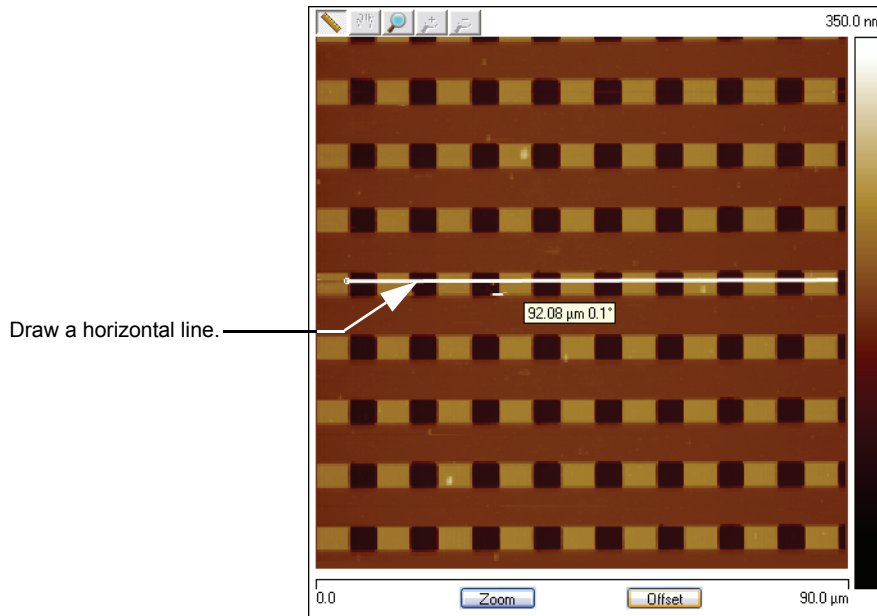
Draw a horizontal line.

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 **Measure Vertically at 90mm Scan Size**: Page 459**.**

6. Based upon the results in the above step, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

7. Select **Calibrate** > **X-Y**. The **OptoXY Closed Loop Calibration** dialog box displays (see Figure 19.10e).

Figure 19.10e   Closed Loop Scanner Calibration Dialog Box
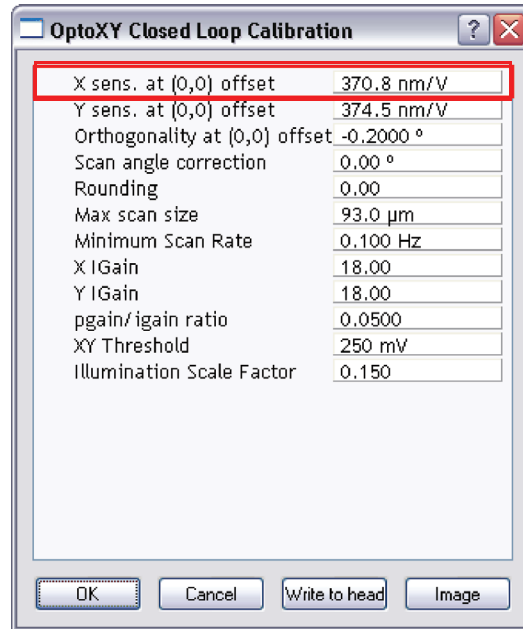
8. Multiply the quotient obtained in Step 6, above, by the **X sens at (0,0) offset** value shown on the **OptoXY Closed Loop Calibration** dialog box.

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 **OK**. This closes the **OptoXY Closed Loop Calibration** dialog box.

11. Repeat Step 4 to check your change.

19.10.3 Measure Vertically at 90μm Scan Size

1. Return to the image of the calibration reference.

2. Select two features that are widely spaced in the vertical direction and draw a vertical line connecting like portions of features (top edge-to-top edge, etc.). See Figure 19.10f. The SPM displays the calculated distance between features.

Figure 19.10f Calibration Vertical Reference
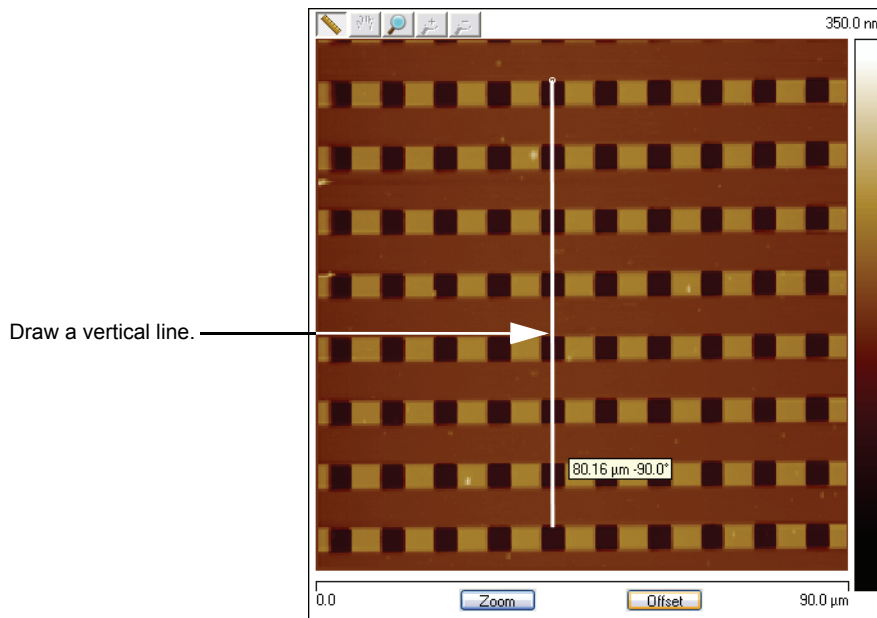
3. Verify that the microscope's calculated distance agrees with the known vertical distance. If there is significant disagreement between the two, fine tuning is required; go to the next step. If the displayed distance agrees with the known distance, no further calibration is required.

