MultiMode™ SPM Instruction Manual

Version 4.31ce

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CAUTION: Some early-model AFM tipholders may short out power supplies when used with MM-SPMs. If you suspect your tipholder is from an older AFM, check with Digital Instruments or your international representative before using. **2-9**

ATTENTION: Certains modèles de support de bras de leviers pour AFM de contact peuvent endommager les alimentations électriques quand ils sont utilisés avec un MultiMode (MM-SPMs). En cas de doute, contacter Digital Instruments ou son représentant légal pour verification. **2-9**

WARNHINWEIS: Einige ältere Ausführungen des AFM-Spitzenhalters können einen Kurzschluß verursachen falls sie mit einem MultiMode-AFM verwendet werden. Bitte wenden Sie sich im Zweifelsfall an Digital Instruments bzw. an die für Sie zuständige DI-Vertretung. **2-9**

WARNING! During and prior to set up of the laser, it is especially important to avoid looking directly at the laser beam or at the laser spot. The laser head should never be plugged into the microscope control electronics unless the head is installed in the Z-stage mount. Care should be taken when highly reflective samples are inserted onto the chuck. Avoid looking at all reflected laser light. Operators should use care to avoid staring into beams that may be reflected from sample surfaces. **5-2**

ATTENTION! Avant d'utiliser le laser, et durant tout le temps pendant lequel il fonctionne, il est impératif de ne pas regarder directement le faisceau. La sonde laser ne doit jamais être branchée sur l'électronique de contrôle du microscope, tant que la tête 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. **5-2**

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. **5-2**

WARNING! Staring at a bright beam or reflection can result in eye damage. Be sure that you are using a magnifier with a laser filter installed. **5-10**

AVERTISSEMENT! Fixer un faisceau lumineux puissant ou sa réflexion peuvent entrainer des dommages au niveau des yeux. Il est impératif d'utiliser un filtre laser. **5-10**

WARNUNG! Der direkte Blick in den Laserstrahl oder dessen Reflektion kann Augenschädigungen hervorrufen. Bitte stellen Sie auf jeden Fall sicher, daß ein optisches Gerät, mit dem Sie in den Laserstrahl schauen, mit einem Laserschutzfilter ausgestattet ist. **5-10**

WARNING! To avoid eye damage due to high-level laser light, do not place highly reflective or metallized objects into the head area while the laser is on. **5-13**

AVERTISSEMENT! Pour prévenir tout accident au niveau des yeux, dû à la lumière du laser, il est recommandé de ne pas placer d'objet très réfléchissant ou métallique sous le faisceau laser lorsque celui-ci est allumé. **5-13**

WARNUNG! Um Augenschädigungen aufgrund von Laserlicht zu vermeiden achten Sie bitte darauf, daß keine reflektierenden oder mit Metall beschichteten Gegenstände im Bereich des AFM-Kopfes benutzt werden, solange der Laser eingeschaltet ist. **5-13**

CAUTION! When imaging fluid samples, use extraordinary precautions against spillage. Fluids must NOT be spilled on or around the sample stage, electronic boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise, damage may result! In the case of a spill, immediately clean and dry all affected surfaces carefully. **7-1**

ATTENTION! En milieu liquide, prendre toutes les précautions pour éviter une fuite qui pourrait atteindre la platine porte -échantillon, le boitier électronique ou tout autre partie électronique du microscope. Eviter tout contact avec un liquide corrosif. **7-1**

WARHINWEISS! Falls Sie Proben in Flüssigkeiten abbilden, lassen Sie äußerste Vorsicht walten, damit keine Flüssigkeit verspritzt wird. Flüssigkeiten dürfen nicht auf die oder nahe der Probenhalterung, der Elektronikbox oder anderen Komponenten, die elektronische Bauteile enthalten, verspritzt werden. Vermeiden Sie bitte, korrosive Flüssigkeiten auf freiliegende Oberflächen zu verspritzen; andernfalls wären Beschädigungen die Folge! Falls Sie Flüssigkeit verspritzt haben, säubern und trocknen Sie alle betroffenen Flächen sorgfältig. **7-1**

IMPORTANT: Do not attempt to operate the standard air tipholder in a fluid environment. Standard tipholders have exposed electrical signal lines that could short circuit if exposed to a conducting fluid. **7-2**

ATTENTION: En milieu liquide, ne pas utiliser le support de bras de levier standard prévu pour une utilisation à l'air. Le support de bras de levier standard comporte des fils électroniques non protégés qui peuvent provoquer un court-circuit en cas de contact avec un liquide conducteur. **7-2**

WARNHINWEISS: 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. **7-2**

CAUTION! When imaging fluid samples, use extraordinary precautions against spillage. Fluids must NOT be spilled on or around the sample stage, electronic boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise, damage may result! In the case of a spill, immediately clean and dry all affected surfaces carefully. **7-4**

ATTENTION! Lors d'un travail en milieu liquide, prendre toute précaution pour éviter des fuites. Les liquides ne doivent pas se répandre sur la platine porte échantillons, le boîtier électronique ou toute autre partie du microscope contenant de l'électronique. Eviter toute fuite de liquide corrosif sur les surfaces exposées. Le non respect de cette recommandation peut entraîner des dommages. En cas de fuite, nettoyer et sécher immédiatement les surfaces touchées. **7-4**

WARHINWEISS! Falls Sie Proben in Flüssigkeiten abbilden, lassen Sie äußerste Vorsicht walten, damit keine Flüssigkeit verspritzt wird. Flüssigkeiten dürfen nicht auf die oder nahe der Probenhalterung, der Elektronikbox oder anderen Komponenten, die elektronische Bauteile enthalten, verspritzt werden. Vermeiden Sie bitte, korrosive Flüssigkeiten auf freiliegende Oberflächen zu verspritzen; andernfalls wären Beschädigungen die Folge! Falls Sie Flüssigkeit verspritzt haben, säubern und trocknen Sie alle betroffenen Flächen sorgfältig. **7-4**

CAUTION! When imaging fluid samples, use extraordinary precautions against spillage. Fluids must NOT be spilled on or around the sample stage, electronics boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise, damage may result! In the case of a spill, immediately clean and dry all affected surfaces carefully **8-26**

ATTENTION! Lors d'un travail en milieu liquide, prendre toute précaution pour éviter des fuites. Les liquides ne doivent pas se répandre sur la platine porte échantillons, le boîtier électronique ou toute autre partie du microscope contenant de l'électronique. Eviter toute fuite de liquide corrosif sur les surfaces exposées. Le non respect de cette recommandation peut entraîner des dommages. En cas de fuite, nettoyer et sécher immédiatement les surfaces touchées. **8-26**

CAUTION! Do not add excessive amounts of fluid or the cell will overflow and damage the piezo tube scanner. **8-29**

ATTENTION! N'ajoutez pas trop de fluide. Sinon, la cellule débordera et endommagera le scanner du tube piezo. **8-29**

CAUTION! When imaging fluid samples, use extraordinary precautions against spillage. Fluids must NOT be spilled on or around the sample stage, electronics boxes, or other components containing electronic parts. Avoid spilling all corrosive fluids on exposed surfaces; otherwise, damage may result! In the case of a spill, immediately clean and dry all affected surfaces carefully. **8-29**

ATTENTION! Lors d'un travail en milieu liquide, prendre toute précaution pour éviter des fuites. Les liquides ne doivent pas se répandre sur la platine porte échantillons, le boîtier électronique ou toute autre partie du microscope contenant de l'électronique. Eviter toute fuite de liquide corrosif sur les surfaces exposées. Le non respect de cette recommandation peut entraîner des dommages. En cas de fuite, nettoyer et sécher immédiatement les surfaces touchées. **8-29**

WARHINWEISS! Falls Sie Proben in Flüssigkeiten abbilden, lassen Sie äußerste Vorsicht walten, damit keine Flüssigkeit verspritzt wird. Flüssigkeiten dürfen nicht auf die oder nahe der Probenhalterung, der Elektronikbox oder anderen Komponenten, die elektronische Bauteile enthalten, verspritzt werden. Vermeiden Sie bitte, korrosive Flüssigkeiten auf freiliegende Oberflächen zu verspritzen; andernfalls wären Beschädigungen die Folge! Falls Sie Flüssigkeit verspritzt haben, säubern und trocknen Sie alle betroffenen Flächen sorgfältig. **8-30**

CAUTION: Because TappingMode cantilevers are relatively stiff, Force Mode can potentially damage the tip and/or surface. DI recommends using Force Plot only with extreme caution. **11-13**

ATTENTION: Les bras de leviers pour TappingMode étant relativement rigides, l'utilisation du mode “Force” peut endommager la pointe ou l'échantillon. Digital Instruments recommande d'utiliser le mode “Force Plot” avec une extrême précaution. **11-13**

WARNHINWEIS: Da TappingMode-Meßspitzen relativ steif sind, kann im Force Mode unter Umständen die Meßspitze oder die Oberfläche beschädigt werden. DI empfiehlt, den Force Plot nur unter größter Vorsicht anzuwenden. **11-13**

CAUTION: Because TappingMode cantilevers are relatively stiff, Force Mode can potentially damage the tip and/or surface. DI recommends using Force Plot only with extreme caution. **11-16**

ATTENTION: Le pointes Tapping étant relativement raides, l'utilisation du mode "Force" peut endommager la pointe ou l'échantillon. Digital Instruments recommande d'utiliser le mode "Force Plot" avec une extrême précaution. **11-16**

WARNHINWEIS: Da TappingMode-Meßspitzen relativ steif sind, kann im Force Mode unter Umständen die Meßspitze oder die Oberfläche beschädigt werden. DI empfiehlt, den Force Plot nur unter größter Vorsicht anzuwenden. **11-16**

CAUTION: If employing stiffer cantilevers than those normally used, the sample may be damaged by the cantilever tip. To minimize this damage, make the Setpoint as low as possible. **11-34**

ATTENTION! L'utilisation d'un levier plus rigide que ceux utilisés habituellement peut entraîner un endommagement de l'échantillon par la pointe. Afin de réduire les risques d'endommagement, ajuster la valeur cible du rétro-contrôle (Setpoint) à sa valeur la plus faible possible. **11-35**

WARNHINWEIS! Falls Sie mit Cantilevern höherer Federkonstanten arbeiten, als normal üblich, kann die Probe durch die Spitze beschädigt werden.. Um eine evtl. Beschädigung zu minimieren bzw. zu vermeiden, benutzen Sie bitte einen Arbeitspunkt (Setpoint), der nur geringfügig über der freien Deflektion des Cantilevers liegt. Die Differenz zwischen Setpoint im „Feedback-Menü" und der freien Deflektion des Cantilevers (Photodetektorsignal A-B) sollte minimal sein. **11-35**

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. **12-6**

ATTENTION: 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. **12-6**

WARNHINWEIS: 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. **12-6**

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. **13-12**

ATTENTION: Lors d'un travail en mode 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. **13-12**

WARNHINWEIS: 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. **13-12**

WARNING: Do not insert a conducting object (e.g., screwdriver) into the Phase Extender box while it is engergized. **13-14**

ATTENTION**:** Ne pas insérer d' objet conducteur (par exemple: un tournevis) dans le boîtier d'extension de phase (Phase Box) quand celui-ci est sous tension. **13-14**

WARNUNG: Stecken Sie keine leitfähigen Teile (zum Beispiel Schraubenzieher) in die Phase Extender Box, während diese eingeschaltet ist. **13-14**

CAUTION: When closing the System.par file after viewing it, if you are asked to SAVE the file or to SAVE CHANGES, be sure to say **No**. **13-15**

ATTENTION: Si vous avez besoin de consulter le fichier "System.par," à sa fermeture lorsque l'ordinateur demande SAUVEGARDER (SAVE) ou SAUVEGARDER LES CHANGEMENTS (SAVE CHANGES), répondre toujours **NON**. **13-15**

WARNHINWEIS: Falls Sie sich mit einem Editor die Datei „system.par“ anschauen und beim Verlassen des Editors gefragt werden, ob Sie die Datei speichern („SAVE“) bzw. die Änderungen speichern wollen („SAVE CHANGES“), antworten Sie bitte mit „Nein“ („No“). **13-15**

CAUTION! Do NOT splash solvent on the scanner tube or wiring at the center of the scanner body—certain components (e.g., wiring insulation) may be dissolved, causing scanner failure! **15-45**

ATTENTION! Ne pas éclabousser le tube en céramique piézo-électrique ou le montage à l'intérieur du tube avec un solvant. Certains composants pourraient être dissous, entraînant une défaillance du tube. **15-45**

WARHINWEIS! Verwenden Sie keine Lösungsmittel auf dem Scanner-Röhrchen oder den Kabelanschlüssen am Piezo - manche Komponenten (z.B. Isolation der Anschlußdrähte)könntensichauflösenundeineFehlfunktiondesScannersnachsichziehen. **15-45**

Chapter 1 *Introduction to the MultiMode™ SPM*

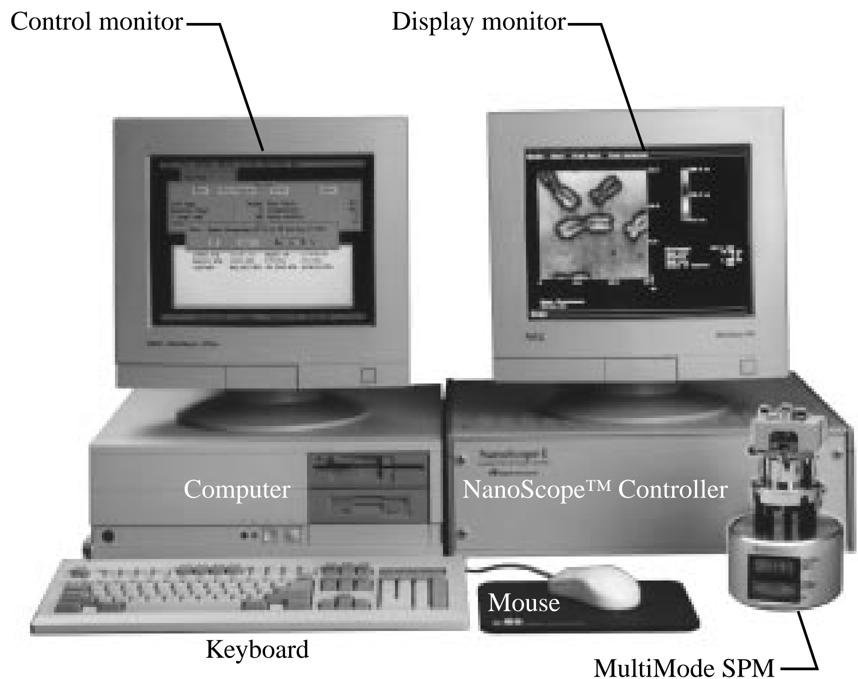

Figure 1.1. MultiMode™SPM system components.

1.1. Introduction

The MultiMode™ scanning probe microscope (MM-SPM) is one of Digital Instruments' original, mainstay designs and remains a highly dependable unit; several hundred MultiModes have been shipped to laboratories and businesses worldwide. The unit is designed for imaging small (approx. 1.5 cm dia.) samples using a series of interchangeable scanners and is able to provide images from the atomic scale to 175 µm in size. This manual is designed to assist operators with using the MM-SPM and has been rewritten from earlier editions. The reader is also referred to the *Command Reference Manual*.

The MM-SPM is designed around a stationary probe. That is, samples are scanned back and forth beneath the probe. (This is opposite to other of Digital Instruments' designs—e.g., Dimension Series SPMs—which mount samples stationary while scanning the probe back and forth above them.) Typically, samples are fixed to round 1.5 cm metal disks ("pucks"), then magnetically attached to the top of the scanner tube. As the scanner moves back and forth, the sample moves with it, allowing the probe to extract information from the sample surface much like a phonograph needle plays a vinyl record.

Because the size of features imaged with SPM is often below the visible wavelength of light, all information gathered from sample surfaces is electronically derived and rendered. Electronic controls have evolved through the years from an early array of switches and knobs to the present software control system, designated NanoScope™, version 4.x. Digital Instruments has divided its SPM software into a two-function architecture: **Real Time** and **Off-line**. The **Real Time** software functions are dedicated to running the actual microscope, changing the size and location of scans, controlling gains, etc. Images produced from scans may be analyzed and/or modified using the microscope's **Off-line** functions. These yield sectional profiles, correct for noise and artifacts, analyze for depth, roughness, grain size and power spectral density, among many other things. One major advantage to the NanoScope design is that both modes may be run simultaneously. That is, the microscope can save ("capture") images in **Real Time** mode while the operator is busy analyzing earlier images in the **Off-line** mode, making the MM-SPM a maximum-productivity tool.

How does it work?

This manual is designed to assist users of the MM-SPM in obtaining microscopic images by leading them through the most basic steps. No previous knowledge is assumed, and the reader is encouraged to forge ahead, even when full understanding of the microscope's workings may seem out of reach. Initially, the microscope may be treated as a "black box" by following instructions in this manual; however,

a deeper understanding of how SPM works will prove invaluable to the operator. Our advice: *experiment*! Consult the *Command Reference Manual* for more information regarding software controls.

Six Rules of Safety

Here is a summary of precautions to follow during your learning phase. If you follow the six rules below, the MM-SPM can come to little harm and you may feel free to experiment boldly.

- *1. Read the manuals!*

  Even if you have prior experience with the MM-SPM, be sure to read chapters 1-5 in this manual before doing any imaging work. Each of the remaining chapters are dedicated to specific types of imaging. Chapter 6 provides an introduction to contact AFM, which is a good place for beginners to start. Other chapters may be read as required. Also consult the *Command Reference Manual* for a complete explanation of software controls.

- *2. Follow good "rules of engagement."*

  "Engagement" refers to the process of bringing the tip and surface together. This is harder than it sounds, and the software routine for controlling the process is complex. Some probes (especially crystal silicon TappingMode™ probes) are prone to breakage if engaged too quickly or too hard. Ensure that engagement settings never exceed the limits of safety (see Chapter 5) and never attempt to engage manually using coarse adjustment screws.

- *3. Never move the head while imaging.*

  The head contains the tipholder, laser and photodiode array. An X-Y translation stage is provided for moving the head and tip several millimeters across the sample for coarse adjustment. Even for relatively smooth samples, the head should NEVER be moved with the tip engaged. This almost always results in tip breakage. Always disengage first before using the X-Y stage to move the tip.

- *4. Never leave your controller ON while the computer is turned OFF.*

  Operators are advised to turn OFF their controller and computer when finished with imaging. If the controller is left ON for an extended period without an energized computer, damage to the scanner may result. (This is especially true if the scan has been heavily offset in X and Y.)

- *5. Do not unplug cables to/from energized hardware. Turn OFF first.*

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

- *6. Check all connections before hardwiring external equipment.*

  External equipment which is hardwired into the MM-SPM, such as for EFM and ECSTM imaging, requires special cautions. To prevent damage to your microscope, always check connections carefully against documentation before energizing the system. For more information, see *Support Note 210*.

The first step to gaining a working knowledge of your microscope is to learn about its various parts and what they do. Chapter 2 provides a quick tour of the MM-SPM hardware and principles of operation.

1.2. Microscope Specifications

The MultiMode SPM, proven in over 500 installations, is the highest resolving SPM in the world. The microscope can be fitted with any of several scanners, depending upon the imaging requirements. Generally, the smaller the scan, the smaller the scanner used. This is especially true of atomic-scale scans, which are most often conducted with “A” or “E” scanners. Larger scans are normally performed using “J” scanners. Following is a list of basic specifications:

1.2.1. Image size and resolution.

Images consist of raster-scanned, electronic renderings of sample surfaces. There are three default sizes: 128 x 128 pixels, 256 x 256 pixels, and 512 x 512 pixels. In addition, six width-to-height aspect ratios may be specified by the user: 1:1, 2:1, 4:1, 8:1, 16:1, and 32:1. Thus, it is possible to obtain “strip scans” which require less time to capture.

The controller provides 16-bit resolution on all three axes, with three independent 16-bit digital-to-analog converters (DACs) in X and Y for control of the scan pattern, scaling and offset. This configuration provides 16-bit resolution of the lateral scanning motion at any scan size, and the ability to perform atomic resolution imaging throughout the full lateral range of the scanner. The patented digital feedback is governed by integral and proportional gain controls, providing immediate response to scanning parameter changes.

The MultiMode can scan up to 200 µm laterally (in X and Y) and 10 µm vertically (Z axis). A table summarizing each scanner’s capabilities is provided in Chapter 2.

1.2.2. Scanning techniques with the MultiMode SPM.

The MultiMode is so called because it offers multiple SPM modes, including AFM, ECAFM, ECSTM, STM and TappingMode. While many early SPMs offered only one dedicated operating mode (e.g., STM), the MultiMode was the world's first multiple-use SPM. It remains one of Digital Instrument's most versatile instruments. A complete range of Atomic Force Microscopy (AFM) and Scanning Tunneling Microscopy (STM) techniques is available with the MultiMode SPM. Some of these techniques are available only through Digital Instruments.

- **Contact AFM**— Measures topography by sliding the probe's tip across the sample surface. Operates in both air and fluids. See Chapter 6.
- **TappingMode™ AFM**— Measures topography by tapping the surface with an oscillating tip. This eliminates shear forces which can damage soft samples and reduce image resolution. TappingMode is available in air and fluids (patented). This is now the technique of choice for most AFM work. See Chapter 8.
- **Phase Imaging**— Provides image contrast caused by differences in surface adhesion and viscoelasticity. Requires an Extender™ Electronics Module (patent pending). See Chapters 8 and 13.
- **Non-contact AFM**— Measures topography by sensing Van der Waals attractive forces between the surface and the probe tip held above the surface. Provides lower resolution than either contact AFM or TappingMode.
- **Magnetic Force Microscope (MFM)**— Measures magnetic force gradient distribution above the sample surface. Performed using LiftMode to track topography (Extender Electronics Module recommended). See Chapter 13.
- **Electric Force Microscope (EFM)**— Measures electric field gradient distribution above sample surfaces. Performed using LiftMode to track topography (Extender Electronics Module recommended). See Chapter 13.
- **Surface Potential Microscopy**— Measures differences in local surface potential across the sample surface. Performed using LiftMode to track topography (Extender Electronics Module only). See Chapter 13.
- **LiftMode™**— A combined, two-pass technique that separately measures topography (using TappingMode) and another selected property (e.g., magnetic or electric force), using the topographical information to track the probe tip at a constant height above the surface (patented). See Chapter 12.
- **Force Modulation**— Measures relative elasticity/stiffness of surface features (patented). Force modulation is only one of several types of force imaging which are possible. See Chapter 11.
- **Lateral Force Microscopy (LFM)**— Measures frictional forces between the probe tip and sample surface. See Chapter 10.

- **Scanning Tunneling Microscopy (STM)**— Measures topography of surface electronic states using a tunneling current which is dependent on the separation between the probe tip and a conductive sample surface. An optional Low-Current STM Converter allows operation in the subpicoamp tunneling current region which can be useful when scanning poorly conductive samples. Tunneling spectroscopy may also be performed. See Chapter 9.
- **Electrochemical Microscopy (ECSTM and ECAFM)**— Measures the surface structure and properties of conducting materials immersed in electrolyte solutions with or without potential control. See ECSTM/ECAFM manuals.
- **Lithography**— Use of a probe tip to mechanically scribe or indent a sample surface. May be used to generate patterns, test surfaces for microhardness, etc. Performed using AFM and STM. See the *Command Reference Manual* and *Support Note 225*.

Most of these imaging techniques are discussed in this manual. If you do not find sufficient information here, refer to Digital Instruments World Wide Web site (http://www.di.com) to order the necessary support notes or obtain technical support.

1.2.3. Controller Electronics and Auxiliary Channels

The MultiMode utilizes a NanoScope IIIa Controller having a digital signal processor (DSP) with a 20 MHz peak rate for arithmetic operations. The MultiMode is equipped with four auxiliary digital-to-analog converters (DACs). Three DACs have ±10 V outputs, and one DAC has a ± 12 V and ±220 V outputs; all four channels have 16-bit resolution.

In addition, there are two ±10 V analog-to-digital converters (ADCs) having 14-bit resolution and software-selectable filters. One ADC has four-way mutliplexing.

All Digital Instruments SPMs may be attached to an optional Signal Access Module (SAM), which provides direct access (via BNC connectors) to all input and output signals between the controller and the microscope. Generally, the SAM is useful for customized use or modification of the SPM.

Chapter 2 SPM Fundamentals for the MultiMode

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2.1. Hardware

This section provides a quick tour of the MultiMode SPM and its hardware. The MultiMode SPM consists of seven major components: SPM, controller, computer, keyboard, mouse, display monitor and control monitor. Mouse movements automatically transfer the cursor between monitors, enabling the operator to seamlessly switch between control and display functions.

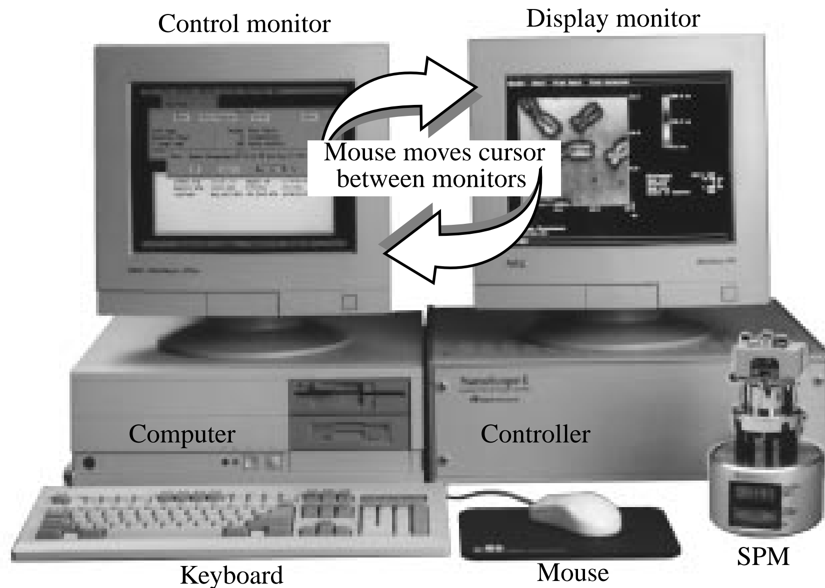


Figure 2.1. MultiMode SPM system hardware.

2.1.1. MultiMode SPM

The heart of the system is the SPM itself, shown below in figure 2.2.

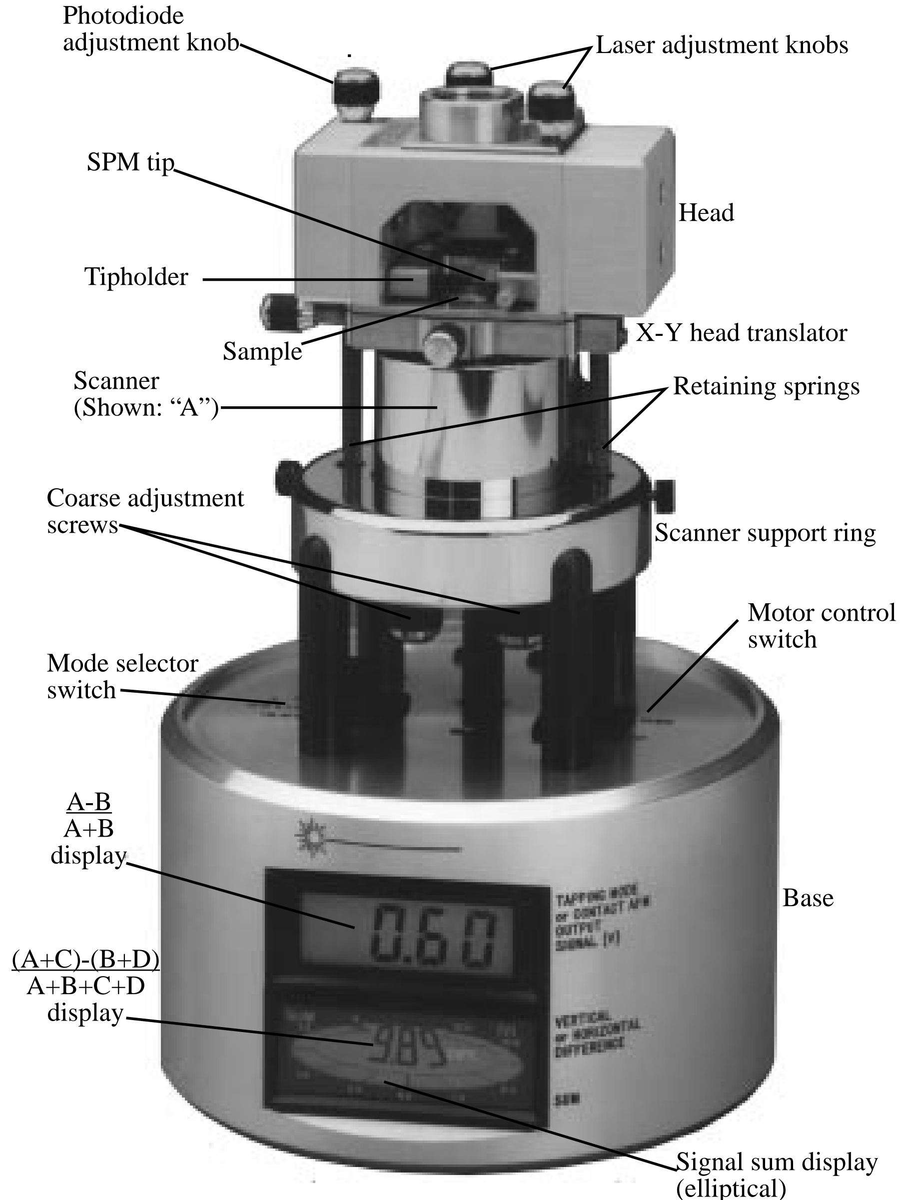


Figure 2.2. MultiMode SPM.

2.1.2. SPM Head

Figure 2.3 below shows a MM-SPM head with various adjustment knobs. The head and attached X-Y stage are kinematically mated to the scanner via three contact points. A pair of retaining springs hold down the head, allowing it to be raised and lowered using adjustment screws threaded through the scanner body. On older models, two screws are manually adjusted by the operator; the rearmost screw is motorized and under computer control. Newer, “vertical” scanners use single-screw adjustment.

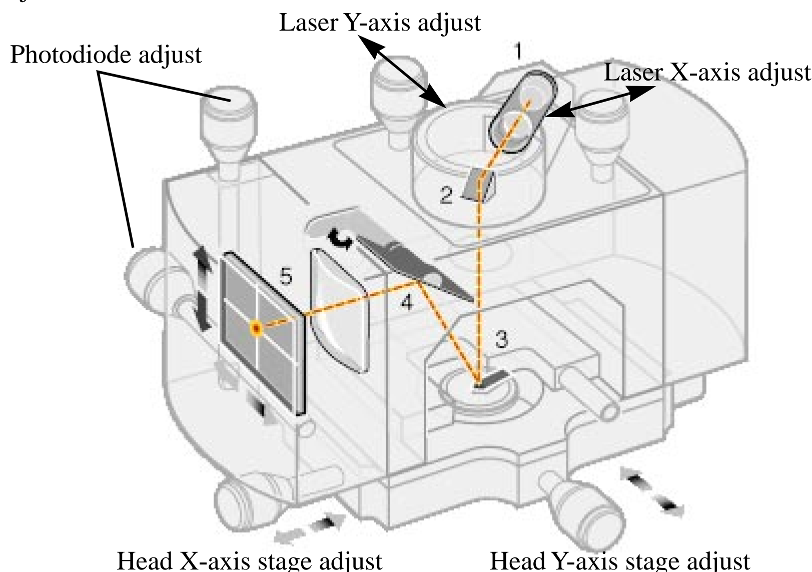


Figure 2.3. MultiMode SPM head and major components: laser (1); mirror (2); cantilever (3); tilt mirror (4); photodetector (5).

Photodiode array— The four elements of the quad photodiode (position sensitive detector) are combined 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 directly in the contact AFM. It is fed into an RMS converter (or phase module if attached) for Tapping mode operation. Similarly, the amplified differential signal between the sum of two left photodiodes and the sum of the two right photodiodes provides a measure of the torsion in the cantilever and is used in Lateral Force Microscopy (**Image data type: Friction**). Figure 2.4 shows the arrangement of the photodiode elements in the MultiMode head.

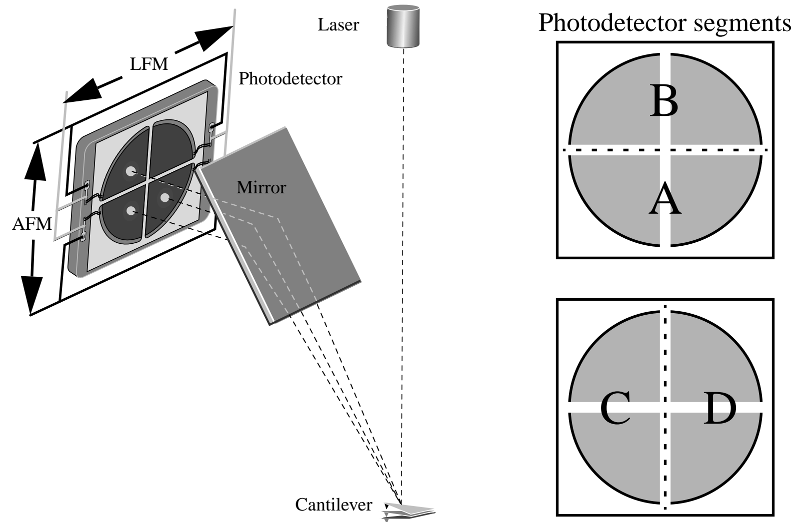


Figure 2.4. Quad photodetector arrangement. Different segments of the photodetector are used for generating AFM and LFM signals.

2.1.3. Scanners

Figure 2.5 below shows the various, interchangeable scanners. The maximum scan size and resolution of images depend upon the choice of scanner (see chart). Longer scanners, e.g., type "J," yield larger scan sizes; shorter scanners, e.g., type "A," offer smaller images down to the atomic-scale. Smaller scanners tend to be more noise-free at acoustic frequencies because of their compact size and rigidity. Larger scanner offer wider scans, while requiring extra noise dampening precautions at smaller scan sizes of high resolution.

Figure 2.5. Various scanners available with the MultiMode SPM. Left-to-right: AS-130V (vertical "J"); AS-200 ("K"); AS-130 ("J"); AS-0.5 ("A"). Not shown: AS-12 ("E") All scanners are interchangeable.

Because each scanner exhibits its own unique piezo properties, each has its own parameter file. When scanners are changed, the parameter file for the new scanner is changed along with it, ensuring maximum accuracy at any scan size. Loading new parameter files requires only a few seconds. The chart below describes the range capabilities of each MultiMode SPM scanner.

Model	Scan Size	Vertical Range
AS-0.5 ("A")	0.4µm x 0.4µm	0.4µm
AS-12 ("E")	10µm x 10µm	2.5µm
AS-12V ("E" vertical)	10µm x 10µm	2.5µm
AS-130 ("J")	125µm x 125µm	5.0µm
AS-130V ("J" vertical)	125µm x 125µm	5.0µm
AS-200	200µm x 200µm	8.0µm

Figure 2.6 depicts the electrode configuration used on one type of scanner piezo tube. Electrodes are oriented as shown when the MultiMode is viewed from the front. With the **Scan angle** parameter in the control panel set to **0.00**, the fast-scan direction is in the direction of the X-axis.

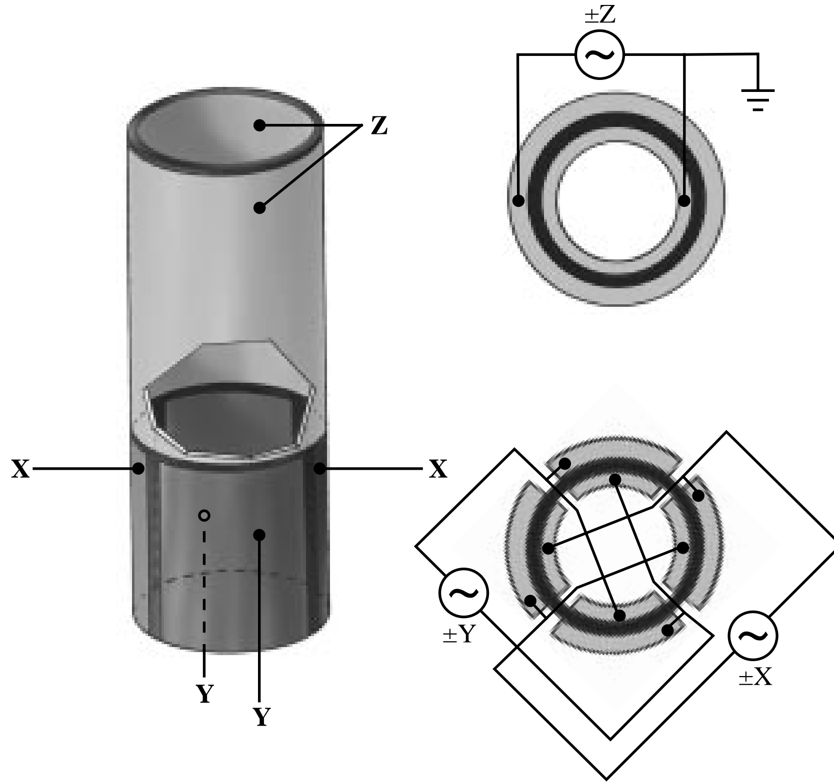


Figure 2.6. Typical scanner piezo tube and X-Y-Z electrical configurations. AC signals applied to conductive areas of the tube create piezo movement along the three major axes.

AC voltages applied to the scanner crystal's X-Y axes produce a raster-type scan motion as represented in figure 2.7. The horizontal axis presented on the display monitor is referred to as the "fast axis" (in this example, the X-axis) and scans at a **Scan rate** entered by the user. The orthogonal axis is known as the "slow axis" (in this example, the Y-axis).

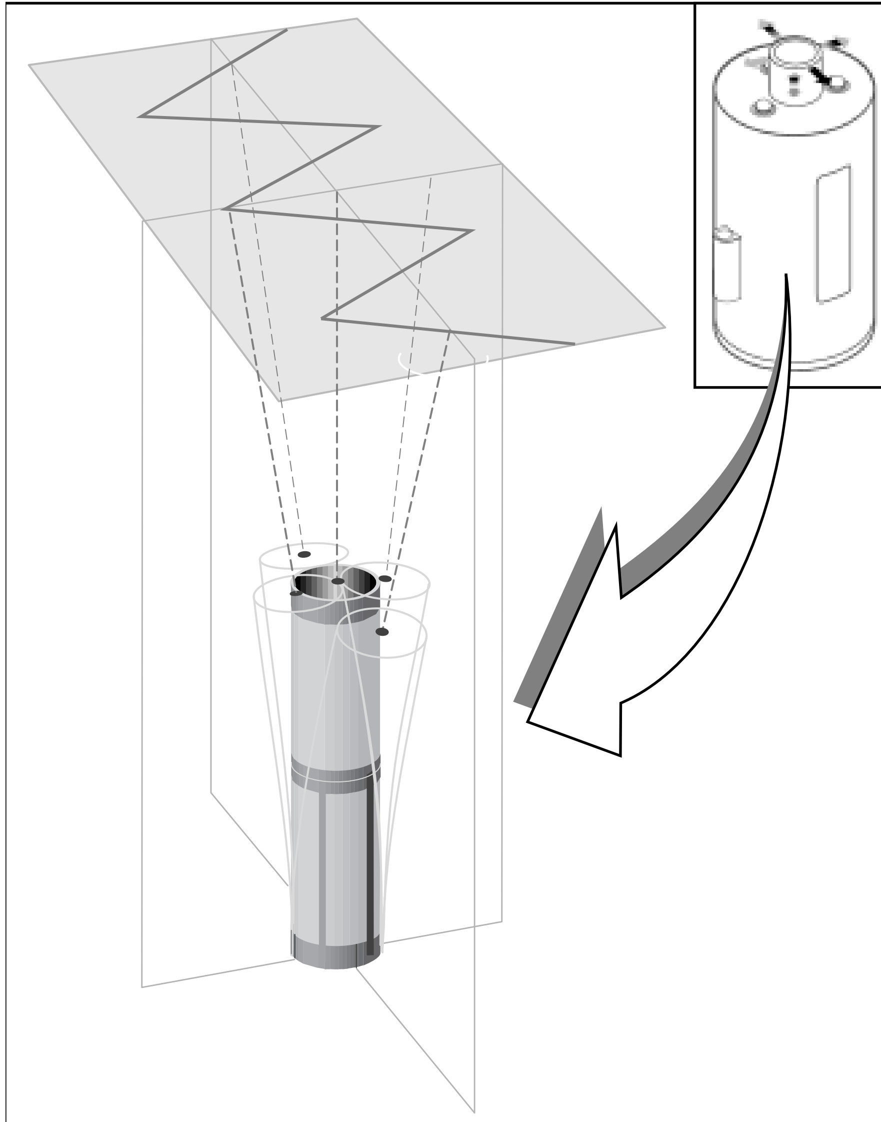
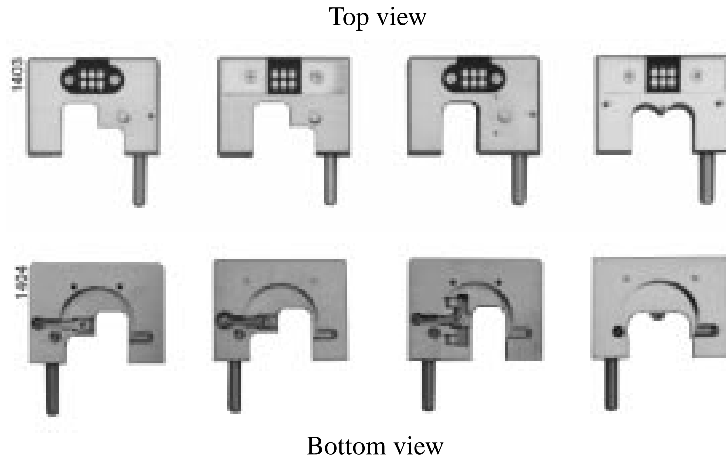


Figure 2.7. Voltages applied to the X- and Y-axes produce a raster scan pattern. Either axis may be designated as the "fast axis."

2.1.4. Tipholders

The sample and mode of SPM to be performed dictate the choice of tip and tipholder. For example, if contact AFM is to be used for imaging, a silicon nitride cantilever mounted in a standard tipholder is the usual choice. If TappingMode is to be used for imaging a biological specimen in fluid, a special fluid cell is employed. STM utilizes a special tipholder, having a tiny tube holder adapted for holding wire tips. Examples of each tipholder are shown below in figure 2.8.

Top view



Bottom view

Figure 2.8. Various tipholders utilized with the MultiMode SPM.Left-to-right: contact AFM and TappingMode; EFM; force modulation;STM.

Notes

- STM tipholder shown now superceded by model MMSTMC.

CAUTION: Some early-model AFM tipholders may short out power supplies when used with MM-SPMs. If you suspect your tipholder is from an older AFM, check with Digital Instruments or your international representative *before* using.

ATTENTION: Certains modèles de support de bras de leviers pour AFM de contact peuvent endommager les alimentations électriques quand ils sont utilisés avec un MultiMode (MM-SPMs). En cas de doute, contacter Digital Instruments ou son représentant légal pour verification.

WARNHINWEIS: Einige ältere Ausführungen des AFM-Spitzenhalters können einen Kurzschluß verursachen falls sie mit einem MultiMode-AFM verwendet werden. Bitte wenden Sie sich im Zweifelsfall an Digital Instruments bzw. an die für Sie zuständige DI-Vertretung.

2.1.5. Probes

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

Wire Probes

STM probes usually consist of wire, cut and/or etched to produce atomically sharp tips at one end. Usually these are made from tungsten or platinum-iridium alloy wires. A potential is established so that electrons flow between the tip and sample. A similar type of wire probe is used for lithography. These often consist of ordinary tungsten STM tips and/or a wire with a tiny diamond fixed to its end. Lithography tips are used for mechanically deforming sample surfaces in the form of controlled dents and scratches. Essentially, the tip serves as a scribe or punch. It may be used to test surfaces for microhardness, etch patterns or explore material characteristics.

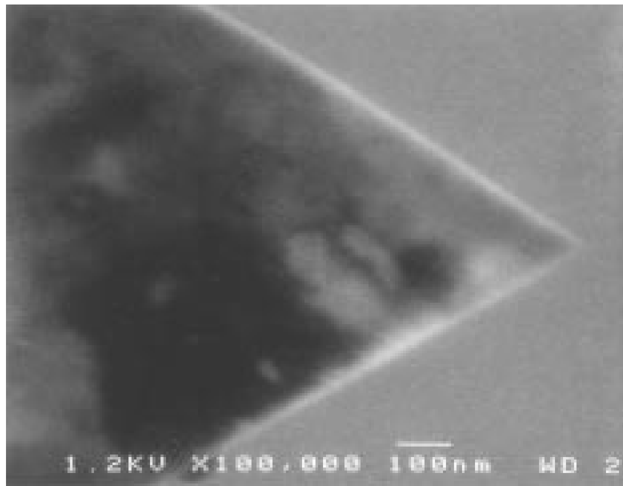


Figure 2.9. Diamond tip mounted on wire for microhardness testing and lithography work.

Cantilevered Probes

Most SPM work is done using cantilevered probes. These consist of a flexible cantilever extending from a rigid substrate, to which a tip has been attached. In contact AFM, the cantilever's flexibility acts as a nanometric spring, allowing the tip to measure surface forces. In TappingMode, the probe is oscillated up and down at its resonant frequency while its amplitude and phase are monitored.

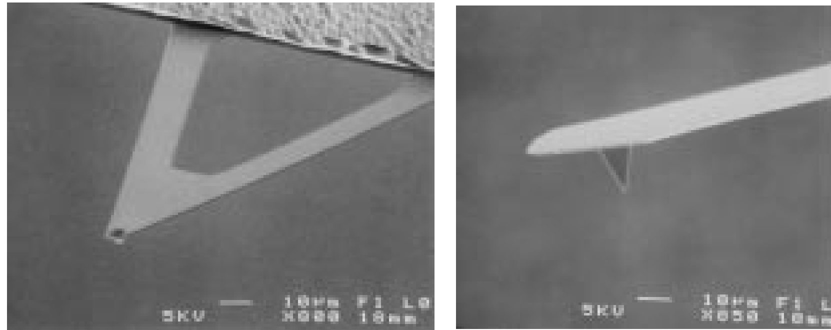


Figure 2.10. Two types of cantilevered probes: silicon nitride (left), and crystal silicon (right).

Cantilevered Probes—silicon nitride

Most contact AFM is conducted with silicon nitride tips. These tips exhibit excellent flexibility, making them easier to use and more "forgiving" than stiffer crystal silicon cantilevers. They are offered in a variety of sizes and coatings, allowing the user to match them to the sample being imaged. One characteristic of silicon nitride tips is that they are easily captured by the sample's surface tension (capillary) properties; that is, trapped within a microscopic layer of condensed atmospheric water vapor on the sample surface. This surface tension effect exerts considerable force at the probe's atomically sharp tip. Although this may prove unproblematic on harder samples, it is frequently enough to deform softer samples. Adjustment to the SPM's **Setpoint** parameter can offset much of this force; however, it may still prove troublesome on delicate samples. Silicon nitride tips may also be operated in TappingMode, although they are not optimal for this purpose.

Cantilevered Probes—TappingMode™

Digital Instruments' answer to minimizing contact AFM forces is TappingMode, a proprietary form of AFM. In this instance, a stiff crystal silicon probe is oscillated to its resonant frequency. Because the tip describes a high-frequency (e.g., 100-plus KHz), oscillating arc, it possesses sufficient energy to break free of surface tension

forces. The probe is considerably stiffer than silicon nitride, making it more brittle and less forgiving. Thus, the operator must be more cautious while setting up the tip and sample.