4. Using the results from the above step, divide the known distance by the distance displayed next to the line drawn a few steps earlier.

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

5. Select **CALIBRATE > SCANNER > X-Y** to display the **OptoXY Closed Loop Calibration** dialog box (see Figure 19.10e).

6. Select the **Y sens at (0,0) offset** parameter.

7. Multiply the quotient obtained in Step 5, above, by the **Y sens at (0,0) offset** value shown on the **OptoXY Closed Loop Calibration** dialog box.

8. Enter the new value to adjust the scanner's slow axis to more closely match calculated distances with actual feature distances.

9. To save the new parameter value, click **OK**.

10. Repeat Step 3 to check your change.

19.10.4 Scan angle correction

Scan angle correction is an additive adjustment to align the closed loop fast scan axis with the open loop fast scan axis.

To determine the value to enter for **SCAN ANGLE CORRECTION**:

1. Place on the stage a sample containing at least one straight line feature. A two-dimensional array of repeated features is ideal. No particular alignment of the sample is required to establish a value for **SCAN ANGLE CORRECTION**.

2. With the CLH in open loop mode, set **Scan** > **SCAN ANGLE** to **0°** and measure (see note below) on the display image the angle between the straight line feature and the fast scan axis.

3. Withdraw the tip from the sample.

4. Toggle **Scan** > **XY CLOSED LOOP** from **OFF** to **ON**.

5. Re-engage the tip with the sample. Repeat step 2 in closed loop mode, noting the angle between the chosen sample reference line and the X- (fast scan) axis.

6. Enter the difference, angle (closed) - angle (open), shown in Figure 19.10g, as the value of **OptoXY Closed Loop Calibration** > **SCAN ANGLE CORRECTION** (see Figure 19.10h).

Figure 19.10g   Scan Angle Correction Determination
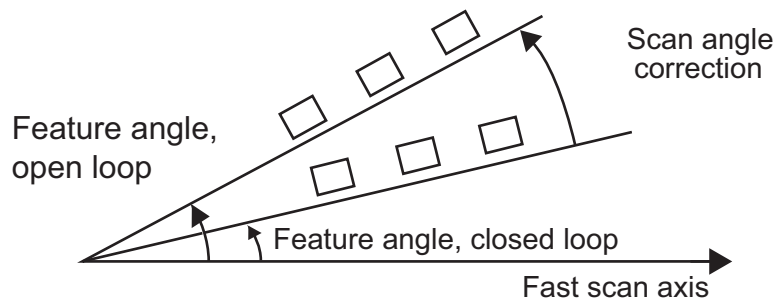
Figure 19.10h Scan Angle Correction
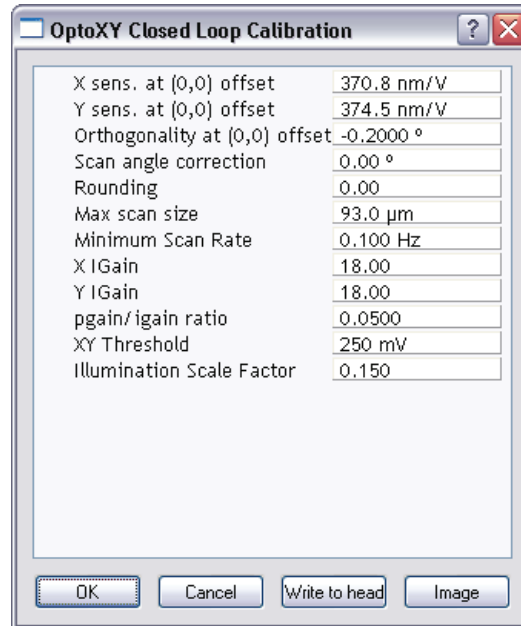
19.10.5 Other parameters

New parameters in the **OptoXY Closed Loop Calibration** panel are detailed below.

ROUNDING

Rounding defines the percentage of the fast scan time not captured or shown on the Display Window. A rounding of 0.1 corresponds to 10% longer scan lines than displayed in the image. This function allows mechanical systems to stabilize after the scan direction changes. This function distorts the linearity corrections made on the head or scanner.

Range and Settings: 0 - 0.50

> **Note:** This parameter should be zero for short, slow scans and increased only for long, fast scans.

MAX SCAN SIZE

A software limit on the largest scan along the fast scan axis.

Range and Settings: a positive number of microns. Typical value:100μm.

> **Note:** **MAX SCAN SIZE** applies only to closed loop operation and is set to **100MM** at the factory. It is not necessary to ever adjust it. Reducing **MAX SCAN SIZE** from its default setting restricts scan size. Increasing **MAX SCAN SIZE** from its default setting cannot damage the scanner and never results in a scan size larger than is obtained in open loop mode with 440V (the maximum allowed) applied across the piezoelectric scan tube. You might increase **MAX SCAN SIZE** to 110μm, for instance, if you determine your particular

scanner is capable of 110μm scan size in open loop mode and the extra scan size is valuable to you. The disadvantage of making this too large is that the XY feedback loop may reach the edge of the scan or sensor range. If this occurs, the edge of each line may have several pixels that are all collected at the same point.

MINIMUM SCAN RATE

The **MINIMUM SCALE RATE** sets a lower limit for the **SCAN RATE** parameter on the **Scan** panel.

Range and Settings: `0.001–10 Hz`; default = `0.100 Hz`

X IGAIN

The integral gain for closed loop scanner control along the X-axis (the fast scan axis if the scan angle is 0°)

Range and Settings: 0.0625 — 128. Typical values: 15.

Y IGAIN

The integral gain for closed loop scanner control along the Y-axis (the slow scan axis)

Range and Settings: 0.0625 — 128. Typical values: 15.

XY THRESHOLD

The RMS amplitude of high pass filtered X- or Y-scan error signal that is interpreted as evidence of control loop oscillation, so triggers reducing **X IGAIN** or **Y IGAIN** to reachieve control.

Range and Settings: 0 — 500mV. Typical value: 250mV; adjust higher if needed.

PGAIN/IGAIN RATIO

Proportional gain as a fraction of integral gain, applied to both X and Y axes.

Range and Settings: 0.05 — 1.00. Typical value: 0.3.

19.11 Z Calibration

The height and Z sensor signals require separate calibrations. Because the Z sensor is linear, only one calibration constant is required. However, the height sensitivity (**Z SCAN SENS**) varies as a function of offset, so multiple calibration points are necessary. We use **Z OFFSET** = **0** and **Z OFFSET** = **±50V**. The silicon calibration references distributed by Veeco have specified vertical features accurate to within ± 2%. The calibration reference is referred to throughout the examples provided in this section.

> **Note:**   Refer to the label on your calibration reference sample to verify the measurement employed. Older systems may have samples with a different Z value.

19.11.1 Engage Surface

1. Set up the microscope for **TappingMode** imaging.

2. **ENGAGE** and begin scanning in Contact mode.

3. Find a square pit and center the pit in the image using a **Scan size** of approximately **10MM**.

4. Verify that the **Z Center Position** value shown next to the image display is close to **0 volts** (**±5 VOLTS**), shown in Figure 19.11a.

Figure 19.11a   Z Piezo Voltage (position)
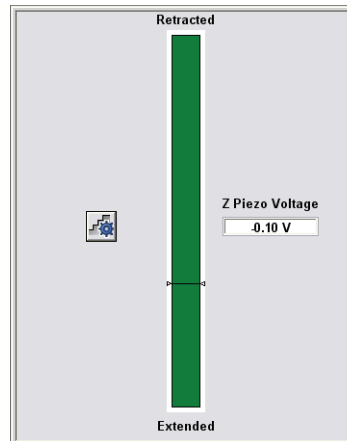
5. If the **Z Center Position** value is not close to zero, use the **Microscope** > **Step Motor** > **TIP UP** and **TIP DOWN** buttons to adjust (see Figure 19.11b).

Figure 19.11b   Step Motor
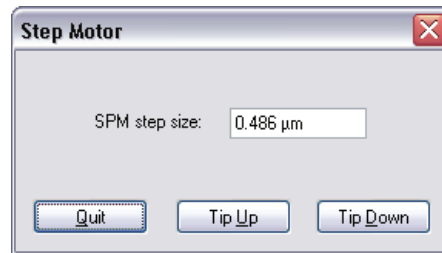
19.11.2 Capture and Correct an Image

1. Set **Channel 1 Data type** to HEIGHT and **Channel 2 data type** to HEIGHT SENSOR.

2. Capture an image by selecting **Capture** from the NanoScope tool bar, or click the CAPTURE icon.

3. When the image is captured, load the image by double clicking it in the browser bar.

4. Click the height image (CHANNEL 1) to select it.

5. Remove all tilt and scan line errata from the image by selecting **Modify** > **Flatten** or click the **FLATTEN** icon on the NanoScope tool bar.

   a. Set the **FLATTEN ORDER** parameter in the input box to **1ST**.

   b. Go to the display window and draw a stopband over the pit as shown in Figure 19.11c.

Figure 19.11c   Draw a Stopband
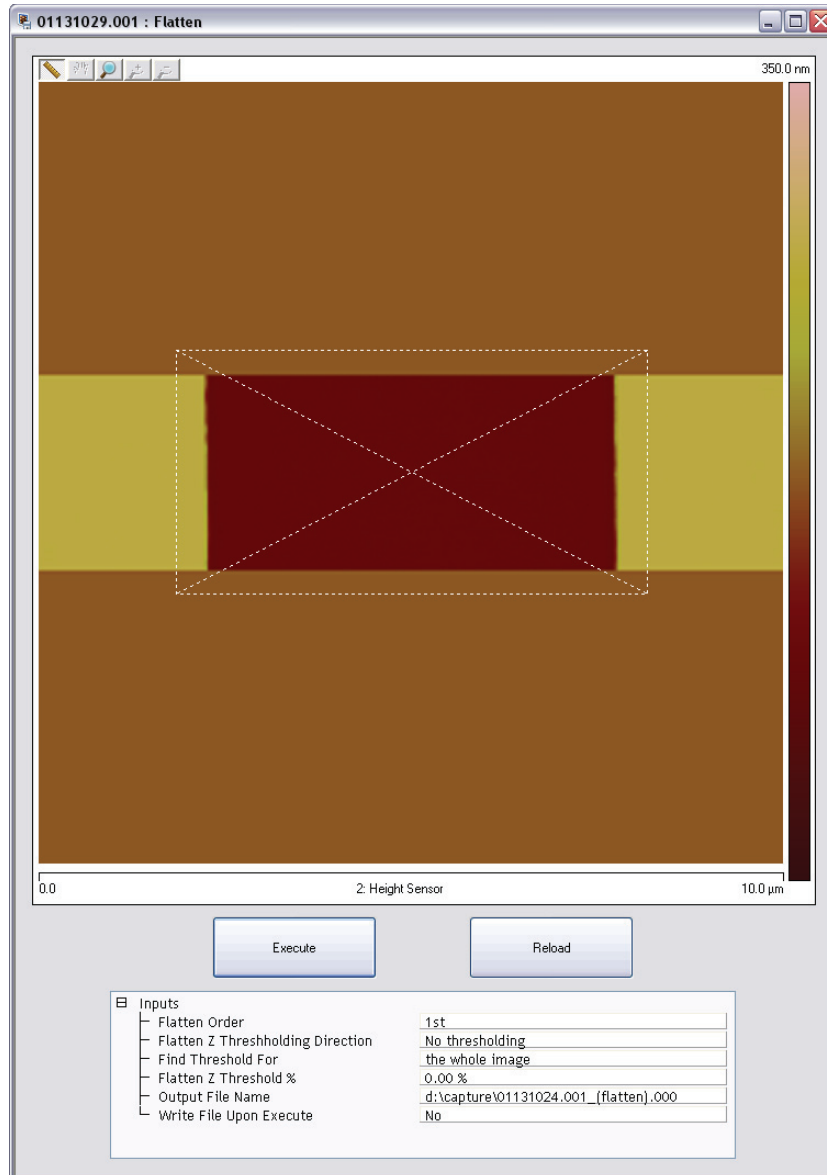
   c. Click **EXECUTE** to complete the flattening procedure.