Cantilevered Probes—MFM

Another variation of the TappingMode tip is the MFM probe. This is basically a crystal silicon TappingMode probe having a magnetic coating on the tip. (Such tips are sold by Digital Instruments under the name NanoProbe™.) As the magnetized tip oscillates through magnetic fields on the sample surface, it modulates the cantilever's phase and frequency. These are monitored, providing a measure of magnetic field strength and providing images of magnetic domains.

Cantilevered Probes—EFM

Similar to MFM (see above), EFM is also conducted using NanoProbe tips. Tips may be electrically connected to the microscope's circuitry to obtain surface potential maps of the surface, or oscillated while monitoring phase changes due to electrostatic forces. These techniques yield images of the sample's electrical domains.

Notes

- Both MFM and EFM may be conducted using the MM-SPM alone; however, best results are obtained with an Extender Electronics Module attached. For more information regarding this attachment, contact Digital Instruments.

Specialized Probes

As the field of SPM continues its explosive growth, new probes are constantly introduced. Here are a few examples of specialized probes:

- Scanning capacitance microscopy (SCM)— Tip acts as an RF antennae to image microcapacitance phenomena. MFM tips may be used for SCM.
- Chemical doped tips— Tips doped with a chemical species of interest to the investigator. The chemically doped tip measures chemical bonding forces on sample surface, images receptor sites on biological membranes, etc.
- Thermal imaging— Tip incorporates a tiny thermocouple to image heat.
- Focused ion beam (FIB)— TappingMode tips up to 6 µm in length cut from crystaline silicon with a focused ion beam. Improved aspect ratio gives them improved angular resolution on steep side walls.

2.2. Control Mechanisms and Feedback

To produce quality images, the SPM must be capable of controlling the tip-sample interaction with great precision. This is accomplished with the use of an electronic feedback loop, which safeguards the tip and sample by keeping forces between them at a user-specified **Setpoint** level. Although signal processing varies according to the image mode used, the feedback loop performs essentially the same function.

2.2.1. A brief history of SPM control mechanisms.

The first SPMs were scanning tunneling microscopes (STMs), which use tunneling current to monitor tip-sample separation. By monitoring the flow of electrons from tip to sample (or vice versa), the tip's height above the surface can be precisely maintained. The tip-sample separation is typically maintained at several atomic diameters, or about 10 angstroms. As tip-sample separation decreases and increases due to feature height, the tunneling current increases and decreases respectively, obeying an exponential relationship.

A diagram portraying the tunneling effect during STM is shown in figure 2.11. In this example, electron activity describes a zone several angstroms wide. Because electrons flow exponentially across the gap, depending upon tip-sample separation, dramatic differences in current are shown as the tip-sample distance is varied slightly. This monitoring mechanism remains the most sensitive used in SPM, achieving greater resolution than any other method.

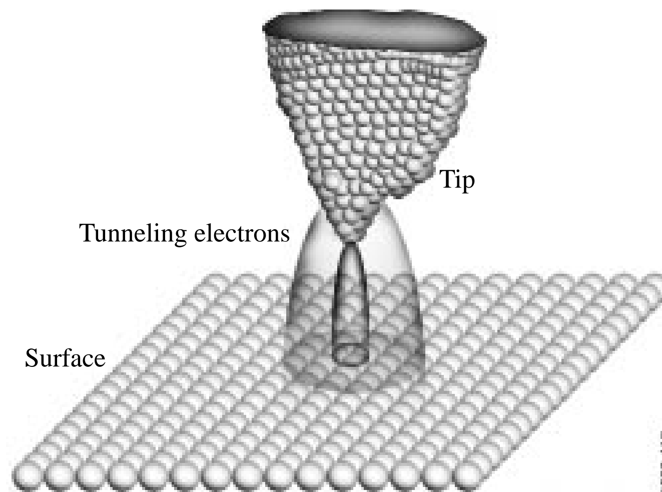


Figure 2.11. Tunneling phenomenon between tip and sample. Electron flow varies exponentially with tip-sample distance.

STM's biggest difficulty is that it requires electrically conductive samples. Soon after its introduction, a method was sought which would allow non-conducting samples to be imaged. Efforts lead to the first contact AFMs, which continued using tunneling current as the monitoring mechanism. Figure 2.12 portrays an early contact AFM scheme. In this scheme, a contact probe was scanned over the surface. As the tip encountered features, it moved up and down. Positioned directly above the surface of the contacting probe's cantilever was an STM probe. Fluctuations in current between the STM tip and contact probe were used as the feedback mechanism and to render an electronic image. The main disadvantage of this method was difficulty in aligning the contacting tip's cantilever and the STM tip directly above it.

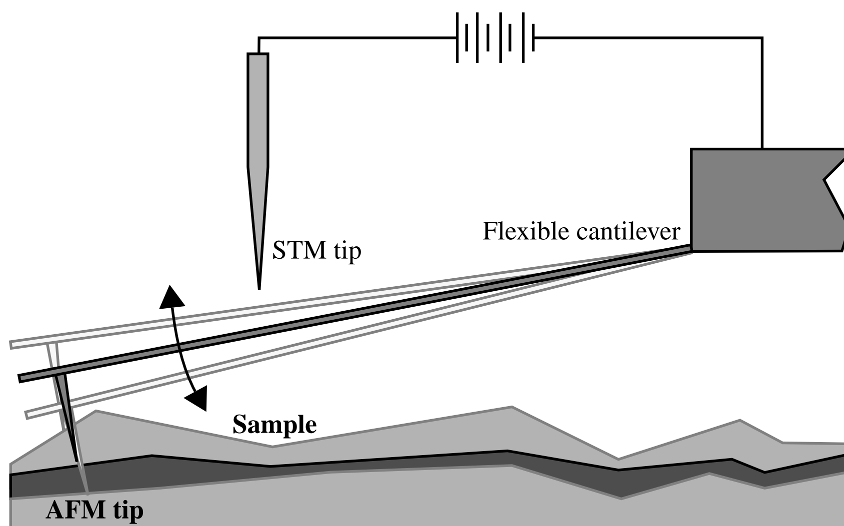


Figure 2.12. Early contact AFM which allowed imaging non-conductive samples. In this scheme, a contact AFM tip was monitored using the STM tip directly above it.

Preceding the first SPMs, some profilometers had relied upon optical methods to monitor the rise and fall of a sharp stylus over sample surfaces. This approach offered good sensitivity by reflecting a laser beam off the end of the stylus and into a photodetector to obtain an "optical lever" capable of detecting even the smallest

movements (figure 2.13). This approach was then applied to SPMs. A related method utilized interference to detect shifts in interference fringes.

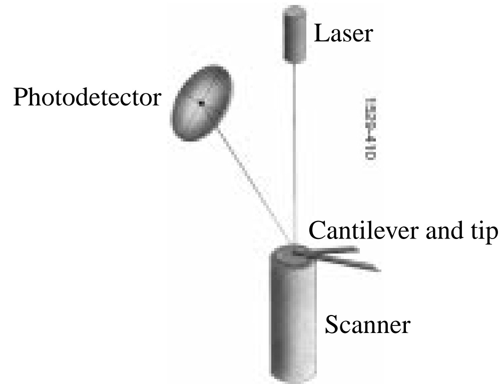


Figure 2.13. Optical lever for monitoring tip movement.

Laser beam movement is monitored over two axes: vertically and horizontally. As the tip traces various surface features, its upward and downward movement shifts the beam between upper and lower photodiode components, creating voltage differences which are electronically rendered into height information. Lateral movements of the beam are also monitored, corresponding to frictional phenomena on the surface.

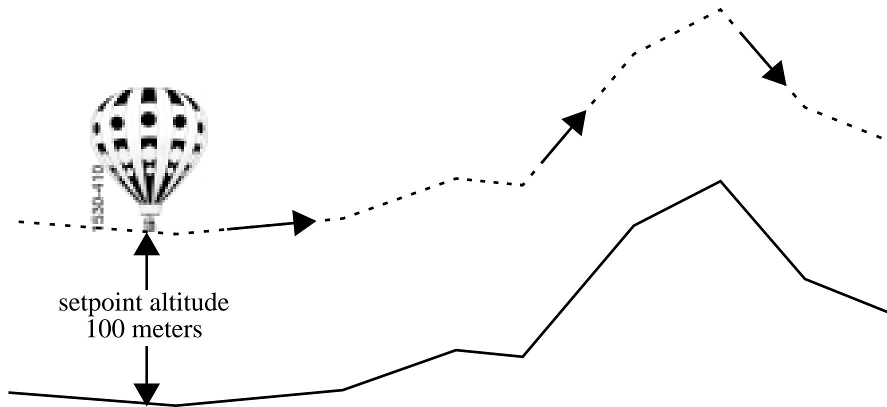
2.3. Feedback Gains

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

2.3.1. Proportional and Integral Gain—An Analogy

To better understand gains and how they control SPM probes, consider the analogy of a hot air balloon carrying three balloonists. Each rider controls a separate valve on the balloon's gas burner. The valves are mounted in parallel, such that if any one valve is open, gas flows to the burners, causing the balloon to rise. Similarly, each balloonist may turn their burner off to reduce altitude. Mounted beneath the balloon's gondola is a camera, which automatically takes a photograph of the ground below. The balloon's objective is to obtain detailed photographs of the surface. To

obtain the highest resolution images, the balloon must track the surface as closely as possible without crashing into it. This poses a dilemma to the balloonists: how to tightly control the balloon's position relative to the ground.



Because the balloon will drift slightly up and down due to the effects of wind and temperature, the balloonists must establish some minimum altitude as a safety zone. Let us call this the "setpoint" altitude, and let us assume that it is set at an altitude of 100 meters.[1]

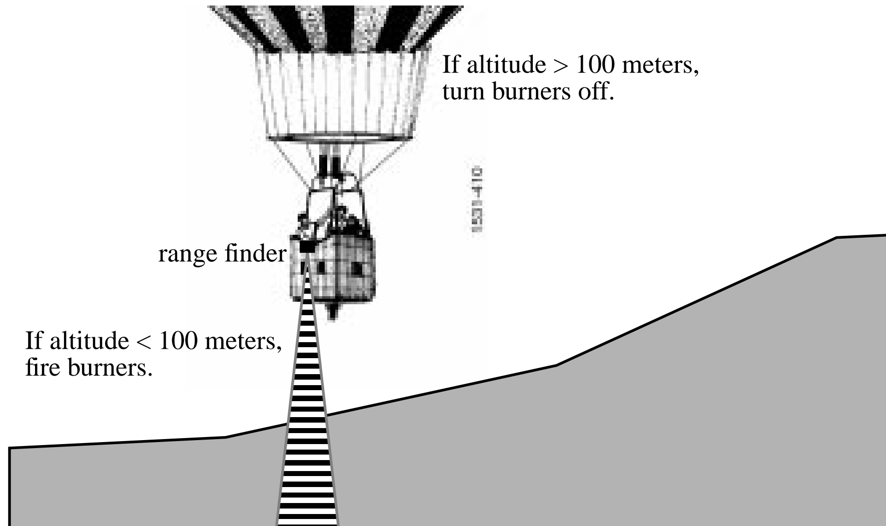
When the terrain is flat, the problem is simplified. The balloonists need only ensure a constant supply of gas is supplied to the balloon's burners to keep the balloon aloft. As the terrain becomes hilly, the task becomes more complex. If the terrain rises, the balloonists must respond by firing the burners to lift the balloon. As the balloon clears the hill and terrain drops away, the balloonists must turn the burners off to reduce height and continue tracking the terrain. The type and intensity of the balloonists' responses to terrain can be modeled in terms of three types of feedback: *proportional*, *integral* and *LookAhead*.

2.3.2. Proportional Gain

Proportional gain means that something is done proportionally in response to something else. In the case of our first balloonist, Peter, this means producing hot air in proportion to the balloon's altitude above the terrain: where the terrain rises

1. For the sake of simplicity, "setpoint" in this analogy applies to the balloon's altitude; however, setpoint in SPM is applied to tip-sample forces, *not* the tip's height above the surface.

sharply, Peter uses large amounts of gas to lift the balloon; where the terrain is relatively flat, Peter supplies a small, steady amount of gas to maintain the setpoint altitude above the surface.



A simple feedback loop emerges in this analogy: let us say Peter uses a range finder every 30 seconds to determine the distance between the balloon and ground. If the balloon is below its setpoint altitude, he fires the burners. If the balloon is above its setpoint altitude, he turns off the burners to lower the balloon. The higher the proportional gain, the more Peter reacts to changes in altitude. For example, at a proportional gain of **1**, if the balloon is 25 meters too low, he opens his valve at 10 liters per second; if the balloon is 50 meters too low, he opens his valve at 20 liters per second. The proportional gain value serves as a multiplier such that at a proportional gain of **2**, the gas flow rates are doubled from a proportional gain of **1**, and so on. Although this sort of feedback gain works well for simple, linear models, it does not function as well for nonlinear models. There remains always some residual error which causes the system to approach, but not quite reach, the target state.

Assuming that the balloonists wants to get as close as possible without crashing, the response will depend upon, among other things, the balloon's speed over the terrain. When the balloon is being carried swiftly, it is necessary to apply feedback earlier to compensate. (That is, more gas must be used earlier.) On the other hand, if there is little or no wind, the balloon may achieve a closer tracking of the terrain. There may also be sufficient knowledge of the terrain to anticipate its rises and falls. In order to compensate for these effects, *integral* and *LookAhead* gain feedbacks may also be employed. These are discussed next.

2.3.3. Integral Gain

Integral gain is used to correct the cumulative error between a system and its target state. In the case of the balloon, it is not enough to use only proportional gain. As we have seen, the balloon will maintain a constant error around the setpoint altitude if it relies on proportional gain alone. It is also necessary to consider whether the total error between the balloon's actual altitude and its setpoint altitude is increasing or decreasing over some interval of time. To correct for cumulative error, our second balloonist, Irene, utilizes *integral gain*.

Let us assume that Peter announces the balloon's altitude every 30 seconds from his range finder. Irene uses a stopwatch and clipboard to record the amount of error at each measuring interval, averaging the error over a preceding interval of time (e.g., 3 minutes). Irene fires the burners based upon her observations: if she notices that the running average error puts the balloon below the setpoint altitude, she fires the balloon's burners, if she notices that the average error puts the balloon above the setpoint, she turns the burners off. The effect of integral gain feedback is to reduce total error by addressing error over a longer period of time. This tends to smooth out the short-term, fluctuating effects of proportional gain while narrowing the error closer to the setpoint value. Unfortunately, if the integral gain is set too high, there is a tendency to overshoot the setpoint. Therefore, integral gain is highly sensitive and must be used carefully.

2.3.4. LookAhead™ Gain

Finally, the third balloonist, Larry, employs yet another type of gain to ensure optimal tracking over the terrain: *LookAhead* gain. For our example, Larry uses a map to anticipate the rise and fall of the terrain. When his map indicates a mountain, he opens his valve to fire the burners and lift the balloon. When a valley is indicated on the map, he turns his burner off to lower the balloon. The effect of LookAhead gain is to keep the balloon within the proper altitude zone so that proportional and integral gains will perform better by maintaining the balloon closer to its proper setpoint altitude. When the terrain is comprised of regular rises and falls, the LookAhead balloonist is at his best, easily anticipating rises and falls. In these instances, LookAhead gain can be maximized. Conversely, when the terrain is rough and broken, the LookAhead balloonist must struggle with the balloon's sluggish response to anticipate every irregular rise and fall, and may actually make control of the balloon more difficult. In these instances, LookAhead gain should be minimized or turned off.

2.3.5. Completing the Analogy—Feedback Gains in SPM

Feedback gains used to control an SPM's probe tip are not far removed from those controlling a hot air balloon. In the case of a probe tip, the objective is quite similar: the operator assigns a setpoint value corresponding to a certain amount of tip-sample force, then adjusts gains to track the surface as closely as possible while maintaining the setpoint. Instead of gas-fired burners, however, the Z-axis piezo crystal uses voltage to retract and lower the probe. In addition, such parameters as **Scan rate** must be figured in. Just as a balloonist would find it difficult to closely track rough terrain in a fast-moving balloon, the microscopist must frequently adjust **Scan rate** and **Setpoint** to track samples successfully.

2.3.6. Setpoint

In our ballooning example, "setpoint" referred to the target altitude to be maintained. In scanning probe microscopy, "setpoint" refers to how much tip-sample force is maintained. There are two ways of defining setpoint, depending upon whether one is referring to contact AFM or TappingMode. In contact AFM, setpoint is determined by the amount of cantilever flexion—as the setpoint increases, the cantilever flexes more and tip-sample forces increase. In TappingMode, setpoint is determined by the RMS amplitude of the oscillating tip—as setpoint decreases, RMS amplitude decreases, but tip-sample forces increase[1].

2.3.7. The SPM Electronic Feedback Loop

Just as the balloonists in the example above want to get close to the ground without crashing into it, the SPM is designed to tightly control the tip's position relative to the sample surface. In the case of contact AFM, this usually means applying a very light force to the tip—just enough to trace surface features, but not so much force that the tip is broken off or the surface damaged. In the case of TappingMode, it means holding the tapping force (measured in terms of the oscillating probe's amplitude) to the setpoint level.

In the earliest SPMs (which were scanning tunneling microscopes), the tip was scanned at a constant height above a very flat sample surface (e.g., cleaved graphite) while the tip's current was monitored. Because tunneling current flows exponentially as a function of tip-sample distance, the image was rendered from mapped current values at X-Y coordinates. This gave a height rendering of features based upon current flow. As long as the sample was atomically flat, the tip could be scanned safely above it and an image produced. Unfortunately, this arrangement did not work well for rougher surfaces: the tip would crash into raised features, damaging itself and/or the surface.

The next generation of SPMs added a Z-axis piezo crystal to the arrangement and used a feedback loop to profile the sample's features. Now, instead of using tip-sample current flow to produce an image directly, the current was used instead as the feedback signal to activate the Z-axis piezo. This allowed the tip to be lifted and lowered, keeping tip height constant over surface features and accommodating rougher samples. But how were images produced? Instead of using the tip-sample current directly to render an image, the feedback loop was monitored indirectly. This process allowed the feedback loop to protect the tip and sample while giving quality images. In addition, the feedback circuit could be monitored at various points to access new types of information about the tip-sample interaction.

As SPM evolved beyond its scanning tunneling roots, the feedback circuit was modified to accommodate new types of imaging. The first major change arrived with contact AFM, which permitted non-conducting surfaces to be imaged. Tunneling current was now used indirectly to monitor a cantilevered tip as it profiled sam-

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1. At first glance, this may seem counterintuitive. However, recall that an oscillating tip in TappingMode attains its fullest amplitude when it is in free air and not interacting with a sample. As the oscillating tip is brought against the sample, its RMS amplitude decreases due to damping effects. The harder the tip is pressed into the sample, the more RMS oscillation is reduced. Thus, requesting a **Setpoint** of **0.00** in TappingMode commands the system to press the tip against the sample so hard that the cantilever cannot oscillate at all. In TappingMode, reducing setpoint increases tip-sample forces...the opposite of contact AFM.

ples. Although this method allowed imaging of non-conducting samples, it was unreliable due to adjustment difficulties with the STM probe and cantilever flexion.

The next great leap in SPM design which presaged the present state of the art was the introduction of light beam deflection. In one design, a laser is employed to configure a “light lever” similar to those used with surface profilometers in industry. As the tip encounters surface features it flexes, causing the incident laser beam to move across a photodiode detector. Another design relies upon interferometric measurements; however, this design has been superceded by the light lever’s simpler, more reliable design.

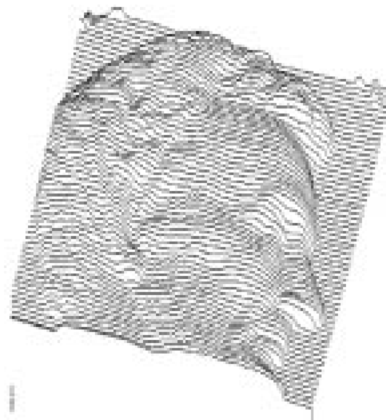
As new modes of SPM have been added to the field, the analysis of feedback signals has evolved to keep up with changes. The next section discusses how signals are actually processed inside the NanoScope to render images.

2.3.8. More about Feedback and Images

Digital Instruments’ unique digital signal feedback architecture is described in numerous patents filed with the US Patent Office. In summary, the basic feedback processes may be broken down as follows:

LookAhead gain

In the example above, having a record of previous flights over terrain enabled three balloonists to better anticipate the rises and falls of the terrain below them. Similarly, the feedback controller relies upon data from the previous (immediately adjacent) scan line to anticipate local features. It is easier to image samples which contain regular, periodic features (e.g., gratings) since scan lines change relatively little from scan-to-scan. Consider, for example, scan lines tracing the surface of a penny.



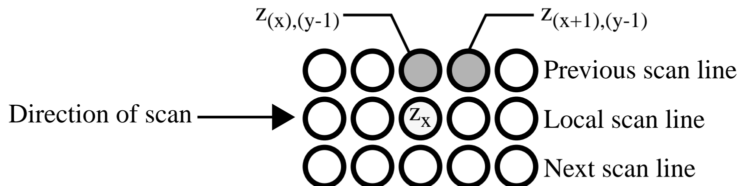
Although this scan is much larger than normally found in SPM, it illustrates how an adjacent, lagging scan line can be used to determine local scan lines on regular surfaces. In most places (e.g., the forehead), each scan line changes little from the line next to it. In some local areas (such as under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

The entire purpose of **LookAhead gain** is to take full advantage of regular features by using every line to anticipate the next one. In NanoScope software, the **LookAhead gain** value may be adjusted between a range of **0** (off) to **16** (maximum). As values are adjusted upward from **0**, the **LookAhead gain** is weighted to apply more data from the adjacent (lagging) line. Although **LookAhead gain** is relatively useless for random surfaces, it is a tremendous help on regular surfaces.

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

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

That is, the **LookAhead**-weighted integral gain, G_{int}^{LA}, is calculated by subtracting the adjacent pixel's Z-axis value from the one immediately next to it, then multiplying this difference by the **LookAhead gain** value[1] and summing the product with the current data value. A diagram of the affected pixels appears as shown here:



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

Integral gain *and average error*

1. Although the **LookAhead gain** value can be set by the operator to values between **0** and **16**, these are *not* the values plugged into the equation. This is a digital signal feedback process and the actual value multiplied varies between 0 and 2^{24}. A similar rule holds also for both **Integral** and **Proportional gain**.

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

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

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

Proportional gain

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

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

As suggested in the equation, by the time proportional gain is figured in, the bulk of error correction has already been completed. This tends to make **Proportional gain** a less "touchy" control when compared to **Integral** and **LookAhead gain**. Nevertheless, the system can be driven into oscillation whenever gains are excessive, including **Proportional gain**.

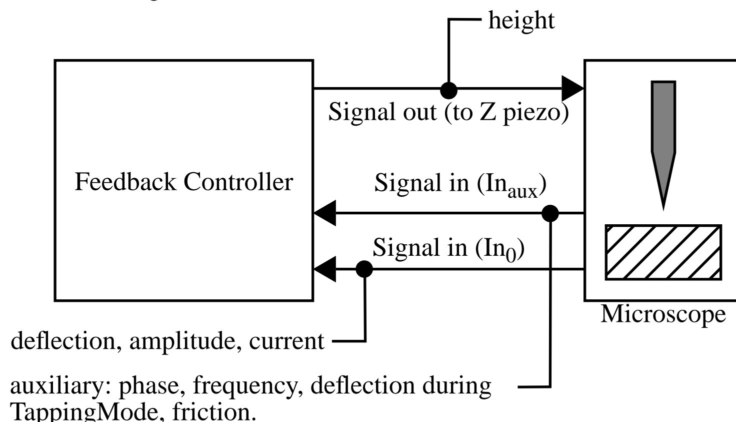
REMINDER: Gain values entered on the **Real Time / Feedback Controls** panel do not directly translate to any real quantity, but are merely self-referencing; e.g., a **Proportional gain** value of **2.0** is not the same as **Integral** or **LookAhead gain** values of **2.0**.

2.3.9. What *Data Type* of image?

SPM technology at Digital Instruments has rapidly grown beyond its scanning tunneling roots to encompass numerous types of microscopy. This includes: ECSTM, contact AFM, ECAFM, TappingMode in air, TappingMode in fluids, amplitude and phase magnetic force microscopy (MFM), surface potential and field gradient electric force microscopy (EFM), lateral force microscopy (LFM), force modulation imaging, scanning capacitance microscopy (SCM), thermal imaging, and force volume imaging. In addition, there are numerous variations and combinations of the above; new types of SPM are added continually as the field expands. Each of these variations reveals something unique by using Digital Instruments' feedback system to process and extract signals in slightly different ways.

The NanoScope system allows up to three simultaneous image channels, plus auxiliary channels. Each of the image **Channel** control panels (**Channel 1**, **2**, and **3**) contains a **Data type** parameter specifying the type of image to be shown on that channel. The **Data type**, in turn, is determined by the currently selected microscope (**Real Time / Microscope / Select**) and **AFM mode** shown on the **Other Controls** panel. For example, although **Height** data can be displayed for most types of imagery, only TappingMode displays **Amplitude** data. Similarly, only contact AFM displays **Friction** data. Whenever the **AFM mode** and **Data type** parameters are changed, some new portion of the feedback signal is utilized and/or processed differently. Some users tap the NanoScope's auxiliary channels to generate new type of images from the feedback system.

To better understand what is being viewed when selecting different **Data type**s, consider the diagram below:



As this diagram shows, different portions of the feedback loop are being accessed, depending upon what is selected at the **AFM mode** and **Data type** parameters. During STM, the signal coming into the feedback controller (at line In_0) is **Current**. In

contact AFM, the same line now conveys **Deflection** voltage. In TappingMode, the same line conveys **Amplitude** data. In addition, the auxiliary channels utilize line In_A through In_D.

Notes

- Although there are four separate auxillary lines (In_A through In_D), the controller can only access one at a time, plus the In_0 line.

The controller is designed to handle two input signals simultaneously (In_0 and one auxiliary line). Because it is extracted from a different (output) portion of the feedback loop, a third channel can be used to simultaneously extract **Height** data without affecting the input signals. This allows a maximum of three **Data types** for any one sample. For example, a sample being imaged with contact AFM can show **Height**, **Deflection** and **Friction** all at the same time.

2.4. Control Parameters and Feedback

The feedback scheme described in Section 2.3 above provides a general platform for imaging sample surfaces; however, vast differences in samples require additional controls to obtain optimal images. The NanoScope imaging system utilizes over 1000 parameters. The vast majority of these are never seen by users, but those that are common should be reviewed. This section provides a brief description of some of the more commonly used parameters.

2.4.1. Reexamining the Control Loop

Recall that the NanoScope control system performs two main functions: 1) it generates drive voltages to control the X-Y scans of the piezoelectric transducer; 2) it maintains an incoming analog signal from the microscope detection circuitry at a constant value. This is done by way of a closed-loop feedback control system. The computer is programmed to read voltage from a comparator circuit through an analog-to-digital (A/D) converter. It is programmed to keep the two inputs of the comparator circuit equal (0 volts). An output voltage generated by the computer continuously moves the piezoelectric transducer in the Z direction to correct for differences read into the A/D converter. This closed-loop feedback control is the heart of the imaging portion of the control station.

SPMs are capable of displaying either the input (difference), or output (correction) applied to the Z-axis piezo. The Nanoscope III control system can also display information from a second A/D converter. This second input can be friction between the AFM tip and the sample, data from an electrochemical cell, or one of four user-selectable inputs.

2.4.2. General Description of Main Menu Items

This section is a general overview of various Real Time menu items. Refer to the *Command Reference Manual* for more information about these settings.

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

Aspect ratio— Dermines whether the scan is to be square (**Aspect ration 1:1**), or non-square (**Aspect ratio 2:1, 4:1, 8:1, 16:1** or **32:1**). The **Aspect ratio** parameter is only displayed if the **Non-square scan** option is selected in the **Scan Controls** panel (upper-left panel button).

X offset; Y offset— These controls allow adjustment of the lateral scanned area and the center of the scanned area.

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

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

Number of samples— Sets the number of pixels displayed per line and the number of lines scanned per frame.

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

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

Integral gain and **Proportional gain**— Controls the response time of the feedback loop. The feedback loop tries to keep the output of the SPM equal to the setpoint reference chosen. It does this by moving the piezo in Z to keep the SPM's output on track with the setpoint reference. Piezoelectric transducers have a characteristic response time to the feedback voltage applied. The gains are simply values that magnify the difference read at the A/D convertor. This causes the computer to think that the SPM output is further away from the setpoint reference than it really is. The computer essentially overcompensates for this by sending a larger voltage to the Z piezo than is truly needed. This causes the piezo scanner to move faster in Z. This is done to compensate for the mechanical hysteresis of the piezo element. The effect is smoothed out due to the fact that the piezo is adjusted up to four times the rate of the display rate.

2.4.3. User Example

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

- Display both the input and the output of the feedback loop. This means setting the display to show both **Height** data and the appropriate microscope signal (STM = **Current**, contact AFM = **Deflection**; TappingMode AFM = **Amplitude**).
- Engage the microscope and reduce the gains until they are close to zero. The input display data will become larger in Z, and the height data will blur or become smeared. Raise the gains using the arrow keys until the input voltage is minimized.
- Try increasing and decreasing the scan rate parameter. This will increase or decrease the traveling velocity of the tip. Note that it will be necessary to increase the gain settings at faster scan speeds and decrease the gains at slower scan speeds.

2.4.4. Review of General Operating Concepts

The AFM system is comprised of two main components: 1) the scanner; 2) the AFM detection system. The scanner houses the piezoelectric transducer. The piezo element physically moves the sample in the X, Y and Z direction. The detection system consists of a laser which generates a spot of light that is reflected off of a microfabricated cantilever onto a mirror and finally into a photodetector (see figure 2.14). The position of the spot is determined by circuitry which generates a voltage from the difference between the photodiode segments (A - B). The circuit outputs a voltage ranging from +10V to -10V depending on the position of the spot on the two photodiodes.

The AFM system maintains the tip at the end of the cantilever in contact with the sample surface. The sample is scanned under the tip in X and Y. Features on the sample surface deflect the cantilever, which in turn change the position of the laser spot on the photodiodes. This position change is read by the feedback loop. The feedback loop moves the sample in Z to restore the spot to its original position. Refer to figure 2.14 (opposite page).

Figure 2.14.A. A flat portion of the sample surface is scanned beneath the tip left-to-right, maintaining the laser beam at the center of the photodiode array.

Figure 2.14.B. As the tip encounters a raised feature, the cantilever is pushed up, deflecting the laser beam upward onto the "A" portion of the array. With the "A" photodiode receiving an increased portion of the laser light, its voltage increases while portion "B's" decreases (A > B).

Figure 2.14.C. The Vertical Deflection (A-B) voltage differential is sensed by the feedback electronics, causing a dropped voltage to the Z piezo crystal—the piezo retracts. As the Z piezo retracts, the cantilever recenters the laser beam onto the photodiode array (A = B).

Figure 2.14.D. As the tip encounters a decline in the sample topology, the tip drops. This directs more of the beam onto the "B" portion of the photodiode array. With the "B" photodiode receiving an increased portion of the laser light, its voltage increases while portion "A's" decreases (A < B).

Figure 2.14.E. Again, the Vertical Deflection (A-B) voltage differential is sensed by the feedback electronics, increasing voltage to the Z piezo crystal—the piezo extends. As the Z piezo extends, the tip is pushed down until the laser beam recenters on the photodiode array (A = B).

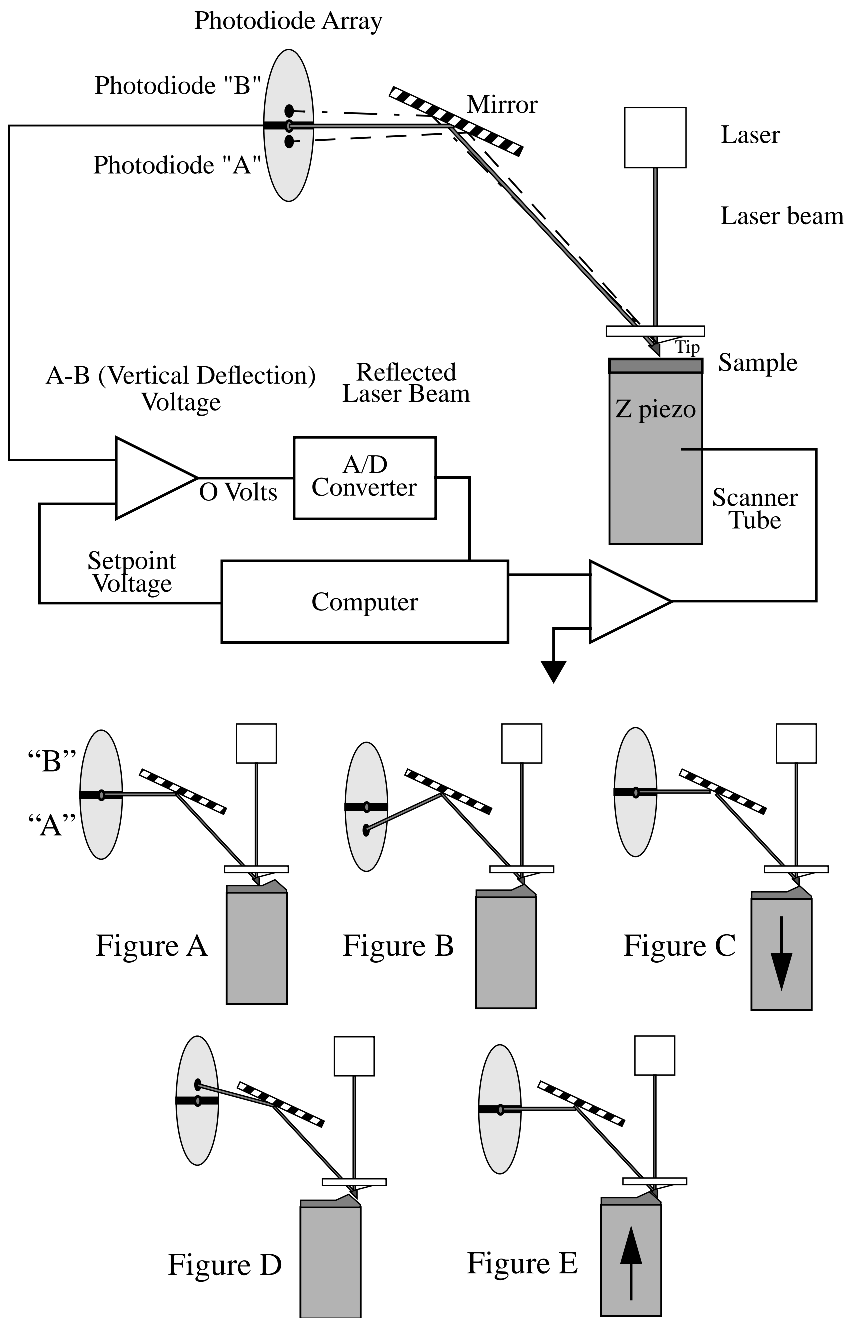


Figure 2.14. Contact AFM concepts (Figures A—E exaggerated.)

The AFM always first engages in the repulsive region of its operating range. In other words, the cantilever needs to exert a positive pressure on the sample surface. The AFM block diagram shows the relationship between the cantilever movement and the laser spot on the photodiode array. The diagram shows that the spot moves up (more on "A") when the cantilever is pushed up. The initial setup is to have the **Vertical Deflection** (A-B) voltage about 2-3 volts *more negative* than the **Setpoint** voltage. Digital Instruments recommends starting with the **Setpoint** voltage set to 0 volts and the **Vertical Deflection** (A-B) set to -2 volts. The reason for this is that 0 volts is the middle of the control range. The indication of a good engagement is a distinct jump of about 1V from the **Vertical Deflection** (A-B) voltage to the **Setpoint** voltage.

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

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

Integral gain— This number is multiplied times an accumulated average of A/D readings. This is the low frequency feedback control.

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

2.5. Review of TappingMode AFM

2.5.1. General Operating Concepts

One advantage of TappingMode AFM is an absence of frictional forces which exert torque on the cantilever. Unlike traditional contact AFM, the feedback loop keeps a vibrating cantilever at a constant amplitude, rather than keeping a cantilever at a constant deflection. The tip on the cantilever is modulated through mechanical excitation at its resonance. A laser beam is reflected off of a microfabricated cantilever, onto a mirror, then reflected onto a photodiode array. The laser spot oscillates vertically across the array as a result of the vibrating cantilever. The signal from the photodiodes is rectified, then lowpass filtered into a DC voltage (**RMS Ampl.**). The magnitude of the RMS amplitude is proportional to the amount of cantilever motion.

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

The engagement of TappingMode AFM requires that the setpoint voltage be smaller than the RMS voltage, which is set automatically by the software. The tip is lowered until the RMS reaches the setpoint.

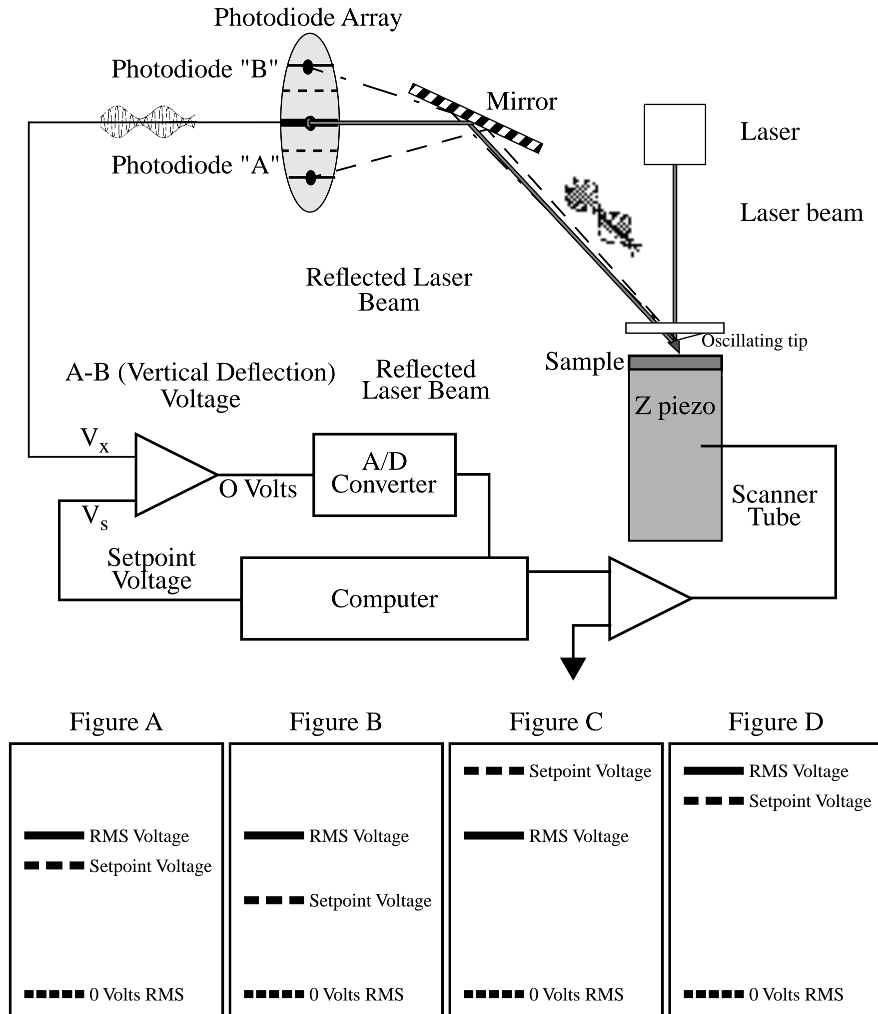


Figure 2.15. TappingMode AFM concepts. (See sect. 2.5.2 for explanation.)

Figure 2.15 shows the relationship between the RMS and the setpoint voltage during the engage cycle. The initial setpoint voltage is determined by the computer rather than the user. The computer sets the setpoint equal to 95 percent of the RMS amplitude. The tip is then lowered until the RMS matches the setpoint. The computer then tests for true engagement as follows: 1) the motor halts the tip's descent; 2) the setpoint is lowered slightly; 3) the feedback control monitors movement of the Z piezo. Depending upon the tip's relationship to the sample, one of the two following conditions will result:

1. A small Z piezo movement. This indicates that the cantilever is truely engaged with the sample surface.

2. A large Z piezo movement. This indicates that the cantilever is being damped by air trapped between the cantilever and sample surface (not in contact with the actual, solid surface)—this is a false engagement condition. The setpoint is readjusted and the engage cycle repeated until the computer reads a small change in Z when the setpoint voltage is lowered further. One symptom that this condition is occuring is when the “tip travel µm” display stops momentarily, then starts again.

2.5.2. Optimizing the TappingMode AFM signal after engagement.

The figures on the bottom of figure 2.15 show the relationship between the RMS and the setpoint voltages.

There are some basic rules to remember:

1. The setpoint voltage is always lower than the RMS voltage (figure A).

2. The difference between the RMS voltage when the tip is off the surface, and the setpoint voltage dictates the amount of damping or “tapping force.” The larger the difference, the greater the tapping force (figs. A, B).

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

The initial setup for TappingMode AFM is to:

1. Tune the cantilever at its resonance. Quit out of the **View / Cantilever Tune** software routine after the resonance is found.

2. Set the desired RMS voltage. This is usually about 1 to 4V.

3. Engage the microscope.

2.6. Terms and Abbreviations

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

AFM— Atomic force microscopy; atomic force microscope.

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

bias— Electrical potential applied to a tip or sample which causes electron flow to ensue from one to the other. (STM and EFM only.)

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

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

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

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

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

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

ECAFM— Electrochemical atomic force microscopy; electrochemical atomic force microscope.

EFM— Electric force microscope; electric force microscopy. Method of SPM used to image electric forces on samples.

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

error— Difference between actual tip-sample force measured at the detector and the setpoint force.

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

feedback— Process of self-correction between probe's actual, real time height-surface force and its intended height-surface force based upon the probe's signal.

fluid cell— Accessory used for imaging materials in fluid, consisting of a specialized tipholder and o-ring.

force modulation— SPM mode used to image visco-elastic properties of materials.

integral gain— Amount of correction applied in response to the average error between setpoint force and actual force measured by the detector.

LFM— Lateral force microscopy; lateral force microscope. Frictional measurements of surfaces based upon a tip's lateral and torsional response.

LiftMode™— Two-part, proprietary method of imaging surfaces consisting of a surface scan to obtain height data, followed by a second scan to extract other information about the surface (such as magnetic force or elasticity) while the tip profiles the previous Z path at a constant height. The two images are subtracting from each other to yield an image uninfluenced by topography.

LookAhead gain— Amount of correction applied in response to the error signal between setpoint force and actual force measured by the detector, based upon recorded information from the adjacent scan line.

MFM— Magnetic force microscope; magnetic force microscopy.

NanoScope™— Trademark name applied to Digital Instruments' SPM products.

probe— Integrated mechanical device used to image surfaces, including a substrate, cantilever and tip.

proportional gain— Correction applied in response to the error signal between setpoint force and actual force measured by the detector, in direct proportion to the error.

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

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

STM— Scanning tunneling microscopy; scanning tunneling microscope.

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

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

spring constant— Amount of force required to bend a cantilever some given amount.

TappingMode™— Proprietary mode of SPM exclusive to Digital Instruments which utilizes an oscillating probe to obtain nanometric images. Advantages include negligible surface impacts, high resolution and sensing of magnetic, electric and chemical forces.

tipholder— Removable appliance for mounting SPM probes. On MultiMode SPMs, the tipholder is installed within the head of the microscope. On Dimension Series SPMs, the tipholder plugs onto the end of the scanner tube.

Chapter 3 *Setup & Installation*

3.1. Installing the MultiMode SPM

Set up the computer, main controller and two monitors as described in this chapter. If MultiMode software is not already installed on the system, install the software on the Intel PC using the supplied 3.5" floppy disks. The MultiMode scanning probe microscope control program should be installed in a special directory (C:\SPM) which must be entered to run Z.EXE.

3.2. Component List

If this is a first-time installation, verify that you have received all necessary components. The MultiMode SPM system includes the following:

- MultiMode SPM base
- NanoScope™ SPM controller
- *MultiMode SPM Instruction Manual* (this document)
- *Command Reference Manual*
- *MultiMode parameter files on 3 1/2" diskette*[1]

1. NOTE: The calibrated scanner's parameter files are located in the hard disk's EQUIP directory.

Component List (cont.)

- NanoScope controller-to-MultiMode SPM cable, 37-to-25 pin, D-type.
- Fiber optic illuminator, Fiber-Lite, Model 190
- Vibration isolation pad
- Scanner calibration reference: XYZ, 10µ x 10µ, 180 nm vertical (all scanners); Mica sample ("A" scanners); 1 µ XY grating ("E" scanners)
- Package of 10 contact mode cantilevers - silicon nitride type
- Package of 10 TappingMode cantilevers - single crystal silicon
- MultiMode SPM head

Figure 3.1. MultiMode SPM head.

- MultiMode SPM scanner with retaining springs
- Tweezer kit containing: SM3-SA; SA-2A; 88 MM; 7-SA.
- 45X magnifier with mounting stand
- Sample disks (pucks): 20 large, 20 small.
- Sample adhesives
- Noise reduction hood
- Intel computer
- Monitors: 14" (NEC XV15) and 17" (NEC XV17)

3.2.1. Unpack The System

The NanoScope system is normally shipped in five separate boxes. Each monitor is shipped in its own box, the computer and controller are shipped in separate boxes, and the MultiMode SPM, cables and hardware are shipped in one box.

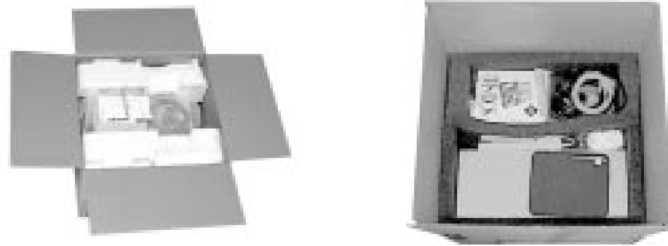
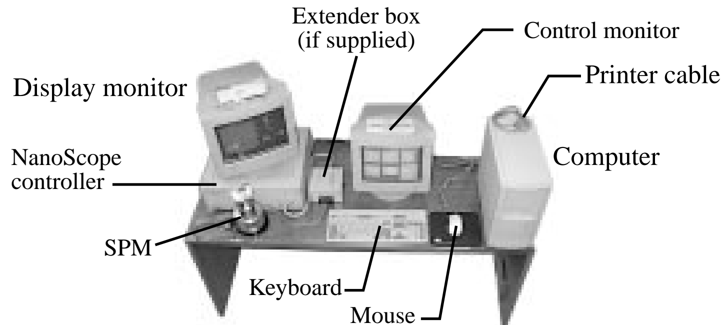


Figure 3.2. Each monitor is shipped separately (left). The SPM is packed in a separate box (right). Heavy hardware should be removed carefully from each box with the help of 2-3 people.

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

2. First remove the computer and controller. The controller may be used to support the display monitor[1] as shown in the setup below.



1. The display monitor is the larger (17-inch) of the two monitors.

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

Cabling

4. Before connecting cables, review the following precautions:

- DO NOT power anything until all cables are connected and double-checked. Power cords should be connected to their source of power last.
- DO NOT route cables through pinch-points or over sharp edges. The table should be pulled away from walls so that cables will not be pinched. The cable between the controller and SPM carries up to 440 volts and must be protected from chaffing.

5. Connect computer cables (monitors, keyboard, mouse, controller and printer) as shown below.

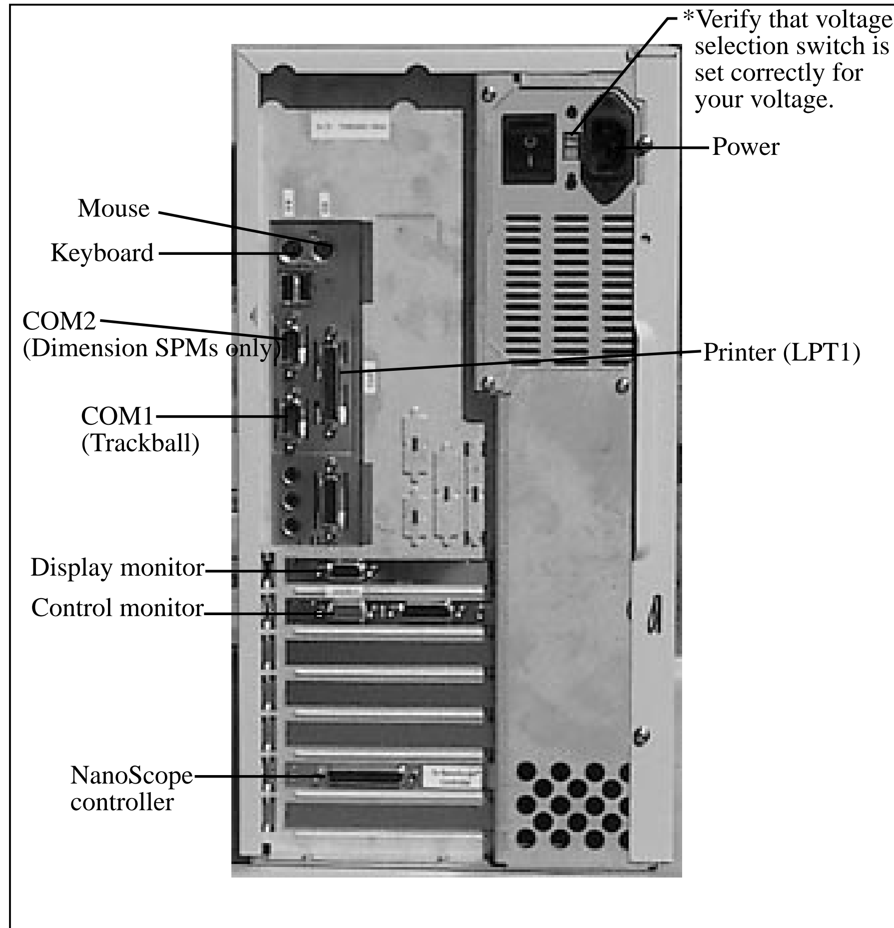


Figure 3.3. Rear view of computer on standard MultiMode systems.

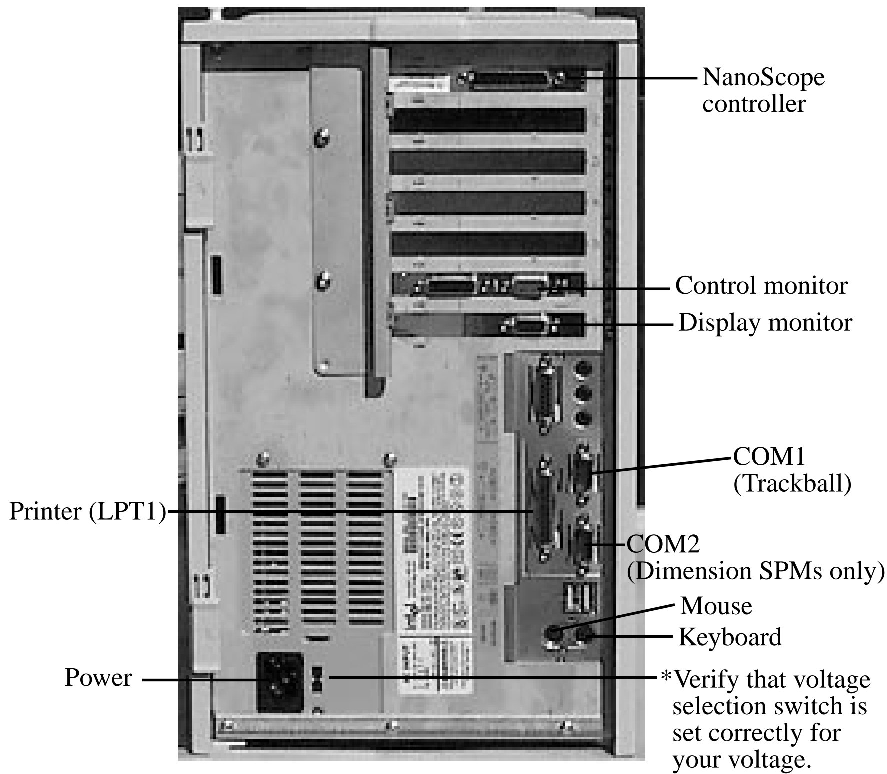


Figure 3.4. Rear view of computer on *ce* certified (European) MultiMode systems.

Note that computers are not equipped with network cards from DI, but may be added by the user. Printers are also user-supplied. Serial ports COM1 and COM2 may be configured by the user for peripheral equipment.

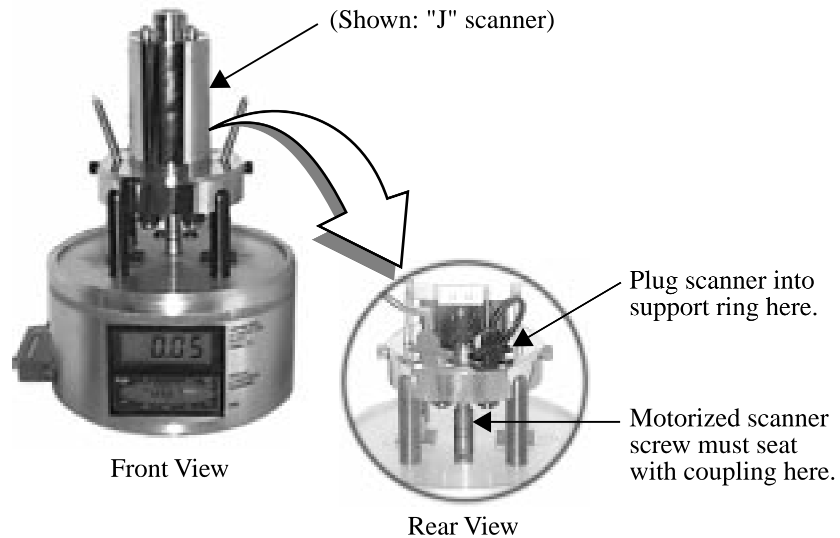
6. Connect cables to the back of the NanoScope controller: the power cable should be connected to the controller's receptacle on one side, the controller-to-computer cable on the other. DO NOT connect the power cable to a source of power yet.

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

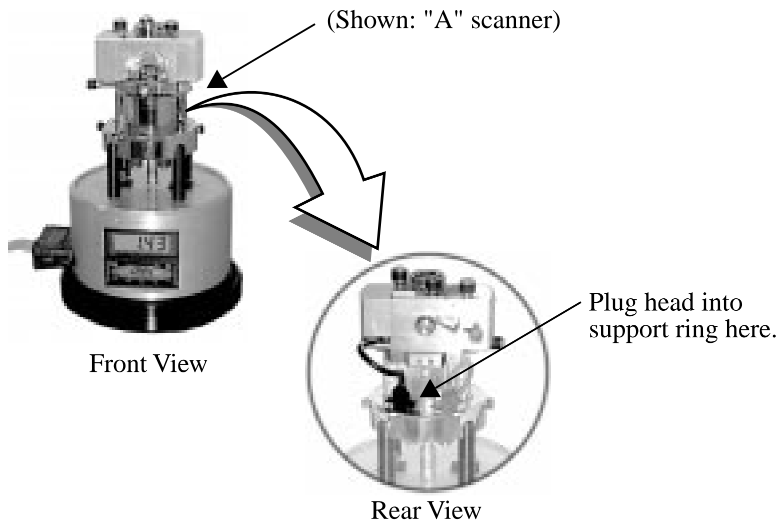
Notes

- If an Extender™ Electronics Module is included, install it now between the SPM and controller. Verify that it has been properly configured per instructions provided in Chapter 13 of this manual BEFORE powering the NanoScope controller.

8. Install the scanner on the support ring with the scanner cap upright. If the motorized scanner screw will not seat securely into the coupling on the base, use the toggle switch on the SPM base to rotate the screw slightly until it aligns and seats with the coupling. Attach retaining springs to the support ring by inserting into holes and securing with screws. Plug the scanner into the receptacle on the support ring as shown below:



9. Carefully set the MultiMode's head atop the SPM scanner. The head should mate kinematically with the scanner's contact balls. Secure the head using both retaining springs by stretching the springs up and hooking them over posts located on both sides of the head. Plug the head into the receptacle on the support ring as shown below:



10. Recheck all cable connections. Review the steps above one last time to ensure that all connections are properly made. Be especially certain that the controller cable is NOT plugged into the parallel port of the computer or damage to the electronics will result.

3.2.2. Vibration isolation

The microscope must 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 imaging is sensitive to ordinary room vibrations.

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

Another effective vibration isolation system consists of a large mass, 20 or more pounds, suspended from elastic "bungee" cords. The mass should stretch the cords at least one foot, but not so much that the cords reach their elastic limit. The SPM should be placed on the large mass having a natural frequency of about 1 Hz or less both vertically and horizontally. Test this by gently pushing on the mass and measure the rate at which it swings or bounces. A ready-to-use tripod isolation system

is also available from Digital Instruments (model TRVI). Experience suggests that air tables often have poor horizontal isolation. If an air table is used, it may require additional horizontal isolation for atomic-scale images.

As a final note, the best way to reduce coupling from vibrations is to eliminate as many sources of vibration as possible. Remember that vibrations can be transmitted to the SPM 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 noises (including conversation) can disrupt atomic images, so it is best to isolate the SPM as much as possible. Use the noise reduction hood supplied with the system to alleviate acoustic noise.

3.2.3. System Power Up & DOS Start-Up

Verify that all cables are interconnected, especially the SPM controller to the computer. Power up the two monitors, followed by the NanoScope controller, then the Intel computer using the switch in front. Using a power strip switch allows you to power up the entire system at the same time.

Chapter 4 Cantilever Preparation for the MultiMode™ SPM

The MultiMode microscope comes furnished with etched silicon cantilever substrates for TappingMode AFM and silicon nitride cantilevers for contact AFM modes. In both cases, the cantilever probes should be inspected under the microscope when being used for the first time, to get a better understanding of how the probes and substrates are connected and taken apart. The procedure for removing individual substrates from the wafer varies depending on the wafer. It will be easier to accomplish this task with the aid of a stereo microscope having 50—70X magnification.

4.1. Silicon Cantilever Substrates

The silicon cantilever substrates used in TappingMode™ can be removed from the wafer with the following procedure. Note that the cantilevers are stored tip-side-up and that the silicon is very brittle. Contacting the cantilever during this operation will almost certainly break it off of the substrate.

1. Inspect the wafer with an optical microscope to get a feel for the orientation of the cantilever substrates and to inspect the cantilevers themselves. (A 10-70X stereo microscope is useful for this task.) The cantilevers are tip side up as viewed in the wafer holder. Included with the MultiMode microscope is a wafer tool kit which contains tweezers and wafer handling tools.

2. Disconnect the substrate from the bulk of the wafer by pressing down gently on the non-cantilever end of the substrate. Alternatively, using the sharp pointed tweezers, carefully break the 2 substrate supporting arms connecting the substrate to the

silicon wafer frame. The supporting arms connecting the substrate to the bulk of the wafer will shatter as pressure is applied. See figure 4.1 for clarification.

It may be convenient to break several substrates from the wafer at one time. Extras may be safely stored in a specially prepared closable container. At the bottom of the container, place X4-grade, GEL-PAK™ adhesive strips. Place the substrates, tips facing up, on the adhesive to permit easy removal of the substrates when needed. Cover the container when not in use.

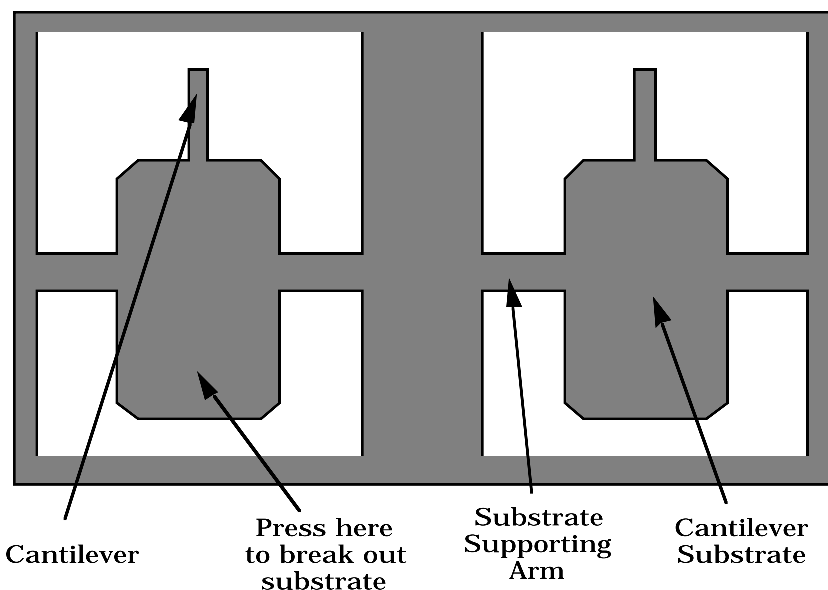


Figure 4.1. Silicon cantilever substrates in wafer.

4. Use the curved, sharp-pointed tweezers to remove the cantilever substrate from the wafer container. Grasp the sides of the substrate, away from the lever and probe tip. It may be helpful to tip the substrate to one side to help grasp it in the tweezers. Be very careful about avoiding any contact with the probe lever, since it will immediately snap off. Silicon is very brittle.

4.1.1. Tip Shape of Etched Silicon Probes

Etched silicon probes provide the highest aspect ratio and most consistent tip sharpness of the probes supplied at present. There are some subtleties in general shape that should be understood to gain the best advantage from the etched silicon tips when imaging samples with steep walls over steps of 100 nm to several microns in height.

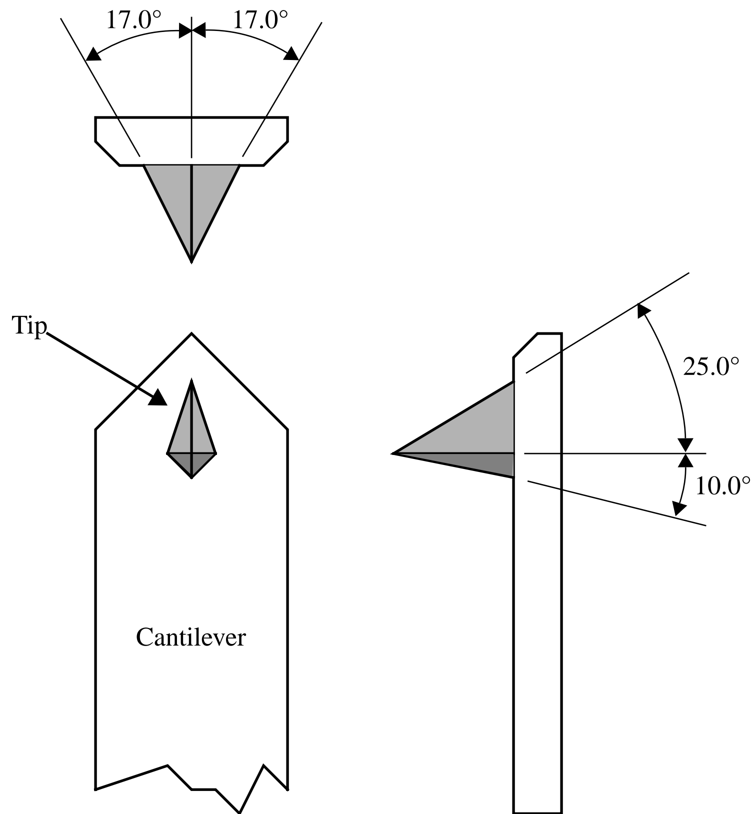
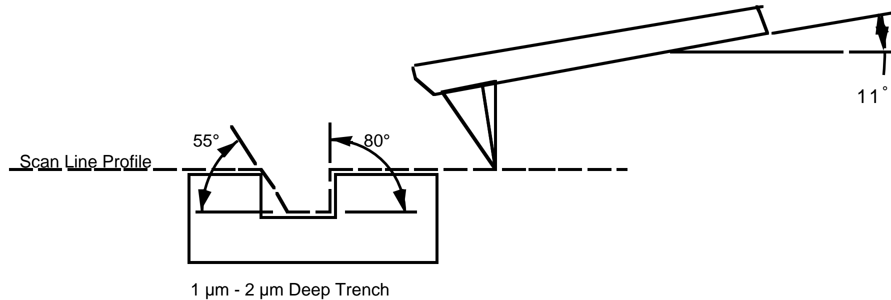


Figure 4.2. Silicon cantilever—theoretical tip shape.

The present process creates a tip which has symmetry from side to side with a 17± 2 ° half cone angle (refer to top of figure 4.2). Front-to-back, along the length of the lever, the tip is asymmetric (refer to lower-right of figure 4.2). Another factor affecting the interaction of the tip shape with the surface is the substrate mounting angle. Along the front edge of the tip, the half angle is nominally 25°, and at the back edge of the tip the half angle is approximately 10°, with both of these numbers not accounting for the tilt of the substrate. With the mounting angle of the substrate taken into account, the front is 35° and the back 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.

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

Scan direction = 0 deg.



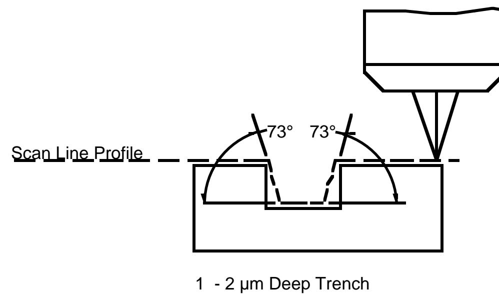
Note: Any wall angle on the left wall that is > 55 deg. will be shown as 55 deg. in the image.

Figure 4.3. Silicon probe tip profile artifact - front to back

The best orientation of the sample to measure sidewall angles, uses the back edge of the tip (that which faces back towards the cantilever substrate) to measure step angles (refer to figure 4.3). Using the back edge, step angles approaching 80 degrees can be measured routinely, depending on the step height. 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 edges of the tip could contact the other faces of the small opening. Wall angle measurements are best measured in open areas for these reasons.

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

Scan direction = 90 deg.



Note: Any wall angle that is > 73 deg.
will be shown as 73 deg. in the image.

Figure 4.4. 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 (figure 4.4).

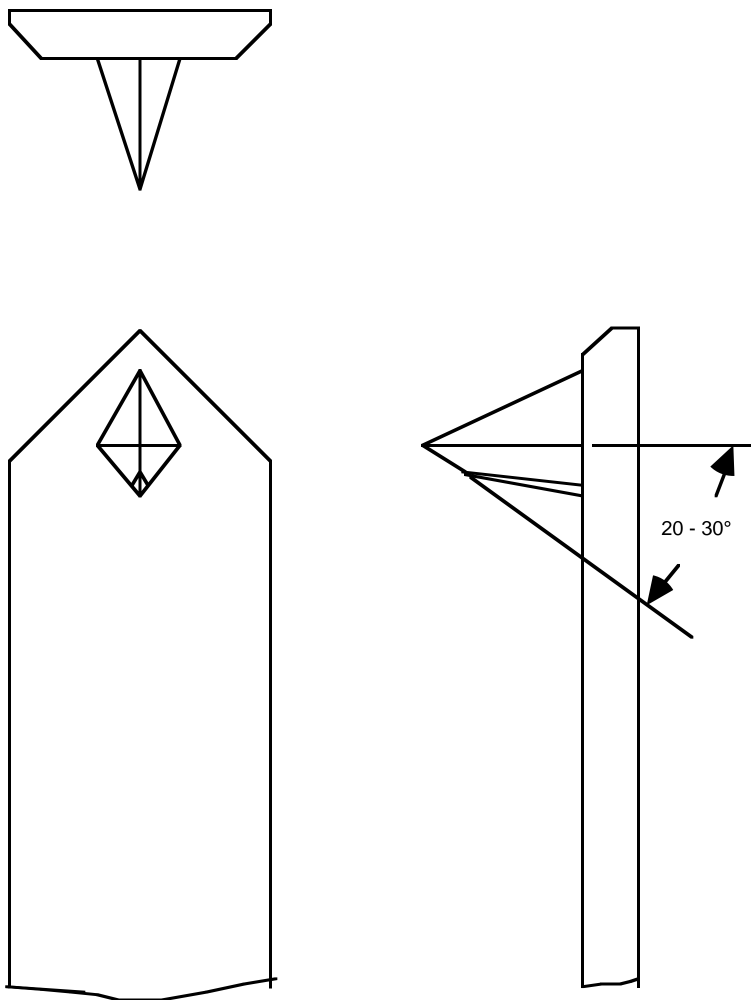
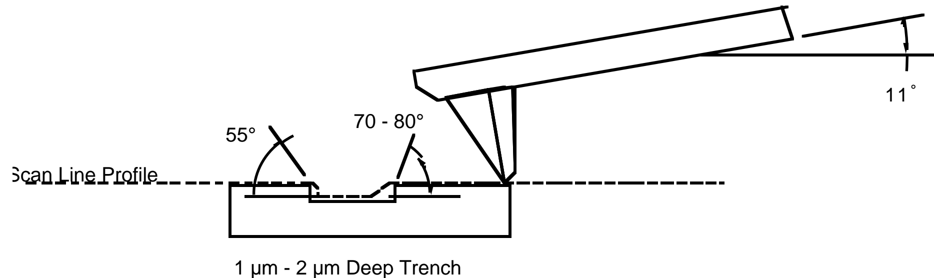


Figure 4.5. Silicon probe—common shape artifact.

Due to the nature of the etching process that shapes the tip, there is often a short angled ridge near the highest point of the tip. The exact length of the ridge is variable but rarely exceeds 0.5 µm in total length. It is inclined steeply, so that for reasonably flat surfaces the highest point is the only one which interacts with the surface. Depending on the tip in use, sample features of approximately 0.5 µm in height can begin to produce artifacts of an apparent shallow slope over scan fields of larger than 1-2 µm.

Subsequent scan line produced by using
the realistic probe tip shape



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

Figure 4.6. Common silicon probe profile—resultant scan artifact.

Shown above in figure 4.6 is the resultant effect of the angled back ridge on the step angle measurement for a deeper trench depth. This will be tip and topography dependent.

The above discussions for figures 4.2–4.6 are in relation to microscopic scale shape characteristics. There is one further detail which can come into play affecting the wall angle over shorter (nominal 100 nm) step height measurements: 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 point of the tip to a length of 100 nm from the end of the tip. It is formed in such a manner that the radius of curvature of a silicon tip can be in the range of 5–10 nm (on a very good tip).

4.2. Silicon Nitride Cantilever Substrates

When using the MultiMode microscope for the first time, start with the provided strip of cantilevers and skip to step 4, which follows. Step 4 provides instructions on how to separate substrates from strips.

1. Verify that the wafer is oriented with the tips facing upward (gold coated surface down). Inspect the wafer with an optical microscope. We suggest using a 10 -70X stereo microscope to become familiar with the styles and orientation of the cantilevers on the probe substrate.

2. Remove the Pyrex strips by resting the silicon ring on a glass slide or ruler, then applying downward pressure with the tweezers until the strip breaks free from the silicon ring. While doing this, be very 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 this occurs accidentally.)

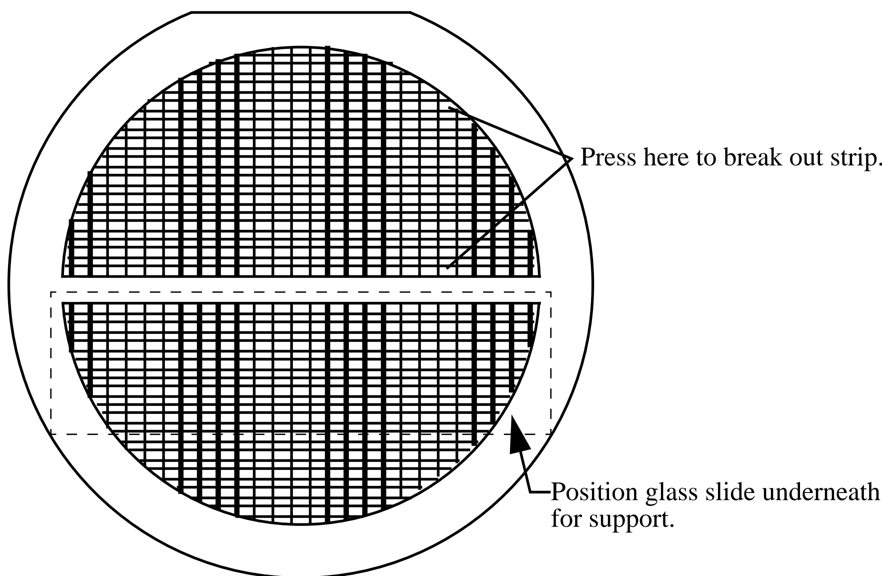


Figure 4.7. Silicon nitride cantilevers in a wafer.

3. Place the strip down on a white piece of paper to inspect it under the microscope. When doing this, be careful 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 should be a saw-cut almost through the Pyrex.

4. 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, as shown in Figure 4.8. The saw cut should be approximately on the edge of the slide. 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. It should pop right off. Repeat this process until as many cantilever substrates as required have been removed.

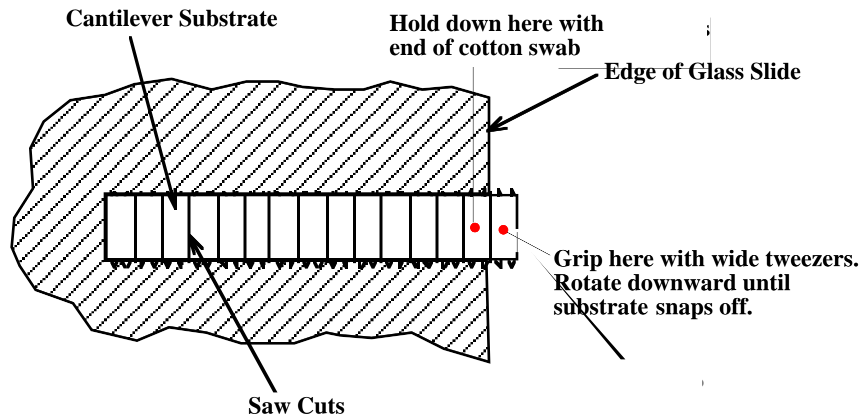


Figure 4.8. Substrate break-off.

Extra substrates are easily stored in a covered container. The shipped substrates are held on X0-grade, GEL-PAK™ adhesive strips. The strips are used to permit easy removal of the substrates. If GEL-PAK adhesive strips can't be found, a simple substitute is the adhesive area from a Post-it note. When placing the substrates on the adhesive, be careful that the cantilevers are on the top side of the substrate.

5. Each substrate has two cantilevers on each end of the substrate. Both 100 and 200-micron length cantilevers with two different leg widths are provided. When ready to use a cantilever substrate, it may be desirable to remove the unused cantilevers from that substrate, but it is not necessary. For most applications, use the 100-

micron cantilever with the wider legs. For atomic scale images, the 200-micron triangular cantilever with the wider legs can yield good results.

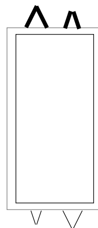
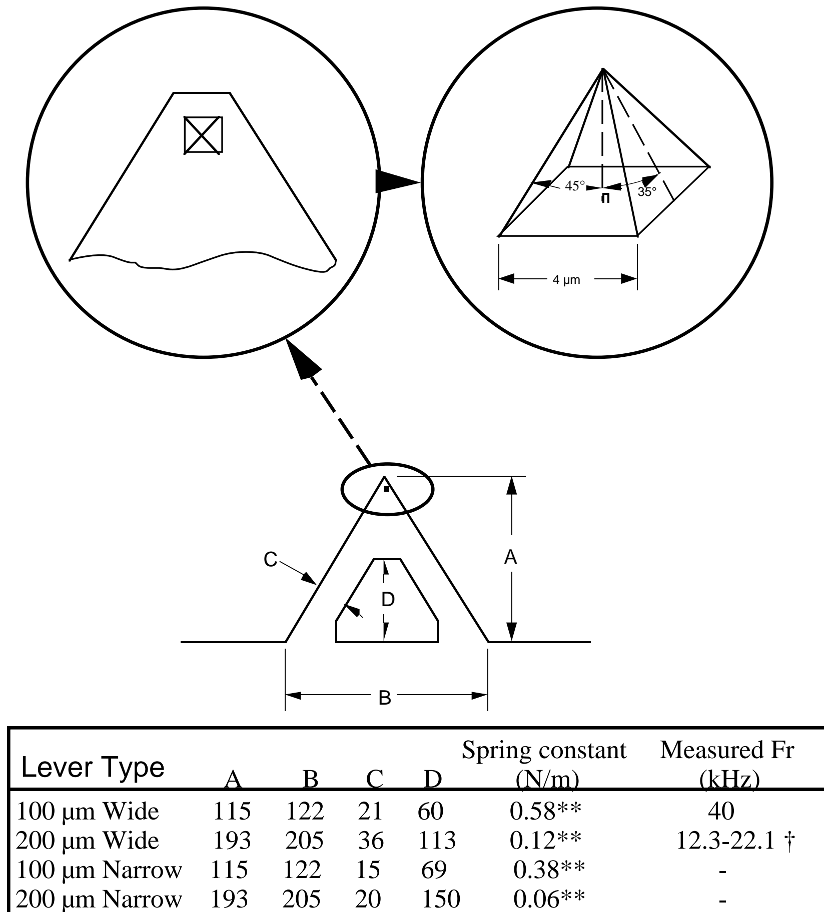


Figure 4.9. Substrate shown with the 100 and 200 µm long, wider cantilevers on top and the 100 and 200 µm long, slimer cantilevers on the bottom.

4.2.1. 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. Bear in mind that the probe tip is approximated by a pyramid formed by intersecting <111> planes in silicon. The approximate shape of the tip is shown in the upper right in figure 4.9. In the same figure are dimensions and approximate values for spring constants and resonant frequencies.



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**	-

Figure 4.10. Silicon nitride cantilevers—specifications and tip shape.

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° (figure 4.10) 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.

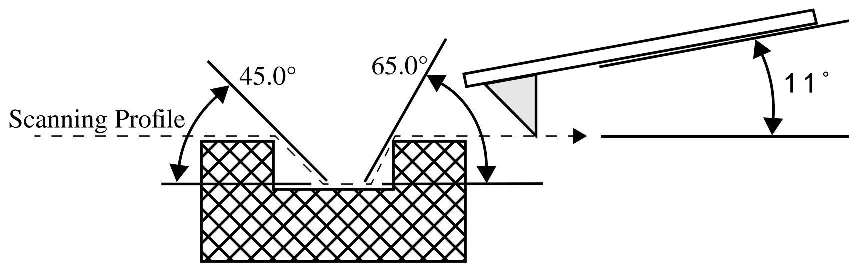


Figure 4.11. Silicon nitride cantilevers—sidewall profile effect.

There is one last detail that is useful to know about silicon nitride cantilever probes. There are two types 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 there is a slight cusp formed 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 5 Head, Probe and Sample Preparation

Preparing to Image with the MultiMode SPM

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

Other chapters in this manual describe how to perform specific types of imagery. The table below outlines where you will find additional information for each type of imagery. If you are new to SPM and want to practice, we suggest you begin with contact AFM in Chapter 6.

For Specific Information Regarding:	See Chapter:
Contact AFM in air	6
Contact AFM in fluids	7
TappingMode in air and fluids	8
Scanning tunneling microscopy (STM)	9
Lateral force microscopy (friction)	10
Force imaging: force modulation, etc.	11
Interleave scanning	12
Magnetic force microscopy (MFM)	13
Electric force microscopy (EFM)	14

This instrument uses a semiconductor diode laser emitting a maximum 1.0 mW beam at 670 nm. The light is emitted downward and normally reflects back into the system's optics from the back of the cantilever probe. Note that the laser is powered when the SPM head is plugged into the microscope's support ring and the **Mode** switch is set to either **AFM & LFM** or **TM AFM** (TappingMode). (As a safety precaution, the MultiMode SPM head features an internal switch to shut power off to the laser when the head is tilted. Operators should use care, however, to avoid staring into beams reflected from sample surfaces.)

WARNING! During and prior to set up of the laser, it is especially important to avoid looking directly at the laser beam or at the laser spot. The laser head should never be plugged into the microscope control electronics unless the head is installed in the Z-stage mount. Care should be taken when highly reflective samples are inserted onto the chuck. Avoid looking at all reflected laser light. Operators should use care to avoid staring into beams that may be reflected from sample surfaces.

AVERTISSEMENT! Avant d'utiliser le laser, et durant tout le temps pendant lequel il fonctionne, il est impératif de ne pas regarder directement le faisceau. La sonde laser ne doit jamais être branchée sur l'électronique de contrôle du microscope, tant que la tête 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.

5.1. Initial Preparation for Contact AFM Imaging

5.1.1. Prepare the sample.

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

Sample Preparation

If it is your first time operating the microscope, we recommend that you image the calibration sample provided with the instrument (usually a 10μm-pitch grid of 180nm step height).

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

Provided with the instrument are red and white colored “sticky tabs” which are 2 sided adhesive patches. Peel off a “sticky tab” from the provided sheet, and place it on the steel small sample puck, then peel off the red-and-white paper. This leaves a patch of the two-sided adhesive on the steel sample disk, which will hold the sample chip to the disk. Using tweezers, place the small sample to be imaged firmly on the “sticky tab” adhesive (figure 5.1). Alternatively, a small sample can be glued down to the sample puck using cyanoacrylate glue (superglue). Place the small sample disk atop the scanner.

Figure 5.1. Gently press the sample onto the sticky tab until secured.

5.1.2. Load the sample.

Remove head and load sample.

If the head is not already removed, do so now by unfastening the retaining springs on either side and unplugging the head's micro-D connector. Gently lift the head off and set aside. This will expose the top of the scanner tube. Mount the sample puck with the calibration standard atop the scanner tube. An internal magnet supplied with most units holds the puck down.

Figure 5.2. MultiMode base with scanner mounted on support ring.

Reinstall the head.

With the sample in place, remount the head by gently lowering it over the scanner tube while checking for clearance. The top of the sample should protrude no more than a few millimeters above the top of the head's X-Y translation stage. Secure both retaining springs and plug the head's connector into the support ring (figure 5.3).

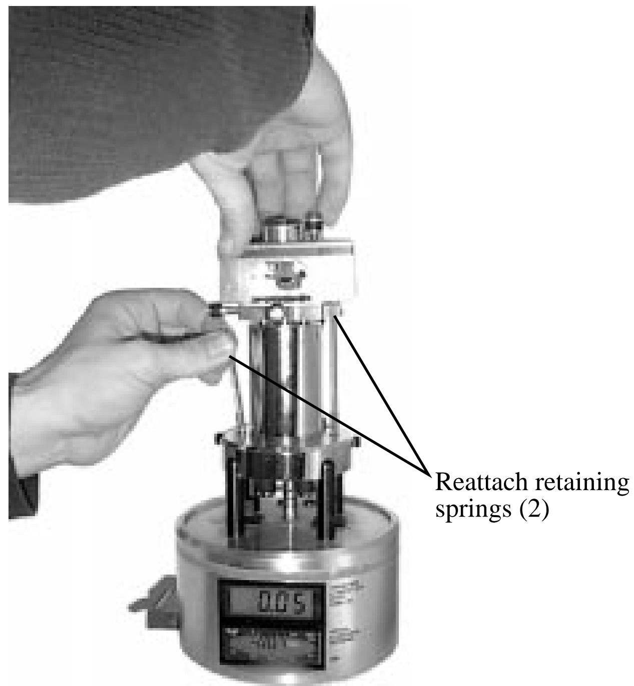


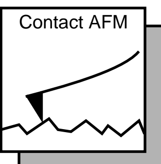
Figure 5.3. The head is held securely using retaining springs.

Check head for free vertical movement.

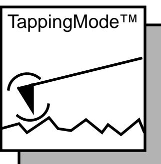
Verify basic function of the motorized Z-axis by toggling the **Up** switch on the MultiMode base (figure 5.4). This activates the leadscrew at the rear of the unit to lift the head upward. If you are using a standard, three-screw scanner, the two forward screws will also have to be rotated to keep the head level while lifting. (Single-screw, vertical scanners require only that the rear, motorized screw be rotated to raise and lower the head.) To verify rotation of the motorized screw, feel the flexible coupling on the base with your finger while the tip **Up** / **Down** switch is toggled.

Figure 5.4. The tip is quickly raised and lowered using the *Up / Down* toggle switch on the MultiMode SPM's base.

5.1.3. Load a probe into the tipholder.



For contact AFM, install a silicon nitride probe tip in the AFM tipholder. Figure 5.5 shows the AFM tipholder. Detailed procedures for silicon nitride cantilever substrate preparation are given in Chapter 4, including a description of the procedure to break out substrates from the wafer. Ensure the gold-plated side of the substrate is placed down towards the substrate mount, with the nitride film side attached to the cantilever oriented up away from the substrate mount.



The procedure for installation of TappingMode, single crystal silicon probe tips is essentially the same as for contact AFM (see above paragraph). The substrate should be face-up, with the probe's cantilever pointing away from the AFM tipholder. This ensures that the cantilever and tip are facing toward the sample once the tipholder is mounted in the head. Refer to Chapter 4 for a description of the procedure to break out each substrate from the wafer.

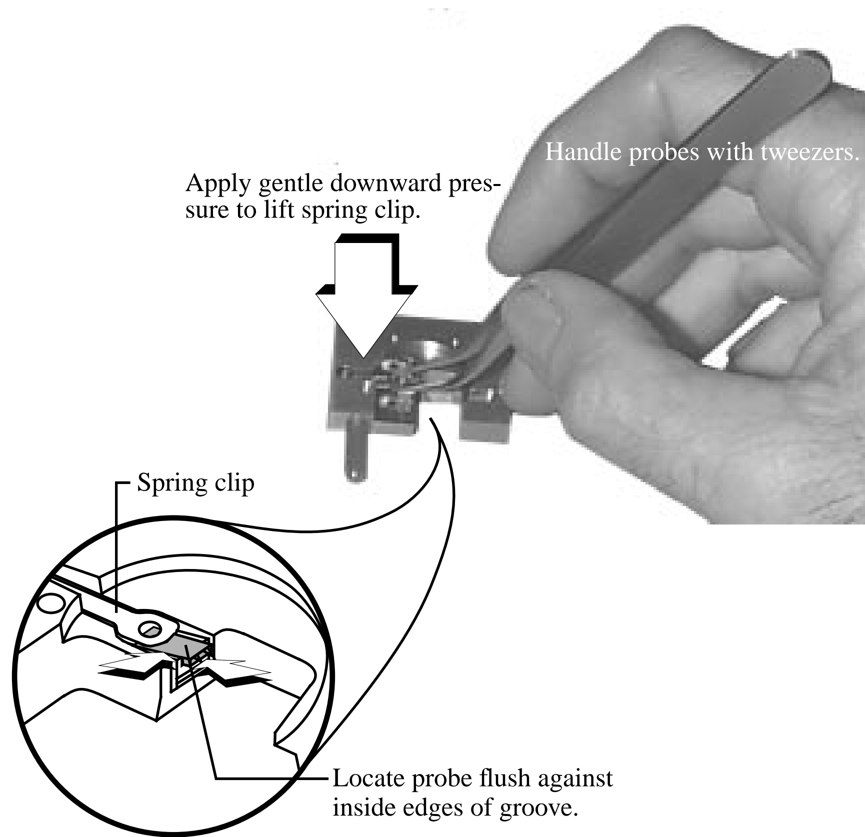


Figure 5.5. Slide the probe carefully into the tipholder’s groove. Gentle downward pressure against the tipholder will lift the spring clip for probe insertion.(Shown: underside of AFM tipholder.)

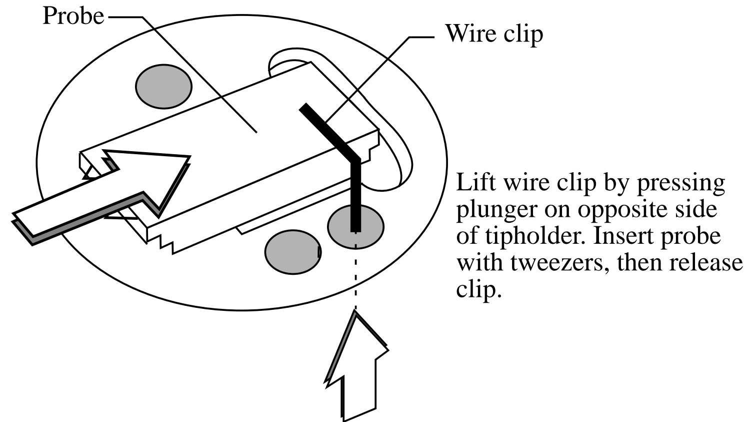


Figure 5.6. Fluid cell probe installation is similar to AFM/LFM tipholders. Lift wire clip by pressing plunger on opposite side to insert probes. (Shown: underside detail of fluid cell.)

To load a probe into the tipholder.

Refer to figure 5.6. Turn the tipholder upside down with the groove facing up as shown. Apply gentle upward pressure against the plunger to lift the spring clip. With the spring clip lifted, carefully slide the probe into the tipholder's groove until it is located squarely against the innermost edges, then lower the spring clip by releasing pressure against the plunger. This will hold the probe securely in the tipholder's groove. Check that the probe's substrate is flush with the back of the groove and flat against one side (this keeps the probe's cantilever oriented in the correct direction). If identical probes are loaded the same way each time, aiming the laser onto the cantilever will be much quicker and easier.

CAUTION! The spring clip is extremely fragile and must be handled with great care to prevent bending.

ATTENTION! Les embouts de ressort sont extrêmement fragiles et doivent être manipulés avec une extrême précaution.

WARNHINWEIS! Die Haltefeder ist sehr empfindlich und muß sehr vorsichtig behandelt werden, um ein Verbiegen zu vermeiden.

5.1.4. Install the tipholder.

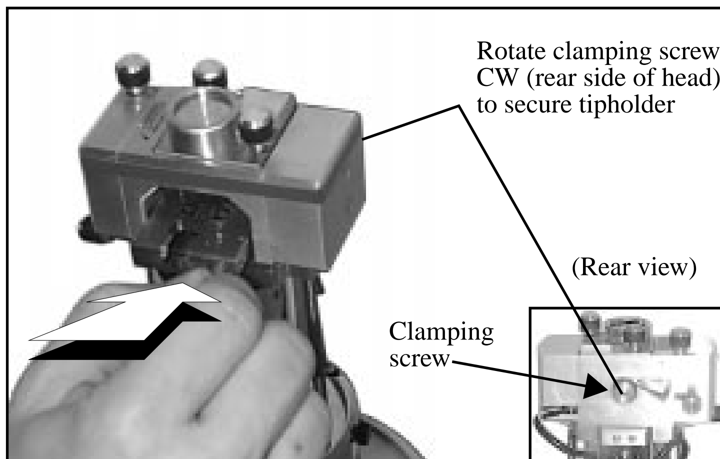


Figure 5.7. Install tipholder in head without touching the sample. Secure tipholder using clamping screw at rear of head.

Once the tipholder is loaded with a probe (see Section 5.1.5 above), the tipholder is placed inside the SPM's head and clamped into position. First, verify that the head is sufficiently raised to clear the sample with the tip. (The top of the sample should protrude no more than 1-2 mm above the plane of the head's bottom hole.) Insert the loaded AFM tipholder into the MultiMode's head by lifting the tipholder carefully over the sample. Do not touch the sample with the tipholder. Press gently forward, then lower the tipholder (figure 5.7).

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

5.2. Laser Alignment

This section describes two methods for aligning the laser, mirror and photodiode for all modes except STM. (STM does not use a laser.) The first method uses a high-powered monocular magnifier (supplied with each MultiMode) to observe the laser spot's position on the cantilever. The second method uses a strip of paper to observe the laser's position. The choice of method is largely a matter of personal preference. Before beginning, verify that the mode switch on the MultiMode SPM's base is switched to **AFM & LFM** or **TM AFM**. If the switch is toggled to **STM**, the laser will be turned off.

5.2.1. Method 1: the magnifier method

This operation is performed using the high-powered monocular magnifier (shipped with each MultiMode) or a similar magnifying system, under low light conditions. First, the laser spot is positioned onto the cantilever. The photodiode is then positioned to maximize the signal and the spot is fine-adjusted onto the very tip of the cantilever.

WARNING! Staring at a bright beam or reflection can result in eye damage. Be sure that you are using a magnifier with a laser filter installed.

AVERTISSEMENT! Fixer un faisceau lumineux puissant ou sa réflexion peuvent entrainer des dommages au niveau des yeux. Il est impératif d'utiliser un filtre laser.

WARNUNG! Der direkte Blick in den Laserstrahl oder dessen Reflektion kann Augenschädigungen hervorrufen. Bitte stellen Sie auf jeden Fall sicher, daß ein optisches Gerät, mit dem Sie in den Laserstrahl schauen, mit einem Laserschutzfilter ausgestattet ist.

1. Position the magnifier (figure 5.8).

Using the 45X magnifier, focus on the cantilever substrate. There are two ways you can do this. One way is to view the substrate from the side through the gap between the cantilever mount and the bottom of the head shell. This is a useful vantage point with the glass cantilever mount, as the edge of the substrate can be clearly seen. Also, this is the same position used to view the cantilever-to-sample separation when the tip is brought close to the surface; therefore the same magnifier position can be used for both operations. With the metal cantilever mount, it is preferable to do the alignment by looking down on the substrate from an elevated angle. Whichever viewing angle is used, the alignment process is the same.

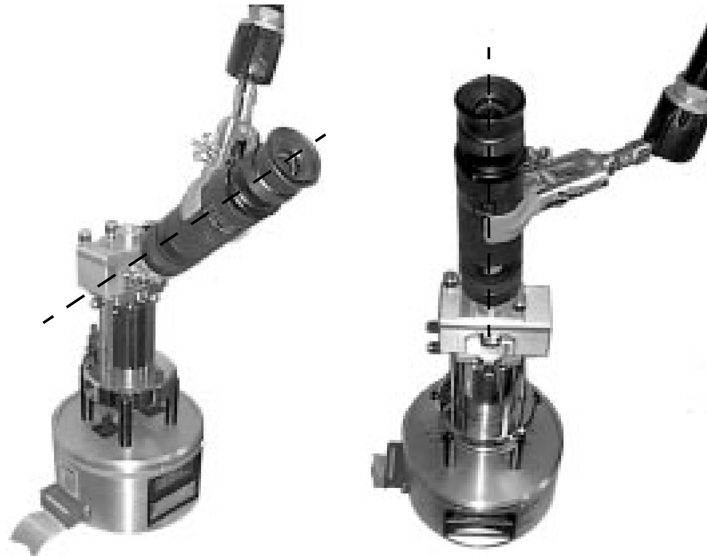


Figure 5.8. Two ways of positioning a magnifier when aligning the laser. Through the head's front window (left), and overhead (right).

2. Identify the key features.

There are many laser light reflections, particularly from the tipholder, the sample, and various metal surfaces. It is helpful to identify the key features before starting the alignment. Make sure that the adjustment screws are positioned such that the cantilever is well above the sample, so reflections off the sample do not make it hard to locate the cantilever. Compare the view through the magnifier to the earlier view of the cantilever.