   d. Right click the image, select **CHANNEL 2** (**Z SENSOR**) and click **EXECUTE** again to flatten **CHANNEL 2**.

19.11.3 Measure Vertical Features

With the image corrected, its vertical features may now be measured. This is performed using **Depth** analysis to utilize more data points.

1. Select the **Analysis** > **Depth** command or click the **DEPTH** icon on the NanoScope tool bar. This loads the **Depth** analysis screen shown in Figure 19.11d.

2. Right click the image and select **CHANNEL 2**, **HEIGHT SENSOR**.

Figure 19.11d   Depth Analysis Screen
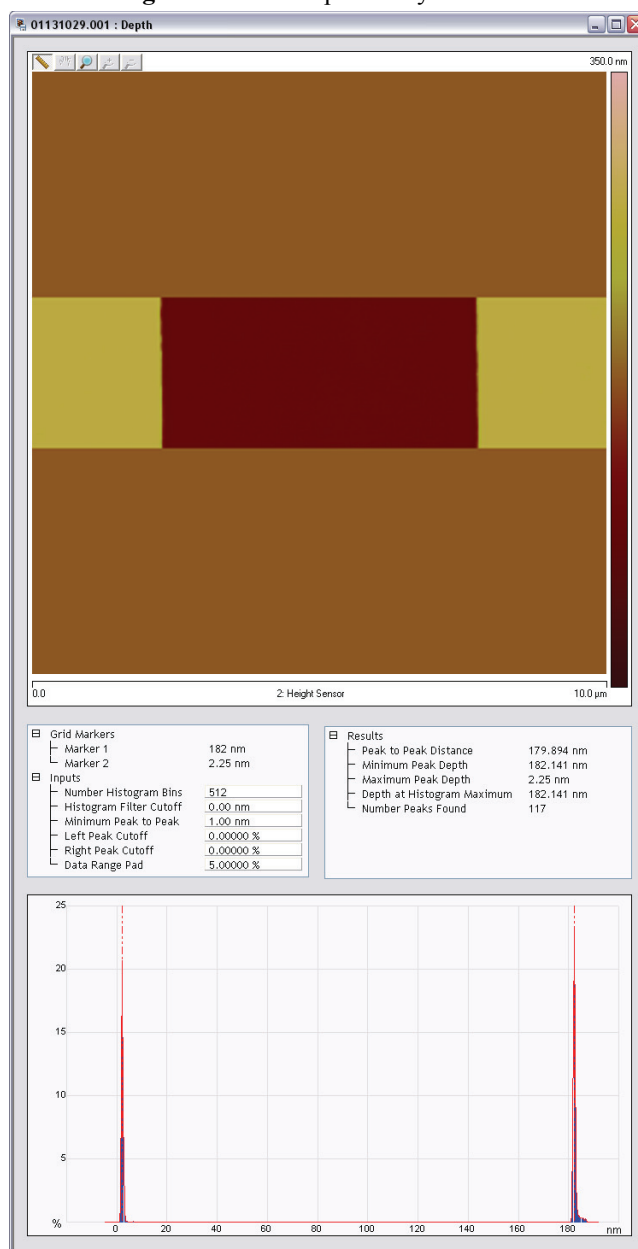
3. If desired, part of the image may be selected for analysis by drawing a cursor box surrounding that region. Otherwise, the whole image will be used (see Figure 19.11d).

> **Note:** A histogram of the height data within the drawn cursor box displays in the window, showing two prominent peaks. These peaks correspond to two elevations on the surface: the bottom of the pit and the top surface. There should be a line cursor on each peak.

4. If the two peaks do not appear in the display, increase the **Histogram filter cut off** in the **Inputs** dialog box to **2-10ɴᴍ**. If the two peaks are too close together, increase the **Minimum Peak to Peak** in the **Inputs** dialog box.

5. In the **Results** dialog box located to the right of the image in the **Depth** panel, check the **Peak to Peak Distance** and record it for future reference.

19.11.4 Correct Z Sensitivity

If the depth of the pit on the 10µm silicon calibration reference deviates significantly from the specified depth, correct the **Zscan sens.** parameter in the **Z Calibration** dialog box.