3. Position the laser spot on the substrate.

The laser spot can be moved around with the two positioning knobs on the top of the head. With the right-positioning screw, move the spot so it is on the cantilever substrate. This is easy to see with the silicon nitride cantilevers because the glass substrate will glow and there will be a lot of diffused red light scattered around the internal area of the head.

4. Find the cantilever with the laser spot (figure 5.9).

While observing the substrate through the magnifier, slowly move the laser spot lengthwise along the substrate with the X-axis positioning screw until the spot is barely off the edge of the substrate (**1**). The laser spot may be visible on the sample just off the substrate edge. With the rear-positioning screw, move the spot along the edge of the substrate (**2**). Very small movements of the positioner are enough to pass over the cantilever, so move slowly with small motions. Since the substrate is mounted by hand, there is usually a small amount of angle between the substrate and the axes of the positioners, so the best way to look for the cantilever is to make small motions along the edge of the substrate and to move the spot back and forth a little bit lengthwise for each small increment of edgewise motion. This way, it is possible to stay adjacent to the edge without moving away from the substrate or back onto it. In this fashion, move along the edge of the substrate. If a corner of the substrate is reached without finding the cantilever, move back the other way.

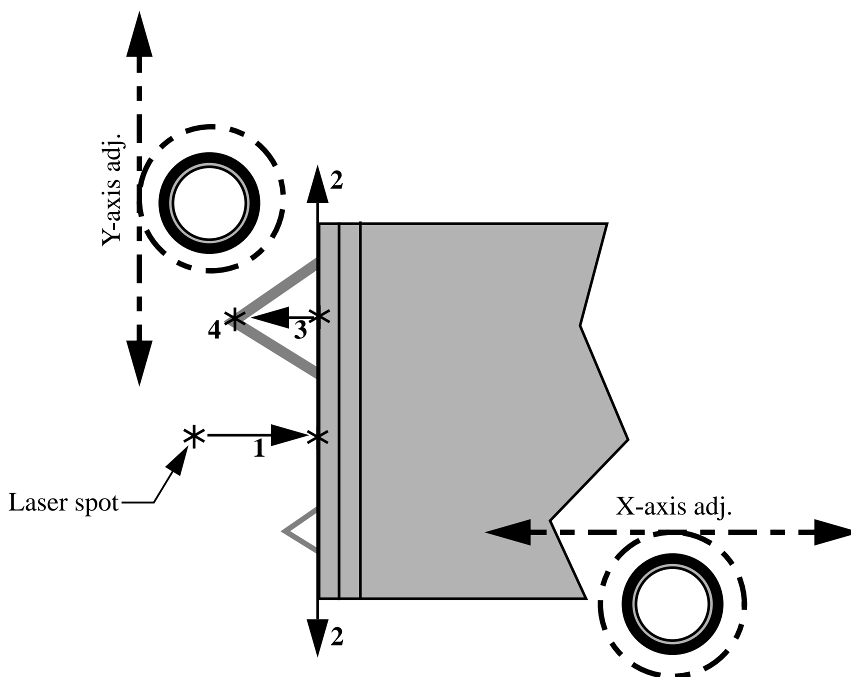


Figure 5.9. Laser spot positioning.

5. Position the laser spot on the tip of the cantilever.

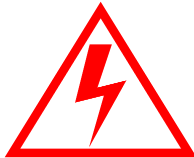
With the silicon nitride cantilevers and a glass cantilever mount, a small bright red dot appears to be hanging in mid-air right off the edge of the substrate when the laser beam is on the cantilever. With the metal mounts with either cantilever, the cantilever should be visible when the spot is near it. With very small motions of the positioners, move the spot around on the cantilever until the spot is near the tip.

5.2.2. Method 2: The "paper" method

Some users prefer to align the laser by observing light patterns reflected or diffracted from the surface of the cantilever onto a piece of paper. This is known as the "paper" method of laser alignment. Interpreting light patterns requires some experience, but is not especially difficult. The paper method can quickly verify with a high level of confidence whether the laser spot is on the tip of the cantilever.

Figure 5.10. Paper method of laser alignment using a slip of paper inside SPM head.

WARNING! To avoid eye damage due to high-level laser light, do not place highly reflective or metallized objects into the head area while the laser is on.



AVERTISSEMENT! Pour prévenir tout accident au niveau des yeux, dû à la lumière du laser, il est recommandé de ne pas placer d'objet très réfléchissant ou métallique sous le faisceau laser lorsque celui-ci est allumé.

WARNUNG! Um Augenschädigungen aufgrund von Laserlicht zu vermeiden achten Sie bitte darauf, daß keine reflektierenden oder mit Metall beschichteten Gegenstände im Bereich des AFM-Kopfes benutzt werden, solange der Laser eingeschaltet ist.

Inside the SPM Head

In this version of the paper method, the user inserts a narrow (1 cm wide) slip of paper into the head and observes patterns reflected from the top side of the cantilever (figure 5.10). Once learned, this is a very quick method for obtaining laser alignment. Because the slip of paper prevents light from reaching the photodetector, the sum signal cannot be monitored while using this method.

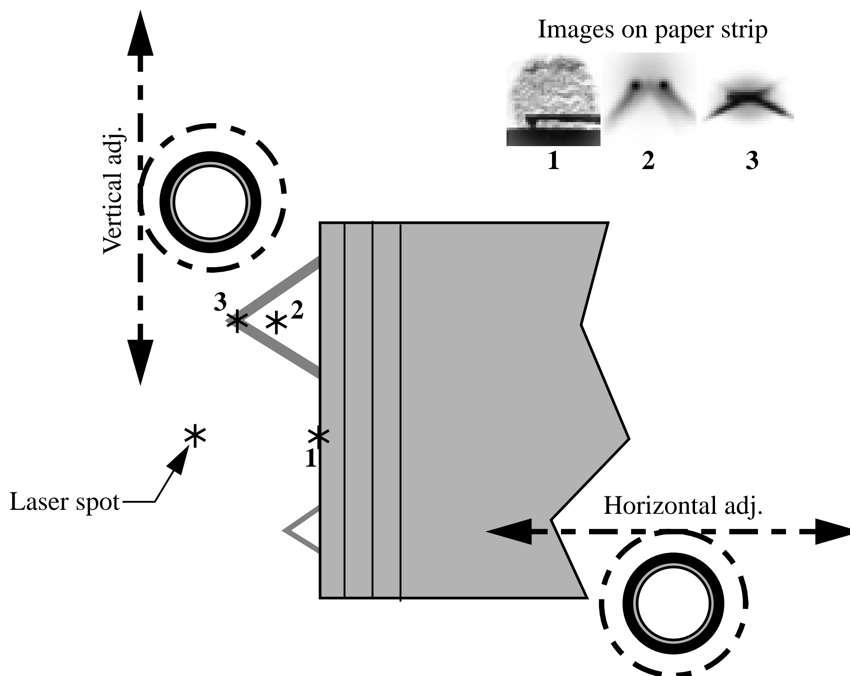


Figure 5.11. Directional control of laser spot using adjustment knobs. Three spot locations are shown along with their resultant reflection patterns.

1. Position the laser beam off the cantilever substrate (figure 5.12).

Look at the cantilever substrate through the large cutout in the front of the optical head. Use the laser positioners on the top of the optical head to position the laser beam off the end of the cantilever substrate. To be certain the beam is off of the substrate, vary the beam position in X until the laser light is reflected brightly from the substrate, then turn the X-adjustment screw counterclockwise until the bright reflection disappears. This may be easier to see if the cantilever is one or two millimeters above the sample surface. Insert a 1 cm wide slip of white paper in front of the photodiode.

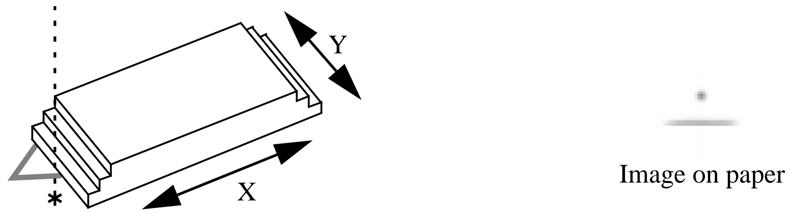


Figure 5.12. Laser spot off probe and resultant reflection pattern. Intensity may vary according to reflectivity of sample.

2. Position the laser beam in X (figure 5.13).

Position the laser beam so that a small amount of light is reflected from the edge of the cantilever substrate. The purpose of this step is to position the beam such that most of the laser light is falling off the end of the substrate; therefore, it is important that the amount of reflected light be small.

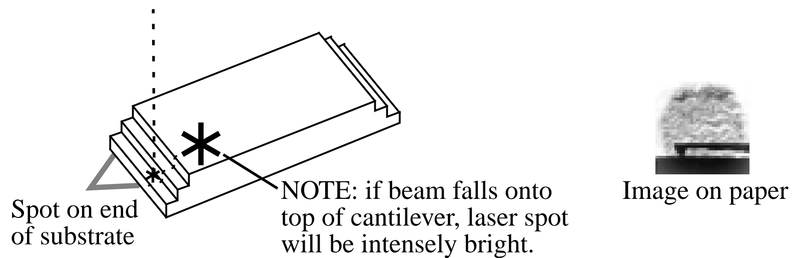


Figure 5.13. Locate the laser spot on the edge of the cantilever.

3. Position the laser beam in Y.

Using the Y adjustment, move the beam parallel to the edge of the substrate while looking for a reflection on the paper slip. Typically, the beam will reflect off both of the legs of the silicon nitride cantilevers, so there will be two spots reflected which may or may not show on the paper at the same time. In either case, the object is to position the beam halfway between the two legs. Note that the separation between the legs will be greater on the 200-µm cantilevers than on the 100-µm cantilevers.

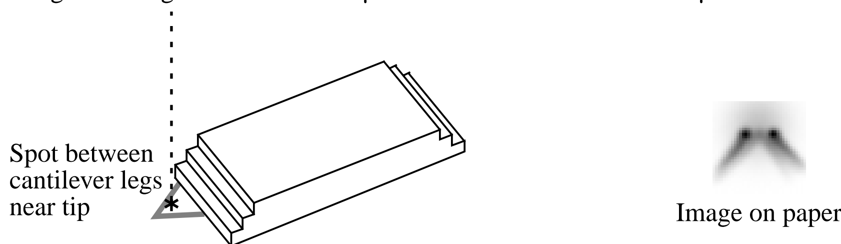


Figure 5.14. Position the laser spot between cantilever legs.

Notes

- The procedure is similar for the silicon cantilevers but because the cantilever is a single beam, there will only be one laser spot reflected onto the paper.

4. Align the laser beam with the end of the cantilever (figure 5.15).

For silicon nitride cantilevers: move the laser beam out onto the end of the cantilever by turning the X adjustment counterclockwise while looking at the reflected spot on the slip of paper. The spots reflected from the legs should merge into a single, well defined, bar-shaped spot. Verify that the laser beam is aligned with the end of the cantilever by moving the beam back and forth in Y. If more than one reflected spot is observed, then again position the beam between the two legs and move out in X. Repeat until only a single, bright, well-defined spot is observed when the position of the laser beam is varied in Y. If the reflected spot is dim or less well defined, the laser beam may be improperly aligned. Attempting engagement under these conditions may result in damage to the cantilever or sample.

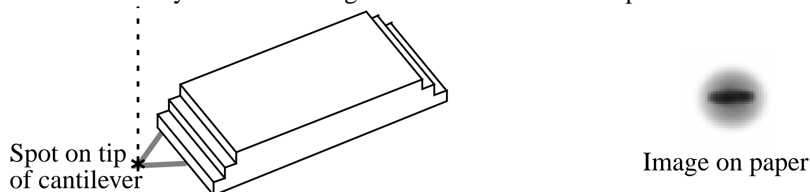


Figure 5.15. Alignment is complete when the laser spot is located on or near the end of the cantilever.

Good images can usually be obtained any time there is a good bar-shaped pattern reflected off the back of the cantilever; however, the following suggestions for fine-tuning the alignment of the laser spot position may enhance the image quality in some cases. The position of the laser spot on the cantilever affects the sensitivity of the cantilever deflection signal. The closer the spot is to the tip, the higher the sensitivity. However, the strength of the deflection signal decreases and optical interference problems increase as the laser spot is moved to the end of the cantilever, because the cantilever is triangular in shape and the laser beam begins to spill off the cantilever as the beam is moved towards the tip. This results in a trade-off between signal strength and sensitivity.

Increased sensitivity is desirable when imaging atoms. So, when imaging atoms, turn the laser diode X adjustment slightly counterclockwise to move the laser beam closer to the end of the cantilever. Stop when the reflected bar-shaped pattern on the piece of paper begins to shorten as the beam spills off the cantilever. It is important to keep the entire laser beam on the cantilever when imaging samples with tall features. For these and most other samples, a good, solid, bar-shaped reflection on the piece of paper provides the best results.)

For silicon cantilevers, the paper method will be slightly different because the silicon cantilever is a single beam rather than an open triangle (refer to figure 5.16). The general method is as follows:

1. Locate the laser spot on the substrate.

2. Move the spot along the Y-axis to locate the center of the cantilever.

3. Move the spot off the substrate and onto the cantilever.

4. Locate the spot on the center of the cantilever.

5. Move the laser spot to the end of the cantilever.

Throughout the procedure, reflections from the cantilever can be interpreted on a slip of paper. Use figure 5.16 below as a guide.

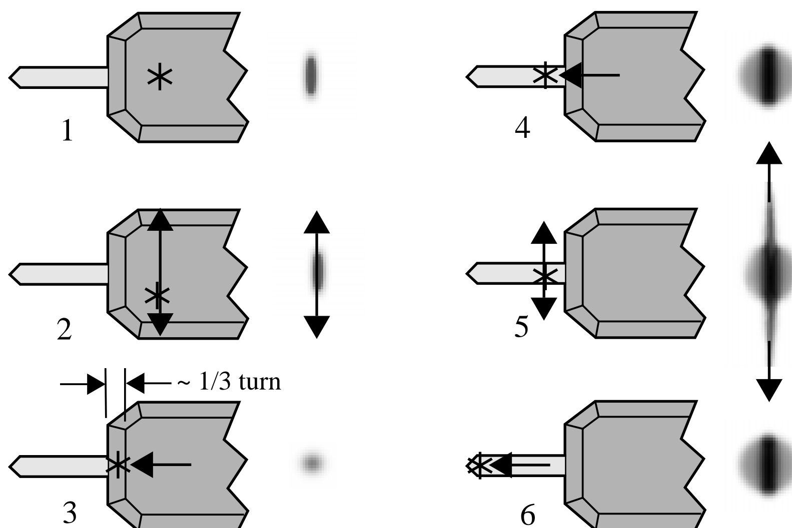


Figure 5.16. Procedure for aligning silicon cantilevers using a slip of paper: 1) locate beam on substrate; 2) find center of substrate; 3) move toward cantilever; 4) locate cantilever; 5) find center of cantilever; 6) move to end of cantilever.

6. Align the photodiode with the reflected beam.

Contact AFM

Once the laser beam is aligned with the cantilever, it is necessary to position the photodiode relative to the reflected spot. This is done by first adjusting the mirror lever on the rear of the head, then adjusting the photodiode adjustment knob such that the SUM signal displayed on the elliptical bar graph is maximized. This is a much less sensitive adjustment than the laser beam alignment, so the maximization need only be approximate. If the laser beam has been properly aligned, the maximized SUM signal should be over 4 volts with gold-coated silicon nitride cantilevers. If this is not the case, check the alignment of the laser beam.

TappingMode

For TappingMode: with the microscope toggled to **TM AFM** mode (the LED on the face of the MultiMode base should be green), use the mirror lever on the rear of the head and the photodiode adjustment knob to maximize the SUM signal displayed on the elliptical bar graph, and zero the top/bottom differential signal which is shown on the lower LCD display. If the range of movement of the photodiode stage is inadequate to accomplish this, adjust the mirror angle with the lever in the back

of the head. If the laser beam has been properly aligned, the maximized SUM signal should be 1—3 volts with a silicon cantilever. If this is not the case, check the alignment of the laser-beam.

Projecting Diffraction Patterns with Head Removed (Newer Models Only)

Yet another "paper" method of laser alignment consists of removing the head and interpreting diffraction patterns as light spills around the cantilever (figure 5.17).

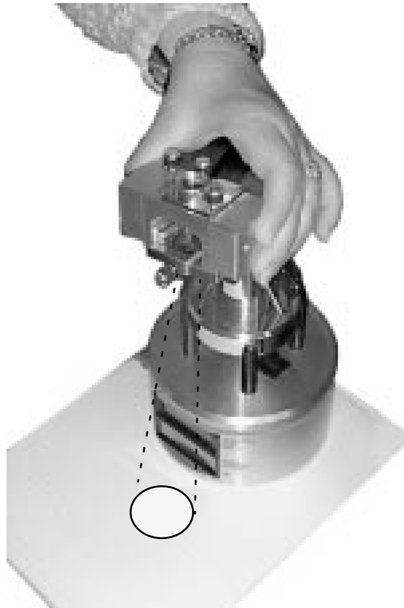


Figure 5.17. Projecting diffraction patterns downward with head removed. NOTE: Microscope shown is a MultiMode™ AFM, however, procedure applies to all current models of small sample microscopes.

Notes

- As a safety measure, all SPM heads are equipped with a cutout switch that automatically turns off the laser when the head is moved (to prevent eye damage). Early model heads feature a magnetic switch which turns off the laser when the head is removed from the scanner. The method described here will NOT work with these early model heads. Later models feature a mercury tilt switch which allows the head to be removed from the scanner without turning off the laser. Always handle the head with caution. DO NOT DROP!

The projection method is very similar to the method described above using a slip of paper. The patterns are also similar, but less distinct.

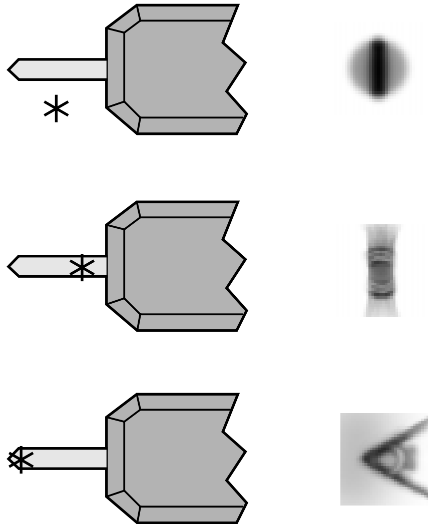


Figure 5.18. Various diffraction patterns projected on paper for silicon cantilever. Patterns for silicon nitride cantilevers are similar.

5.2.3. Maximize the SUM Signal

This section descibes what to do after the laser spot is aligned on the cantilever and assumes knowledge of how to read voltages from the meters mounted on the front of the MultiMode base. If you are unfamiliar with reading the MultiMode's voltage meters, skip ahead to section 5.4, then return to this section. Additional information is provided in each of the various chapters on imaging.

After the laser beam is aligned on the tip of the cantilever using one of the methods described above, move the mirror lever on the back of the head (figure 5.19) to maximize the SUM signal. Next, adjust the photodiode positioner to set the output signal to the desired value.

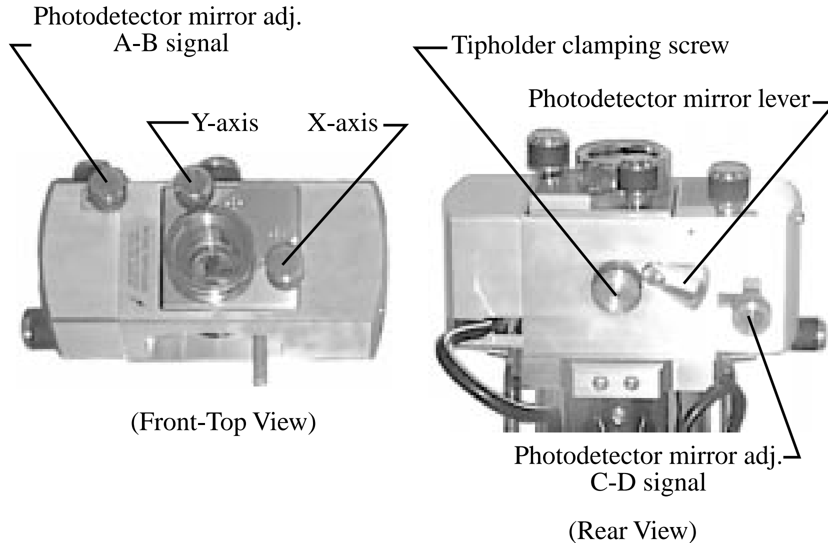
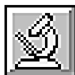


Figure 5.19. Signals produced by the photodetector are optimized using the head's various adjustments.

This adjustment is much less sensitive than the laser position adjustments. The maximized value should be approximately **5.0—9.0 Volts** for silicon nitride cantilevers. The value of this signal varies with many factors. It is important to note that it is possible to see a large response on the elliptical bar graph without having the laser beam on the cantilever, so it is important to visually verify that the laser beam is on the cantilever and not rely on the elliptical bar graph alone. Attempting to engage with the laser beam improperly aligned will usually destroy the cantilever.

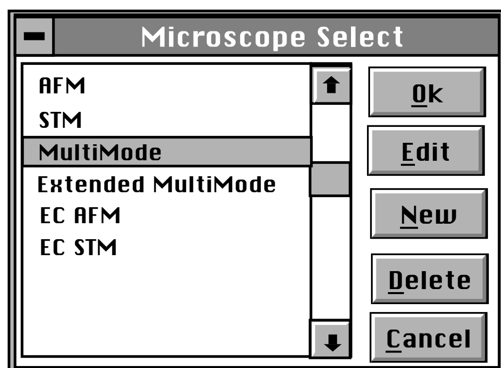
5.3. Start the Microscope Program



From the DOS prompt, change to the C:\SPM directory. Start the SPM microscope's Z.EXE program in C:\SPM by entering "**Z**." To proceed to the Real Time functions, use the mouse to click on the **Real Time** icon on the tool bar.

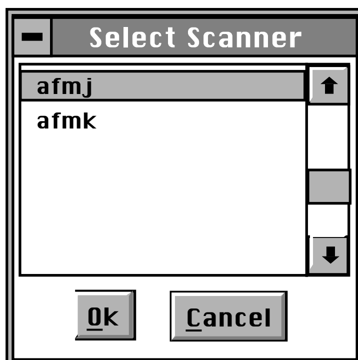
5.3.1. Select microscope and scanner parameters.

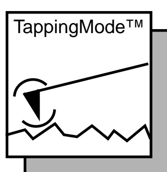
This step needs to be completed only when your NanoScope system has more than one micorscope or more than one scanner. Set the microscope head configuration by using the mouse in the **d** / **Microscope Select** menu**.**



Click on **Ok** using the mouse to close the **Microscope Select** dialog box.

Set the scanner configuration by using the mouse to select a scanner in the **Microscope / Scanner** menu.

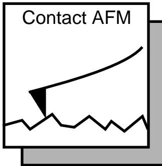




For TappingMode, set the **Microscope Mode** to **Tapping.** This is done by clicking on **Microscope mode** in the **Other Controls** panel. After this is completed, return

to the **Scan Controls** panel to access the most important control settings used for imaging.

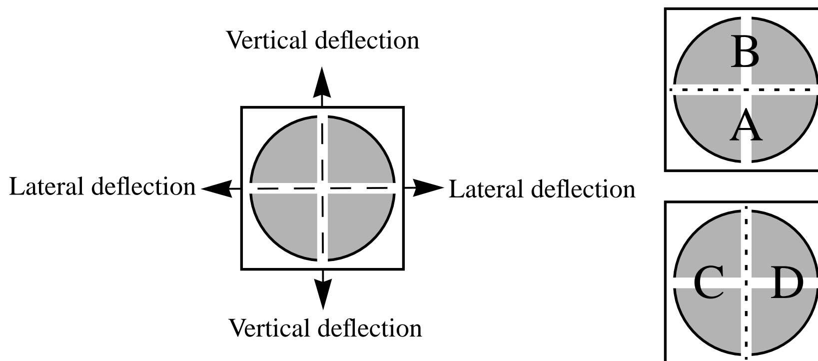
For contact AFM, set the **Microscope Mode** parameter on the **Other Controls** panel to **Contact.** After this is completed, click on **Scan Controls** to again bring up the most important control settings used for imaging.



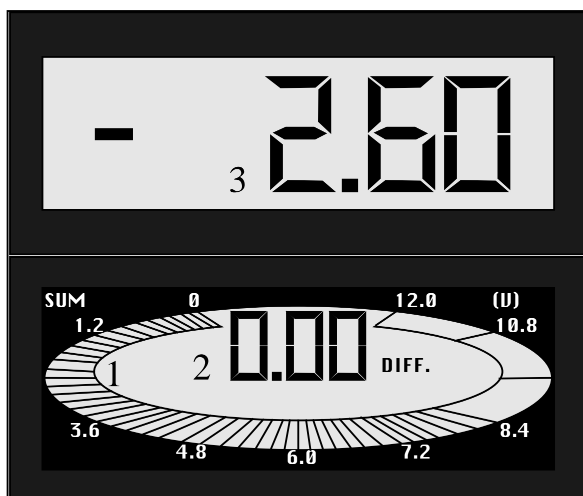
5.4. MultiMode SPM Voltage Meters

A complete description of the signals coming into and out of the MultiMode SPM is available in Support Note 210, *NanoScope Signal Access Module (SAM)—Description and Use*. The SAM is normally used for accessing these signals directly. A brief description of the SPM's voltages and their interpretation using the meters on the front of the base is provided here for quick reference.

The MultiMode SPM base is equipped with meters which indicate voltage coming from the four-segment photodetector. The photodetector array is represented here:



The MultiMode SPM's bottom, elliptical (**SUM**) meter 1 indicates the *total voltage generated by the photodetector*. That is, the combined voltage of photodetector segments. This is displayed during all modes (except STM when all meters are off).



TAPPING MODE
or CONTACT AFM
OUTPUT
SIGNAL (V)

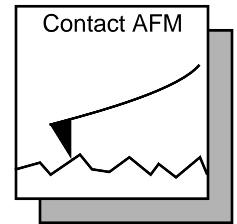
VERTICAL
or HORIZONTAL
DIFFERENCE

SUM

The bottom digital meter 2 reads *differences in voltage between various segments of the photodetector*. With the mode switch toggled to **AFM & LFM**, it indicates the voltage difference (C - D), that is, the left segments minus the right segments. With the mode switch toggled to **TM AFM** (TappingMode), it indicates the voltage difference (A - B), that is, the bottom segments minus the top segments.

The topmost digital meter 3 indicates the *output signal of the SPM*. Depending upon the mode selected, the topmost meter reads either the (A - B) voltage difference (mode switch toggled to **AFM & LFM**), or the RMS voltage (mode switch toggled to **TM AFM**).

Chapter 6 Contact AFM



6.1. Introduction to Contact AFM

This chapter covers procedures for operating the MultiMode SPM in contact AFM mode. It is assumed the operator has previously prepared a contact AFM probe and aligned the MultiMode head per instructions provided in Chapter 5 of this manual. Specific information regarding tip preparation is provided in Chapter 4; Appendix A of the *Command Reference Manual* contains further information regarding tips.

6.2. Preparation Prior to Imaging

6.2.1. Adjust the Detector Offsets

Verify that the MultiMode head has been fitted with a contact AFM probe tip per instructions provided in Chapter 5 of this manual. The laser beam should already be positioned on the back of the cantilever. This will provide a starting point for adjusting the laser sum value.

To adjust the detector setting, first review the operation of the various adjustment screws. The detector adjustment screws are at the left side of the head. The upper-left screw moves the laser spot up and down as shown on the computer screen detector graphic, and side to side (long axis) on the dark red laser spot screen directly on the SPM head. The lower-left screw moves the laser spot right-to-left as

shown on the computer screen detector graphic and top-to-bottom (short axis) on the dark red laser spot screen mounted into the SPM head.

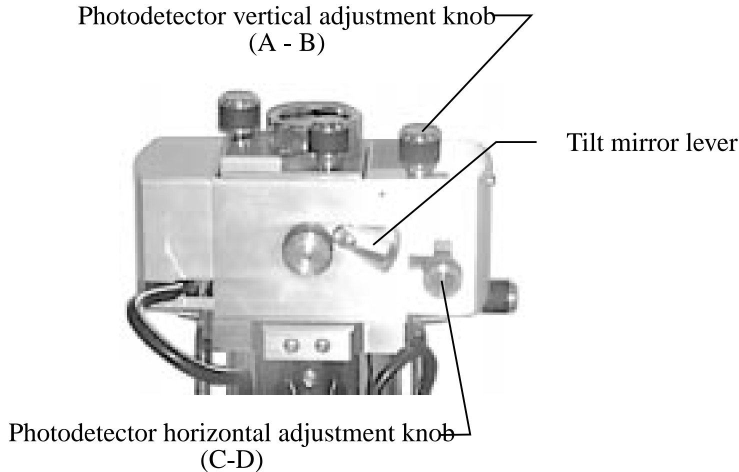


Figure 6.1. Photodetector mirror adjustments—rear view.

Refer to figure 6.2. For laser aligning screws atop the SPM head, the right-front screw moves the laser spot left-to-right (horizontally or along the X-axis). Turning this screw clockwise moves the laser spot to the right. The left-rear laser aligning screw moves the laser spot top-to-bottom (vertically along the Y-axis). Turning this screw clockwise moves the laser spot rearward.

Notes

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

For initial adjustment, center the spot on the cantilever as described in Chapter 5.

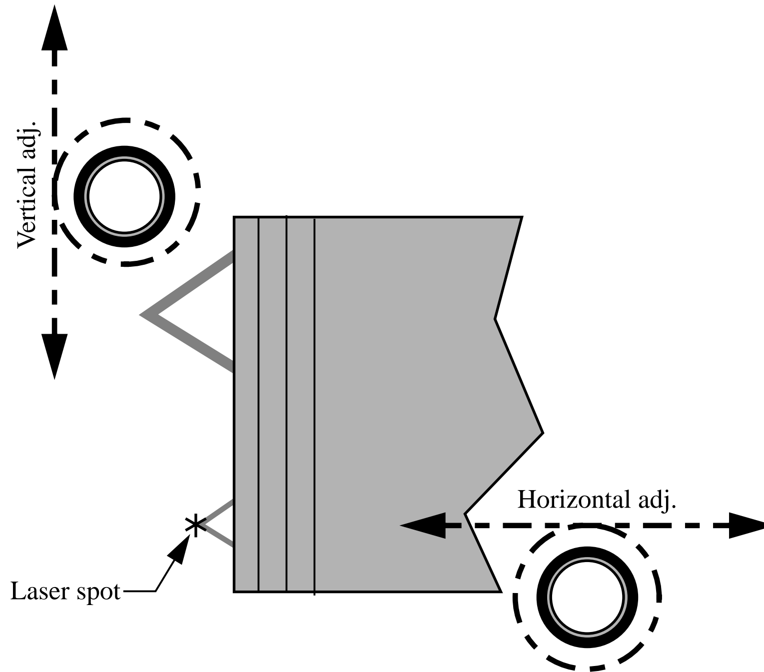


Figure 6.2. Laser adjustment knobs—top view.

6.2.2. Signal settings

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

Notes

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

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

6.2.3. Adjust tip height above sample surface

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

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

6.3. Suggested Initial Control Settings

6.3.1. Initial Settings: *Scan*, *Feedback* and *Other Controls* panels

Before making changes to **Scan Controls** panel screen parameters, go to the **Other Controls** panel (**Panels / Other**) and verify that the **AFM Mode** parameter is set to **Contact**.

Click on **Panels / Scan**. Set the **Scan size** as large as desired (up to approximately 85-100 µm with a "J" scanner) and set the **X** and **Y offsets** to 0.0. Set the **Scan angle** to **0.0 degrees**, **Scan rate** to **2-3 Hz** (for single crystal silicon contact cantilevers 450 µm long you may want to set this to **1.5-2 Hz**). When starting, set the **Number of samples** to **256** to expedite setup; later, the value may be increased to **512** for better image clarity. Make sure the **Slow scan axis** is **Enabled**. Set the **Z Limit to** 440 volts (if not already set)

Next, click on **Panels / Feedback.** Set both **Integral** and **Proportional gain** to **2.0** each and the **Setpoint** to **0.0 Volts**. The **Feedback Controls** screen should appear as shown below.

Feedback Controls	
Integral gain:	2.00
Proportional gain:	2.00
LookAhead gain:	0.00
Deflection Setpoint:	0.00 V

Figure 6.3. ***Feedback Controls*** **settings for initial setup (contact AFM).**

6.3.2. Other Controls Panel

Using the mouse, select **Panels / Other.** Verify that the **AFM mode** is set to **Contact.** Set **Units** to **Metric**, **Color table** to **2**, and **Input attenuation** to **1x.** (See example below.)

Other Controls	
Microscope mode:	Contact
Z limit:	2.000 µm
Units:	Metric
Color Table:	2

Figure 6.4. ***Other Controls*** **settings for initial setup (contact AFM).**

6.3.3. *Interleave Controls* panel

Next, go to **Interleave Controls** by clicking with the mouse on **Panels / Interleave**. Verify that the **Interleave mode** field is set to **Disabled**. (Do not attempt to set **Interleave mode** to **Enabled** at this point.)

6.3.4. *Channel 1*, *2* and *3*

On the **Channel 1** panel, set **Data type** to **Height** (see below). Set **Z range** to a reasonable value for the sample. (E.g., for the 180 nm step height calibration sample a reasonable Z range setting would be 300 nm initially). **Line direction** can be set to either **Trace** or **Retrace**. On the **Channel 2 panel,** set **Data type** to **Off** to disable the secondary channel.

Channel 1	
Data type:	Height
Data scale:	300 nm
Line direction:	Trace
Scan line:	Main
Realtime Planefit:	Line
Offline Planefit:	Full
Highpass filter:	Off
Lowpass filter:	Off

Figure 6.5. ***Channel 1*** **panel for initial setup (contact AFM).**

6.4. *Initiate the* ***Engage*** *Command*

Go to the upper menu bar selection **Motor**, and click on **Motor**, followed by **Engage** (or click on the **Engage** icon). A pre-engage check, followed by Z-stage motor sound should be observed. If for any reason the engage aborts because the SPM head is still too far away from the surface, click on the **Abort** button and readjust the screws to start the tip closer to the sample surface. Assuming the tip is better positioned before engaging again, an image should begin to appear on the image monitor.

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

6.4.1. Adjust *Setpoint* with *Force Calibration*

The most accurate way to minimize the **Setpoint** value is by using Force Calibration (**Real Time / View / Force Mode / Plot**). This consists of obtaining a force plot, then adjusting the **Setpoint** value until the tip almost pulls free of the surface. In this way, it is possible to minimize tip-sample forces and reduce wear on both the tip and surface.

Getting a good Force Calibration plot.

Force plots are discussed in detail in Chapter 11 of this manual. For standard force calibration plots, start with the following parameter values:

Panel	Parameter	Setting
Z Scan Controls	Scan Size	500 nm —1 µm[a]
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	9.00 Hz
	Number of samples	512
	Average count	1
	Display mode	Both
	Trigger mode	Off
	Start mode	Calibrate
Channel 1	Data type	Deflection
	Z range	(Set to maximum)

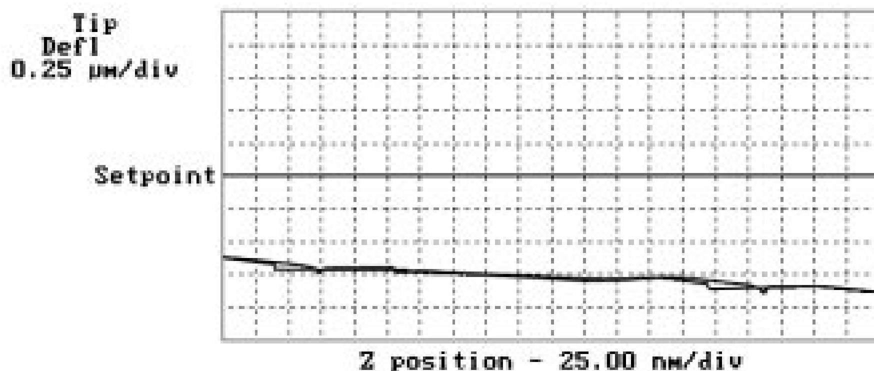
a. A good Z-axis scan size is from 500 nm to 1 µm. Scans larger than 1 µm may diminish details and make them hard to see. Scans smaller than 500 nm may be too small and make it difficult to pull the tip clear of the surface.

What is happening?

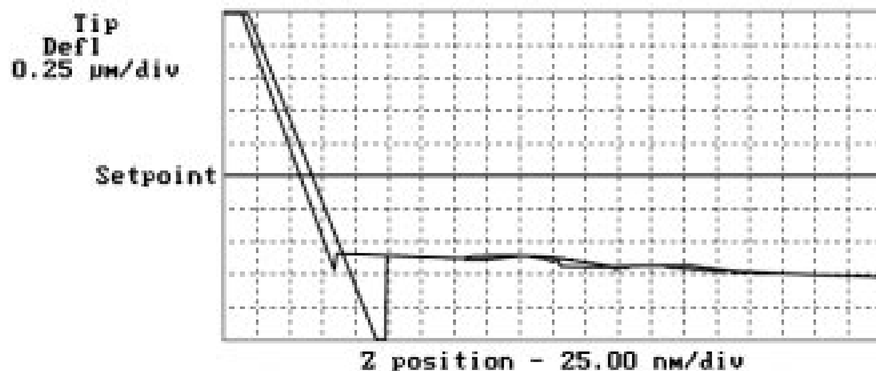
The piezo is being retracted from the **Z scan start** position to the **Z scan start**-plus-**Scan size** position. The **Z scan start** point is at the leftmost portion of the plot. The **Z scan start**-plus-**Scan size** point is the rightmost portion of the plot. This corresponds to the **Z Center Position** that was being used while scanning just before starting Force Calibration. This means that the **Z scan start** is recalculated at every cycle to Force Calibration. The **Scan size** will be automatically changed if its value is bigger than the difference between the **Z Center Position** and the fully retracted position.

Procedure

When you first enter forcecal the graph will probably look like this:

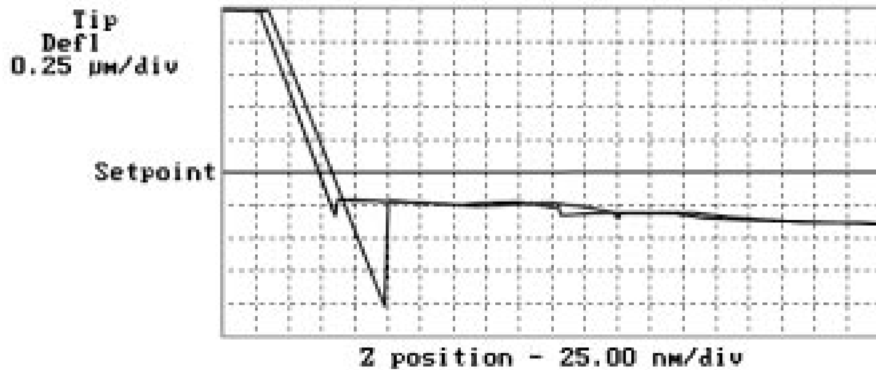


The tip is being moved up and down above the surface without touching it. If the line is railed at the top or bottom of the image click on the **Setpoint 0** button in the **Feedback Controls** panel. Then you should see the above graph. To get it to touch the surface you need to increase the **Scan start**. Doing this with an arrow key works well. After a few key presses, you should see a graph similar to the one shown here:



If the line slants to the bottom of the graph and stays there, the tip is sticking to the surface and not popping off the surface when the tip is fully retracted. To get the tip clear of the surface, increase the **Scan size** until you can see it come off.

Now adjust the **Setpoint** for imaging. Having the setpoint anywhere above the line where the tip is off the surface will work. The farther it is above, the more force is placed onto the sample.



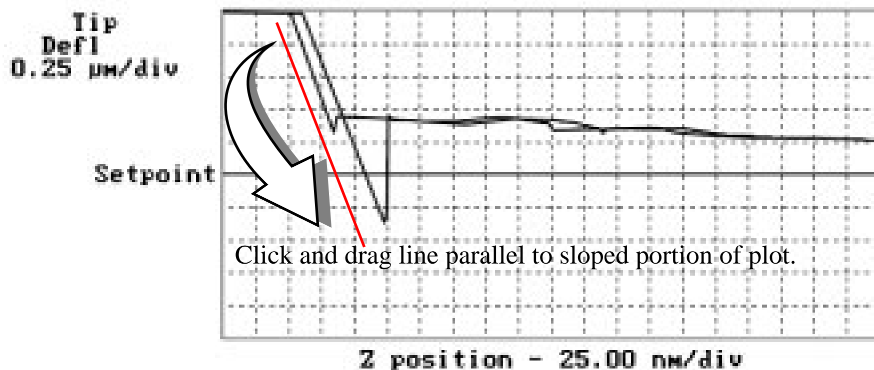
To adjust tip-sample force to the minimum amount, you can run in the area where the tip is actually pulling up but the liquid layer is holding the tip on the surface. However, this is not a stable way to image: if the tip pops free of the surface, the **Setpoint** must be increased to reattach the tip.

To minimize tip-sample forces, lower the **Setpoint** until the tip pulls free of the surface and record the **Setpoint** value: this is the pulloff value. Increase the **Setpoint** again to regain the surface, then lower the **Setpoint** to a point just above the pulloff value. At this point, tip-sample forces should be at their minimum.

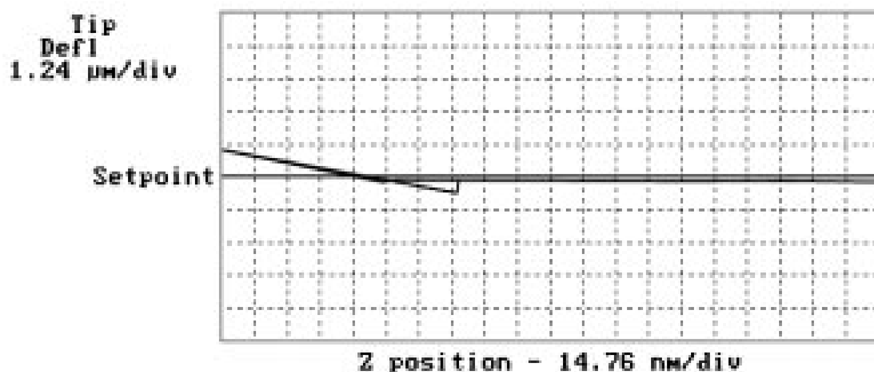
If **Setpoint** is the only parameter requiring adjustment, skip ahead to section 6.5 below; otherwise, go to the next section to adjust the SPM's **Sensitivity** parameter.

6.4.2. Adjust *Sensitivity* (if required).

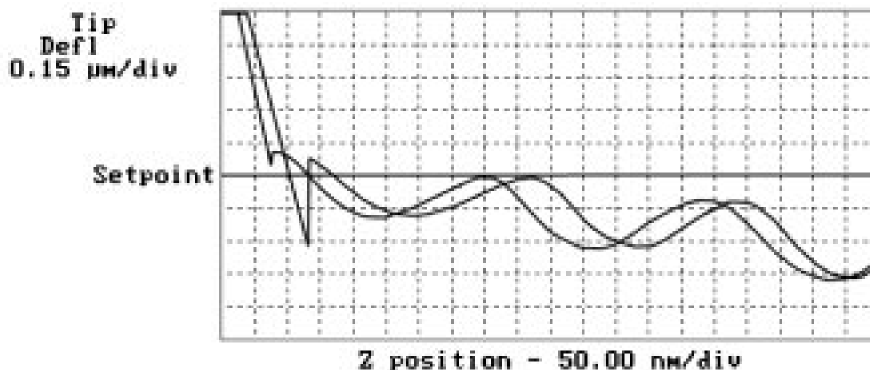
If imaging in **Deflection** mode instead of **Height** mode, adjust the detector's **Sensitivity** parameter to the cantilever as described in this section. Use the mouse to draw a line parallel to the part of the plot where the tip is on the surface. To clear the screen, click the mouse's right button while in the graph.



If you want to see more graph range you can change the **Input attenuation** to **8x**. The graph will not change in appearance until you make the graph range larger. It can now be made eight times larger. With this you can see the bottom of those big pulldowns. Be sure to change the **Input attenuation** back to **1x** before imaging.



If there are periodic waves in the plot of the tip in free air, these are caused by optical interference. Some of this is acceptable but it can be bad enough to be a problem. The only way to fix this without changing tips is to adjust the aim of the laser. This can be done while in **Force Cal**.



6.5. Beyond the Basics of AFM Operation

The above section provided a quick introduction to setup of the MultiMode microscope in the contact AFM mode. Although a great deal can be accomplished from this starting point, there is far more to the operation of the AFM. More regarding tip-sample interactions can be learned in Chapter 1 of the *Command Reference Manual*. Section 11.2 in this instructional manual, "Contact AFM Force Plots," contains valuable information regarding the precise measurement of tip-sample forces.

Cantilever selection is an important area and it will become more important as tip and cantilever technology continues to develop. A clear understanding of the parameters in the Real Time control panel allows the user to tune the microscope to a wide variety of samples. Force mode imaging provides a great deal of information if the user fully understands its nuances. For more information regarding force mode imaging, see Chapter 11 in this manual.

6.5.1. Cantilever selection

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

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

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

Cantilever Type	**k (N/m) (narrow legs)**	**k (N/m) (wide legs)**
100 micron (triangular)	.38	.58
200 micron (triangular)	.06	.12

Table 6.1. Cantilever spring constants

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

6.6. Optimization of Scanning Parameters

Careful selection of the scan parameters is important to the successful application of contact AFM. In most cases, the optimal parameter selection depends on the sample. All parameters in the Real Time control panel are discussed in the NanoScope III *Command Reference Manual*. The user is encouraged to review these descriptions carefully. This section analyzes the effects of the most important parameters.

6.6.1. Data type

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

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

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

6.6.2. Gain settings

The **Integral**, **Proportional**, and **LookAhead** gains in the **Feedback Controls** panel control the feedback on the piezo height. The feedback loop tries to keep the deflection signal constant by adjusting the height of the piezo tube. If the gains are high, as they should be for **Height** data, the piezo height will change to keep the cantilever deflection nearly constant. If the gains are low, as they should be for topographical **Deflection** data, the cantilever will deflect from its nominal position as features in the sample are encountered. In general, the **Integral** and **Proportional gain** can be set to **2–3** to start scanning. To optimize the gains for height data, increase the **Integral gain** until the piezo begins to oscillate, then eliminate the oscillations by reducing the gain with two or three clicks of the left-arrow key. Repeat the process for the **Proportional gain**. Piezo oscillations typically cause high frequency wavy lines in the Real Time image. For deflection data, engage the microscope with the gains high, then lower them as much as possible once the system is scanning.

The **LookAhead gain** includes information from the previous scan line to determine the current gain setting. It should only be used on samples with step-like features which are oriented perpendicular to the fast scan direction. Otherwise, it should be left at **0**.

Notes

- **LookAhead gain** controls are not implemented on some software releases. For more information, contact Digital Instruments' software department.

6.6.3. *Scan size* and *Scan rate*

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

6.6.4. Setpoint

The **Setpoint** parameter defines the desired voltage (and, therefore, the desired deflection of the cantilever) for the feedback loop. The setpoint voltage is constantly compared to the present photodiode cantilever deflection voltage to calculate the desired change in the piezo position. When the gain values are high, as they should be when the **Data type** is set to **Height**, the Z piezo position changes to keep the photodiode output signal close to the **Setpoint**; therefore, the cantilever deflection remains nearly constant. When the gain values are low as they should be when the **Data type** is set to **Deflection**, the piezo height does not change, and the photodiode signal varies around the **Setpoint** value.

In contact AFM, increased **Setpoint** yields higher tip-sample forces. The **Setpoint** can be adjusted to increase or decrease the cantilever deflection and, therefore, the contact force of the tip on the sample. The **Force Calibration** command in the **Real Time / View** menu allows the setpoint to be adjusted while viewing a graph of the tip position versus the deflection voltage. Using this procedure, which is described in detail in the next portion of this section, the contact force of the tip on the sample can be minimized. This is especially important on soft materials such as biological samples.

6.6.5. Lowpass filter

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

6.6.6. Highpass filter

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

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Chapter 7 Contact AFM in Fluids

7.1. Fluid Operation—Overview

An ever-increasing realm for SPM technology is the imaging of samples in fluid. This may be prompted from a desire to minimize surface forces on delicate samples, the need to observe biological specimens in their natural, fluid environments, and/or the necessity to make real time observations of samples undergoing electrochemical reactions (ECAFM). In order to conduct ECAFM observations with electrical potentials, it is necessary to connect an external potentiostat unit. For more information, contact Digital Instruments.

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

ATTENTION! En milieu liquide, prendre toutes les précautions pour éviter une fuite qui pourrait atteindre la platine porte -échantillon, le boitier électronique ou tout autre partie électronique du microscope. Eviter tout contact avec un liquide corrosif.

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

Essentially, the procedure for observing samples under fluid is the same as that for contact AFM in air; however, special hardware is utilized to contain the fluid. In addition, minor adjustments must be made to correct for refractive effects as the laser beam transits air-fluid boundaries. One feature of imaging samples under fluid is that attractive forces due to surface tension effects are eliminated. This enables the sample surface to be imaged with a minimum of cantilever tip force—a decided advantage when imaging biological specimens and delicate materials.

Images of submerged samples may be obtained in TappingMode; however, the drive frequency and amplitude of the cantilever must be reduced due to viscous damping of the fluid. For more information, see Chapter 8. Chapter 8 includes a procedure (section 8.8.3) that should be studied for *all* types of fluid imaging.

This section describes contact AFM operation of the MultiMode SPM in fluid, including loading the cantilever into the tipholder, mounting the tipholder into the head, aligning the laser onto the cantilever and then engaging the tip with the sample. There is also a discussion of methods of preparing the sample for fluid operation. This section assumes familiarity with contact AFM operation of the MultiMode in air. If you are not familiar with air operation of the MultiMode, please first follow the procedures outlined in Chapter 6 before attempting to operate the AFM under fluid.

7.2. Fluid Operation—Hardware

IMPORTANT: Do not attempt to operate the standard air tipholder in a fluid environment. Standard tipholders have exposed electrical signal lines that could short circuit if exposed to a conducting fluid.

ATTENTION: En milieu liquide, ne pas utiliser le support de bras de levier standard prévu pour une utilisation à l’air. Le support de bras de levier standard comporte des fils électroniques non protégés qui peuvent provoquer un court-circuit en cas de contact avec un liquide conducteur.

WARNHINWEISS: 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.

Fluid imaging requires a special tipholder (Figure 7.1). The fluid tipholder interchanges with the standard tipholder in a matter of a few minutes.

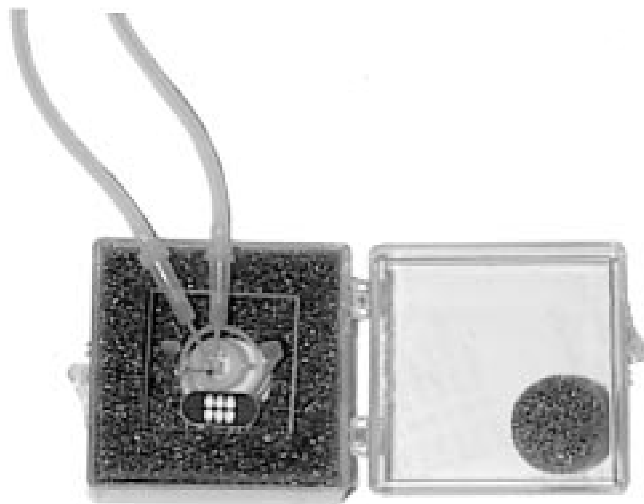


Figure 7.1. Fluid (TappingMode) Tipholder in Case

The fluid tipholder consists of a small glass assembly with a wire clip for holding an AFM cantilever substrate. The glass surfaces provide a flat, beveled interface so that the AFM laser beam may pass into the fluid without being distorted by an unstable fluid surface.

7.3. Notes on Sample Mounting

In many cases, it may be desirable to image a sample under a drop of fluid without use of a closed fluid cell. Simply attach the sample to a 12 mm puck, then put the drop of fluid onto the sample and lower the AFM fluid tipholder into the drop. Without the o-ring, this approach poses a potential spill hazard to the microscope's electronics and must always be undertaken with extreme caution. To help protect the scanner, always use the large size (12 mm) puck. If the sample surface is hydro-

phobic, it should hold a fairly substantial droplet without difficulty. If the sample surface is hydrophilic and/or porous, it may tend to absorb fluids and wet the scanner. In this case, it will have to be encapsulated within a fluid cell.

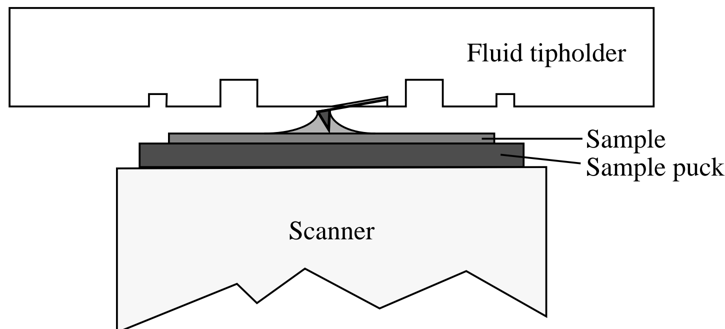


Figure 7.2. Imaging a Sample Covered by a Drop of Fluid

Attach the sample rigidly to the puck. Samples may be secured to the puck with adhesive tape or epoxy. For non-critical applications, Devcon 2-Ton Epoxy works well. For applications where contamination control is more critical, try a more inert, solvent-free epoxy like Master Bond EP21LV or EP21AR (phone number 201-343-8983) or various hot melt adhesives. Be sure to 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 also desirable to image a sample quickly without waiting for an adhesive to cure, you may find it sufficient to hold the sample to the sample holder using double-sided tape. We recommend against using any cyanoacrylate glue (like Superglue) for mounting samples in fluid.

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

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

WARHINWEISS! Falls Sie Proben in Flüssigkeiten abbilden, lassen Sie äußerste Vorsicht walten, damit keine Flüssigkeit verspritzt wird. Flüssigkeiten dürfen nicht

auf die oder nahe der Probenhalterung, der Elektronikbox oder anderen Komponenten, die elektronische Bauteile enthalten, verspritzt werden. Vermeiden Sie bitte, korrosive Flüssigkeiten auf freiliegende Oberflächen zu verspritzen; andernfalls wären Beschädigungen die Folge! Falls Sie Flüssigkeit verspritzt haben, säubern und trocknen Sie alle betroffenen Flächen sorgfältig.

7.4. Fluid Operation—Procedure

7.4.1. Load the Fluid Tipholder with a Cantilever Substrate

The cantilever substrate is held in a small pocket on the bottom side of the tipholder by a gold-plated, stainless steel wire clip. The wire clip is held against the cantilever substrate by a tiny coil spring mounted on the top of the tipholder. To load a cantilever substrate into the holder, turn the tipholder upside down and gently lift the wire clip upward by pressing from beneath. With the wire clip raised, slide a cantilever substrate under the wire and into the pocket. Check that the cantilever substrate is squarely set against one side of the pocket and flush against the back. Avoid scratching the tipholder's glass surface with the tweezers or the cantilever substrate, especially in the area under the cantilever itself. Check that the cantilever substrate is held firmly by the wire.

7.4.2. Install the Tipholder on the Microscope Head

Verify that the sample is attached firmly to a 12 mm puck and loaded atop the scanner. The head must be raised adequately to clear the sample with the tip. If the head is positioned too low, toggle the tip **Up** switch and use coarse adjustment screws to raise the head adequately. Insert the tipholder carefully into the head, careful not to contact the sample.

Figure 7.3. Tipholder Inserted into Microscope Head

7.4.3. Align the laser

Laser alignment is accomplished by either of the procedures described in Chapter 5 in this manual. Note that alignment of an immersed cantilever is slightly different than in air, since refractive effects cause the beam path to bend slightly. The basic process is essentially the same, however. The following instructions give an abbreviated procedure, focusing on the differences or additional techniques required for fluid operation.

7.4.4. Watch Out for “False” Reflections

The fluid tipholder is constructed such that the cantilever substrate rests flat on an angled, glass surface. The angled glass surface produces a false laser reflection, even when the laser is not aimed at the cantilever. This reflection from the glass surface does not affect operation of the MultiMode, but it can be a source of confusion when aligning the laser on the cantilever. Ignore this fainter reflection and watch instead for the much brighter reflection from the cantilever.

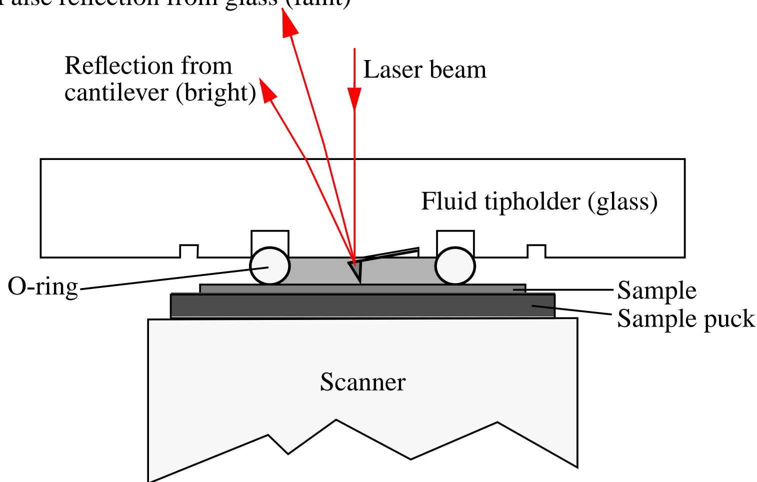


Figure 7.4. False Reflections from Bottom Surface of Fluid Tipholder

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

7.4.5. Align the Laser on the Cantilever

Now use either of the techniques for aligning the laser onto the cantilever that are discussed in Chapter 5 of this manual. Initially, most users find using the optical magnifier the easiest.

7.4.6. Install the Protective O-ring and Flood the Fluid Chamber

The o-ring is used to protect the SPM scanner tube from spilled liquids. It should be inserted into the gland (recessed groove) which is ground into the underside of the tipholder. The o-ring is designed to slide up into the gland as the tipholder is lowered until the tip accesses the sample surface.

Fill a syringe with the fluid to be used for imaging, then chase out all air bubbles. (Injected air bubbles can become trapped inside the fluid chamber and disrupt imaging.) Connect the syringe to the inlet tubing on the tipholder. The outlet tube should be connected to a container where fluid can drain. Push enough fluid through the fluid chamber to flood it completely, but do not clamp the outlet tube.

7.4.7. Lower Tipholder into Fluid

Carefully tighten the clamping screw to secure the tipholder, making certain that the o-ring is positioned properly between the sample and tipholder. If the o-ring attempts to shift off, remove the tipholder and try repositioning the o-ring. If shifting continues, see Chapter 15 in this manual for help. Try clamping the outlet tube. Clamping is not necessary and may effect noise in the image.

7.4.8. Readjust Laser Aiming (If Required)

The presence of fluid may cause the laser spot to refract slightly, requiring slight readjust of the laser aiming screw to move the laser spot back onto the end of the cantilever.

Occasionally, air bubbles will become trapped near the cantilever which interfere with the laser beam's optical path. If this occurs, it may be impossible to get a good reflection from the cantilever after flooding the tipholder with fluid. In this case, use the syringe to force liquid quickly through the cell to try to break the bubbles loose from the tip.

7.4.9. Adjust the Detector Offsets and Setpoint

Turn the detector mirror adjustment screws to center the laser spot on the laser detector as described in section 3.3.1. Typical starting conditions might be to set the vertical deflection signal to roughly -1.0 V and the **Setpoint** to 0 V. Bear in mind that 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.

Typically, samples are softer in liquid than in air, so be careful that there is not too large a difference between the setpoint and the vertical deflection signal before engaging. Also, it may be desirable to reduce the setpoint once engaged to obtain minimum tracking force.

7.4.10. Check Scan Parameters

Check that the various scan parameters such as **Scan rate**, **Scan size**, **Integral gain** and **Proportional gain**, etc. are reasonable.

7.4.11. Engage the Surface

While monitoring closely through the 25X magnifier, use the coarse adjustment screws and motor **Down** switch (on the MultiMode base) to bring the tip just above the level of the sample surface. Under the **Real Time / Motor** menu, click on **Engage** (or click on the **Engage** icon). The motor will begin to move the SPM head and probe down towards the sample. When the cantilever reaches the surface, the system should automatically stop, beep, and then begin to image the sample. In the event that the system engages immediately or before the tipholder actually reaches the surface, try increasing the **Setpoint** approximately 2.0 Volts, then try again.

If other difficulties are encountered, see the Troubleshooting section of this manual.

7.4.12. Adjust Scan Parameters

Once engaged, adjust the scan parameters to obtain the best image. This procedure will be similar to operation in air except for the following cautions:

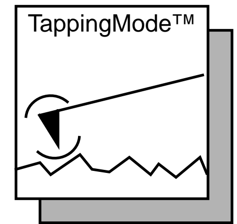
1. Samples are often softer in fluid and so adjusting the applied force can be much more critical. To avoid sample damage, adjust the **Setpoint** as low as possible. To do this reduce the setpoint using the cursor keys until the cantilever pulls off the surface and the **Z Center Position** on the display monitor jumps to **Limit** (-220 V). Increase the **Setpoint** slightly until the cantilever begins to touch the surface again and an image appears. Another way is to use the **Force Calibration** command to select the **Setpoint** and estimate the contact force, as described in Chapter 11. Note that since the cantilever will typically adhere to the sample surface much less in fluid, it is often possible to image at much smaller contact forces in liquid than in air.

2. The optimal **Integral** and **Proportional gains** and **Scan rate** may be different from air operation since the dynamics of the cantilever will change in fluid. Set the two gains as high as possible (starting with the **Integral gain**) without causing oscillations to appear in your image. Choose a **Scan rate** that is sufficiently slow to image without distorting your data.

7.4.13. Clean and Dry Parts When Done

When sample imaging is complete, drain the fluid tipholder and carefully remove it from the head while avoiding spills. Rinse and dry the tipholder and o-ring to prevent the buildup of salts or other contaminants on these parts. When cleaning the tipholder, take great care to avoid scratching the glass surfaces in the center of the tipholder.

Chapter 8 TappingMode AFM



8.1. Introduction to TappingMode™ AFM

This chapter covers procedures for operating the MultiMode SPM in TappingMode for both air and in fluids (section 8.8). It is assumed that the operator has previously prepared a TappingMode probe tip and aligned the SPM head per instructions provided in Chapter 5 of this manual. Additional information regarding cantilever preparation is provided in Chapter 4.

Note:

TappingMode is disabled for users of NanoScope E configurations.

8.2. Basic Principle of TappingMode

Figure 8.1 represents a cantilever oscillating in free air at its resonant frequency. A piezo stack excites the cantilever's substrate vertically, causing the tip to bounce up and down. As the cantilever bounces vertically, the reflected laser beam is deflected in a regular pattern over a photodiode array, generating a sinusoidal electronic signal. The signal is converted to a root mean square (RMS) amplitude value, which is displayed in AC volts on the topmost ("TappingMode Output Signal") meter located on the front of the MultiMode base.

Figure 8.2 represents the same cantilever at the sample surface. Although the piezo stack continues to excite the cantilever's substrate with the same energy, the tip is deflected in its encounter with the surface. The reflected laser beam ("return sig-

nal") reveals information about the vertical height of the sample surface and some characteristics of the sample material itself. These material characteristics may include elasticity ("hardness"), magnetic and/or electric forces present.

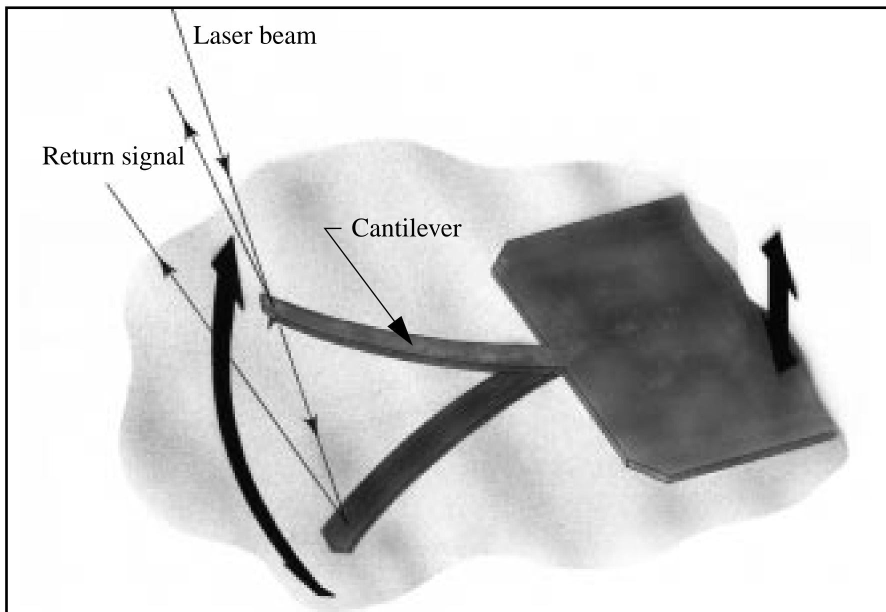


Figure 8.1. Tapping cantilever in free air.

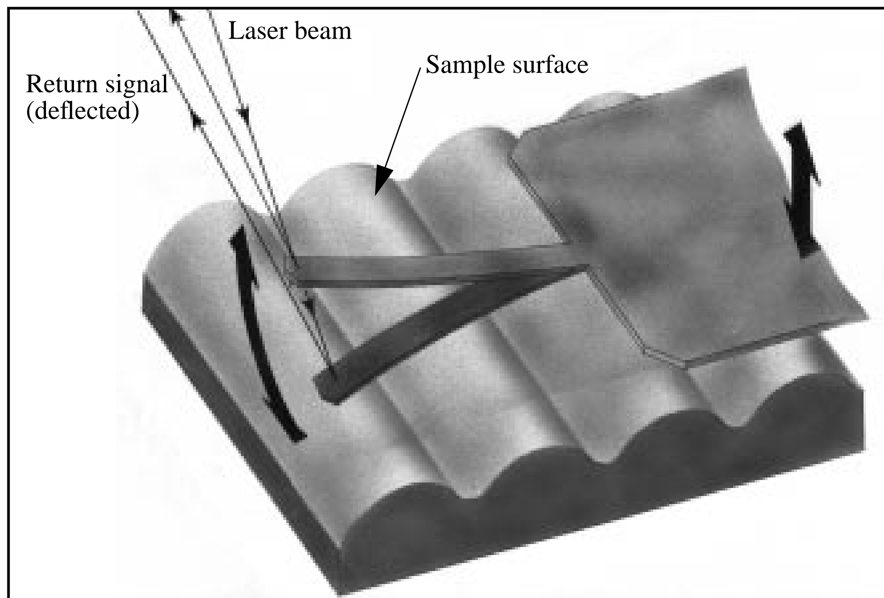


Figure 8.2. Tapping cantilever on sample surface.
Note deflection of cantilever and return signal (exaggerated).

8.3. Preparation Prior to Imaging

8.3.1. Switch to TappingMode and check parameters

The microscope must be switched to TappingMode. Go to the **Real Time / Other Controls** panel and set the **AFM mode** parameter to **Tapping**. Toggle the selector switch on the left side of the MultiMode base to **TM AFM**; the tiny LED indicator on the front of the base should glow green.

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

Panel	Parameter	Setting
Scan Controls	Scan Size	**25 µm**
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	1.00 Hz
	Number of samples	256
	Slow scan axis	Enabled
	Z limit	440 V
Feedback Controls	Integral gain	0.300
	Proportional gain	0.300
	LookAhead gain	0.00
	Setpoint	[a]
	Drive frequency	200–400 kHz[b]
	Drive amplitude	50—800 mV
Other Controls	AFM mode	Tapping
	Input attenuation	1x
	Engage Setpoint	1.00
Interleave Controls	Interleave mode	Disabled

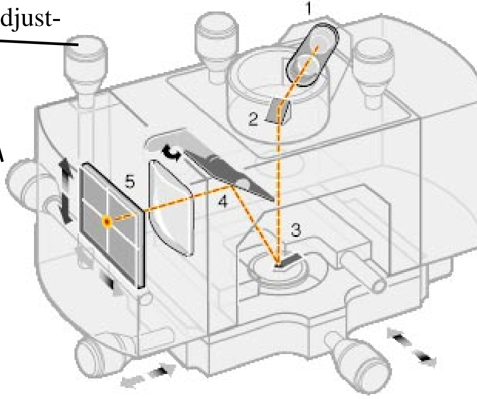
a. Setpoint is initially set by the software during engagement. It may be adjusted up or down afterward.

b. The **Drive frequency** varies according to the length and stiffness of each cantilever. TappingMode cantilevers are shipped in boxes stamped with their resonant frequency. The **Drive frequency** should be set close to (± 50 kHz) this value.

8.3.2. Adjust laser and photodetector

Verify that the tipholder has been fitted with a TappingMode, single crystal silicon probe and aligned per instructions provided in Chapter 5 of this manual. Photodetector voltage values are displayed on meters mounted on the front of the MultiMode base. The laser photodetector is adjusted using the photodetector adjustment knobs on the left-top and left-rear of the head (see Figure 8.3 below). Recall that

RMS amplitude is an AC signal which does not have any real meaning until cantilever tuning is completed (see section 8.3.3). The laser spot will need to be approximately centered prior to entering the **Cantilever Tune** routine.



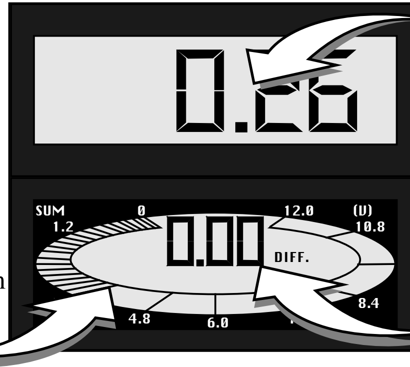
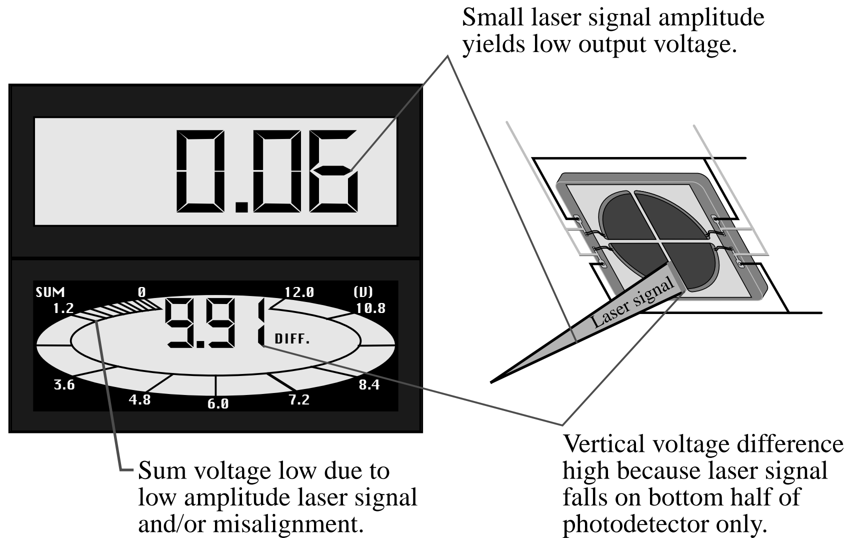


Figure 8.3. Photodetector adjustment knobs and SPM voltage meters.



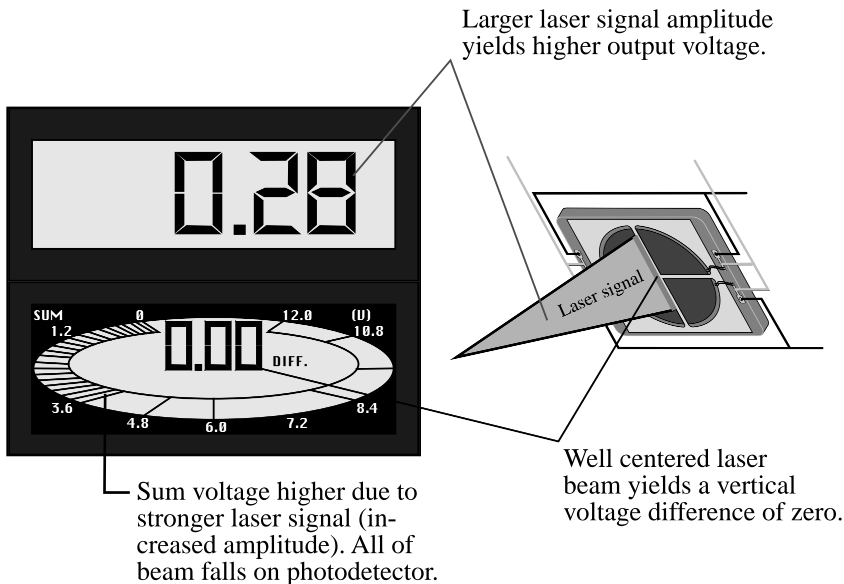


Figure 8.4. Voltage meters on the MultiMode base reveal a great deal about the amplitude and alignment of the TappingMode laser signal on a tuned tip operating at its resonant frequency.

8.3.3. Additional preparations

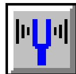
In TappingMode, the AC voltage signal representing RMS amplitude is used to provide the dynamic feedback signal for surface height tracking. The vertical deflection signal (displayed on the "Vertical or Horizontal Difference" meter) should be close to zero (± 1.0 V) prior to running **Cantilever Tune**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

Panel	Parameter	Setting
Channel 1	Data type	Height
	Z range	0—4.50 µm[a]
	Line direction	Trace
	Scan line	Main
	Real-time Planefit	Line
	Off-line planefit	Full
	High-pass filter	Off
	Low-pass filter	Off

a. A **Z range** value of 4.50 µm is the maximum for most "J" scanners. Always start with a low **Z range** value if feature height is unknown, then increase until the image is optimized. On the 180 nm step height calibration sample, a reasonable **Z range** setting would be **300 nm** initially

8.3.4. Tune the cantilever

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



1. Click on **View / Sweep / Cantilever Tune**, or use the Cantilever Tune icon. Several panels will appear...

Auto Tune Controls

Auto Tune Controls
Start frequency: 100.000 kHz
End frequency: 500.000 kHz
Target amplitude: 2.000 V
Peak offset: 0.00 %
Auto Tune

Sweep Controls

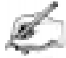
Graph Controls
Sweep width: 400.000 kHz
Drive frequency: 300.000 kHz
Sweep sample count: 256

Main Controls
Amplitude setpoint: 0.00 V
Drive amplitude: 300 mV

Motor
Interleave Controls

...while the **Frequency Sweep** ((a plot of cantilever response as a function of applied vibrational frequency) is shown on the display monitor. The two main panels, **Sweep Controls** and **Auto Tune Controls,** allow the operator to either manually or automatically tune the cantilever. For most purposes, the **Auto Tune** function will suffice. To tune the cantilever automatically, simply click on the **Auto Tune** button; the computer and controller will do the rest, setting such parameters as **Setpoint** and **Drive amplitude** automatically.

At times, it may be useful to tune the cantilever manually, for example, to purposefully offset the drive frequency above or below the resonant frequency as is often done during MFM and EFM imaging.



Note:

More than one type of cantilever exists. Different types have different dimensions and therefore different resonance frequencies. Check the box used to ship the cantilever to see what its resonant frequency is, or refer to *Appendix A* in the *Command Reference Manual*.

Tuning cantilevers manually

The parameter values, especially the drive frequency and the sweep width, given in the following example apply to one type of cantilever. The nominal parameter values may vary depending upon the actual cantilever used.

For initial set-up, the **Sweep Controls** screens should be set to the values shown in the example below.

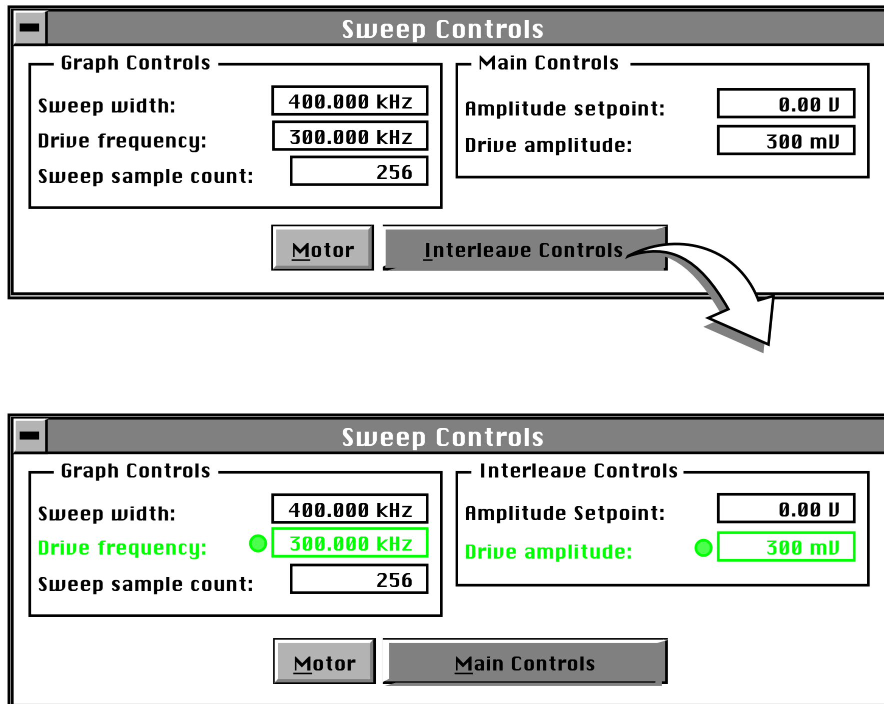


Figure 8.5. ***Sweep*** **control panels for** ***Main Controls*** **(top) and** ***Interleave Controls*** **(bottom).**

2. Set parameters in the **Sweep Controls** panel as follows:

- Set the **Drive frequency** parameter to a value near the center of the range of the resonance frequencies specified for the cantilever. For example, if the frequency range is specified as 240—420 kHz, select a drive frequency of **330 kHz**.
- Start with a **Drive amplitude** of about 300 mV. It is possible to detach the cantilever from the substrate by applying too large of a drive amplitude; therefore, it is important to exercise some caution when adjusting the **Drive amplitude** parameter. Note that the **Drive amplitude** is reset automatically by the **Auto Tune** command.
- Set the **Sweep width** to about the same value as the **Drive Frequency,** or at least some value large enough to cover the frequency range specified for the wafer.
- Zero the **Amplitude Setpoint**. Note that the **Amplitude Setpoint** will be reset automatically by the **Auto Tune** command.

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

- Increase the **Drive amplitude** to **600 mV**.
- Increase **Sweep width** to the maximum value.

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

4. After identifying the maximum amplitude peak with the lowest frequency in the frequency response plot, center the peak on the frequency sweep plot shown on the display monitor. This is most easily accomplished with the **Zoom In** and **Offset** subcommands in the menu bar of the display monitor. The **Offset** subcommand shifts the plot by setting the center frequency equal to the cursor position. The **Zoom In** subcommand stretches the plot by decreasing the sweep width and shifting the center frequency value.

If the top of the resonance peak is off the plot, increase the **Amplitude Setpoint** until it appears. Continue to **Zoom In** and center the peak until the peak coincides with the vertical center line within about 10 Hz. The value displayed for center frequency is now used as the resonant frequency of the cantilever.

In reality, the system will work well in TappingMode if the drive frequency is at, or below, the peak in the resonance plot. The **Drive Frequency** can be decreased to the point where the vibrational amplitude reaches 90 percent of the maximum value. It is often preferable to operate at a frequency lower than the resonant frequency due to shifting of the resonant frequency upon approach of the tip to the surface.

8.3.5. Setting the *Drive amplitude* and *Setpoint*

After tuning the cantilever to its resonant frequency, the free-vibration amplitude must be specified. The desired operating amplitude depends on the sample, and other scanning conditions.

Prior to engagement, the **Amplitude Setpoint** parameter is used to scale the cantilever response (vertical) axis of the frequency sweep display graph. It does not actually adjust the operating feedback setpoint, since the computer automatically determines the initial engagement setpoint during engagement.

Only after engagement does the Setpoint parameter in the control panel define the operating amplitude to be maintained by the feedback loop. Perform the following operations to adjust the oscillation drive amplitude and the frequency sweep graph vertical axis scaling.

1. Increase the **Drive amplitude** until the peak amplitude in the Frequency Sweep plot reaches the desired amplitude. A good amplitude would be 1—3 volts. In general, the peak amplitude will reach 3 volts with the drive amplitude set to under 500 mV.

The amplitude value can be determined by adjusting the amplitude setpoint value until the center frequency crosses in the middle (horizontally) of the cantilever tune graph. You can also **Quit** the **Cantilever Tune** menu and adjust the **Drive amplitude** until the RMS value is appropriate. The actual setpoint value adjusted by the

user prior to engage is meaningless because the operating setpoint is determined automatically during engage by the control program.

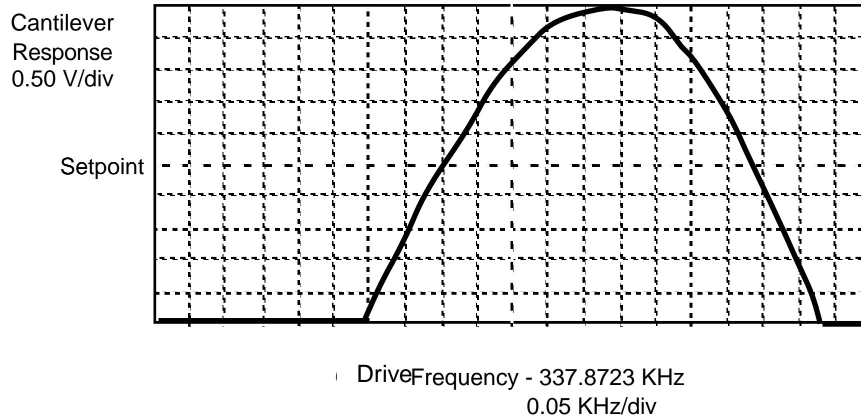
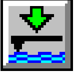


Figure 8.6. Sample starting control point.

2. Click on **Quit** and the parameters set in the **Cantilever Tune** control panel will now appear in the **Feedback Controls** panel. The value of the RMS amplitude displayed on the laser signal display will now hold steady at the amplitude that coincided with the selected **Drive frequency**. After the microscope is engaged, the RMS amplitude value displayed on the display monitor will match the **Setpoint** parameter specified in the **Feedback Controls** panel.

8.4. Engaging The Microscope



After cantilever oscillation has been adjusted and the operating point is defined, the microscope is nearly ready to engage. The following items remain to be done:

1. Recheck all control panel parameters. For the calibration standard, set control panel parameters as shown in Figure 8.7. The feedback gains and the scan rate are the most important parameters. Start with the **Integral gain** set to **0.50** and the **Proportional gain** set to about **1.20** and set the **LookAhead gain** (if supported) to zero. The scan rate should be set below 2 Hz. Verify that **Input attenuation** is set at **1x** on the **Other Controls** panel before imaging.

Scan Controls Settings

Scan Controls	
Scan size:	25.0 µm
X offset:	0.00 nm
Y offset:	0.00 nm
Scan angle:	0.00 deg
Scan rate:	1.51 Hz
Number of samples:	256
Slow scan axis:	Enabled
Z limit:	440 V

Figure 8.7. Suggested *Scan Controls* settings during TappingMode set-up.

Other Controls Settings

Other Controls

Units:	Metric
Color table:	2
AFM mode:	Tapping
Input attenuation:	1x
Engage Setpoint:	0.90
Z modulation:	Disable

Figure 8.8. Suggested *Other Controls* settings during TappingMode set-up

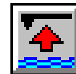
2. Move the sample to the area of interest using the stage manipulator.

3. Use the meters to verify that the vertical deflection is between -1 and +1, the RMS amplitude (topmost meter) is 1—2 volts, and the sum voltage is greater than 1 volt.

4. The operator should now be ready to engage. Go to the upper **Real Time** menu bar and click on **Motor** followed by **Engage**. You should now see a pre-engage check, followed by Z-stage motor sounds. If for any reason the engage aborts because the tip is still too far away from the surface, use the coarse adjustment screws to bring tip and surface closer together. If all goes well after re-engaging, a well formed image will begin to appear on the display monitor.

If it becomes necessary to image another part of the sample, execute a **Withdraw** command before moving the X-Y translation stage; otherwise, the tip may be damaged. After the tip is engaged, the control panel values can be readjusted to provide the desired scan parameters. Refer to section 8.6 below for scan parameter optimization.

8.5. Withdrawing the Tip



Select **Withdraw** from the **Motor** menu. The SPM will stop scanning, then ascend approximately 7 microns. If more clearance between tip and sample is desired, toggle the **Up / Down** switch on the top-right side of the MultiMode base. Never withdraw samples without carefully observing that the tip has adequate clearance during the entire sample removal sequence.

8.6. Beyond Basics with Resonating Techniques

This section discusses some of the subtle aspects of the operation of the MultiMode in TappingMode. Without a thorough understanding of principles associated with cantilever resonating techniques, distorted data may be generated. Understanding the **Cantilever Tune** process and the effects of Real-time scan parameters is very important to effective operation of the microscope. It is also very important to understand similarities and differences between the force calibration mode of contact AFM and force calibration mode while in TappingMode.

8.6.1. Subtleties of Cantilever Oscillation

The response of the cantilever to inputs plays an important role in the operation of the MultiMode microscope 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 its vibrational amplitude. The cantilever drive system pumps energy gradually into the cantilever oscillation.

Figure 8.9 shows a typical response curve of the cantilever amplitude as a function of time. To demonstrate the conflicting requirements, the performance of the system will be analyzed at two operating points.

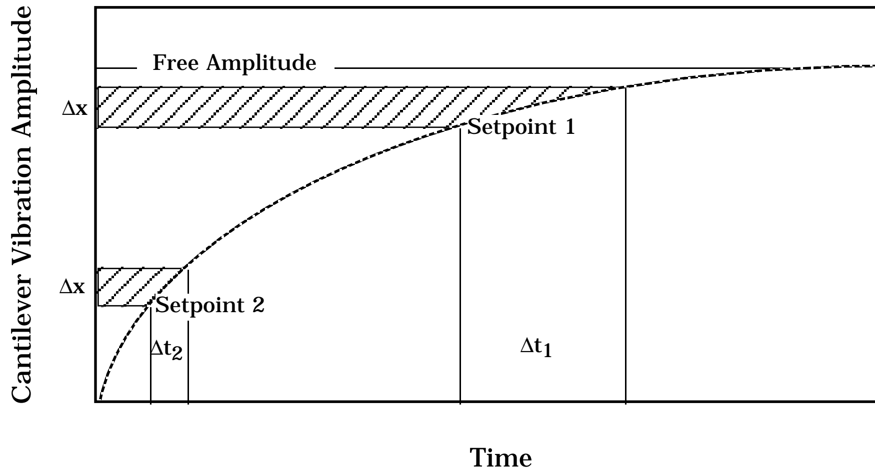


Figure 8.9. Cantilever response curve.

At Setpoint 1 the operating point is only slightly lower than the free vibration amplitude. This has the advantage of dissipating very little energy to the sample surface. (The drawback is that the system takes longer to recover from a given perturbation in the amplitude.) Consider the case 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 will be slow in responding to the error created by going off of the step. At operating Setpoint 2 the cantilever amplitude builds up more rapidly. Therefore, the feedback system will sense the error caused by going off of the step and respond more rapidly. Unfortunately, more energy is transferred to the sample surface while scanning at this operating point.

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

It should be noted that this effect is only a problem when the tip encounters a low point in the sample. The amplitude of the cantilever vibration decreases very quickly when taller portions of the sample are encountered. As a result, the system

response can be markedly different depending on whether the tip is climbing or descending a feature in the sample. For this reason, the **Scope Mode** can be very useful when setting scan parameters. As the tip descends, features can be evaluated by comparing the **Trace** and **Retrace** in the Scope Mode. Figure 8.10 shows the effects of poorly selected scan parameters on a calibration standards 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. It does, however, track the surface closely when moving out of the pit.

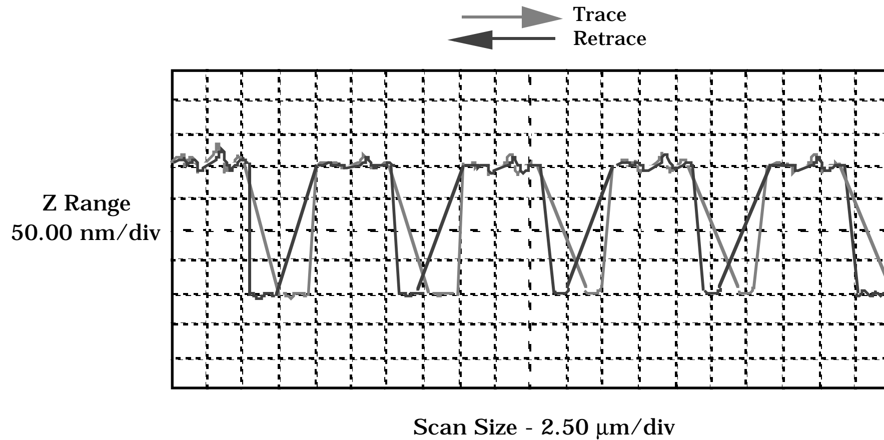


Figure 8.10. Scope trace with high *Scan rate.*

Figure 8.11 shows 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/Retrace lines now coincide closely.

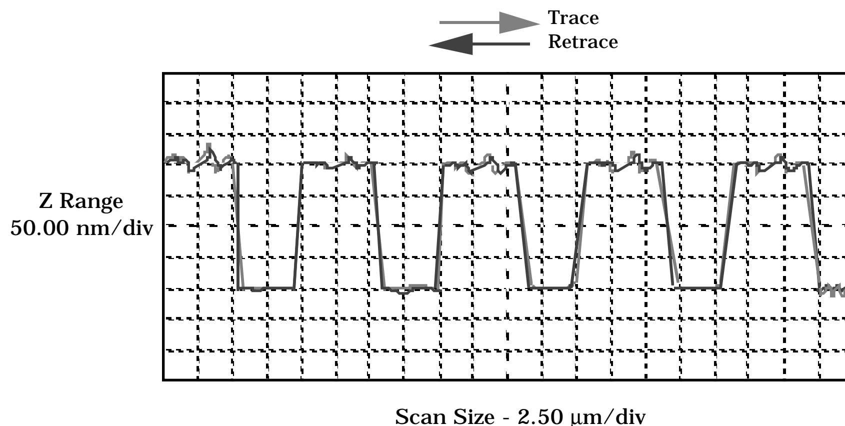


Figure 8.11. Scope trace with correct *Scan rate*.

8.6.2. Tuning 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 resonance frequency of the cantilever, but the Drive frequency should be further tuned to improve scanning performance.

It has been determined that the microscope produces better data in TappingMode when the Drive frequency is set lower than the resonance peak of the cantilever. The Drive frequency should be set such that it coincides with a 10 percent decrease in the vibration amplitude. Figure 8.12 shows the suggested operating region. It should be noted that this is a suggestion based on our observations; users are certainly encouraged to experiment with the microscope and decide what produces the best results.

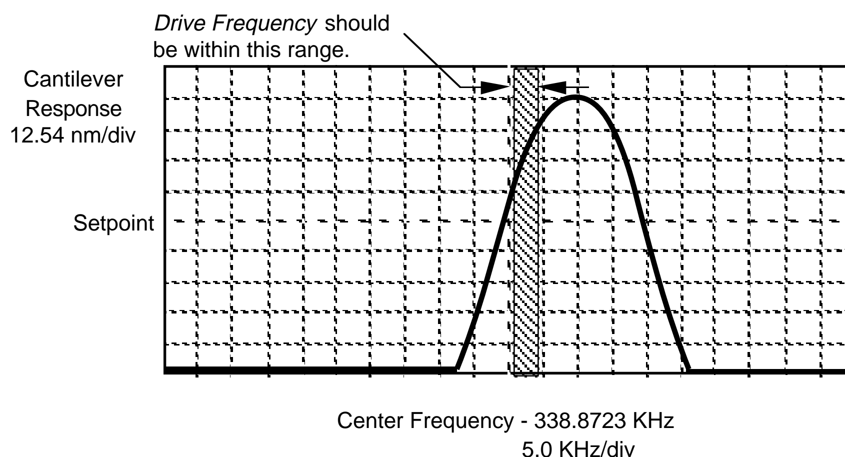


Figure 8.12. Example of *Cantilever Tune* frequency sweep.

8.6.3. Optimization of scanning parameters

Careful selection of the scan parameters is important to the successful application of the MultiMode in TappingMode. In general, the effects of the various scan parameters are the same for the TappingMode as they are for contact AFM mode. The user is encouraged to review section 5.2. which discusses parameter optimization for contact AFM.

This section focuses on parameters specific to TappingMode. As with other operating modes, the optimal parameter selection depends greatly on the sample. All of the parameters in the real-time control panel are discussed in the *Command Reference Manual*. The user is encouraged to review all parameter descriptions. This manual analyzes the effects of the most important parameters.

8.6.3.1. *Data Type* (*Channel* panels)

Data type is the first parameter to set because the settings 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 that is collected by the system. **Height** data corresponds to the change in piezo height needed to keep the vibrational amplitude of the cantilever constant. **Amplitude** data describes the change in the amplitude directly. The **Sensitivity** parameter in the **Force Calibration** control panel must be determined, as described in the discussion of the Force Calibration mode, before Amplitude data is accurate.

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

TappingMode microscopy should not be conducted 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 essentially the derivative of the height; this is commonly referred to as the error-signal mode. The error-signal mode provides a sensitive edge detection technique. With the dual screen mode it is possible to capture both Height and Amplitude data simultaneously.

8.6.3.2. Gain settings

The **Integral**, **Proportional**, and (on some software versions) **Look ahead gains** on the **Feedback Controls** panel must be high enough to force the feedback system to track the sample surface. When scanning in TappingMode, the Integral and Proportional gains must be set to lower values than are used in the contact mode. Oscillation usually occurs with Integral gains of 1—2. The **Proportional gain** can usually be set a factor of 5—10 times higher than the **Integral gain**. To optimize the gains, increase the **Integral gain** until the piezo begins to oscillate, then eliminate the oscillations by reducing the gain with two or three clicks of the left arrow key. Repeat the process for the **Proportional gain**. Piezo oscillations typically cause high frequency wavy lines in the Real-time image.

8.6.3.3. *Scan Size*, *Scan rate*, and *Setpoint*

As discussed above, the **Scan size**, **Scan rate**, and **Setpoint** values have dramatic effects on the data. As in contact mode, the **Scan rate** must be decreased as the **Scan size** is increased. **Scan rates** of **0.5—1.0 Hz** should be used for large scans on samples with tall features. High scan rates help reduce drift, but they can only be used on flat samples with small scan sizes.