1. Open the **Z Calibration** dialog box by selecting **Calibrate** > **Scanner** > **Z**.

2. Divide the actual depth of features by the measured depth (indicated in **Depth** analysis by the **Peak to peak distance**):

$$\frac{\text{actual depth}}{\text{Peak to peak distance}}$$

3. Multiply this quotient by the **Zscan sens.** value in the **Z Calibration** dialog box, and replace the former **Zscan sens.** value with the new result.

4. Click **ok** to accept the new **Zscan sens.** value.

19.11.5 Recheck Z-axis Measuring Accuracy

After executing the steps above, recheck the Z-axis measuring accuracy of the SPM by repeating the steps outlined above until you obtain accuracy of 1 to 2%.

19.11.6 Z Sensor Sensitivity

Repeat Sections **Capture and Correct an Image**: Page 465, **Measure Vertical Features**: Page 467 and **Correct Z Sensitivity**: Page 468 using **Channel 2**. This time you will adjust **Z sensor sens** rather than **Z scan sens**.

19.11.7 Calculate Retracted and Extended Offset Deratings

Piezoelectric materials exhibit greater sensitivity at higher voltages. In the steps outlined above, the Z-axis was calibrated while scanning near the middle of the piezo voltage range (i.e., **Z Center Position** ~ `0V`). In this section, you will calibrate the Z-axis piezo while extended and retracted to determine how much the Z sensitivity varies with **Z Center Position**.

1. If you are not already **ENGAGE**d, select **ENGAGE** under the **Microscope** pop-down menu or click the **ENGAGE** icon. See **Engage Surface**, page 464.

2. Use the **Microscope** > **Step Motor** > **TIP UP** button until the **Z Center Position** reads `50V` (±5V).

   Note: By using the motor to move the tip up, the feedback loop forces the Z-axis piezo to extend to continue its tracking of the surface.

3. Refer to the steps above (**Capture and Correct an Image**, page 465 through **Measure Vertical Features**, page 467) to determine the measured depth of the calibration standard with a `+50V` **Z Center Position**.

4. Record the measured depth. If the depth measured by the extended piezo is correct within two percent, skip to Step 9.

5. Select **Calibrate** > **Z** to display the **Z Calibration** window, shown in Figure 19.11e.

6. Click the **Extended offset der** parameter.

7. Perform the following calculation:

$$(1 + \text{current offset der}) \bullet \frac{\text{actual depth}}{\text{meas. depth}} - 1$$

For example, if the current offset equals 4% and the measured depth equals 185nm, then:

$$(1 + 0.04) \bullet \frac{\text{actual depth}}{\text{meas. depth}} - 1$$

8. Enter the new percent value from above (example was 12%) for the **Extended offset der** parameter in the **Z Calibration** panel.

9. The procedure for calculating and setting the **Retracted offset der** is exactly the same as for the **Extended offset der**; however, the piezo must be **retracted** to `−50V`. To calculate the **Retracted offset der**:

   a. Use the **TIP DOWN** button in the **Step Motor** panel to retract the piezo.

   b. Retract the **Z Center Position** to `−50V`.

   c. Repeat Step 3 - Step 8 above.

CALIBRATE > SCANNER > **Z** opens the **Z Calibration** panel, shown in Figure 19.11e, which includes definition of the applicable range of Z-piezo voltages [Z-sensitivity (in nm/V)] is doubled in CLH closed loop mode. The voltage values entered and displayed to induce retraction or extension of the tip are -50V and +50V, respectively, in CLH closed loop mode, rather than the usual ±100V of open loop mode. All **Z Calibration** panel parameters apply to both open loop and closed loop operation.

Figure 19.11e   Z Calibration Panel
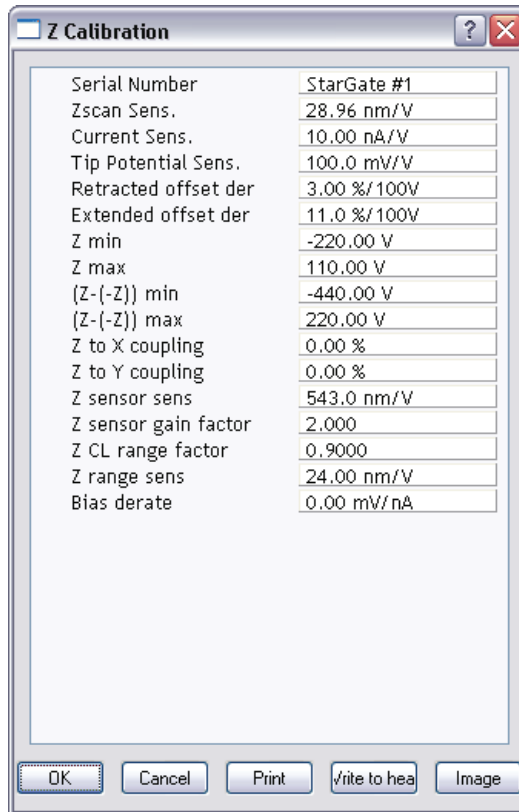
ZMIN

The lowest voltage allowed to be applied to the negative terminal of the Z-piezoelectric actuator.

Range and Settings: -220 — +210V. Typical value: -130V.

ZMAX

The largest voltage allowed to be applied to the positive terminal of the Z-piezoelectric actuator.

Range and Settings: -210 — +220V. Typical value: +110V.

	CAUTION:	**ZMIN** and **ZMAX** are set at the factory or during installation of the CLH scanner and are based on the safe operating range of the Z-axis piezoelectric tube. Lowering **ZMIN** or raising **ZMAX** to outside the factory-set range can result in irreparable damage to the scanner. Consult with Veeco *first* if you believe **ZMIN** or **ZMAX** should be adjusted.