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

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

8.6.3.4. *Lowpass filter* (*Channel* panels)

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

8.6.3.5. *Highpass filter* (*Channel* panels)

The **Highpass filter** invokes a digital two-pole highpass filter which removes low frequency effects such as bow (caused by Z hysteresis). As with the **Lowpass filter**, it also operates on the digital data stream regardless of scan direction. This parameter can be **Off** or set from **1** through **9**. Settings of **1** through **9**, successively, lower the cut-off frequency of the filter applied to the data stream.

Note

- It is important to realize that in removing low frequency information from the image, the highpass filter distorts the height information in the image. As a result, this filter should be **Off** when accurate height information is desired.

8.7. Force Calibration (Z Scan Controls) Mode

Force Calibration mode in TappingMode is similar to Force Calibration mode in contact AFM mode; however, there are some notable differences. The **Force Calibration** command is in the **Real-time / View** menu. In **Force Calibration** mode, the X and Y voltages applied to the piezo tube are held at zero (unless a DC offset is in effect) and a triangular waveform is applied to the Z electrodes of the piezo tube. As a result of the applied voltage, the sample is moved up and down relative to the oscillating tip as shown in Figure 8.13. The **Z scan start** parameter sets the offset of the piezo travel while the **Z scan size** parameter defines the total travel distance of the piezo. Therefore, the maximum travel distance is obtained by setting the **Z scan start** to +**220 volts** with the **Z scan size** set to **440 volts**. In practice, the maximum scan size should not be applied to the piezo because the cantilever is delicate and may break if too much deflection is induced.

CAUTION: Use caution when working in **Force Calibration** mode**.** This mode can potentially damage the tip and/or surface.

ATTENTION: Digital Instruments recommande d'utiliser le mode "**Force Calibration**" avec une précaution. L'utilisation du mode "Force" peut endommager la pointe ou l'échantillon.

WARNHINWEIS: Seien Sie vorsichtig, wenn Sie im Force Calibration Mode arbeiten. Dieser Meßmode kann unter Umständen Meßspitze oder Probe beschädigen.

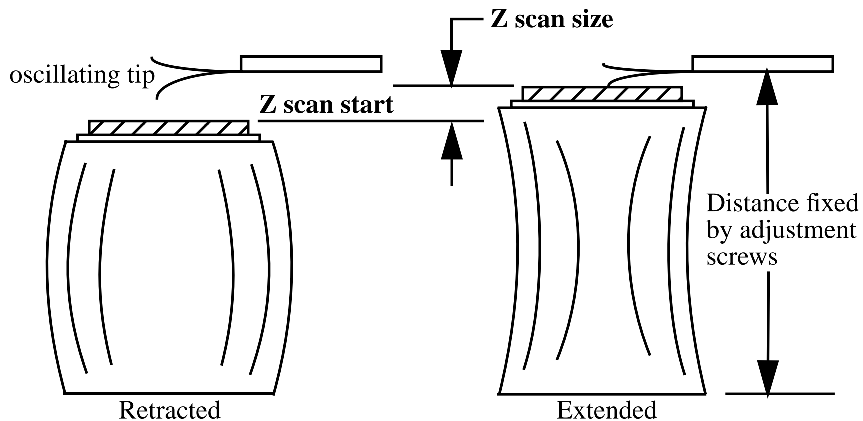


Figure 8.13. Piezo travel in *Amplitude Cal.* mode

As the piezo moves the sample up and down, the RMS value of the cantilever-deflection signal is monitored. The Force Calibration plot (a plot of the RMS signal

as a function of the voltage applied to the piezo tube) shows on the display monitor. The control panel containing the parameters used to control the microscope in the Force Calibration mode is visible on the control monitor's top menu bar.

Force Calibration mode is used to compare the amplitude of cantilever oscillation on the surface to the free-air amplitude. Other uses of Force Calibration mode include characterizing the forces on the cantilever tip, diagnosing the performance, and calibrating the RMS amplitude voltage as a function of the oscillation amplitude of the cantilever.

8.7.1. The *Force Calibration* plot

Figure 8.12 depicts a typical **Force Calibration** plot for TappingMode operation. The vertical axis of the graph represents the RMS of the cantilever deflection signal. The voltage applied to the Z electrodes of the piezo tube is plotted along the horizontal axis. The left side of the graph is equal to the Z scan start voltage while the right side of the screen is equal to the quantity (**Z scan start** minus **Z scan size**). The Force Calibration plot represents the RMS signal for one complete extension/retraction cycle of the piezo. The **Z scan rate** parameter in the Force Cal control panel defines the rate at which the piezo completes an extension/retraction cycle; therefore, the rate at which new curves are displayed.

The right side of the plot reflects the free vibration amplitude of the cantilever. At this piezo position the vibration amplitude is not influenced by the sample. Eventually as the piezo extends the vibrating cantilever toward the sample, the probe contacts the sample surface and the vibration amplitude decreases. After the tip begins to contact the sample surface, each nanometer decrease in the cantilever position decreases the vibrational amplitude of the cantilever by two nanometers. The vibrational amplitude of the cantilever continues to decrease as the probe lowers. When the piezo turns around and begins to retract the sample, the vibrational amplitude of

the cantilever begins to increase. It continues to increase until the tip is free of the surface, then it levels off at the free-standing amplitude.

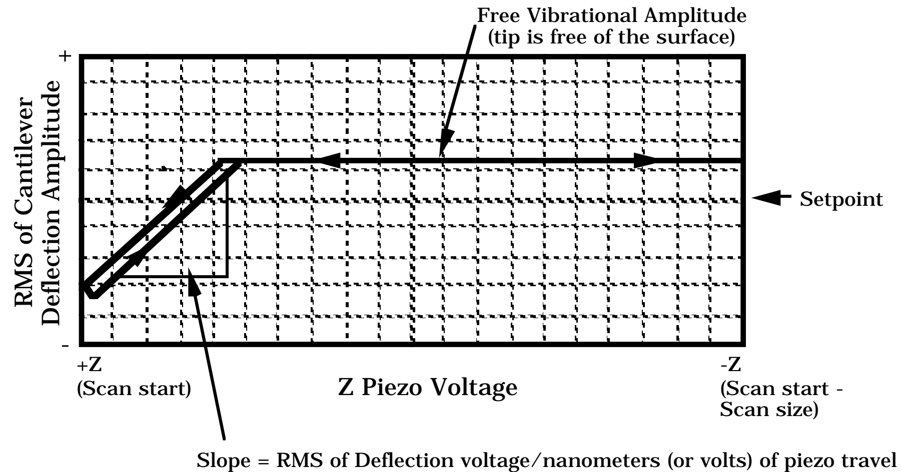


Figure 8.14. Typical *Force Calibration* curve.

The display monitor also presents a linear scale of the Z piezo voltage. The scale ranges from +220 volts at the "extended" end to -220 volts at the "retracted" end. The range of the piezo travel, as defined by the **Z scan start** and **Z scan size** parameters in the Force Calibration control panel, is represented by two white lines on the scale. The lower, white line corresponds to the **Z scan start** parameter in the control panel while the spacing between the lines corresponds to the **Z scan size**. The average voltage applied to the Z electrode on the piezo tube just prior to entering the **Force Calibration** mode is represented by the blue, "Z center" line.

8.7.2. The *Force Calibration* control panel—additional features

The Force Cal control panel is shown in figure 8.13. The setpoint voltage used in the feedback loop can be adjusted from this panel. With the **Tip Up** and **Tip Down** buttons, the stepper-motor can be used to adjust the position of the vibrating cantilever relative to the sample. The **Capture** button stores the Amplitude Calibration plot for Off-line viewing. It should be noted that some of the parameters influence the Real-time operation of the microscope while most of the menu parameters affect only **Force Calibration** mode.

The input sensitivity of the MultiMode head must be defined properly to keep the values on the vertical axis of the Amplitude Calibration plot accurate. The **In**

sensitivity parameter should be set to **0.125** by using the **Real-time / Microscope / Calibrate / Detector** command for the MultiMode microscope.

Z Scan Controls

Graph Controls		Other Controls	
Z scan start:	100 nm	Trigger mode:	Off
Z scan size:	500 nm	Trig threshold:	0.00 V
Z scan rate:	4.88 Hz	Trigger channel:	Amplitude
X offset:	0.00 V	Z step size:	2.40 V
Y offset:	0.00 V	Step threshold:	7.53 V
Number of samples:	512	Start mode:	Calibrate
Average count:	1	End mode:	Extended
Sensitivity:	0.0379 V/nm	Indent Feedback:	Disabled
Units:	Metric		

Figure 8.15. ***Force Cal.*** **control panel—TappingMode.**

Most of the parameters in the control panel have the same function as the Force Calibration parameters used for contact AFM, which are discussed in Chapter 6 of this manual. Those parameters not discussed in Chapter 6 are discussed in the following paragraphs:

Setpoint— The RMS value of the cantilever deflection voltage maintained by the feedback loop in the Real-time mode can be adjusted by changing this parameter. In the TappingMode **Force Calibration** menu, this parameter defines the centerline of the vertical, “Tip Amplitude” axis of the amplitude calibration plot shown on the display monitor. Changing the **Setpoint** shifts the amplitude curve on the graph, because the centerline value is always equal to the Setpoint. For example, if the Setpoint is at 3.0 Volts, the RMS amplitude axis of the graph will be centered around 3.0 Volts; raising the Setpoint to 4.0 Volts will shift the amplitude curve down by one volt, so the graph will be centered at 4.0 Volts. Changing the value of this parameter in the **Amplitude Calibration** mode also changes the Setpoint parameter in the Real-time control panel.

Drive frequency— This item defines the frequency at which the cantilever is oscillated. This frequency should be very close to the resonant frequency of the cantilever. Changing the value of this parameter in the **Force Calibration** menu also changes the **Drive frequency** parameter in the Real-time control panel.

Drive amplitude— 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 the **Force Calibration** mode also changes the **Drive amplitude** parameter in the Real-time control panel. It is possible to fracture the cantilever by

using too high of a drive amplitude; therefore, it is safer to start with a small value and increase the value incrementally. If the amplitude calibration plot consists of a flat line all the way across, changing the value of this parameter should shift the level of the curve. If it does not, the tip is probably fully extended into the surface and the tip should be withdrawn before proceeding.

Sensitivity— This item relates the vibrational amplitude of the cantilever to the Z travel of the piezo. It is calculated by measuring the slope of the RMS amplitude versus the Z voltage when the tip is in contact with the sample as shown in figure 8.12. The NanoScope system automatically calculates and enters the value from the graph after the operator uses the mouse to fit a line to the graph. This parameter must be accurately calibrated before Amplitude data obtained in Lift mode will be accurate.

Note

- Sensitivity can only be set on channel one.

8.7.3. Examples of amplitude calibration plots

Figure 8.16 shows a typical amplitude calibration plot. Depending on the sample and the operating conditions, the curve may include other notable features. This section presents examples of some of those conditions.

8.7.3.1. Very high contact force

Figure 8.16 shows a curve produced when the tip is too close to the sample. This is a very dangerous condition because the cantilevers used in the TappingMode are very stiff and brittle. If they are driven into the sample surface so that the oscillation amplitude changes to zero, they will break.

DI recommends using **Force Calibration** only when the tip is known to be bad, or when it is important to obtain experimental information shown in **Force Calibration**

The flat portion on the left side of amplitude curve in figure 8.18 occurs because the tip is so close to the surface that it no longer vibrates. As the piezo extends the sample further and 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 and the cantilever or tip can be shattered if the deflection continues.

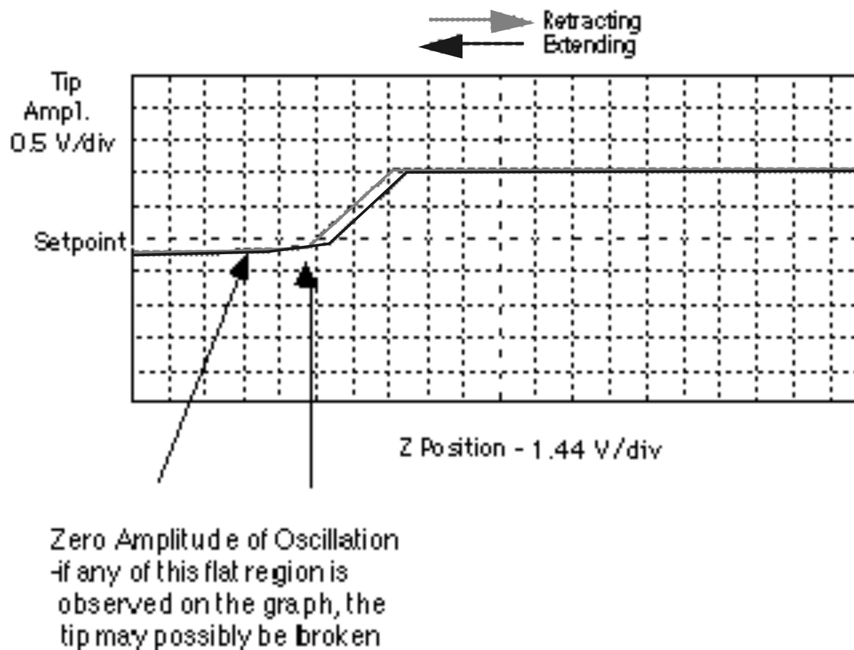


Figure 8.16. Amplitude force plot with high contact force.

This situation can be avoided by reducing the value of **Z scan start** so there is no flat portion on the left side of the curve. Rapidly increasing the value of **Z scan size** is very dangerous since the total vibrational amplitude of the cantilever is small relative to the total Z travel of the long-range scanner. The total amplitude of the cantilever oscillation is usually less than 100 nanometers while the "J" scanner travels up to 6 microns along the Z axis. In general, the **Z scan size** parameter should be much smaller than the value entered into the **Force Cal.** menu of contact AFM.

8.8. TappingMode in Fluids—Introduction

Operation of TappingMode in fluid provides the same advantages of TappingMode in air, with the additional ability to image samples under native liquid conditions. In fluid TappingMode, the probe is oscillated so that it only intermittently contacts the sample surface. This can reduce or eliminate frictional forces that often damage soft or fragile samples in contact mode. The following sections provide general instructions for TappingMode imaging in fluid, then provide practical instructions for imaging two different biological samples. Instructions should be adjusted for different samples and imaging conditions.

Before attempting TappingMode in fluids, it is recommended that the user become familiar with standard TappingMode operation in air (see above sections) and contact AFM in fluid (Chapter 7).

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

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

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

8.8.1. Acknowledgments

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

8.8.2. Overview

TappingMode in fluid combines the advantages of TappingMode with the ability to image samples in native liquid environments (e.g., physiological buffer solutions).

Digital Instruments would appreciate receiving any further suggestions and is anxious to hear about new sample preparation techniques and experience with TappingMode in fluid.

8.8.3. Fluid cell preparation

Required Materials

- TappingMode Fluid Cell, Model MMTFC
- Cantilevers (100µm, narrowed-leg, Oxide-Sharpened Silicon Nitride tips, Models NP-S or NP-STT, work well)
- Deionized water
- Source of filtered (0.2 µm), compressed air or dry nitrogen (chromatographic-grade)
- UV lamp, high-intensity; Oriel Mod. 6035 pencil-style spectral calibration lamp or equivalent (optional for cantilever cleaning).
- 1 cc and 5cc syringes or micropipettes (200 µl pipette tips)
- Fluid cell liquid lines (silicone tubing and fittings)
- Fluid cell o-ring (optional see Section 8.8.5.)
- Hemostats or similar clamping device (for liquid lines)
- Filter paper

8.8.3.1. Clean fluid cell and o-ring.

To reduce contamination problems and to obtain high-quality images, the fluid cell and o-ring must be as clean as possible. The following cleaning procedure works well:

- While soaking in warm, soapy water, place a few drops of liquid dish soap on the fluid cell and o-ring, then rub gently with a cotton swab or finger. Take care not to scratch the glass surface with any abrasive material.
- Rinse completely free of detergent with distilled water.
- Blow dry the fluid cell with 0.2 µm-filtered, compressed air or dry nitrogen until all moisture is evaporated.

8.8.3.2. Select and install cantilever.

Best results are usually obtained with the 100 micron, narrow-legged cantilevers with oxide-sharpened silicon-nitride tips (Model NP-S or NP-STT). Some users also use electron beam deposited (EBD) tips for fluid work. Contact Digital Instruments for information on making or purchasing EBD tips. Try oxide sharpened tips to start. Good results have also recently been obtained with etched silicon cantilevers (standard air TappingMode cantilevers, Mod. ESP or FESP), used at very small oscillation amplitudes. Users should experiment to find which cantilevers work best for their sample.

Install a cantilever snugly into the fluid cell's tipholder. Check that the spring clip holds the cantilever tightly.

8.8.3.3. Clean cantilever with UV light if desired.

The AFM's resolution can be limited by contaminants on the cantilever tip. Here is a procedure to remove organic contamination from the tip:

Place the fluid cell with installed tip face-up on a clean surface. Position a UV lamp very close (3-5 mm) to the fluid cell and allow the unit to be irradiated for 15-30 minutes at full intensity.

8.8.4. Sample mounting—Method 1 (with an o-ring).

Secure a sample substrate (cover glass or mica) to a magnetic stainless steel sample puck using double-sided adhesive. Load the prepared sample substrate onto the AFM scanner.

8.8.4.1. Install o-ring on sample.

Place the o-ring onto the sample substrate. It should be positioned so that it forms a seal around its periphery and does not overlap any edges.

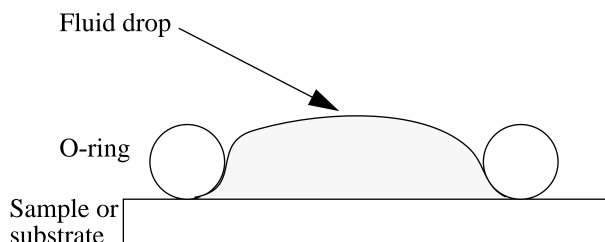


Figure 8.17. Place the o-ring on the sample and slowly fill with fluid using a micropipette.

CAUTION! Do not add excessive amounts of fluid or the cell will overflow and damage the piezo tube scanner.

ATTENTION! N'ajoutez pas trop de fluide. Sinon, la cellule débordera et endommagera le scanner du tube piezo.

8.8.4.2. Fill o-ring with liquid.

Drip enough liquid solution into the o-ring to form a small pool, as shown in Figure 8.17. Use filter paper to wick away small droplets from the top of the o-ring until the top is dry. Verify that the o-ring is not leaking around its edges.

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

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

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

8.8.4.3. Pre-wet the fluid cell.

Occasionally air bubbles will form in the fluid cell and block laser light. This step can reduce the chance of forming bubbles.

Before installing the fluid cell into the head, insert a syringe or pipette filled with liquid solution into a fluid port. Flood the fluid cell port with buffer solution, allowing liquid to drip out the bottom of the cell.When you have finished, leave the buffer-filled syringe inserted. A small amount of buffer solution should be held to the bottom of the cell by surface tension, as shown in Figure 8.18.

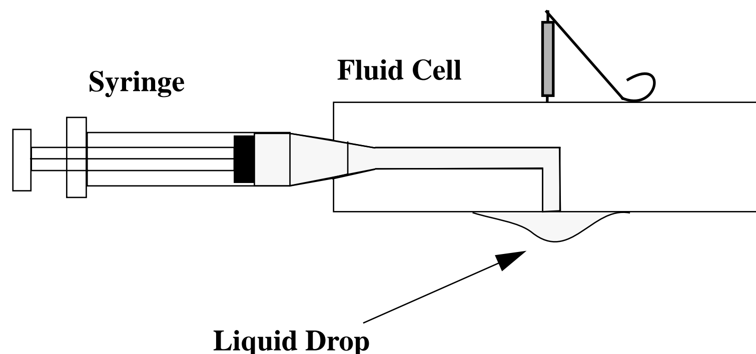


Figure 8.18. Flush the fluid cell before installation to reduce bubble formation.

8.8.4.4. Install SPM head and fluid cell.

Place the SPM head onto the scanner and secure the head stabilizing springs. Plug the head's 15-pin connector into the scanner support ring. Use the three scanner screws to lift the SPM head so that there will be sufficient clearance to install the fluid cell without breaking the cantilever. Make sure the head is level side-to-side and that the head is tilted slightly forwards so that it will be level when the tip contacts the surface. Carefully install the fluid cell into the SPM head, lowering the fluid cell onto the o-ring until seated securely. Tighten down the clamp to hold the fluid cell in place. If you have purchased a vertical-engage scanner, leveling problems are eliminated.

8.8.4.5. Fill the fluid cell with liquid.

Attach a drain line to the other fluid cell port. Slowly flush the fluid cell with buffer solution from the syringe. Check for leaks and wick away any spilled liquid with filter paper until SPM components are dry.

8.8.4.6. Remove bubbles and clamp off fluid cell lines.

Observe the fluid cell and cantilever through the viewing port using an optical microscope. Bubbles inside the fluid cell near the cantilever can interfere with the laser beam—these should be removed.

To remove bubbles from the cell, rapidly push liquid through the cell with a syringe. If sufficient force is applied, the bubbles will be washed away. To prevent the introduction of air and spillage, be certain to maintain a good seal between the syringe and fluid cell port.

When the fluid cell has been filled and bubbles have been removed, clamp off the drain line with a pair of hemostats or similar clamp.

8.8.5. Sample mounting—Method 2 (without an o-ring).

The fluid cell may be used without the o-ring. Place the fluid cell close to the sample surface, and slowly inject fluid until a liquid drop forms which is held by surface tension between the sample surface and the glass cantilever holder.

Use Loctite 770 (recommended adhesive) to affix a teflon cover over the steel sample puck (teflon should slightly extend over the edge of the sample puck) and then use 2 component epoxy to attach the mica to the teflon. The hydrophobic teflon will help confine the solution without installing an o-ring.

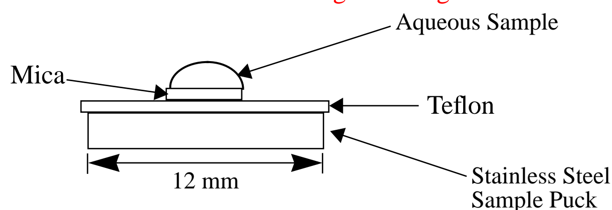


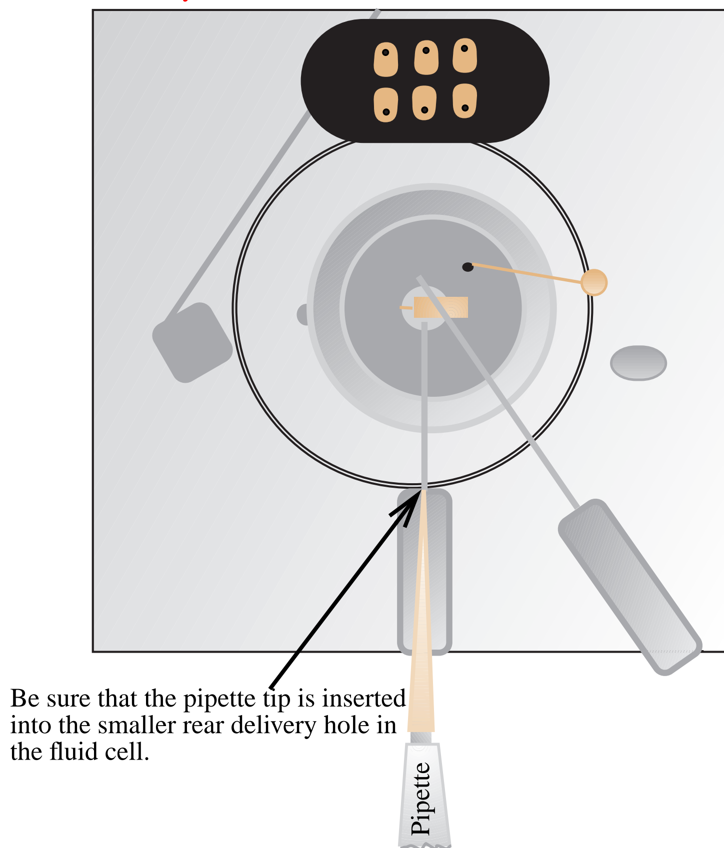
Figure 8.19. Stainless steel sample puck with teflon cover.

CAUTION! Do not use a sample volume that exceeds 50 µl as excess fluid may be squeezed onto the AFM scanner. Do not pre-wet the fluid cell when using the o-ring-less method.

To use this method of fluid imaging, align the SPM head and mounted fluid cell (cantilever installed) with the dry sample puck first before applying the aqueous sample. Allow 0.5 mm clearance between the tip and dry substrate surface. Once the optics and laser are aligned, remove the SPM head and fluid cell. Take the dry sample substrate off the scanner and apply the sample in a volume of approximately 25-40 µl. The liquid should form a small dome over the mica and not spread onto the surrounding teflon. Carefully replace the prepared sample puck. Slowly mount the SPM head and fluid cell onto the sample. Since the aqueous sample will change the light deflection, the laser photodetector will need to be re-adjusted. Course adjustments are made by tilting the deflection mirror while fine adjustments are made with the photodetector adjustment knobs on the left-top and left-rear of the head. Adjust the photodiode position to give a deflection signal ≈ 0 V.

An alternative method to removing the SPM head after alignment is, with the dry sample substrate in place on the scanner, slowly deliver 25-50µl of sample solution

into the fluid cell using a standard micropipette (e.g. Eppendorf, Gilman) with a 200µl capacity pipette tip. The end of the pipette tip fits into the fluid cell port holes (refer to figure below). Inspect from the side to make sure the fluid is well confined to the mica area only.



8.8.5.1. Aim the laser on cantilever and adjust photodiode position.

Instructions for adjusting the laser beam and photodiode through fluid are provided in previous sections. Adjust the photodiode position to give a deflection signal of around 0 V.

8.8.5.2. Check that the microscope is dry.

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

Note

Over time, evaporation of the fluid may necessitate replenishing the fluid cell using a standard micropipette. The pipette will fit conveniently into the fluid chamber port.

8.8.6. TappingMode operation in fluid.

At this point in the procedure, the sample should be loaded in the AFM and the fluid cell filled with fluid. The laser should be aligned on the cantilever and the photodiode centered to give a deflection signal near 0 V.

8.8.6.1. Choose TappingMode operation in software.

In the **Other Controls** screen, set the **AFM mode** parameter to **Tapping**. If using a Dimension or BioScope system, switch the **Z modulation** parameter to **Enabled**.

8.8.6.2. Set initial scan parameters.

For initial imaging, try the following settings:

- Drive frequency = Tune to a peak between 8-10 kHz.
- Drive amplitude = 500 mv (should correspond to ≈0.5 V rms amplitude)
- Scan speed = 1 Hz
- Integral gain = 0.5 (MMAFM); 1.5 (BioScope)
- Proportional gain = 0.75
- Z modulation = Enabled (BioScope and Dimensions only)
- Z range = 50 nm
- First Image Data type = Height
- Second Image Data type = Amplitude
- Third Image Data type (optional) = Phase; Height retrace

The user should try to adjust and optimize these settings for each imaging condition and sample.

8.8.6.3. Select the drive frequency using the *Cantilever Tune* command.

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

Enter the **View / Sweep / Cantilever Tune** menu to select a drive frequency. Users of TappingMode in air will notice that there is no single, well-defined resonance peak, but a number of broader peaks when viewing a wide cantilever sweep. The user must manually select a peak that corresponds to the resonant frequency of the cantilever being used. For the short, narrow Si_3N_4 cantilever, the resonant frequency in fluid is a broad peak centered around 10 kHz. Best results are achieved by tuning the cantilever to a peak between 7-12 kHz. Higher and lower frequencies have also been used depending on the type of cantilever employed. Start with a **Sweep width** of 20 kHz and a **Sweep setpoint** of 9 kHz in the **Sweep Control** menu.   A typical **Cantilever Tune** screen is shown in Figure 8.20.

Note: Make certain the cantilever is very close to the sample surface when tuning the cantilever under fluid.

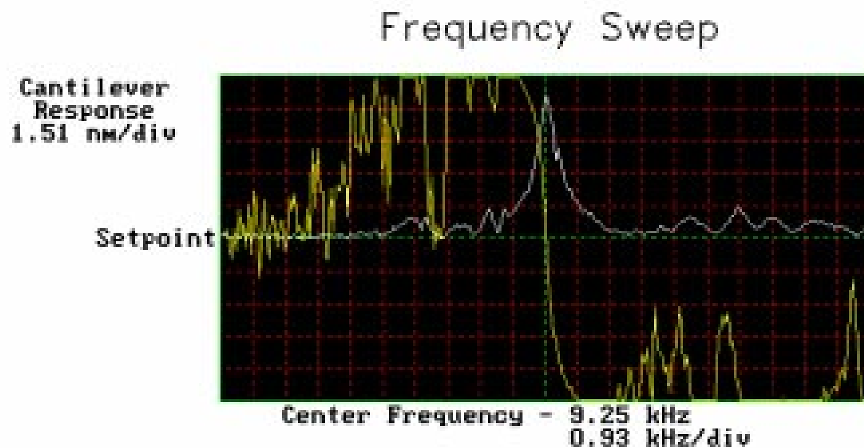


Figure 8.20. Typical *Cantilever Tune* curve for 100µm, narrow-legged silicon nitride cantilever in fluid. The user must manually tune using *Zoom in* and *Offset* functions.

8.8.6.4. Adjust the *Drive amplitude*.

Adjust the **Drive amplitude** until a desired cantilever RMS amplitude is obtained. For samples with small height variations, try an RMS amplitude of around 0.5 V. Usually, this requires a **Drive amplitude** of **250-500 mV**. For rougher samples try larger RMS voltages (1-2 V). Experiment to find what level of cantilever oscillation gives best results for specific applications. Good results are usually not obtained on soft samples if it is necessary to use a **Drive amplitude** above **1.0 V**.

Note

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

8.8.6.5. Recenter the photodiode.

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

8.8.6.6. Engage.

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

8.8.6.7. Adjust the setpoint.

Usually the best topographic images are obtained at setpoints 10-20 percent less than the cantilever RMS amplitude before engaging. The setpoint may be optimized in one of two ways, using the Force Cal (**View / Force Mode / Plot**) command or by optimizing the image quality. Both techniques are described below.

Plotting Force

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

Note

- The slope of the Force Cal curve shows the sensitivity of the fluid TappingMode measurement. In general, higher sensitivities will give better image quality. If the sensitivity is very poor, check the mounting of the sample and fluid cell.

Optimizing Image Quality

The **Setpoint** can also be adjusted by simply monitoring the image quality. Select a **Scan size** of **500** nm. Increase the **Setpoint** in small increments until the cantilever pulls off the surface and the Z center voltage goes to -220 V. 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.)

8.8.6.8. Optimize other scan parameters.

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

8.8.7. Troubleshooting tips.

8.8.7.1. Cantilever Tune plot looks bad—fluid cell/loose tip

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

8.8.7.2. O-ring slides across sample surface, causing drift

To reduce movement of the o-ring which can cause drift in AFM images, the fluid cell needs to be set up such that there is minimal lateral movement of the optical head with respect to the sample once the o-ring is installed. This is best accomplished by keeping the head level while positioning the tip as close as possible to the surface *before* installing the o-ring so that the tip's lateral movement is minimized during engagement. Moving the positioning screws at the bottom of the optical head should also be minimized once the o-ring is installed. Further suggestions for solving this problem consist of the following:

1. The new vertical engage scanner allows the tip to approach samples without the lateral offset caused by older, three-point supports. Thus, stress on the o-ring from lateral movement during engagement is not present. Call DI for more details.

2. Lightly coating the area of the o-ring which contacts the sample surface with white petrolatum or vacuum grease will allow the o-ring to slide across the surface, minimizing lateral stress. This also forms a better fluid-tight seal between the o-ring and sample. However, some solvents (i.e., nonpolar organic solvents) may dissolve some of the lubricant into the fluid.

3. Replace the o-ring with a slice of thin-walled glass, plastic, or stainless steal tubing. The diameter and thickness of the ring of tubing should be chosen so that it does not contact the inner or outer walls of the circular groove in the glass cantilever holder. This gives the optical head room to move laterally during engagement and for positioning the tip over the sample surface. The height of the ring of tubing must be chosen so that it is not too tall so that the tip cannot reach the sample surface, or too short so that the ring does not reach the bottom of the glass cantilever holder before engagement. Glue the ring of tubing to the steel sample puck or to the sample to prevent leaks.

Note

- When using the ring of tubing in the fluid cell, fluid is blocked from exiting and cannot be circulated through the cell.

4. When positioning the o-ring on the surface, adjust the positioning knob at the base of the optical head to move it slightly forward. This will counter some of the lateral stress on the o-ring which comes from the optical head moving back during engagement.

Note

- Remember that to prevent leaks when using the standard o-ring, it is best to place the o-ring on the sample before placing the optical head on the scanner, instead of inserting the o-ring into the groove in the glass cantilever holder before placing the optical head on the scanner.

8.8.7.3. Laser sum signal absent or weak—air bubbles

Check that all bubbles are removed from the cantilever. Bubbles may attach themselves to the cantilever, causing the laser beam to be diffracted. Bubbles can sometimes be removed by forcing fluid through the fluid cell. Degassing your imaging fluid prior to use in the AFM will solve bubble problems.

8.8.7.4. Poor image quality

Contaminated Tip— Some types of samples may adhere to the cantilever and tip (e.g., certain proteins). This will reduce resolution giving "fuzzy" images. If tip contamination is suspected to be a problem, it will be necessary to protect the tip against contamination. See section 8.8.6 for a method of reducing tip contamination.

Dull Tip— AFM cantilever tips can become dull during use and some unused tips may be defective. If imaging resolution is poor, try changing to a new cantilever. Also, check the cantilever type being used. Oxide sharpened silicon nitride cantilevers are usually much sharper than standard silicon nitride cantilevers. The user may also wish to try electron beam deposited (EBD) tips to improve resolution. Contact Digital Instruments for details on making or purchasing EBD tips.

Multiple Tip— AFM cantilevers can have multiple protrusions at the apex of the tip which result in image artifacts. In this case, features on the surface will appear two or more times in an image, usually separated by several nanometers. If this occurs, change or clean the AFM tip.

8.8.8. Model sample preparations.

This section overviews sample preparation techniques for two different samples: 1) the protein lysozyme; and, 2) DNA. These protocols can serve as a model when developing methods to bind other samples.

8.8.8.1. General notes on sample binding

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

Binding Specimens to Mica

The following procedures will describe how to bind two different samples to a mica substrate. Mica is commonly used because atomically flat substrates can be simply and inexpensively prepared. In aqueous solutions, the mica cleavage surface becomes negatively charged. Specimen binding is usually accomplished using electrostatic attraction between charges on the specimen and those on the mica surface. Proteins, for example, can usually be made to stick to mica by operating at a pH where they exhibit positively charged domains. DNA, on the other hand, is negatively charged and can be bound either by altering the mica surface charge from

negative to positive (using a silanization process) or by dissolving the DNA in a divalent metal counter ion (e.g. Mg^{++}, Ni^{++}). Both of these techniques are discussed separately below.

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

8.9. Lysozyme on mica—A model procedure for protein binding

Protein binding theory

All proteins contain free amino groups that become positively charged at sufficiently low pH. If sufficient free amino groups are located on the outside surface of the protein, then the protein will bind to a negatively charged mica surface. Proteins will become positively charged at pH below their isoelectric point and are then able to bind to mica. The protein lysozyme, for example, becomes sufficiently positively charged to bind to mica at pH 6. This is shown schematically below in Figure 8.21.

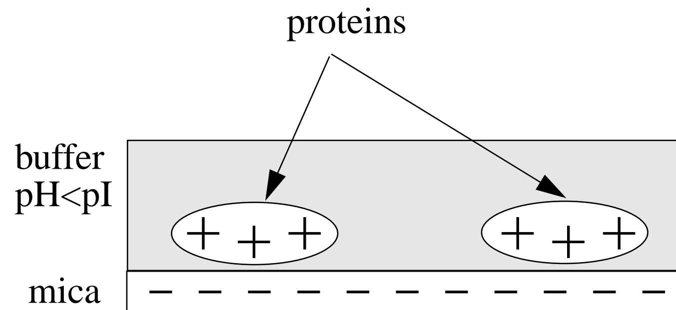


Figure 8.21. Proteins will typically bind to negatively charged mica when the pH is reduced below the protein's isoelectric point, pI.

Protein binding procedure:

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

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

Required Materials

- Deionized water
- Mica substrates
- Lysozyme protein L-6876 from Sigma Chemical
- Phosphate buffer solution, 10 mM KH_2PO_4, 150 mM KCl, pH 6 (buffer may be adjusted for other proteins)
- TappingMode Fluid Cell, Model MMTFC
- Cantilevers (Oxide-Sharpened Silicon Nitride tips, Model NP-S, work well)
- Source of filtered (0.2 µm), compressed air or dry nitrogen
- UV lamp, high-intensity; Oriel Mod. 6035 pencil-style spectral calibration lamp or equivalent (optional for cantilever cleaning).
- Syringes: (1) 1 cc; (2) 5 cc; Micropipettes
- Fluid cell liquid lines (silicone tubing and fittings)
- Fluid cell o-ring (optional see Section 8.8.5.).
- Hemostats or similar clamping device (for liquid lines)
- Filter paper

8.9.1. Dissolve protein.

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

When imaging fluid samples, use extraordinary precautions against spillage.

8.9.2. Prepare fluid cell.

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

8.9.3. Prepare mica.

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

8.9.4. Deposit protein solution on mica.

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

8.9.5. Allow protein to bind to substrate.

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

8.9.6. Rinse unbound proteins from fluid cell.

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

8.9.7. Assemble fluid cell.

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

8.9.8. Clamp fluid drain line.

After the fluid cell has been flushed with buffer solution, reclamp the drain line. This is important for low-noise, low drift imaging. The sample is now ready for TappingMode imaging. A good TappingMode image of lysozyme protein on mica is shown in Figure 8.22

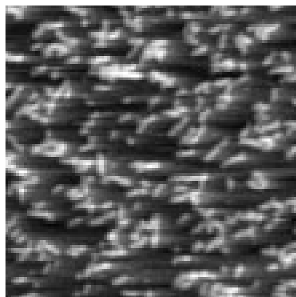


Figure 8.22. TappingMode image of lysozyme in buffer solution using above sample preparation (scan size 500 nm).

Note

- It is also possible to prepare samples inside the fluid cell by flowing the protein solution through the fluid cell. In this case, it may be helpful to engage the tip in contact mode with a zero **Scan size** to protect proteins from binding to the tip.

8.10. Binding DNA to Mica

DNA Binding Theory

DNA and mica are both negatively charged, and so it is necessary to modify the mica surface or the DNA counter ion to allow binding. The counterion method is done by adsorbing the DNA onto the mica in the presence of a divalent (+2 charged) ion, like Ni^{+2}. The divalent ion will serve as a counterion on the negatively charged DNA backbone and will also provide additional charge to bind the mica. This is shown schematically in Figure 8.23.

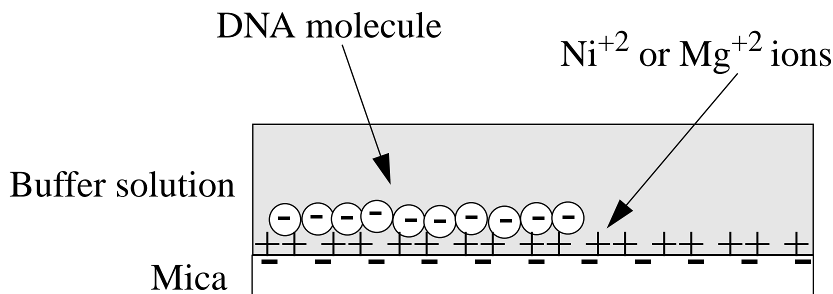


Figure 8.23. Negatively charged DNA may be bound to negatively charged mica in the presence of divalent counterions, such as Ni^{+2}.

DNA binding procedure

The following procedure is adapted from these sources:

- Dunlap, D.D., A. Maggi , M.R. Soria & L. Monaco (1997) "Nanoscopic Structure of DNA Condensed for Gene Delivery." *Nucl. Acids Res.* **25**, 3095.
- Kasas, S., N.H. Thomson, B.L. Smith, H.G. Hasma, X. Zhu, M. Guthold, C. Bustamante, E.T. Kool, M. Kashlev & P.K. Hasma (1997) "*Escherichia coli* RNA polymerase activity observed using atomic force microscopy." *Biochemistry* **36**, 461.
- Lyubchenko, Y.L. & L.S. Shlyakhtenko (1997) "Direct Visualization of Supercoiled DNA *in situ* with Atomic Force Microscopy." *Proc. Natl. Acad. Sci. USA* **94**, 496.

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

Required materials

- Mica substrates
- DNA: BlueScript II SK9(+) double stranded plasmid DNA, 2961 base pairs, 1mg/ml in 10mM Tris, 1mM ethylenediaminetetraacetic acid (EDTA) from Stratagene, La Jolla, CA.
- Buffer solution: 10 mM HEPES and 5 mM $NiCl_2$ pH 7.6 (for loose binding and air imaging), or $NiCl_2$ (for tight binding and fluid imaging)

8.10.1. Dilute DNA in buffer

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

8.10.2. Deposit drop on freshly cleaved mica.

Glue a piece of mica to a metal support as described in section 8.8.5. Cleave mica substrate with a piece of adhesive tape. Place 30 µl of the DNA solution in the center of mica disk. The DNA will bind to the mica within 1 minute.

8.10.3. Prepare AFM and fluid cell

Load prepared sample onto the AFM scanner and assemble the fluid cell, as previously described in Section 8.8.5. It may be helpful to wait for the temperature of the buffer to stabilize (20 minutes or more) before imaging.

The sample is now ready for TappingMode imaging.

8.10.4. Acknowledgments

Digital Instruments, Inc. wishes to express its appreciation to the following individuals for sharing their experience to assist in preparing this section: Monika Fritz, Manfred Radmacher, Magdalena Bezanilla, Helen G. Hansma, Paul Hansma, Jason Cleveland, Jan H. Hoh, Serge Magonov, Chris Johnson, Don Hersch, Tom Kovaleski, Gouliang Yang, Jamie Vesenka, and Eric Henderson.

8.11. Troubleshooting Tips

8.11.1. Cantilever Tune plot looks bad—loose fluid cell or tip

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

8.11.2. 8O-ring slides across sample surface, causing drift

See Chapter 15 (Section 15.10.20) in this manual.

8.11.3. Laser Sum Signal absent or weak—air bubbles

Check that all bubbles are removed from the cantilever. Bubbles may attach themselves to the cantilever, causing the laser beam to be diffracted. Bubbles can sometimes be removed by gently squirting the tip and sample with a stream of fluid. Do not squirt or splash fluid into spaces above the protective skirt.

8.11.4. Poor image quality—contaminated tip

Some types of samples may adhere to the cantilever and tip (e.g., certain proteins). If tip contamination is suspected to be the problem, it will be necessary to protect the tip against contamination. There are at least two ways of accomplishing this:

a) If the sample is adhered to a surface through diffusion (e.g., diffusion of protein onto mica) it may be necessary to first diffuse the sample substance into the substrate, then flush away stray substance by using a straight fluid media. The tip is then lowered into a fluid containing little or no stray substances which may adhere to the tip.

b) If the sample is short-lived and must be imaged quickly, it may be possible to mask the tip against contamination by bringing the tip into gentle contact with an uncontaminated substrate surface. This may be accomplished by first putting the MultiMode into contact AFM mode—switch the **AFM mode** parameter on the Other Controls panel to **Contact**. Engage the substrate surface using a zero scan size.

While the tip is kept in gentle contact with the substrate surface, the sample substance to be imaged may be added to the petri dish and allowed to diffuse/settle onto the substrate. After a diffusion/settling period has lapsed, the tip may be

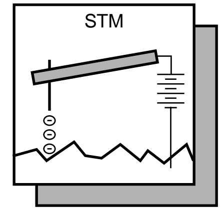
quickly lifted from the substrate surface, switched to TappingMode, then used to image the sample before it becomes contaminated.

8.11.5. Unable to locate particulate samples—attraction to cantilever

Some particulate samples such as proteins may prove difficult to find directly beneath a cantilever if the cantilever has remained stationary during a diffusion or settling period. This may be due to the fact that some types of particulates are more attracted to the cantilever than to the substrate intended to support them. The result is a "shadow" on the substrate directly beneath the cantilever where fewer sample individuals can be located—they are stuck to the cantilever.

If this problem is suspected, simply shift the imaging site to a location outboard of the tip and cantilever. More individual samples should be found there.

Chapter 9 Scanning Tunneling Microscopy (STM)



9.1. 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 (.001 µm), a tunneling current will flow from sample to tip. In operation, the bias voltages typically range from 10 to 1000 mV while the tunneling currents vary from 0.2 to 10 nA. The tunneling current changes exponentially with the tip-sample separation, typically decreasing by a factor of two as the separation is increased 0.2 nm. The exponential relationship between the tip separation and the tunneling current makes the tunneling current an excellent parameter for sensing the tip-to-sample separation. In essence, a reproduction of the sample surface is produced by scanning the tip over the sample surface and sensing the tunneling current. The first STM operated in ultrahigh vacuum on cryogenically cooled samples. In the years following its invention, many variations on the STM theme appeared. P.K. Hansma and J. Tersoff wrote a good review article on the subject, containing experiments, theory, and over 100 references for *The Journal of Applied Physics*, 61 pp. R1-23, Jan 1987. Digital Instruments, Inc. introduced the first commercial STM, the NanoScope I, in 1986.

9.1.1. Overview of STM

MultiMode SPMs shipped before spring 1996 were provided with STM tipholders that fitted to the MultiMode's standard head. Units shipped after this time must be fitted with MultiMode STM converter heads as shown in figure 9.1. These provide improved stability and visual access to the sample.

Figure 9.1. STM converter head.

There are three types of STM converter heads available: a standard version which measures current in the nanoampere range (Model #MMSTMC), a special, low-current version which measures current in the picoampere range (Model #MMST-MLC), and an electrochemistry version (Model #MMSTMEC). The low-current converter head includes an in-line electronics package; information regarding the low-current head is available in *Support Note 222*.

The STM option is generally employed under the following conditions:

- For samples having deeply relieved features, where AFM probes may not be able to penetrate, and/or where feature verticality is very close to 90 degrees.
- On polished samples, where it is necessary to image different layers having similar topography but different electrical conductivities.
- Under conditions where contact with the sample surface is prohibited.

STM relies on a precise scanning technique to produce very high-resolution, three-dimensional images of sample surfaces. The STM scans the sample surface beneath the tip in a raster pattern while sensing and outputting the tunneling current to the NanoScope control station. The digital signal processor (DSP) in the workstation controls the Z position of the piezo based on the tunneling current error signal. The STM operates in both "constant height" and "constant current" data modes, depending on gain settings on the **Feedback Controls** panel. The DSP always

adjusts the height of the tip based on the tunneling current error signal, but if the feedback gains are low (e.g., **Integral gain** < **1.0**; **Proportional gain** < **0.5**), the piezo remains at a nearly constant height while tunneling current data is collected. With the gains high (e.g., **Integral gain** > **1.0**; **Proportional gain** > **0.5**), the piezo height changes to keep the tunneling current nearly constant, and changes in piezo height are used to construct the image. The exponential relationship between tip-sample separation and tunneling current allows the tip height to be controlled very precisely. For example, if the tunneling current stays within 20 percent of the set-point value (the current to be maintained by the feedback system), the variation in the tip-sample separation is less than 0.02 nm.

9.1.2. STM hardware

Some individual STM components are described below:

Converter head and tipholder— The STM converter head (so-called because it "converts" the MultiMode to a scanning tunneling microscope) consists of a rigid ring bisected by a solid support for the tipholder. Because of the converter head's compact construction, it holds the STM tip rigid, minimizing vibrational noise. The converter head features a built-in preamplifier.