Z-(-Z)) MIN

Twice the absolute value of Zmin, computed automatically, requiring **ZMAX** $\geq$ **ZMIN** + 10V.

(Z+(-Z)) MAX

Twice the absolute value of **ZMAX**, computed automatically, requiring **ZMAX** $\geq$ **ZMIN** + 10V.

19.12 Closed Loop Z Calibration

Several additional parameters are used to calibrate the Z-axis in closed loop operation:

Z SENSOR SENS

The sensitivity, in nm/V, when **Z SENSOR GAIN FACTOR** = **1**, of the optical Z-axis sensor. Determine this by measuring a calibration standard with the Z sensor channel. See **Z Sensor Sensitivity**, page 468.

Range and Settings: 0 — 1000nm/V. Typical value: 500nm/V.

Z SENSOR GAIN FACTOR

During the engage, the Z piezo is ramped to find the Z piezo range. The sensor gain is adjusted to set a Z sensor range that is: (Z sensor range factor)*(Z piezo range).The gain of the Z sensor. Make this larger than 1 to ensure that the dynamic range is covered. Note that the resolution of the Z sensor ADC is decreased by this amount.

Range and Settings: 1 — 2. Typical value: 1.5.

Z CL RANGE FACTOR

The range of the Z sensor that is used in Z closed loop mode (ramping). Making this larger increases the chance that part of the ramp may reach the end of the scanner range.

Range and Settings: 0 — 1. Typical value: 0.9.

Chapter 20   Maintenance, Troubleshooting and Warranty

This chapter includes the following sections:

- **Maintenance:**

    - **Cleaning:**

    - **Calibration:**

    - **Fuse Characteristics and Replacement:**

    - **Air Tables:**

    - **Air Pressure:**

    - **Air Table Feet:**

- **Troubleshooting:**

    - **Alarms and Error Messages:**

    - **Common Problems:**

- **Warranty:**

20.1   Maintenance

Generally, the Dimension Icon system requires very little maintenance. A simple schedule of cleaning and calibration check is usually sufficient.

Every three months:

1. Wipe down the major components of the instrument.

2. Clean the SPM stage.

3. Check the air table for proper level and isolation.

4. Check piezo calibration.

20.1.1   Cleaning

Major Components

The Dimension Icon is essentially an assortment of electronic boxes. You should use the same care with this equipment as with other laboratory instruments. Wipe the exterior of the system with clean paper towels or a cloth dampened with water or alcohol. Dispose of wipes in an appropriately labelled solvent-contaminated waste container.

SPM Stage

The surfaces of the granite base, sample chuck and chuck manifold should be wiped down with a lint-free cloth dampened with acetone. To properly perform this:

1. Remove the scanner from the Z stage and place it in a safe place.

2. Remove the sample chuck by lifting it off of the chuck manifold.

3. Disconnect the manifold from the X stage assembly by removing the three Phillips head screws that pass through the X flexure.

⚠️	**CAUTION:**	Do not remove the vacuum hoses on the side of the manifold! The upper hose has a wire inside that will be damaged if the hoses are pulled off.

4. Lift the manifold off of the granite base.

5. Wipe the top of the granite stage base.

6. Wipe the top and bottom of the manifold.

7. Reconnect the manifold to the X stage assembly.

8. Wipe the underside of the sample chuck.

9. Place the chuck back on the manifold.

10. Dispose of wipes in an appropriately labelled solvent-contaminated waste container.

SPM Optics

The video objective on the left side of the stage should be periodically cleaned with a mild glass cleaner or solvent such as acetone or alcohol.

Dimension Scanner

Laser and Photodiode Adjustment Screws

Dust and other contamination can cause the lubrication on the screws to thicken over time, thereby causing the screws to become difficult to turn.

> **Note:** We recommend you clean one screw at a time to avoid mixing up the screws.

1. Remove a screw from the scanner by turning it counterclockwise.

2. Wrap a pipe cleaner around the threads on the screw.

3. Dip the screw in acetone. Unscrew the screw in the pipe cleaner. The bristles on the pipe cleaner are small enough to dig out the accumulated grease in the threads.

4. Clean the threaded insert in the scanner with a Q-tip dipped in acetone.

5. Lubricate the screw and the leading edge of the threaded insert with a small amount of silicon vacuum grease.

6. Thread the screw back into the scanner head.

7. Repeat the cleaning procedure with the other three adjustment screws.

20.1.2 Calibration

A description of the complete scanner calibration is located in a separate chapter. In general the scanner's calibration should be checked every three months. The main change in calibration will be the loss of piezo sensitivity in the first year of use. This is an expected behavior. Loss of 10-15% of the initial scan size is common.

Inspections and Procedures

Noise Test Procedure

Although a noise test is performed prior to shipment and during a new installation, this test is useful to periodically test the instrument's performance in your lab. Increases in the noise level of the instrument are typically due to some sort of environmental change such as the introduction of new equipment in proximity of the Dimension Icon stage. The Dimension Icon system itself is historically not prone to degrade over time, although changes to the environment are a common source of noise.

Because the Dimension Icon is sensitive to vibration, it is often a convenient tool for checking the performance of the vibration isolation system. The following test can be performed under ambient conditions or while an external noise source is applied to the floor.

1. Install a new cantilever and engage in TappingMode on a Si wafer (500nm to 1μm scan size). You can also image the substrate of a silicon probe. The probe can be mounted to a small sample AFM puck with double-sided tape and held to the stage magnetically.