Tips— Probes for the NanoScope STM must be less than 0.012" in diameter to fit into the tipholder. 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 Digital Instruments. 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.

Preamp—Mounted within the converter head is a circuit which contains the preamplifier for the tunneling current and provides interconnections to the MultiMode SPM electronics.

The preamplifier is an FET input amplifier with an input bias current of 25 picoamps, which is small compared to the nanoamps (or fractions of nanoamps) being measured. The preamp is configured such that the tunneling tip is connected through a 1 megohm resistor to ground. The tip is also connected to the input of the amplifier which is wired as a x100 non-inverting amplifier with a cutoff frequency of 15 KHz. The transimpedance gain of the input resistor/preamp combination is 100 mV/nA with an input range from 0 to 100 nA. The noise of the preamp is essentially the Johnson noise in the 1 megohm resistor and the standard filtering is 2 mV rms, equivalent to an input tunneling current of 0.02 nA rms.

A disadvantage of this amplifier configuration is that there is a voltage drop across the 1 megohm resistor which raises the voltage of the tip above ground, reducing the effective bias voltage. The actual bias voltage is equal to:

$$V_{sample} = V_{bias} - I_{tunneling} \times R_{input}$$

This effect is accounted for in the NanoScope III software so the actual bias voltage between the tip and the sample agrees with the menu value.

9.1.3. Fine points of STM operation

Getting the best images on the NanoScope III requires a good interface between sample and tip, reasonable vibration isolation, and proper settings in the software menus. This section provides greater detail on the operation of the STM.

9.1.3.1. STM operating modes

Recall that the two operating modes available on the NanoScope STM are selected with the **Data type** option in the **Channel** panel (refer to figure 9.2). **Height** data reflects the change in tip position required to maintain a constant tunneling current. The DSP senses the tunneling current, calculates the difference from the desired tunneling current, and determines the voltage that must be applied to the piezo tube to keep the tunneling current constant. Due to the known characteristics of the piezoelectric material, 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.

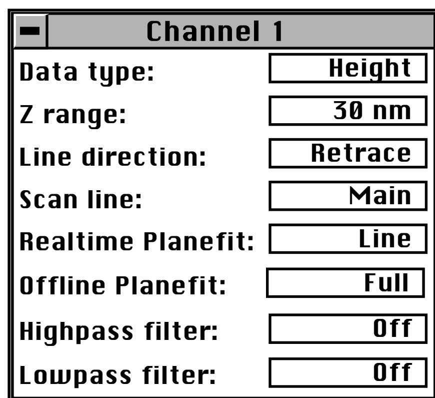


Figure 9.2. Typical STM *Channel 1* control panel parameters.

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. It should be noted that if different chemical species are present in a sample, the height data may not be a direct representation of the topography of the surface of the sample. Different atomic species within a sample may produce larger tunneling currents for a given bias voltage.

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.

9.1.3.2. STM-specific menu parameters

In addition to the **Data type** discussed above, the STM control panels contain three items that are specific to the operation of the scanning tunneling microscope. The **Feedback type**, **Bias**, and **Setpoint** parameters pertain exclusively to the control of the STM.

The **Feedback type** parameters in the **Other Controls** panel determine the transformation performed on the tunneling current prior to the feedback calculations. The three settings select **Linear**, **Log**, or **Boost** 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** and **Boost** selections calculate the error signal as the difference between the log of the instantaneous tunneling current and the log of the setpoint current. The **Boost** mode performs additional operations to optimize the feedback performance for high scan rates over rough surfaces.

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—100 mV** are typical for conductive samples, the allowable setting ranges from **-10.0—10.0 Volts**. Positive settings of the bias voltage induce negative tunneling currents (i.e., electrons flowing from the tip-to-sample).

9.1.3.3. Optimization of STM scanning parameters

The process of selecting and optimizing 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 filters 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. Having **Data type** set to **Height** is usually better for all but atomic-scale scans. In general, height data images are best at higher feedback gains and slower scan rates.

Settings for feedback gains depend on many factors, but perhaps the most important is the **Data type**. If the **Data type** is set to **Current**, the **Integral** and **Proportional gains** should be set as close to zero as possible. The **LookAhead gain** adds information from the previous scan line into the feedback calculation, so it is most useful for samples with long features oriented along the slow scan axis. The gains should be lowered for data captured using the linear **Feedback** type (**Lin**), especially with high **Setpoint** current levels. Large-scale images should be taken at increased gain, except for the **LookAhead gain**, which is best kept to zero (**0**).

When **Data type** is set to **Height**, often the best way to set gains is to view the Real Time scan in **Scope Mode** with the **Slow scan axis** set to **Disabled**. 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** and **LookAhead gains**. High frequency fuzz will appear on the signal when the **Proportional gain** is set too high. Setting the **LookAhead gain** too high will cause oscillations in the scope image and ripples on topview images. Setting the **LookAhead gain** even higher will cause the feedback loop to become unstable. The **Setpoint** current and the **Bias** voltage can also be adjusted using the scope display.

Bias voltages below 20 mV 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 100 mV or even higher may work best. For scans larger than 0.5 µm, it is sometimes better to increase the bias voltage by 50 mV to 100 mV 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 **Setpoint** current 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 **Feedback type** can be set to either **Log**, **Boost**, 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 linear (**Lin**) mode will distort data. For this reason, the **Boost** and **Log** modes—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 **Boost** mode is preferable for large scans with high vertical features such as compact disc stampers or integrated circuits. The proportional and integral gain can be reduced greatly when the **Boost** mode is used. The log input has the advantage of having a gain which is insensitive to the value of the **Setpoint** current.

Large scans cannot be taken at the same scan rate as small scans. When using the large scanners with scans above a few microns, the **Scan rate** should be lowered below 10 Hz. 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 ensure there is no image degradation due to a high **Scan rate**, lower the rate and check for changes in the image. Check the **Scope Mode** 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 **Highpass** and **Lowpass filter** parameters provide the option of filtering the tunneling current signal. A lowpass filter with a cut-off frequency of 25 KHz 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.

9.1.3.4. Tunneling tips

Although the microscope will accept any .010" diameter tip, tips made of platinum iridium (PtIr) and tungsten are used most often. PtIr and tungsten tips are supplied by Digital Instruments. 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. Glass coatings are removed from the very end of the tip by briefly applying a high-bias voltage. The quality of the mechanically formed PtIr tips will vary. Some give beautiful images as soon as tunneling starts; others start well but degrade over time; while others start noisy and stay noisy.

9.1.3.5. 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 or operate the microscope in a glove bag filled with nitrogen or argon. The standard NanoScope microscope heads were not designed to operate in UHV.

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, then select **Engage**.

9.1.3.6. 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 hood or similar cover on.

9.2. Basic STM Operation

9.2.1. Imaging samples

This section explains how to use the NanoScope to image a conductive sample (e.g., the 10-micron, silicon reference supplied with the system).

1. Select the STM option. Click the left mouse button with the cursor on the **Real Time** icon in the menu bar to select the Real Time operating mode. Pull down the **Microscope** menu by clicking on the **Microscope** item in the menu bar, click on the **Select** command, then click on **STM** and **Ok** to place the microscope into STM mode.

2. Attach a sample to a metal puck using an electrically conductive (e.g., silver-based) epoxy such as Dynaloy 325 or equivalent. Ensure that the sample itself is conductive and in electrical contact with the top of the scanner tube (the scanner cap).

3. Insert a new tip in the STM converter head tipholder. The tips come in a small, plastic snap-box filled with foam. Grip the tip with tweezers near the sharp end, then insert the blunt end of the tip into the tipholder. On some older tipholders, it may be necessary to put a slight bend in the tip to help it stay in; newer tipholders feature a curved tube to help grab the tip better. The tip must be inserted so that it protrudes beyond the bottom profile of the tipholder; otherwise, the tipholder will bottom out on the sample surface before the tip engages. Ensure that the tip is tightly held.

4. Place the converter head atop the scanner, checking for clearance between the tip and the sample as the head is lowered. If the tip appears to be too low and threatens to crash into the sample, adjust the scanner screw(s) to obtain more clearance. The head will sit securely atop the scanner under its own weight, assisted by the pull of magnets mounted in the scanner.

5. Verify that the mode switch on the MultiMode's base is toggled to **STM**. The LED on the front of the base should glow blue. For "J" scanners, set the **Scan Controls** panel parameters to the values shown below. It is sometimes helpful to start in Scope Mode (**Real Time / View / Scope Mode**) at a reduced **Scan size**. Once gains are adjusted for the small scan, the **Scan size** may be increased.

Scan Controls	
Scan size:	10.0 µm
X offset:	0.00 nm
Y offset:	0.00 nm
Scan angle:	0.00 deg
Scan rate:	2.44 Hz
Number of samples:	256
Slow scan axis:	Enabled
Z limit:	440 V

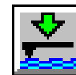
Go to the **Feedback Controls** panel. To prevent crashing the tip into the sample, verify that the **Integral gain** value is set at **1–2**, and that the **Proportional gain** value is set between **2–10**. For now, turn off the **LookAhead gain** (**0.00**). Set the **Bias** to **100.0 mV–1.0 V**, and **Setpoint** to **2 nA**.

Notes

- The Bias and Setpoint parameters must be set according to the conductivity of the sample. Samples which are excellent conductors (e.g., gold) exhibit high electron flow; therefore, the error signal is relatively large. Good conductors can operate with a Bias of 1—50 mV. Conversely, if the sample is a semiconductor, the Bias may have to be set higher (e.g., 100 mV) to obtain an image. Beware of high bias values! If the Bias is set too high for a given conductor, electrical arcing may occur, damaging the tip and/or sample.

6. At this point, the value of the head preamplifier offset may be measured using the **Offset** command in the **Real Time / Microscope** menu. Checking the **Offset** is recommended as a good final check prior to engagement. The procedure is as follows:

- Verify that the tip is withdrawn from the sample surface.
- Invoke the **Offset** command to observe the offset current of the preamplifier in nanoamps. The offset should read ± **0.1 nA**. If the value is too high, check that all cables are plugged in, that the MMAFM switch is set for STM, and that the tip is not touching the sample.



7. Engage the sample surface by selecting the **Motor / Engage** option or by clicking on the **Engage** icon. The display monitor should render an image of the sample surface immediately. Depending on the condition of the tip, the image on the screen will be either clear or noisy. The status bar on the control monitor shows how many microns the tip has traveled. When the tip engages with the surface and tunneling occurs, the computer will beep and an "Engaged" message will appear in the status bar.

The **Z Center Position** bar graph to the right of the image on the display monitor reflects the Z position of the piezo during the scan. Under most circumstances, the white bar 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.

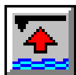
8. After the sample is engaged, the **Scan Controls** and **Feedback Controls** parameters will probably require some adjustment to optimize the image. Carefully and slowly increase the **Integral gain** and **Proportional gain** and vary the **Bias** voltage to see how the image changes. (Sudden changes to these parameters may cause a crash!) Vary the **Scan size** to observe how the image can be magnified, then vary the **X offset** and **Y offset** to see how the image can be moved. Transfer to the **Channel** panels, and instead of displaying an image using height data, select **Current** at the **Data type** field to observe image changes.

Notes

- Generally, the **Date type** field is set to **Height** on the **Channel** panel whenever the Multimode is used in STM mode to image sample surfaces with large fields of view. The **Current** setting is usually selected for imaging atomic features.

9. Click on the **Scope Mode** command in the **View** menu (or use the **Scope Mode** icon) to see what the signal looks like for each scan. Return to **Image Mode** by clicking on the **View / Image Mode** command or the **Image Mode** icon.

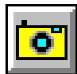
A poor tip—either because it has touched the sample, has contamination on the end, or because of its manufacturing—will render a poor image. In this case, there is little likelihood that the tip will recover and the best procedure is to replace the



tip. Pull down the **Motor** menu by clicking on **Motor** in the menu bar, then click on **Withdraw** (or click on the **Withdraw** icon) to stop scanning, and retract the probe from the surface. After the tip is withdrawn, the “Secured” message will appear in the status bar of the control monitor. Remove the bad tip and install a new one. Verify there is sufficient clearance between tip and sample after installing the new tip—a new tip will extend a different distance out of the tipholder than the old tip.

To Improve Image Quality...

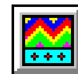
- Vary the **Scan size** and the **X offset** and **Y offset**. Move to a new location on the sample by changing the X offset and/or Y offset of the scan. The X and Y offset parameters define the center of the scan. Often, a better image can be obtained on a different portion of the sample. The X and Y offsets can be changed by altering the values in the control panel, or by using the **Offset** command in the menu bar on the display monitor.
- Alternate the commands **Withdraw** and **Engage** a couple of times. This raises and lowers the tip and may get rid of contamination on the end of the tip.
- Use the **Step Motor** command to single-step the tip up a step or two. Be sure to minimize the **SPM step size** before stepping the motor down. Watch the **Z Center Position** scale on the display monitor 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 10 nA. As a last resort, current may be increased up to 48 nA for a very brief period of time.
- As a last resort, oscillate the tip by setting the **Integral gain** high (about **100**) for a second. The oscillation will show up on the display as a grainy pattern of light and dark. Set the **Integral gain** back down 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.
- If none of the above procedures improves the quality of the signal, replace the tip and try again. If that doesn't work, call Digital Instruments for technical assistance.



10. After a clean image has been obtained, it can be saved onto disk with the **Capture** command in the **Capture** menu (or click on the **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 **Capture** directory. The **Capture Filename** command can be used to define a custom filename or the captured file will be named with a date/time stamp. In either case, a three-digit suffix which is incremented 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, etc.

11. Some simple filtering operations can improve the look of the captured image. Enter the **Off-line** mode and select an image by clicking on it in the file list. There are a number of image processing options available in the **Off-line / Modify** menu. Click on **Modify** in the menu bar to display the menu. The simplest way to remove high frequency noise is using a lowpass filter.



Pull-down the **Modify** menu and click on the **Lowpass** command to apply the low-pass filter to the image and redraw it on the display monitor. Successive **Re-execute** clicks will continue to filter and redraw the image. The entire filtering operation can be undone with the **Undo** command, the filtered image can be saved, or the **Quit** command will return the system to the **Modify** menu. There is also the option of changing the display with the **Color table**, **Color contrast**, or **Color offset** parameters.

Notes

- The **Z range** parameter will have no effect if the **Plot type** parameter in the **Top View** menu is set to **Equal** area.

9.3. 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 with the **View / STS Plot** modes. These are discussed in this section. A related form of imaging, current imaging tunneling spectroscopy (CITS) is described in an appendix of the *Command Reference Manual*.

9.3.1. STS plot modes

In the **STS Plot** modes, 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)— The tunneling current as a function of the bias voltage is displayed. The tip height is held constant while the current (I) versus voltage (V) plot is being acquired. In addition to I versus V, it is also possible to plot the following:

$$\frac{di}{dv} \text{versus V}$$

$$\frac{d\ln(i)}{dv} \text{versus V}$$

$$\frac{d\ln(i)}{d\ln(v)} \text{versus V}$$

STS i(s)— The tunneling current as a function of the tip height is displayed. The bias voltage is held constant while the current (I) versus tip height (S) plot is being acquired. In addition to I versus S, it is also possible to plot $\ln(I)$ versus S.

9.3.2. Operation of STS

In the following sections, the operation of the spectroscopic functions of the NanoScope III 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 to 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. Since the tip is not tracking any topography, the lower gains are acceptable and tend to make the plots more stable.

2. The maximum input range of ±100 nA on the NanoScope with the standard preamps will restrict the current-versus-voltage plots.

3. The spectroscopic functions do not adjust for any loss of bias voltage due to IR losses caused by the input impedance of the preamplifier. This has different effects for the STS i(v) and STS i(s) functions which are discussed individually below:

STS i(v)

The spectroscopic plots do not correct for reductions in bias voltage caused by the preamplifier, which for current-versus-voltage is typically small, anyway. For example, a 1-volt scan on the bias voltage, producing a 50 nA response in the tunneling current would have a 5 percent error at the extremes of the scan (50 nA X 1 MΩ = 50 mV or 5 percent of 1 volt).

Input impedance of the preamplifier leads to reduced bias voltages at increased tunneling currents. This effect can cause the I vs. S plot to be inaccurate. For example, a bias voltage of 20 mV will restrict the upper limit of the tunneling current to 20 nA since that level of current would effectively reduce the bias voltage to zero. Even before the tunneling current gets to 20 nA, the reduced bias voltage will make the measured current appear lower than it should be for a given tip height.

9.3.3. Continuous Imaging Tunneling Spectroscopy (CITS)

For information regarding CITS, see Appendix C in the *Command Reference Manual*.

9.4. Obtaining Images with STM

This section gives suggestions for obtaining STM images. Procedures are included for imaging:

1. Highly ordered pyrolytic graphite (HOPG).

2. A gold calibration ruling.

Note that vibrational stability is crucial to obtaining atomic-scale images. This is achieved through the use of vibration isolation and, more importantly, the use of shorter, more rigid scanners (e.g., "A" or "E" scanners). In some limited cases, it may be possible to obtain atomic-scale images using larger scanners ("J"); however, this often proves difficult or impossible for certain materials.

9.4.1. Imaging highly ordered pyrolytic graphite (HOPG)

This section describes how to use the NanoScope to image graphite atoms in highly ordered pyrolytic graphite (HOPG).

1. The microscope should be placed on a vibration-isolation platform and connected to the controller. (You may find it easiest to perform this at step 5 below.)

2. As it comes from Union Carbide, the HOPG sample is already cleaved and clean enough for tunneling. There is usually no need to re-cleave the sample. If you feel an overwhelming need to cleave the sample again, it can be done by carefully pulling off a layer with sharp tweezers or pressing some sticky tape to the surface and pulling off a layer. You will notice that graphite is slippery and easily dropped. Be careful! Verify that the sample is electrically bonded to the puck.

3. Insert a new tip in the tip-holding tube mounted on the white ceramic cross-member of the converter head. The tips come in the small plastic snap-box filled with foam. The tips are inserted into the foam blunt end first so the sharp end of the tip protrudes from the foam. Grip the tip with a tweezers near the sharp end and insert the blunt end of the tip into the tipholder. The tip should be inserted so that it protrudes about 2 mm and should be fairly tight in the tipholder.

Notes

- Handle the converter head as little as possible during these operations to prevent thermal drift.

4. Place the head on the three magnetic balls mounted on the threaded screws in the scanner. (Make sure that the screws are extended enough so that the tip does not hit the sample.) The head has a hole, a groove, and a flat which mate kinematically with the three magnetic balls. A suggested method of placing the head on the scanner is to slowly lower the head while it is slightly misaligned until its underside touches the screws, then slowly rotate the head until the hole and groove on the underside mate with the screws (all the while watching the tip-sample separation). If it looks like the tip will hit the sample, toggle the motor switch **Up** to raise the head and/or use the coarse adjustment screws.

After the head is securely mounted on the magnetic balls of the base, insert the 15-pin preamp plug into the socket on the scanner support ring. When placing the head down onto the balls, try to prevent it from slamming onto the balls, as excessive pitting in the flat area can cause irregular engagement.

5. Looking at the tip with the 25X magnifier, lower the tip down with the coarse-adjustment screws until the tip is about a tenth of a millimeter or less (about .004

inch or less) above the sample. It is usually easier to use only one coarse-adjustment screw to do this if you do not need to move the tip too far. Try and adjust the screws such that the head is not tilted to one side or the other. If necessary, adjust the rear screw so that there is a minimal amount of tilt in the head from front to back. A reflection of the tip will be visible on the sample. Using the coarse-adjustment screws, lower the tip until there is only a slight gap between the end of the tip and its reflected image — do not bring the ends together. If you do, you will almost certainly have to replace the tip. Don't worry at this point if the head is tilted.

If you haven't placed the microscope on the vibration-isolation platform, do so now. Verify that the mode switch on the left side of the MultiMode's base is toggled to **STM**.

6. Be sure the power to the controller is on. If you have not already done so, start the program by entering "**Z**".

7. Set the scan parameters. Pull down the microscope menu by clicking on the **Real Time / Microscope /Select** command, then click on the scanner being used. Click the **Ok** button to select parameters for the converter head. The "A" scanner is the preferable option for imaging on an atomic scale, but the "E" scanner has also been used effectively.

- To look at graphite atoms, it is necessary to set the **Scan size** to about **5–20 nm**. Use the mouse to change the parameter. Select the parameter and drag the mouse right and left with the left button pressed to change the parameter; the new parameter value will take effect when the mouse button is released. The right or left arrow keys can also be used to set this parameter. Note that the up and down arrow keys move from one item to the next, while the right and left arrow keys change the selected parameter. Numerical parameters may also be set by simply typing over the existing values and pressing the enter key.
- Set the remaining parameters so that the **Feedback Controls** panel is as shown in figure 9.3 below.

8. As a final check, click on the **Offset** command in the **Real Time / Microscope** menu. The offset should be very close to **0.0 nA**. Proceed to the next step if the system reports a reasonable value.

If the offset reads ± **99 nA**, the feedback loop is open (i.e., the microscope or controller cables are not properly installed), or the tip may be in contact with the sample surface. Check this by clicking on the **Withdraw** command and retesting the offset . Remedy this problem before continuing.

Feedback Controls	
Integral gain:	2.00
Proportional gain:	2.00
LookAhead gain:	0.00
Bias:	20.0 mV
Setpoint:	2.00 nA
Analog 1:	0.00 mV
Analog 2:	0.00 mV

Figure 9.3. *Feedback Controls* panel for graphite.

9. Now, let's start tunneling. Click on the **Real Time / Motor** item and then click on the **Engage** command, or use the **Engage** icon. The status bar on the control monitor shows how many microns the tip has traveled. If the travel distance gets up to 250 microns, engagement will halt and the software will prompt you to check the tip alignment. When the tip engages with the surface and tunneling occurs, the computer will beep and an "Engaged" message will appear in the status bar.

If the message "Engaged" occurs immediately after the engage command is given, there is a problem. Either the tip was already touching the surface due to misadjustment of the coarse-adjustment screws, the power to the controller was not turned on, the wrong head was selected, or the 15-pin connector to the scanner was not connected. If the tip was on the surface, you can try backing off the screws and try again. However, after the tip has crashed into the surface, the images will tend to be unacceptably noisy. To try again, click on the **Withdraw** command. The "Secured" message will appear in the status bar of the control monitor. Then, click on the **Engage** command.

Notes

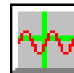
- If version 4.22 or higher software is used, the STM must have the **In polarity** parameter set to **Forward** on the **Microscope / Calibrate / Detector** panel. On older microscopes, the setting didn't matter and may have been set to **Reverse**. This setting will cause false engagement with newer Z.exe programs.

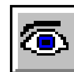
10. At this point, an image should begin to form on the screen. Depending on the condition of the tip, the image on the screen will be either clear or noisy. Let's treat the two cases separately:

- A good signal is one which clearly shows the repeating pattern of the carbon atoms. Compare this image with the graphite image that may be stored in the C:\DATA directory. Check to see how the image repeats on scans from bottom-to-top and top-to-bottom. Usually, when the tip is first engaged, there will be a drift in the piezo which will cause the image to repeat poorly. The most obvious sign of drift is features which are compressed vertically in one frame and vertically elongated in the next frame. This drift will decrease with time.

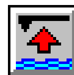
Click on a scan parameter and drag the mouse to change the parameter. Lower the **Integral** and **Proportional gains** near zero and vary the **Bias** voltage to see how the image changes. Vary the **Scan size** to get a feel for how the image can be magnified and vary the X and Y offsets to see how the image can be moved. Instead of displaying an image of the tunneling current, select **Height** at the **Data type** line to display the Z (vertical) signal. You will have to increase the gains and decrease the scan rate to get a good Z-height image.

To use an "E" or "J" scanner on an atomic scale with the **Data type** set to **Height**, the **Z limit** parameter (**Scan Controls** panel) should be decreased to **55 Volts**.





Click on the **Scope Mode** command in the **Real Time / View** menu to see what the signal looks like for each trace across the sample. Return to the **Image Mode** by clicking on the **View / Image Mode** command, or icon.



- A poor tip, either because it has touched the sample, has contamination on the end, or because of its manufacturing, will give a noisy signal. In the worst case, the image will have no pattern at all, and Z on the display will run off scale in both directions. In this case, there is little likelihood that the tip will recover, and the best procedure is to replace the tip. Pull down the **Real Time / Motor** menu, then click on **Withdraw** (or click on the **Withdraw** icon) to stop scanning, and retract the probe from the surface. After the tip is withdrawn, the "Secured" message will appear in the status bar of the control monitor. Before removing the head, it's a good idea to execute the **Withdraw** command several times to protect the sample from being scratched when the head is picked up. Extend the scanner screws a few turns, unplug the preamp, remove the head, and replace the tip. Try to leave the new tip protruding the same amount as the old one. Be sure there is sufficient clearance after installing the new tip, because the new tip will extend a different distance out of the tipholder than the old tip.
- The nominal Z position of the piezo provides useful diagnostic information. The bar graph to the right of the image on the display monitor reflects the Z position of the piezo during the scan. Under most circumstances, the "Z center position" bar should be in the center of the graph to indicate 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. If the "Z center position" reaches the "Extended" end of the scale, the maximum voltage is being applied to the piezo, but the tip still cannot reach the surface. Similarly, the "Retracted" end of the scale means that the maximum voltage of opposite polarity has been applied to the piezo, and the tip cannot be lifted from the surface. The "Retracted" end of the scale is more dangerous, because damage to the tip or sample can result.

If the "Z center position" gets close to either limit, use the **Withdraw** command followed by the **Engage** command to reposition the "Z center position" near the middle of its range. If there is drift in the Z center voltage, use the **Real Time / Motor / Step Motor** command with a small **Step size** to keep the tip within the Z piezo range. This drift will eventually settle out.

EXCEPTION: The **Z limit** represents only a portion of the maximum **Z range** (440 V). When using a **Z limit** of less than **440 V**, version 4.xx software will attempt to recenter the Z-axis if you double-click on the **Z limit** value or change it.

Sometimes the tip will show atomic structure on graphite with noise which is large enough to reduce the definition of the structure. In these cases, it may be possible to reduce the noise without replacing the tip. Any or all of the following procedures may be tried[1]:

- Vary the **Scan size** and the **X** and **Y offsets** (**Scan Controls** panel). Move to a new location on the sample by changing the **X** and/or **Y 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** command in the menu bar on the display monitor.
- Alternate the commands **Withdraw** and **Engage** a couple of times. This raises and lowers the tip and may get rid of contamination on the end of the tip.
- Use the **Real Time / Motor / Step Motor** command to index the tip down a step or two. Be sure to minimize the **Step size** before stepping the motor down. Watch the "Z center position" scale on the display monitor to be ensure the piezo has enough room to retract so the tip will not harm the surface of the graphite. 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 even a moderately noisy tip. Sometimes, though, bad tips can be reconditioned by being driven several steps into the surface and then withdrawn and re-engaged.

1. These suggestions also apply to larger-scale STM imaging and are provided in section 9.2.1 of this chapter.

- Vary the **Setpoint** current over the range **2—10 nA**. As a last resort, you can increase it up to **48 nA** for a very brief period of time.
- As a last resort, oscillate the tip by setting the **Integral gain** high for about one second. The oscillation will show up on the display as a grainy pattern of light and dark. Set the **Integral gain** back down and check if the signal is quieter than before.
- If none of the above procedures improves the quality of the signal, you should cleave the sample, replace the tip, and try again. If that doesn't work, review all parameter settings or call Digital Instruments for technical assistance.

9.4.2. Imaging a gold calibration ruling.

A gold calibration ruling is supplied with systems purchased with the 12-µm range, "E" scanners. This section describes how to image the ruling and assumes you have performed the previous section for imaging graphite.

1. Although it is not as important for large-scale scans, the microscope should be put on a vibration isolation platform. Also, connect the microscope to the controller according to the instructions in the earlier part of this chapter.

2. Attach the calibration ruling to a sample puck with the ruled side facing up. Use carbon glue or silver epoxy to provide a conduction path between the sample puck and the top surface of the ruling. This step is very important because without a conduction path the **Bias** voltage will not be applied to the sample surface and the tip will crash into the sample surface in a effort to detect the tunneling current.

3. Verify that a tip is in the tip-holding tube attached to the cross-member on the converter head.

4. Place the head on the three magnetic balls mounted on the adjustment screws in the scanner. Make sure the screws are extended enough that the tip does not contact the sample.

5. Looking at the tip with the 25X magnifier, lower the tip down with the coarse-approach screws until the tip is about a tenth of a millimeter or less above the sample. This can be done by bringing the end of the tip as close as possible to the reflected image without allowing them to meet. If the tip does touch the sample, replace it, as it will almost certainly be unusable.

6. Be sure the power switch in the right rear of the controller is in the on (up) position. The power lamp on the front panel of the controller will be illuminated.

7. If you have not already done so, start the main program by entering “Z”.

- Go to the **Real Time** mode.
- Click on the **Microscope / Select** command, then click on **Small Sample** ("Base" box), **STM** ("Head" box) and the appropriate "Scanner" selection (e.g., **MMSTME** if the "E" scanner is being used).

Set parameters in the **Feedback Controls** panel as shown in figure 9.4. The **Scan Controls** panel can have its parameters set as follows: **Scan size 100 Volts**; **Z limit 440 V**; **Number of samples 256**; **Scan rate 2.44 Hz**. Set the **Data type** parameter on the **Channel 1** panel to **Height**.

Feedback Controls	
Integral gain:	10.00
Proportional gain:	5.00
LookAhead gain:	0.00
Bias:	500.0 mV
Setpoint:	5.00 nA
Analog 1:	0.00 mV
Analog 2:	0.00 mV

Figure 9.4. STM control panel for calibration ruling

9. Click on the **Real Time / Motor / Engage** command to begin scanning. (See section 9.4.1 for suggestions on adjusting parameters.) At this point, an image should begin to form on the screen; the supplied gold sample has cross rulings of 1µm pitch. Go to the **Scan Controls** panel and increase the **Scan size** item to **10 µm**. If the picture appears grainy or noisy, the tip is probably poor and should be replaced. In any case, you can try the following procedures to improve the image:

- Change the **Integral** or **Proportional gain** parameters. Gain values which are too high will cause graininess in the image, whereas low gains will cause image smearing, particularly where the surface has sharp, abrupt features.
- Vary the **Scan rate**. A **Scan rate** which is too high will cause smearing (like a low gain setting) and lower scan rates may test your patience.
- Vary the **Setpoint** current and **Bias** voltage items.

9.5. Troubleshooting for STM

This section addresses errors or malfunctions encountered during the operation of the MultiMode as an STM. Consult also Chapter 15 in this manual for additional troubleshooting tips.

9.5.1. . Head and microscope-related problems

This section deals with problems related to the scanners or the microscope. If a problem exists with a scanner, try a second one under the same conditions, if possible. Otherwise, the following list of symptoms and cures may be helpful:

9.5.2. . Head engages immediately

If the STM engages immediately after initiating the **Motor / Engage** command, then one of the following probably occurred:

tip on surface— Make sure the tip is not touching the surface of the sample. Adjust the coarse-adjustment screws upward until the tip is far from the surface.

controller off— Verify that power to the controller is on and that the controller is connected to the computer workstation via the beige 25-pin cable.

MulitMode base switch not set for *STM*— Verify that the switch on the left side of the MultiMode base is toggled to **STM**.

head has offset— Follow directions in section 9.4.1 to determine whether the scanner requires adjustment to its offset.

In polarity* parameter set to *Reverse— Check to see that the **In polarity** parameter on the **Detector Calibration** panel (**Microscope / Calibrate / Detector**) is set to **Forward**.

disconnected— Verify that the 15-pin microconnector from the head is plugged into the microscope and the microscope is plugged into the controller.

9.5.3. Tip never engages

If the tip never engages, test for the following:

disconnected—Make sure the microscope is connected to the controller.

binding on drive screw— Feel the rear, motor-driven adjustment screw during engagement to verify that it is rotating. If the motion is erratic, then the screw is

fouled with debris and binding. Remove the screw, clean threads and regrease per instructions provided in section 15.3 in this manual.

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.

bias not applied to sample surface— In some cases (e.g., the calibration reference), the sample consists of a layer of electrically conductive material on top of an insulator. In these cases, a conduction path must be provided between the sample puck and the sample surface. Carbon glue or silver epoxy can be used to connect the bias voltage to the sample surface. With NanoScope software versions 4.23b3 or later, verify that the **In polarity** parameter on the **Detector Calibration** panel (**Real Time / Microscope / Calibrate / Detector**) is set to **Forward**.

9.5.4. Tip crashes

If the tip always crashes into the surface with the **Z Center Position** 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/cm, 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.

clean contacts— The magnetic balls on the ends of the adjustment screws tend to attract small metallic particles which may make the surface gritty. Clean them with adhesive tape and then wipe with alcohol. Also, clean the kinematic mating surfaces on the underside of the head.

check for binding in the motorized screw— Make certain the motorized adjustment screw rotation is smooth and not erratic, which can cause the screw to advance in jumps. If this is the problem, clean the screws as described in section 15.3 in this manual. On newer scanners, the setscrew used to adjust tension on the motorized screw's plastic threaded bushing may need to be slightly loosened.

check sample conductivity— there are two problems associated with sample conductivity. First, the bulk conductivity of the sample may make it more difficult to image. If the resistance of the sample is greater than 1 kΩ/cm, higher bias voltages should be tried. If the resistance is greater than 1 MΩ/cm, bias voltages of 100 mV or more should be used. Samples with resistances of 1 MΩ/cm 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 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.

check the polarity of the Z-axis piezo— Use the **Calibrate** command in the **Real Time / Microscope** menu to review the calibration parameters for the head in use. The **Z polarity** should be set to **Forward**. (**EXCEPTION:** A few rare scanners have serial numbers ending in "RP," reverse polarity; these should be set to **Reverse** instead.)

9.5.5. Problems during *Real Time* operation

This section deals with problems that are associated with the Real Time aspects of the NanoScope III software although they often point to the scanning head or microscope.

9.5.6. 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 larger (e.g., "J") scanners may be less tolerant of sample tilt due to their having small vertical range compared to larger lateral scan range. Note the trend in **Z Center Position** across the sample scan profile using **View / Scope Mode**. Verify that the

sample surface is parallel to the sample puck before mounting it atop the scanner cap. Also verify that the channel panel (e.g., **Channel 1**) has its **Realtime Planefit** parameter set to **Offset** or **None**.

Increase *Z limit* parameter— If set too low, try increasing the **Z limit** parameter value (**Scan Controls** panel).

9.5.7. 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 scanner has been stored in a cold place overnight. Drift can be minimized by keeping the STM in a thermally stable environment. Avoid operating the microscope near windows and air ducts.

tip not tight— Make sure the tip is held tightly in the tipholder. Push and pull on the tip with tweezers to see that it is not loose. (On older tipholders, put a slight bend in the tip to help it mount firmly in the tipholder tube if it seems loose.)

sample hold-down— Verify that the sample is flat against the puck 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.

scanner screws-converter head contact— Clean mating surfaces between the underside of the converter head and the scanner adjustment screws. If dirt or oil are evident, carefully remove with a clean wipe and alcohol. Some scanners utilize plastic threaded bushings in the scanner body through which the adjustment screws are threaded. If the bushings become fouled with grit, or if they are loose, drift may result. If loose, gently tighten setscrews in the scanner body to increase tension on the adjustment screw bushings. (Do not overtighten!) For more information regarding removal and replacement of adjustment screws, refer to section 15.3 in this manual.

stiff rear screw— If the rear screw is difficult to turn (due to an accumulation of dust in the threads, etc.), the drive shaft can wind up while driving the screw and unwind after the stepper motor has stopped. This will cause the tip to continue towards the surface. Clean the threads and screw per section 15.3 in this manual.

9.5.8. *Real Time* image hides features

If the real time image appears flat, but captured images reveal detail, try rotating the **Scan angle** to 90°. The real time image leveling software tends to hide features that are parallel to the fast scan direction.

9.5.9. Image is streaky or wavy

If high-resolution images appear streaky or wavy, it may be a result of the following:

Insufficient vibration isolation— Atomic-scale scans are the most susceptible to vibration in the acoustic and subacoustic frequencies. Verify that the MultiMode is acoustically isolated with a functioning isolation pad and, if required, an acoustic hood. If vibrational noise persists, try moving the microscope to a less noisy location or using an isolation tripod.

Bad tip— Many strange effects have been pondered and hunted down for hours only to find that changing the tip fixes the problem. Try a different tip to see if the problem persists.

Low scan rates— The brute force method for reducing the effects of drift and even low frequency vibration is to simply increase the **Scan rate**. Collect small-scale images at **122 Hz** with 256 x 256 sample points.

9.5.10. 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* or *Boost* feedback**—The most dramatic increase in performance of the feedback over large scans is achieved using the **Log** or **Boost** feedback modes. These make the error signal symmetrical for conditions with the tip too far or too close to the surface. **Boost** mode further optimizes the feedback performance for large scans.

Gain—The settings of the **Integral** and **Proportional gain** parameters also tend to be critical for large scans. They should be just sufficient to produce a slight fuzz on the image, but not so high as to cause 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 **Setpoint** current 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 ($i \approx e^{-S}$).

9.5.11. Image goes white ("A" scanners)

If images go white after a few scans and the **Z Center Voltage** is still within range, check the **Realtime Planefit** parameter on the active **Channel** panel. When using an "A" scanner, it should always be set to **Line**. In general, this will be a problem anytime there is a significant amount of drift in the Z center voltage, but drift is especially prevalent with "A" scanners.

9.6. Overview of Low-Current STM

The following sections provide detailed instructions for performing low-current (sub-picoamp) STM using two Digital Instruments devices: the Low Current STM Converter for the Contact AFM and the LFM (Model #CSTMLC); and, the Low Current STM Converter for MultiMode AFM (Model #MMSTMLC), or with the Extender™ Electronics Module (Model #MMSTMLCE). The reader will find a general description in Section 232.8, followed by detailed instructions for use of Digital Instruments' low-current converters.

9.7. Description

Operation of conventional STMs with current (I_{tun}) in the nanoamp range is characterized by strong tip-sample force interactions. The forces applied to the sample during STM imaging in air actually exceed the forces in atomic force microscopy (AFM). The effects of high STM forces were found when imaging materials which included graphite [1a], inorganic layered compounds[1b], organic conducting crystals [1c] and organic adsorbates on conducting substrates [1d].

For metallic surfaces, the tip-sample gap resistance, $R_{gap} = V_{bias}/I_{tun}$, can be used as a qualitative measure of tip-sample separation. Where

$$V_{bias} = \text{bias voltage}$$

$$I_{tun} = \text{setpoint current}$$

In general, decreasing I_{tun} will increase the tip-sample separation and decrease the tip-sample force.

By operating at R_{gap} in the GΩ range, for example, $V_{bias} = 1.0\text{ V}$ and $I_{tun} = 1\text{ pA}$ the tip-sample distance increases enough to allow less-destructive imaging of a variety of surfaces.

The ability to conduct STM measurements in a wide range of R_{gap} can also be useful for experiments aimed at observing the image variations as a function of R_{gap}.

Picoampere-level STM provides the opportunity to image insulating samples deposited on conducting substrates. Indeed, it was shown that using STM at ultra high R_{gap} allows imaging of the topmost layers of alkanethiol adsorbates on gold surfaces and to reveal earlier unknown surface features.

Picoampere-level STM also opens the possibility of imaging biological macromolecules such as DNA deposited on a non-conducting mica substrate. Though such imaging was explained by lateral conductivity across the water overlayer covering surfaces in air, a true mechanism of electron transfer is not yet established.

9.8. Description of Hardware

The Low Current STM head, which allows STM measurements with I_{tun} in the pA range, is a new product of Digital Instruments, Inc. This head is constructed for operation with our MultiMode and AFM bases.

Low-current STM operation also requires the Picoamp Boost Box, which is installed between the control unit or extender box and the MultiMode or AFM base.

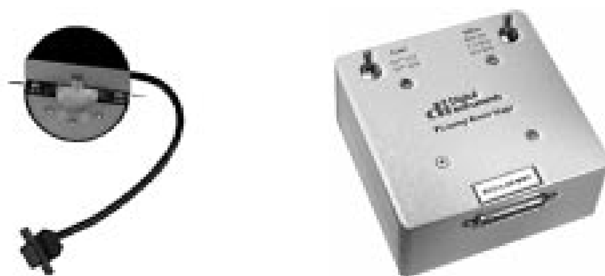


Figure 9.5. Low-Current Converter components: MultiMode head and Picoamp Boost Box.

The gain is switchable between 10^{10} V/A or 10^{11} V/A and the bandwidth is switchable among 400 Hz, 1.5 kHz and 4 kHz. Lower bandwidths allow for lower noise levels which are essential for operation at sub-picoampere I_{tun} .

IMPORTANT: The default calibration value of the detector in standard STM mode is 10 nA/V. This value should be changed to either 0.1 or 0.01 nA/V for operation of the low-current head with gain factors of either 10^{10} and 10^{11} respectively. To changes values, click on **Real-time / Microscope / Calibrate / Detector**. The **In sensitivity** parameter is listed on the **Detector Calibration** panel and can be changed by simply entering the new value.

9.9. Precautions

Due to the low-current head's highly sensitive electronic components, special precautions must be taken.

1. The input stage operational amplifier is extremely vulnerable to electrostatic discharge (ESD) and is easily destroyed. It is recommended that the user be grounded with a wristband at all times that the low current head is handled, especially when tips are installed.

2. The feedback resistor and capacitor in the input stage are extremely vulnerable to contamination. At no time should these delicate components ever be touched with the hands. If these components are accidentally touched, they must be rinsed clean in a highly purified degreasing fluid immediately.

3. Good grounding is essential for low-noise performance. A good contact between the SPM base and the metallic cover (can) included with the low current head is needed to reduce the electrical noise level. A simple grounding kit is included with the converter which includes a length of wire and connecting lugs. Make certain that the can cover and SPM base are electrically connected by the wire and that the can cover is in place. The can cover acts as a Faraday shield.

4. Leakage and offset current:

- Offset current values vary slightly from head to head but are typically in the range of 0.5-0.1 pA. Therefore, minimum operating currents are just slightly below the 1 pA value.

5. Please note that cables connecting the SPM base, the Picoamp Boost Box and the NanoScope controller should not be lengthened if possible.

IMPORTANT: Always disconnect the Picoamp Boost Box and Low-current STM Converter head before performing other types of imaging.

9.10. Installation

The low-current STM converter is designed to be installed onto one of four Digital Instruments devices: 1) the MultiMode SPM (standard); 2) the MultiMode SPM (with Extender™ base); 3) the dedicated AFM; and 4) the dedicated LFM. Generally, installation consists of cable connections and nothing more; however, cabling differs somewhat between the devices.

9.10.1. MultiMode SPM

IMPORTANT! If you are installing on a MultiMode SPM equipped with Extender™ Electronics Module for performing phase modulation MFM and EFM work, you must install the Picoamp Boost box with the jumpers inside set for extended. A label on the outside of the Boost box indicates whether it was set for standard (Model MMSTMLC) or Extender (Model MMSTMLCE) at the factory. If you are not certain which base your MultiMode has, contact Digital Instruments for guidance.

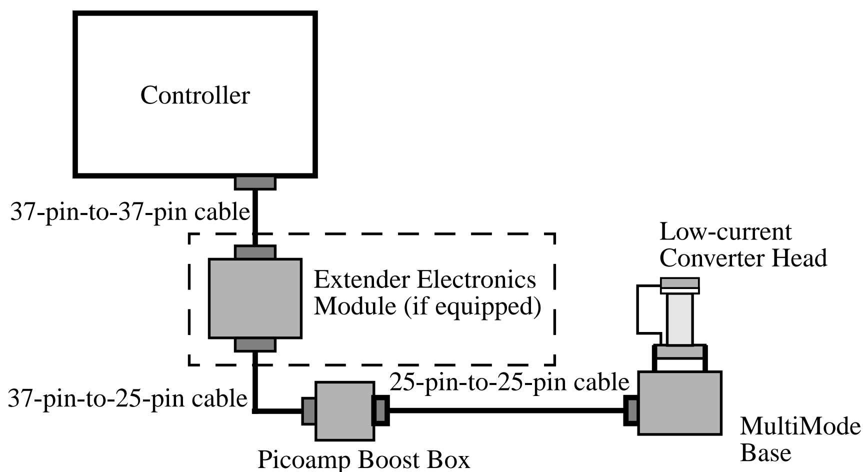
1. Turn off all power to the SPM controller. Disconnect and remove the MultiMode head.

2. Connect the picoamp booster box (male receptacle) to the controller using the 37-pin-to-25-pin connector cable. If an Extender™ Electronics Module is being used with the system, insert it between the controller and picoamp booster as diagrammed below in this section.

3. Connect the picoamp booster to the SPM base using the 25-pin-to-25-pin cable connector.

4. Connect the low-current converter head to the MultiMode's support ring using the micro-D, 15-pin connector cable. Set switches on the picoamp booster to the desired current level and bandwidth.

6. Before repowering the system, double-check all cable connections. If atomic-level scans are intended, verify the cable between the SPM and picoamp booster box is unstrained (a taut cable will transmit vibrations). The cable should rest loosely. Before loading samples and running the SPM, attach the ESD wristband to prevent sudden electrical discharges.



9.11. Operation

Once you check that leakage is absent and the offset current is small, the operation of the low-current STM head is typically checked in the atomic-scale imaging of graphite and the large-scale imaging of a gold-coated grating. The atomic-lattice of graphite is well resolved in the images obtained with I_{tun} in the 2-20 pA and V_{bias} in the 20-100 mV range. Mechanically-sharpened Pt/Ir tips are generally used for such measurements, and a tripod is good enough for the vibrational isolation of the SPM base.

9.11.1. Peculiarities

The transition to low current, however, brings some limitations, which are absent in STM measurements with standard heads. The bandwidth of the amplifier and, consequently, of feedback is substantially narrower than that used for standard measurements in the nA range (12 kHz). Therefore, the feedback mechanism can be less effective at high scan speeds. This must be taken into account, especially during imaging of corrugated surfaces.

However, the low current STM head can be used for imaging of atomically flat surfaces such as graphite and inorganic layered crystals at line frequencies up to 50 Hz and still achieve atomic-scale resolution.

NanoScope II Users: The low-current STM converter head may be used with the AFM base and the NanoScope II version 5.5 software. The software should be run in TipView mode. Readings will be seen in nanoamps; however, actual values are in picoamps.

9.11.2. Illustrative Examples

Different prototypes of the low-current STM head were used for measurements in several laboratories. In addition to the control measurements routinely conducted on graphite, the operation of the low-current STM head has been checked on several layered crystals and alkanethiol layers deposited on gold.

Several images obtained using low-current STM are presented below.

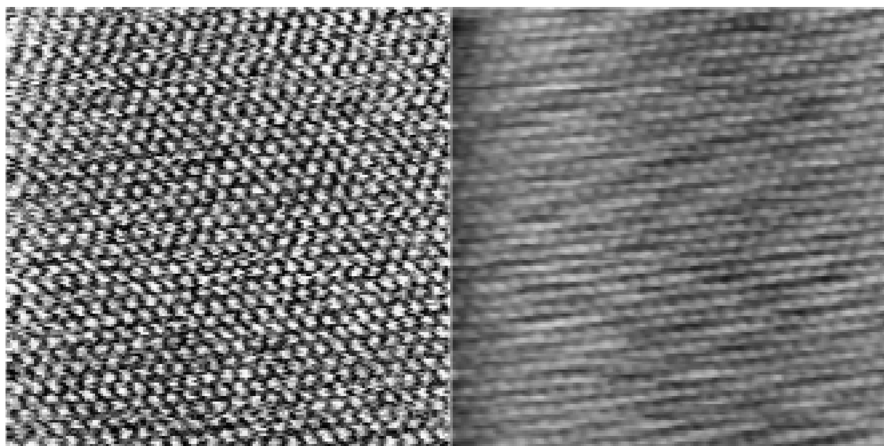


Figure 9.6. STM current and height images of HOPG surface.
Scan size = 6.0 nm, I_{tun} = 1.6 pA, V_{bias} = 29 mV.