2. Set the **Z limit** to **55V** (~1μm). Set the **Samples/line** to **512**.

3. Set the gains as low as possible, while still maintaining a good image with visible surface roughness. Do not lower the **Integral** and **Proportional Gain** below **0.1**.

4. Change the **Scan Size** to **1nm** and capture an image. The Scan rate is not important for this Scan size. You can set the **Scan Rate** to 10Hz to save time. If you have low frequency noise, you may want to set the **Scan Rate** to 2Hz so that you can see at least a few periods (longer time base).

5. Perform a first order **Flatten Auto**. Then, run the roughness measurement. The **RMS value** should be typically **0.5Å**.

An easy way to analyze noise is to use the Section Analysis function. If you draw a line that is perfectly horizontal, the one dimensional Fourier plot will show you what frequencies are contributing to your noise. Remember that when the Scan size is this small, you no longer have an image but rather a temporal graph of the Z displacement.

20.1.3 Fuse Characteristics and Replacement

- **NanoScope V Controller**: 4 fuses. The fuse characteristics are silk-screened on the back of the NanoScope V Controller (see Table 20.1a).

Table 20.1a   Fuse Ratings

Fuse	100V	120V	240V
1	2.5A	2.5A	2.0A
2	800mA	800mA	400mA
3	800mA	800mA	400mA
4	10A	10A	6.3A
ALL FUSES: 250V SLOW BLOW			

- **Dimension Stage Controller**: 1 fuse. The fuse characteristics are silk-screened on the back of the Dimension Stage Controller (see Figure 20.1a).

Figure 20.1a   Stage Controller Fuse Ratings

FUSE RATINGS				
FUSE	100V	120V	220V	230/240
1	1A	0.8A	0.4A	0.4A
USE TIME DELAY FUSES				

Fuse Replacement Procedure

1. Remove all power from the system.

2. Using a screwdriver, press the slot in the fuse holder and rotate the fuse a 1/4 turn counter-clockwise.

3. Gently pull the fuse out.

4. Insert a replacement fuse in the fuse holder.

5. Using a screwdriver, press the slot in the fuse holder and rotate the fuse a 1/4 turn clockwise until the fuse is secure.

6. Reconnect the power.

> **Note:** If the problem persists, contact Veeco.

20.1.4 Air Tables

The VT-1000 air table is manufactured by TMC™ corporation and is referred to in this section. The AH-1000 Integrated Acoustic/Vibration Isolation System includes the VT-1000 air table.

Having a properly installed air table is necessary for a properly operating AFM system. It is very common to see periodic noise in the AFM data due to an under-inflated or improperly balanced air table. It is important to check the air table when you move the instrument.

20.1.5 Air Pressure

The incoming air pressure should be approximately 80PSI. The air table has a built in regulator that is preset for optimum performance.

> **Note:** Incoming pressure higher than 80PSI has not been proven to be harmful. It is just not recommended.

20.1.6 Air Table Feet

The frame of the air table has four legs with feet that support the Dimension stage. The feet should be lowered so that the air table is raised off of the wheels. Periodically check to make sure that the weight of the air table is evenly distributed on all four legs. To do this, simply use the supplied spanner wrench to slightly turn each of the feet in sequence. If the foot you are testing turns easily, there is no weight pressing down on it. Readjust the feet so that each foot is supporting an equal amount of weight.

20.2 Troubleshooting

The Dimension Icon system is very reliable and generally will not need adjustment once properly calibrated, nor is it likely to develop a problem due to use. In most cases any problems that occur can be linked to some external event.

Some typical examples of such events are:

- Lighting storms that cause power transients can damage circuit boards.

- Fluid spills on electrical components can cause damage.

- Improper installation of the air table when relocating the instrument to a new room can cause noise in the data.

- Improper signal cable installation when relocating the instrument can cause problems.

- After relocating the instrument to a new room, the data may exhibit noise. The room itself may have excessive floor vibration or acoustic noise.

- Installing a new board other than what is needed to operate the instrument via the computer can cause the software to not load correctly or a hardware conflict within the computer system.

20.2.1 Alarms and Error Messages

Alarms

The Dimension Icon stage controller will produce a high-pitched noise if it is tilted more than 45 degrees. There are mechanical pumps that need to be kept level in order to properly work.

The Dimension Icon stage controller alarm will also sound if the internal temperature exceeds 40°C. The illuminator inside the controller can cause the inside temperature to rise beyond this point if the ventilation to the controller is restricted. The air inlet screen on the front of the controller may become clogged in a dusty environment.

Software Error Messages

- "**Illuminator port timed out**" – The serial port connected to the Dimension stage controller is not connected or is not communicating properly.

- "**Stage not initialized**" – The serial port connected to the Dimension stage is not connected or is not communicating properly

- "**Stage controller error**" – The serial port connected to the Dimension stage is not communicating properly.

- "**Illuminator Error, Manual override is on**" – The manual control on the front of the Dimension stage controller is being used. Turn the illuminator manual control knob counter-clockwise until a click sound is heard, indicating that it is switched off.

- "**Bad data read from illuminator was 0,S.B. 80**" – The Dimension Icon stage controller power is off. The power supply inside the Dimension stage controller is not working.

- "**Warning focus position not updated**" – You tried to move X-Y stage with no AFM SUM signal.

- "**Safety check failed SUM too low**" – 2 Explanations:

  - You tried to engage with no AFM SUM signal.

  - You tried to move X-Y stage with no SUM signal.

- "**The focus axis is at its limit**" – The focusing objective is either fully extended or fully retracted. This message will appear during the locate tip routine.

- "**Warning: Sample clearance reduced! Unable to move past limit**" – The focusing objective reached the extended limit when moving to the locate tip position. This can happen during STM use due to the lower tip location. The end of the STM probe should protrude no more than 1mm below the end of the tip holder tube. This message can also occur if the sample clearance is set to a very large value. The standard sample clearance distance is 1000µm.

- "**Motor axis is at limit**" – The SPM (Z stage) is fully retracted. This can happen after the stage is initialized. Ignore the message and lower the Z stage by using the Focus Surface controls.

- "**The tip location has been changed to an unsafe distance. If you proceed with this change you will need to re-teach the tip replacement position.**" – This warning appears during the Locate Tip routine if the focusing objective has moved more than 300µm from the previous tip focus position. This is common when switching between silicon and silicon nitride cantilevers due the thickness difference of their substrates. The message will also appear when switching between AFM and STM probes. The intent of this message is a safety check to warn you that you have moved the objective unusually long distance. Select **OK** if you've changed to a different style of probe.

20.2.2 Common Problems

Bent stage flexures – The X or Y stage flexure can be accidentally bent if an object like a tool or tweezers is left on the granite base and the X-Y stage is driven into it. Stage flexures can also become bent due to improper shipping. The X-Y flexures are removed before a new system is shipped for this reason. A bent stage flexure will cause noise in the image due to a vacuum leak between the granite stage base and the chuck manifold. Bent flexures will also cause not repeatable movement in the X-Y stage motion.

No Laser light from scanner – Possible Explanations:

- The software is not running.

- The microscope mode is set to STM in software.

- A microscope other than a Dimension Icon is selected in software.

No stage illumination – Possible Explanations:

- Manual override is on.

- Blown fuse on illuminator circuit board. A spare fuse is provided next to the fuse holder.

No chuck vacuum – Possible Explanations:

- The fuse that powers the vacuum pump is blown. Verify that F1 on the main circuit board in the stage controller is good. Replace with the spare fuse if it's blown.

- The vacuum pump is bad.

- The air and vacuum hoses are not connected.

- One of the four screws on the chuck is missing. The center screw and the four small Phillips screws plug various holes that direct the vacuum to the chuck.

Fluid spills – Fluid spills will cause numerous problems. The most common problem is that the piezo electric element in the Dimension scanner can become permanently damaged. Avoid fluid contact with the end of the scanner at all times.

Cables – The wires inside the cable connector on the Dimension scanner can be broken from the 47-pin plug. Disconnecting or connecting the scanner by pulling on the cable can cause this.

The scanner plug is very durable and typically does not fail due to use. Never connect or disconnect the dimension scanner cable from the stage by pulling on the cable. Always connect and disconnect the scanner by holding on to the plug.

20.3   Warranty

All new catalog-listed standard equipment sold and/or manufactured under Veeco labels is warranted by Veeco to be free of defects in material and workmanship if properly operated and maintained. This one-year warranty covers the cost of necessary parts and labor (including, where applicable as determined by Veeco, field service labor and field service engineer transportation) during the warranty period.

Warranty period takes effect upon date of shipment. Except as detailed below, these warranties extend to parts which are components of standard catalog items and manufactured by persons other than Veeco. The manufacturer warranty covers purchased equipment incorporated into any item supplied by Veeco.

Expendable items, including but not limited to styli, lamps, and fuses, are specifically excluded from the aforementioned warranties and are not warranted. All used equipment is sold on an "as is, where is" basis without warranty, express or implied.

Equipment made or modified to purchaser specifications on special order shall carry the above warranties with respect to material and workmanship, but shall be specifically excluded from any other warranties, express or implied, including those related to performance specifications. Special components shall only carry the original manufacturer warranties.

Warranty Claims

Veeco's obligation under these warranties is limited to repairing or replacing at Veeco's option defective non-expendable parts. Veeco's obligation shall not extend to defects that do not impair service. No claim will be allowed for any defect unless Veeco has received notice of the defect within thirty days following its discovery by purchaser.

Claims for Shipment Damage

No claim will be allowed for equipment damaged in shipment sold under standard terms of F.O.B. Factory. Within thirty days of purchaser's receipt of equipment, Veeco must receive notice of any defect which purchaser could have discovered by prompt inspection of equipment. In any event, Veeco shall have the option of inspection at purchaser's premises or at Veeco's plant before allowing or rejecting the claim.

Documentation Feedback

Veeco is dedicated to the ongoing improvement of our products and technical documentation. If you have any comments regarding this manual or any other Veeco documentation, please e-mail us at: **SBOTechPubs@veeco.com**.

Warranty Eligibility

To be eligible for the above warranties, purchaser must perform preventative maintenance in accordance with the schedule set forth in the manual provided. Veeco assumes no liability under the above warranties for equipment or system failures resulting from improper operation, improper preventative maintenance, abuse or modifications of the equipment or system from the original configuration.

> **Note:** This warranty is in lieu of all other warranties, expressed or implied and constitutes fulfillment of all of Veeco's liabilities to the purchaser. Veeco does not warrant that the system can be used for any particular purpose other than that covered by the applicable specifications. Veeco assumes no liability in any event, for consequential damages, for anticipated or lost profits, incidental damages or loss of time or other losses incurred by the purchaser or any third party in connection with systems covered by this warranty or otherwise.

Service

Field service is available nationwide. Service and installations are performed by factory trained Veeco service personnel. Contact the Veeco Metrology sales/service office for prompt service.

Veeco Instruments Inc.
112 Robin Hill Road
Santa Barbara CA 93117
Attn.: Service Center

Phone: (805) 967-2700
Fax: (805) 967-7717

Index