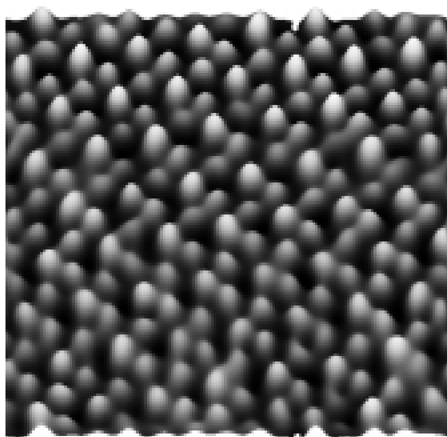


Figure 9.7. STM current image of layered crystal α-$RuCl_3$.
Scan size = 4.48 nm, I_{tun} = 1.5 pA, V_{bias} = 42 mV.

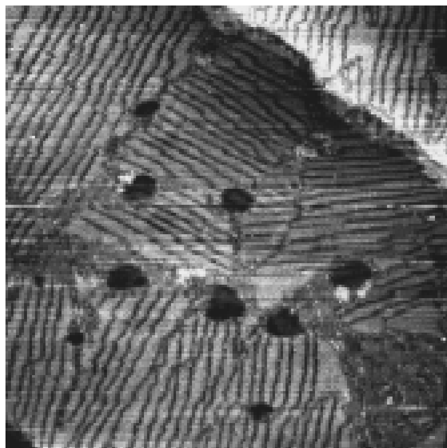


Figure 9.8. STM height image of alkanethiol layer on Au (111) substrate.
Scan size = 178.5 nm, I_{tun} = 2 pA, V_{bias} = 1 V. (Courtesy of Dr. I. Tuzov, NCSU)

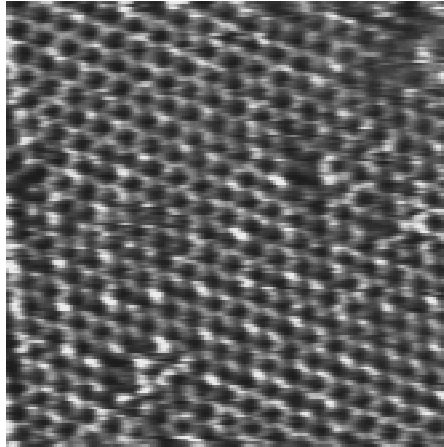


Figure 9.9. Molecular-scale STM current image of alkanethiol layer on Au (111) substrate. Scan size = 10.0 nm, I_{tun} = 13 pA, V_{bias} = 1 V. (Courtesy of Dr. I. Tuzov, NCSU)

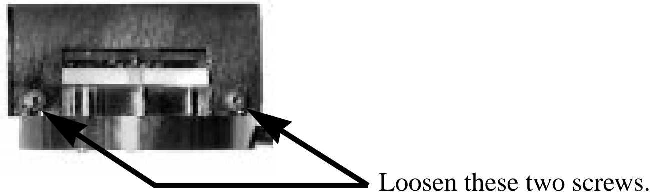
9.12. Servicing the Converter

The Low-current STM Converter features sensitive components which may be damaged if exposed to sudden voltage spikes. Spikes may be due to electrostatic discharge (ESD), or from external voltage sources. Operators should always wear an anti-static, grounding wristband (included in Low Current STM converter kit) while handling the head to protect against ESD.

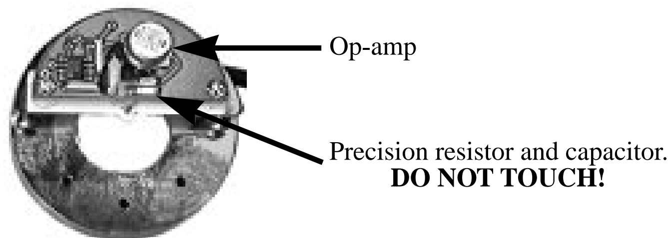
The op-amp is the most vulnerable component; several spares are shipped with the unit. If it is damaged, it may be replaced by doing the following:

1. Turn off the microscope. Disconnect the Low-current STM Converter head by unplugging its cable from the support ring. Set the head on a workbench area grounded against ESD. Ensure that an ESD wristband is being worn.

2. Use the 0.050” allen wrench, included in the converter kit, to loosen the two retaining screws which secure the head’s cover. It is not necessary to remove the screws.



3. Pull the cover straight up and off to expose the PC board and electronics. It will appear as shown below.

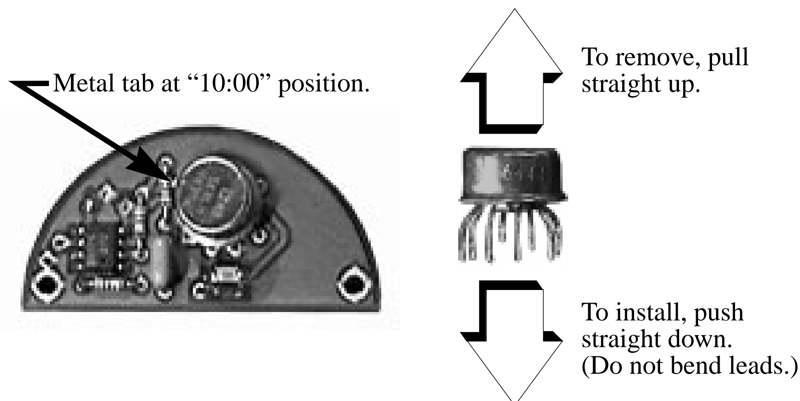


NOTE: Throughout steps #4-6 below, avoid touching the precision resistor and capacitor. Any contaminant from the hands, including body oils, may cause excessive current flow, disabling the head. If these components are touched, they will have to be recleaned immediately using highly purified degreasing compounds.

4. Remove the op-amp by grasping it and pulling straight up. It should slide out of its socket.

5. Remove the new op-amp from its protective package. The op-amp is stored in conductive foam and its leads have been preformed to fit the socket. Be careful not

to bend any of the leads. Orient the op-amp so that the metal tab on the edge of the can is in the “10:00” position as shown below.



6. Verify that each of the op-amp’s wire leads is properly aligned with the appropriate hole, then press the op-amp gently into its socket. Do not bend wire leads.

7. Replace the metal cover on the head. Retighten retaining screws to secure.

9.13. Etching Tungsten Tips

You can purchase tungsten tips from Digital Instruments or make them yourself. This section describes the process of etching tungsten tips.

Materials and Equipment Required:

- Variac auto transformer
- Optical microscope (20-100X)
- Sodium nitrite ($NaNO_2$)
- Distilled water
- Ethyl alcohol
- WD-40 (anti-oxidant)
- Two 50-ml beakers
- Tipholder (see below)
- Platinum wire
- Tungsten Wire, 0.010" Diameter
- Miscellaneous Wire/Clips

9.13.1. Procedure

1. Mix a 5 percent (by weight) solution of Sodium Nitrite in water.

2. Pour ≈ 40 ml of the Sodium Nitrite solution into a beaker.

3. Pour ≈ 40 ml 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 ≈ 1.25 cm 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 ≈2 mm of the tips' surface submerged. More than 2 mm will cause excessive foaming of the solution during etching, and less than 2 mm 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.

Notes:

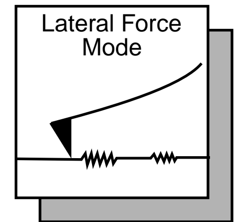
- It possible to vary the tip shape at this point by lowering the voltage on the variac and increasing or decreasing the amount of time the tips are submerged. Longer time gives blunter tips.

12. Dip the tips into ethyl alcohol to clean them. If you plan to keep the tips around for more than a day, dip them into the WD 40 after cleaning.

13. Examine the tips under the optical microscope. Ones 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 the bad ones.

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 40 ml solution.

Chapter 10 Lateral Force Mode



10.1. Introduction

The MultiMode SPM is capable of measuring frictional forces on the surfaces of samples using a special feature 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 AFM mode, it is relatively easy to switch to LFM mode to view and acquire lateral force data. It is important to obtain a good image in AFM mode before switching to LFM mode. The NanoScope system will continue to run the feedback based on the AFM signal and feedback gains in the control panel while LFM data is acquired and displayed. Complete the following steps to switch the system to LFM mode:

1. Set up and run the system in contact AFM mode as described in Chapter 6, assigning the **Channel 1** image to **Data Type: Height** and the **Channel 2** image to **Data Type: Friction**. Set the **Scan angle** to **90.00**

2. Optimize the scan parameters in AFM mode for **Channel 1**.

3. For **Channel 2**, set **Line direction** to **Trace**. This will place high lateral forces on the top of the color bar and low lateral forces on the bottom of the color bar. 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 color bar. LFM data can now be collected and stored.

10.2. LFM Mode in More Detail

The following paragraphs provide more complete information concerning the operation of LFM in friction mode.

10.2.1. Optimal Setup for Frictional Measurements

Scan direction— The cantilever is most susceptible to frictional effects when the scan direction runs perpendicular to the major axis of the cantilever as shown in figure 10.1. The **Scan angle** parameter in the **Scan Controls** panel must be set to **90**° or **270**° to produce this scan direction.

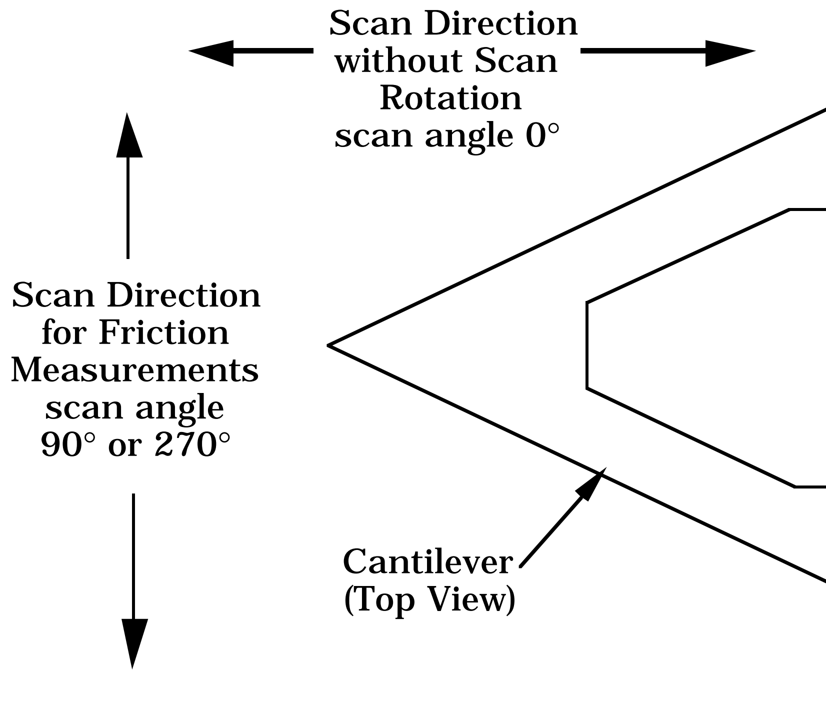


Figure 10.1. Scan angle selection.

The contact mode setpoint voltage will slightly adjust the "gain" of the lateral force signal. By increasing the setpoint, the contact force applied will increase, and so will the frictional or torsional forces in an approximately linear fashion. If the frictional effects are far too large or too small, it will be necessary to resort to changing the cantilever probe used, but if the value is near the dynamic range desired, adjustment of the contact force will produce modest changes in the lateral force or frictional signal.

Tip selection— The analog-to-digital converter on the auxiliary input channel which is used for LFM data has a maximum input range of ±10 volts. This, and the anticipated interaction between tip and sample define the selection of the cantilever to be used for the measurement.

The 200-micron cantilever with wide legs provides a good starting point for frictional measurements. It is flexible enough to provide reasonable signal levels on samples with moderate friction. If the signal exceeds ± 10.00 volts with the 200-µm wide-legged cantilever, one of the stiffer 100-µm cantilevers should be used. If the signal level is too small, the narrow-legged 200-µm cantilever will provide a larger signal.

10.2.2. Determination of friction

The lateral force on the tip is caused by both friction and the tip "running into" or "tripping on" the edges of features. There are a few things the user can do to verify that data obtained in **Data Type: Friction** is a result of friction between the tip and the sample. The topographical information should be compared to the Friction data in both scan directions. The **Scope Mode** can also be very useful when analyzing frictional data.

***Scope Mod*e**— The LFM user should be very familiar with the **Scope Mode** of viewing the data. When using **Scope Mode**, verify that the **Rounding** parameter in the **Scanner Parameters** window (selected with the **Microscope / Calibrate / Scanner** command) is set to zero. In the dual-trace mode, the data from the trace and the retrace of the sample under the tip is shown in an oscilloscope format. The vertical axis represents the differential signal from the left and right photodiodes, while the horizontal axis represents the tip position along the fast scan direction. There are several general features to observe in the scope signal (see figure 10.2 below).

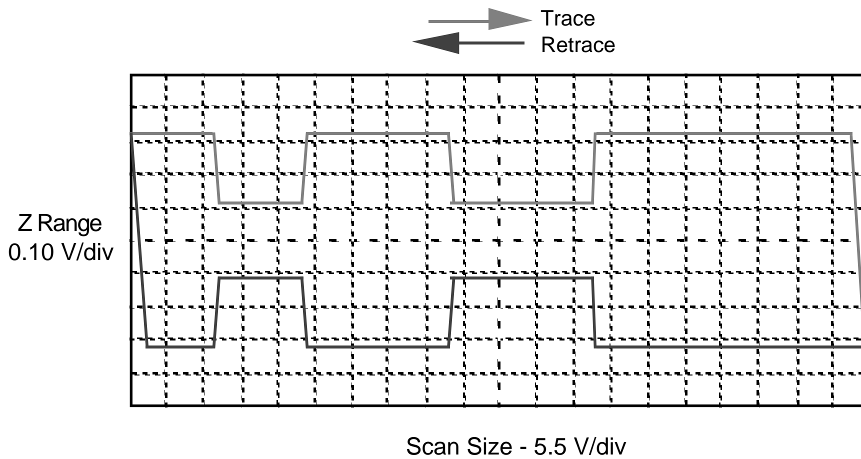


Figure 10.2. Example of a scope trace with low-friction areas.

With the scan direction rotated, the separation between the trace and retrace lines in **Scope Mode** provides a general indication of the amount of friction between the sample and the tip. For example, if there is no friction between the sample and the tip, the trace and retrace will coincide vertically. As the frictional forces between the tip and the sample increase, the cantilever twists to a larger degree and the vertical separation between the trace and the retrace increases. However, there is almost always some friction between the tip and the sample.

The trace and retrace signals will move closer together when regions of low friction are encountered. Figure 10.2 shows a theorized example of the scope trace for a sample with two areas of relatively low friction. Note how the separation between the traces decreases over the low-friction areas as the torsional deflection of the cantilever decreases for both trace directions. When trace and retrace both deflect in the same direction, this is often caused by non-frictional effects; for example, optical interference. In general, samples with areas of varying friction will include step-like features in the **Scope Mode** view.

Compare to topography— Comparing frictional data to topographical data for the same scan area provides another means of deducing the origin of the frictional features. Often, transient frictional features occur at the edges of topographical features. The tip “trips” over the feature, producing the transient frictional feature.

Reverse scan direction— Reversing the scan direction in the **Image Mode** while viewing the friction channel, is also useful in verifying the origin of the data. Remember that changing the **Line direction** parameter in the Real-time control panel changes the direction of the scan during which data is collected. If the data results from friction between tip and sample, the relative signal strengths will invert as the scan direction is reversed. For most color tables, the image produced from the trace in figure 10.2 (i.e., with the **Line direction** parameter set to **Trace**) would be darker in the low-friction areas than it is in the high-friction areas. Conversely, for the same color table, the image produced from the Retrace (i.e., with the **Line** direction parameter set to **Retrace**) would be lighter in the low-friction areas.

10.2.3. Identification of Forces other than Friction

There are a few phenomena that will produce false features in friction data on the auxiliary data channel:

Tripping— "Tripping" occurs when the probe encounters a step on the sample surface. The side of the probe strikes the feature, causing the cantilever to twist. In the **Image Mode** on the auxiliary channel, tripping appears as highlights which coincide with topographical features. In the **Scope Mode** on the auxiliary channel, tripping produces transients similar to those shown in figure 10.3. Regions of high-friction can also produce transients similar to those shown in figure 10.3, so the user must be careful to analyze the data carefully.

Optical interference— Optical interference between the laser light reflected off of the cantilever and secondary sources (e.g., the laser light which spills over the sides of the cantilever and the light transmitted through the cantilever, reflected off the sample and back through the cantilever) produces evenly spaced lines in the image. Spacing of the lines ranges between 1.5 and 2.0 microns and runs perpendicular to the tip.

Rounding— Use the **Calibrate** command in the **Real-time / Microscope** menu to set the **Rounding** parameter to zero before looking at frictional data. In the **Scope Mode,** the Rounding function shifts the trace and retrace lines horizontally and makes it difficult to directly compare the two lines.

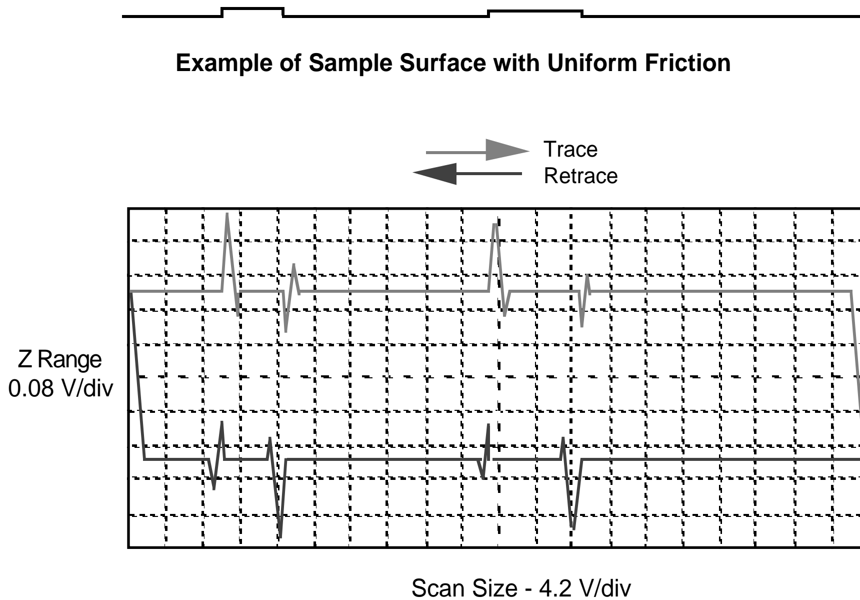


Figure 10.3. Tripping transients in *Scope Mode.*

10.2.4. Example of frictional data

Rarely will samples produce traces like the ones shown in figures 10.2 and 10.3. Typically, combinations of the features discussed above will be present in each trace and retrace. For example, figure 10.4 depicts what the scope mode might show for a sample with two low-friction areas where one is a flat bump; i.e., low-friction foreign material sticking up on a high-friction surface, and where the other low-friction area is even with the sample surface. Notice on the trace that as the tip strikes the edge of the bump (point 1 on the graph), the lateral force increases momentarily and then decreases as the tip begins scanning across the low-friction bump (region 2 on the graph). At the other edge of the bump, the lateral force may decrease somewhat as the tip comes off the bump (point 3 on the graph), and then will increase as the tip scans across the high-friction surface (region 4 on the graph). At the second low-friction area, the spikes due to topography are not present. On the retrace, notice that the sign of the frictional forces will change, but the shape will remain the same. The lateral forces due to topography shift to the other side of the bump and also change sign.

Low Friction Bump
Low Friction Area

Example of Sample Surface with Two Low Friction Areas

Trace
Retrace

Z Range
0.10 V/div

1
2
3
4

Scan Size - 5.5 V/div

Figure 10.4. ***Scope Mode*** **view of sample surface.**

Chapter 11 Force Imaging

11.1. Introduction

Force plots are used to measure tip-sample interactions and determine optimal set-points. More recently, microscopists have begun to plot force measurements across entire surfaces to reveal new information about the sample. This area of SPM promises to open new chapters in materials science, biology and other investigative areas.

11.1.1. Plotting Force in Contact AFM

The **Force Plot** command in the **View / Force Mode / Plot** menu allows the microscopist to quickly check the interaction between cantilever and sample. In **Force Plot** mode, the X and Y voltages applied to the piezo tube are held at zero while a triangular waveform (similar to the one depicted below) is applied to the Z piezo tube.

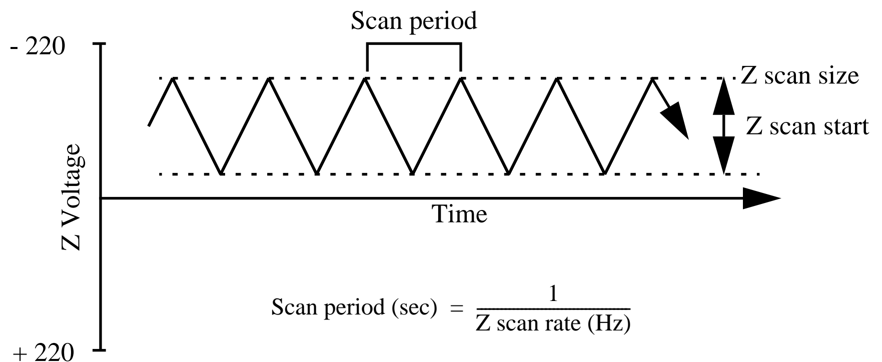


Figure 11.1. Z-axis voltage during force plot scanning.

As a result of the applied voltage, the cantilever tip moves up and down relative to the sample as shown in Figure 11.4. The **Z scan start** parameter sets the offset of the piezo travel (i.e., its starting point), while the **Z scan size** parameter defines the total travel distance of the piezo. Therefore, the maximum travel distance is obtained by setting the **Z scan start** to +**220** volts, with the **Z scan size** set to **440** volts.

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 voltage applied to the piezo tube, shows on the display monitor. The control monitor displays various control panels used to control the microscope in **Force Plot**.

In addition to dedicated force measurement experiments, **Force Plot** may be used to enhance routine topographic imaging. **Force Plot** is frequently used to adjust, calculate, and minimize contact forces between the cantilever and sample.

Force Plot may also be used to diagnose SPM performance and determine sensitivity of the cantilever deflection voltage versus voltage applied to the piezo.

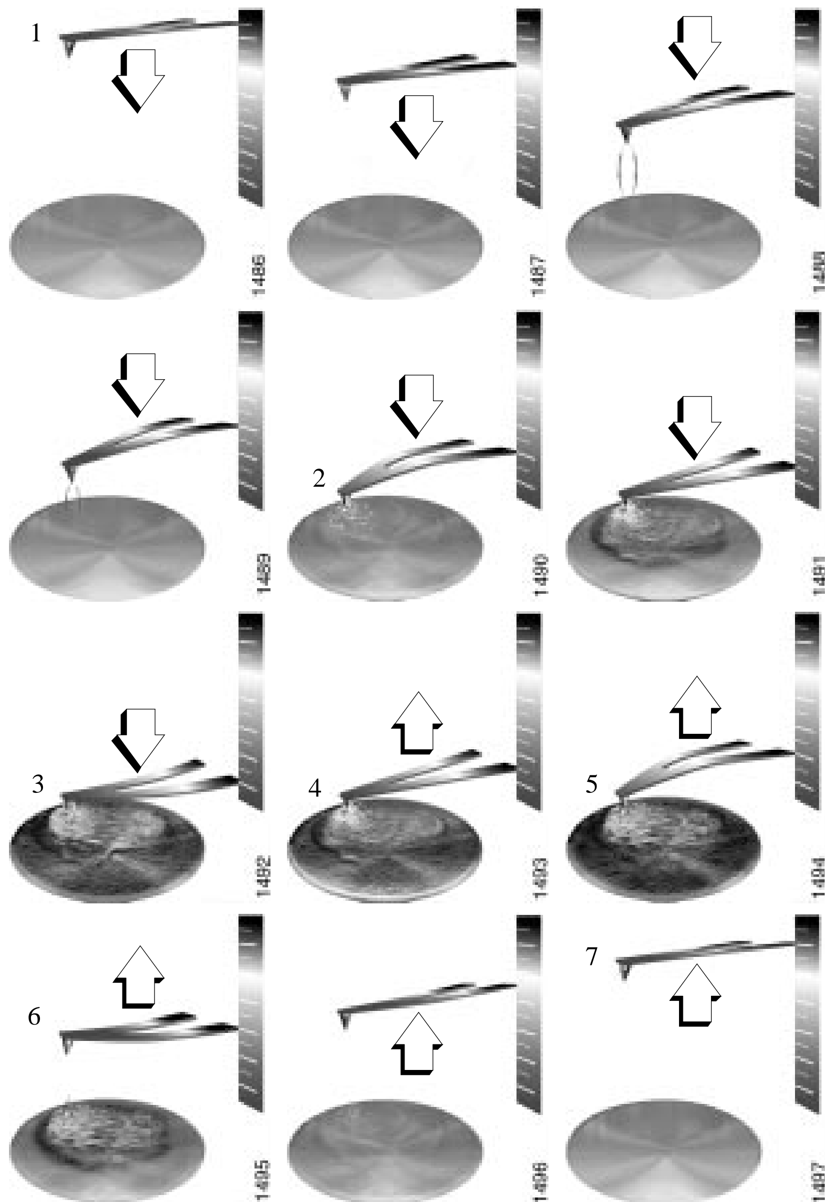


Figure 11.2. Tip-sample interaction during a force plot.

11.1.2. The Force Curve and Piezo Extension-Retraction Cycle

Figure 11.2 above compares portions of the force curve shown in Figure 11.5 to relative positions of the tip and sample at six points. The force curve represents the deflection signal for each complete extension-retraction cycle of the piezo (Figure 11.1). The **Z scan rate** parameter in the **Force Plot** control panel defines the rate at which the piezo completes an extension-retraction cycle (and therefore the rate at which new curves are displayed).

The deflection signal decreases as the piezo turns around and begins to pull the tip away from the sample surface. Typically, the signal continues to drop after the flat, zero deflection point of the force curve. At point 5, the cantilever is not deflected, but due to attractive forces between the tip and the sample, the tip sticks to the sample, and the cantilever is pulled down as the piezo continues to retract. Eventually, the spring force of the bent cantilever overcomes the attractive forces, and the cantilever quickly returns to its non-deflected, non-contact position. This is represented by point 6 on Figures 11.2 and 11.4. Figure 11.3 below represents the effects of tip pulldown due to attractive forces as the tip nears the surface (left), and the sudden, sharp rebound that results as the tip is pulled free (right).

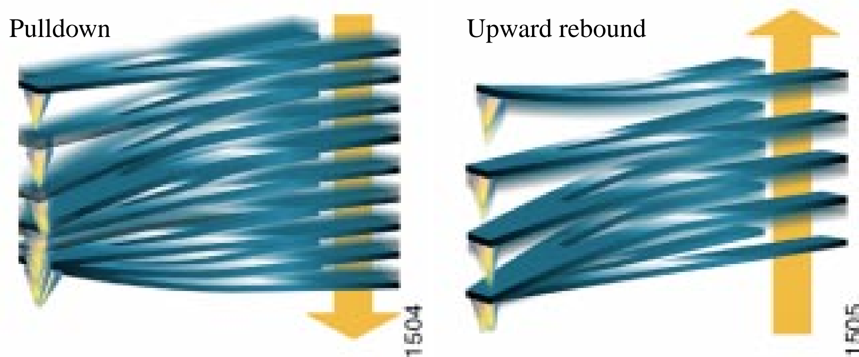


Figure 11.3. As the tip approaches the surface, it is frequently pulled down by attractive forces (left). As the tip lifts off, it sticks to the sample until pulled away, resulting in a sharp rebound (right).

The display monitor presents a linear scale of the Z piezo voltage. The scale ranges from +220 volts at the extended end to -220 volts at the retracted end. The range of piezo travel, as defined by the **Z scan start** and **Z scan size** parameters in the **Force Plot** control panel, is represented by two white lines on the scale. The lower white line corresponds to the **Z scan start** parameter in the control panel, while the spacing between the lines corresponds to the **Z scan size**. The average voltage applied to the Z electrode on the piezo tube just prior to entering the **Force Plot** mode is represented by the “Z center” line.

Notes

- In NanoScope software version 4.2 and later, the tip is retracted by the **Scan size** above the surface when first entering any of the force modes (Calibrate, Step, Indent Volume). Ramping of the Z-axis piezo begins only when the user initiates operation using one of the **Probe** commands (**Run Continuous**, **Run Single**, **Approach Continuous** and **Approach Single**). The mode is indicated simultaneously on the gray status bar at the bottom of the control monitor.

11.2. Contact AFM Force Plots

To minimize or calculate the contact force between the tip and sample, it is important to obtain a good force curve. This procedure is described in detail in Section 6.4.1. Additional information relevant to obtaining force curves is provided in this section.

False Engagement

Figure 11.4 illustrates a force curve due to a falsely engaged tip. Light reflected off the sample is received by the photodiode, causing an increase in the deflection signal until the signal equals the setpoint and causes the system to **Engage** (even though the tip is not on the surface). Interference in the reflected light causes the hump-shaped waveform.

Although the **Tip Down** button can be used to move the tip down to the surface, there exist other ways to correct a false engagement: 1) increase the setpoint and engage again; 2) adjust the photodetector positioner to make the top/bottom differential voltage more negative, then engage the tip again. This has the effect of pushing the cantilever farther up before the setpoint is reached. Chapter 15 on troubleshooting contains tips on false engagement.

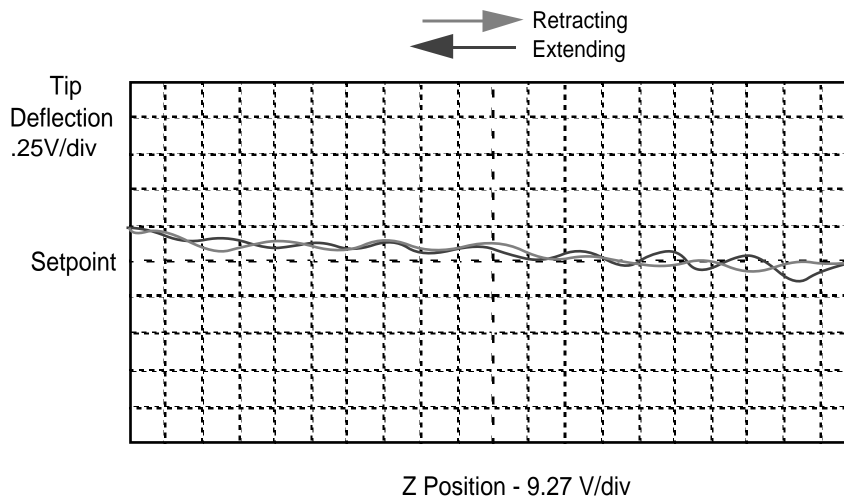


Figure 11.4. False engagement ("J" scanner).

Helpful Suggestions for Working with Force Plots

Motor Control / Tip Up and **Tip Down** buttons provide coarse adjustment of **Z scan start** voltage. With these buttons the SPM head, including tip and piezo, is moved vertically (in much the same way that piezo-travel positioning is adjusted with the **Z scan start** parameter).

Notes

- The **Step Motor** function is generally used only when the scanning range of the Z piezo is exceeded and/or when it is necessary to position a force measurement in the center of the scanner's range. Because the Z-axis leadscrew has some backlash, it may be necessary to rotate the screw several turns by clicking on the **Tip Up** or **Tip Down** buttons before movement is obtained.

In an analogous manner, the photodiode positioner can be used as a coarse adjustment for **Setpoint** voltage. Changing the beam's position on the photodiode by rotating the mirror adjustment knob shifts the force curve on the graph. Moving the photodiode down by rotating the positioner counterclockwise shifts the curve down, just as decreasing the **Setpoint** parameter shifts the curve down. Conversely, rotating the positioner clockwise moves the curve up by moving the photodiode up.

11.2.1. Contact AFM Force Plots

Let’s begin with the simplest of SPM force plots: a contact AFM force plot using a silicon nitride tip. Because of the pliant property (and lower spring constant) of silicon nitride probes, they are sensitive to attractive and repulsive forces. A force plot in contact AFM is shown below. (See also Figure 11.2.)

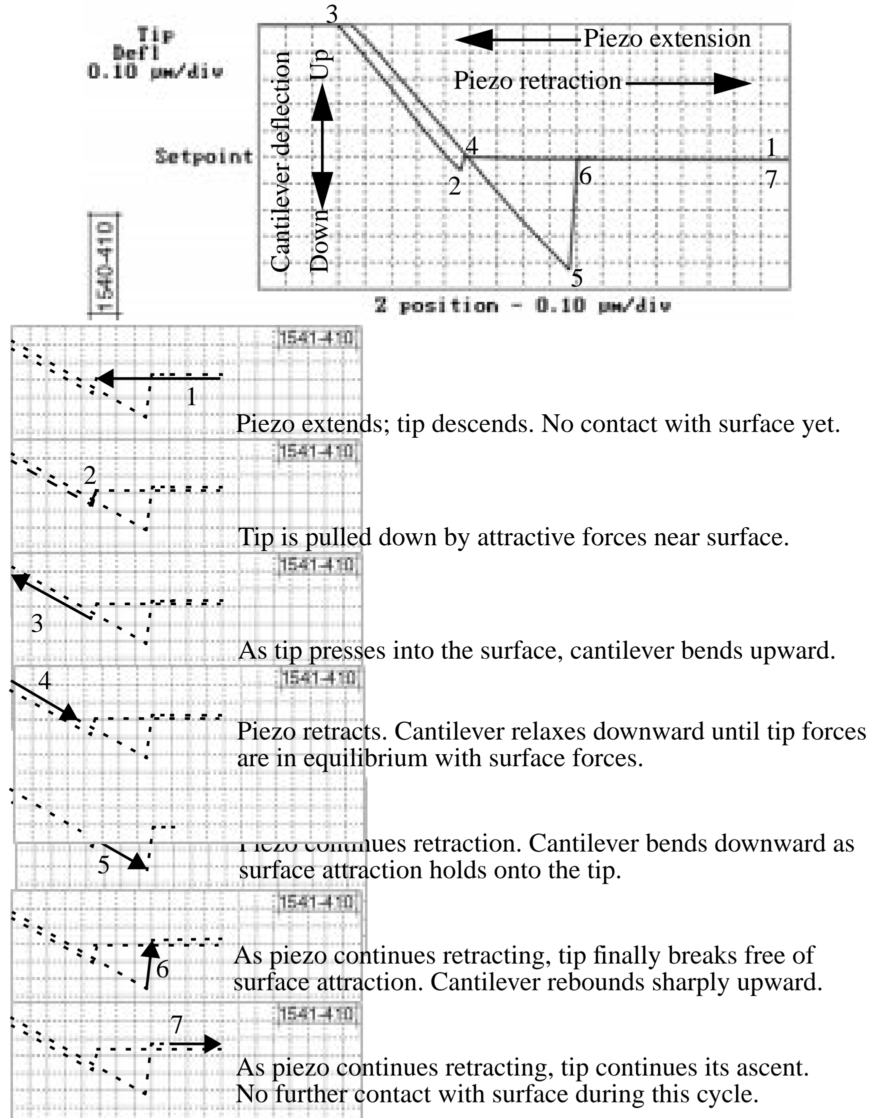


Figure 11.5. Anatomy of a force curve.

Here, the horizontal axis plots the probe's movement relative to the sample. As the probe descends toward the sample, the probe-sample distance decreases. A descent is achieved by extending the Z-axis piezo crystal, which is plotted from right-to-left in yellow on the NanoScope display monitor. As the probe ascends away from the sample, the probe-sample distance increases. An ascent is achieved by retracting the Z-axis piezo crystal, which is plotted from left-to-right in white on the NanoScope display monitor.

The cantilever's deflection is plotted on the vertical axis of the graph: when the cantilever is deflected downward, it is plotted on the graph's downward vertical; when the cantilever is deflected upward, it is plotted on the graph's upward vertical.

The graph reveals at least two very important things:

- **Sample-tip attraction**— As the tip approaches the sample, various attractive forces reach out and "grab" the tip. This is evidenced at point 2 (slight dip) in the graph above. Notice how the tip suddenly plunges toward the sample here during its descent. This is sometimes called the "jump-to-contact" point and is usually due to electrostatic attraction and/or surface tension (capillary) forces.

  Attraction is also evident between points 3 and 4 (sloped line) as the cantilever is pulled away from the sample. If attractive forces are strong enough, the tip will cling to the sample surface as it is pulled clear. Eventually, the sample "lets go" and the tip rebounds sharply upward (white line, between points 4 and 5).

  By knowing the spring constant of the cantilever, it is possible to measure the attractive forces of tip-sample interactions with good precision.

Notes

Although attractive forces appear small, remember that the tip is extremely sharp. Since only a few nanometers of the tip actually touch the sample, even minute forces are distributed over an exceedingly small area, which add up quickly. Many materials are easily dented by the tip under such conditions. Use of force plots may be used to adjust a setpoint which applies minimal force to the sample during contact AFM. (More on this topic below.)

- **Material elasticity**— It is possible to extract some 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 3. As the tip is pressed further and further into the material, the probe's cantilever flexes. The amount of cantilever flexion for a given amount of downward tip movement gives an indication of the material's elasticity.

  For example, if the material is extremely hard, pressing the probe downward will result 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 sometimes possible to obtain quantitative measurements of sample elasticity. (See, for example: Radmacher, *et al.* 1994. *Science*, Vol. 265:1577-1579.)

Two imaging techniques may be employed to measure and display elasticity at multiple points on a sample surface: force modulation (see Section 11.5 below) and force volume imaging (see Section 11.7 below).

11.2.2. Sensitivity Determination

Sensitivity represents the cantilever deflection signal versus voltage applied to the piezo and is normally set from the **Force Plot** mode. The sensitivity must be calibrated before accurate deflection data can be obtained. Sensitivity is equal to *the slope of the force curve while the cantilever is in contact with the sample surface*. Complete the following steps to calculate the sensitivity:

- Obtain a good force curve on the display monitor.
- Position the cursor on one end of the contact portion of the curve.
- Click on the left mouse button to fix the line segment.
- Drag the mouse to position the "rubber band line" parallel to the contact portion of the force curve (see Figure 11.6).

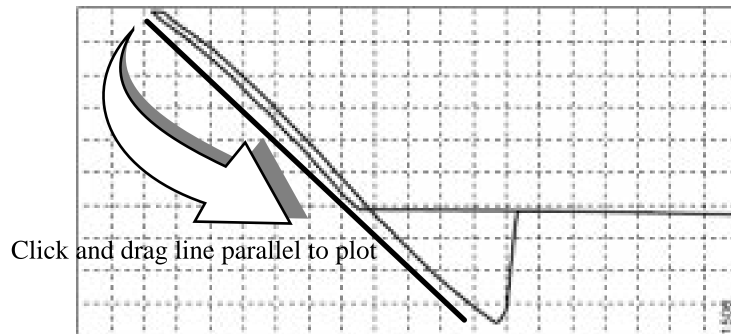


Figure 11.6. Use the mouse to set the *Sensitivity* parameter.

- The second click on the mouse causes the system to calculate the slope of the line segment and enter the calculated value as the Sensitivity in the menu.
- A click of the right mouse button will remove the line segment from the screen.

Note that **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. If **Sensitivity** is calibrated on a material much stiffer than the cantilever, it measures 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 higher 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.

Notes

- 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.

11.2.3. Force Minimization

Force Plot allows the contact force of the cantilever on the sample surface to be minimized. The force curve clearly shows the relationship between the **Setpoint** and the cantilever deflection voltage when the cantilever is off the sample surface. **Setpoint** can be adjusted to set the nominal deflection of the cantilever and, therefore, the nominal force applied by the cantilever during data collection.

The microscope can be run below the point of zero deflection of the cantilever to minimize the contact force of the cantilever on the sample. It is possible to get a negative deflection whenever the cantilever sticks to the surface. The system cannot engage to a negative deflection operating point, because the engagement process requires a setpoint which is greater than the voltage at zero deflection. However, the operating point can be changed after the cantilever has been engaged.

In **Force Plot**, the setpoint can be adjusted to the zero cantilever-deflection point and beyond, while viewing the force curve. The setpoint can be adjusted (most often made more negative) so that it lies between the flat segment of the force curve which corresponds to the zero deflection point, and the tip of the retraction scan where the cantilever pulls off the sample surface V_{CSmin}. The contact force is at its minimum when V_{CSmin} is on the centerline of the deflection-signal axis.

In practice, V_{CSmin} should be a little below the centerline since V_{CSmin} is the point where the cantilever pulls off the surface and operation at this deflection will be unstable. Finally, note that changing the **Setpoint** option in the **Feedback Controls** panel will change the **Setpoint** parameter in its Real Time counterpart when the **Force Plot** routine is exited. After exiting, if the image looks good, the force can be decreased further by lowering the **Setpoint** in small increments until the cantilever pulls off. Resetting the **Setpoint** to a value higher than the top/bottom

differential signal on the upper MultiMode meter display will recapture 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 will provide a low contact force.

If a high initial contact force will adversely affect 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 will experience the relatively high initial engagement force. Once the force is minimized, increase the **Scan 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 is translated to a rougher part of the sample.

11.2.4. 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 nominal contact force of the tip on the sample if the spring-constant, k , of the cantilever is known. The contact force is defined by the equation:

$$F = k(\Delta Z)$$

where ΔZ is the Z distance from the control point to V_{CSmin} in nanometers. An example of how to compute the contact force from the **Force Plot** graph is shown in Figure 11.9.

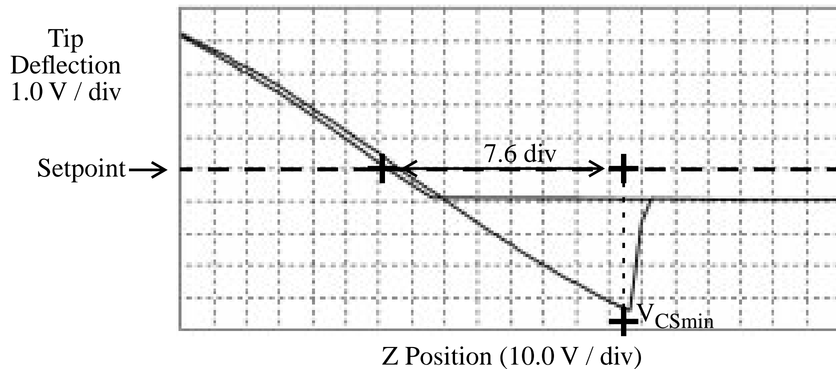


Figure 11.7. Computing contact force.

Recalling that contact force F is equal to $k(\Delta Z)$, we can calculate the contact force from the sample plot above. Let us assume, for example, that the spring con-

stant of the cantilever is $k = \frac{0.6N}{m}$ and that $\text{Z piezo sensitivity} = \frac{12nm}{V}$. The plot above may be measured at the points where the retract portion of the curve intersects the setpoint to tip pull-off (rebound). The distance is then multiplied times the Z piezo sensitivity to obtain ΔZ . In this example:

$$\Delta Z = 7.6 \text{ div} \times 10.0 \text{ V/div} \times 12 \text{ nm/v} = 152 \text{ nm}$$

Therefore, the contact force is calculated as:

$$F = 0.6\text{N}/m \times 152 \text{ nm} \\ = 91.2 \text{ nN}$$

When the **Data type** is set to **Height** with the feedback gains set high, the tip tracks the sample surface with nearly constant deflection of the cantilever. When the cantilever deflection is constant, the force is constant and the force calculation determines the 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**. When collecting deflection data, the feedback gains are ideally set low so the sample stays at a constant position relative to the cantilever holder. In this case, the cantilever deflection (and therefore the force applied to the sample) varies as features on the surface are encountered. The nominal force applied to the surface can be calculated from the force equation. The force applied at other points on the sample can be calculated relative to the nominal force by using deflection data and the spring-constant of the cantilever. Note that the **Sensitivity** parameter must be accurate—that is, previously determined and entered—before the deflection data will be accurate.

An simple alternative to calculating force is to follow these five steps:

1. Set sensitivity using the mouse.

2. Change units to metric.

3. Count vertical units from Setpoint to UCS min.

4. Multiply by Tip Deflection Scale.

5. Multiply by the spring-constant, k.

11.2.5. Interpreting Force Curves

An examination of force curves can prove useful in determining adhesion and hardness characteristics of samples. The examples in Figure 11.8 represent some of the general variations in force curves. For more information regarding force imaging, refer to Digital Instruments' application note *Probing Nano-Scale Forces with the Atomic Force Microscope*.

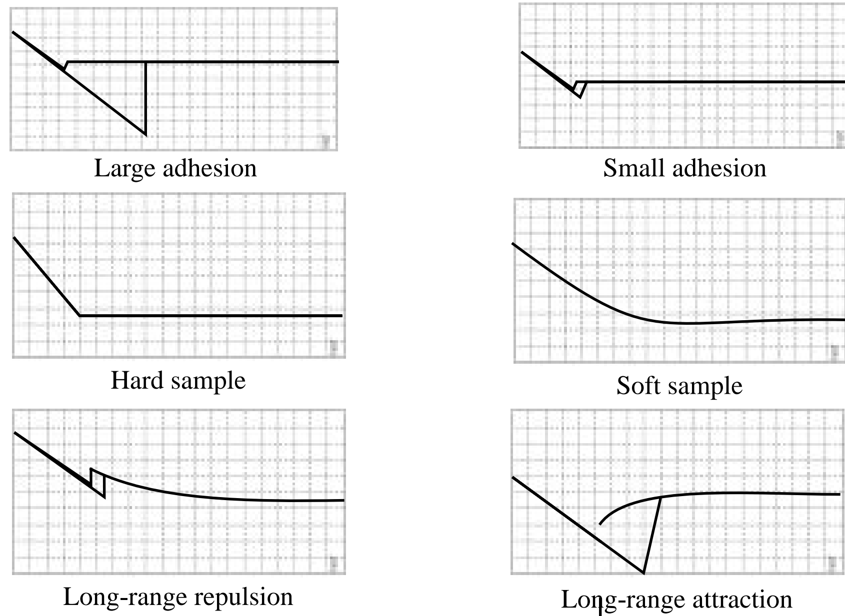


Figure 11.8. Force curve examples.

11.3. Force Imaging in TappingMode

CAUTION: Because TappingMode cantilevers are relatively stiff, **Force Mode** can potentially damage the tip and/or surface. DI recommends using Force Plot only with extreme caution.

ATTENTION: Le pointes Tapping étant relativement raides, l'utilisation du mode "Force" peut endommager la pointe ou l'échantillon. Digital Instruments recommande d'utiliser le mode "Force Plot" avec une extrême précaution.

WARNHINWEIS: Da TappingMode-Meßspitzen relativ steif sind, kann im Force Mode unter Umständen die Meßspitze oder die Oberfläche beschädigt werden. DI empfiehlt, den Force Plot nur unter größter Vorsicht anzuwenden.

Force Mode allows the imaging of forces between the tip and surface, including chemical bonds, electrostatic forces, surface tension, magnetic forces, etc. In TappingMode, forces may be observed by measuring changes in the tip's RMS amplitude, phase, or deflection. The user may represent force plots in one of two forms: **Force Plot** and **Force Volume** (see Section 11.5). Both are essentially the same, with **Force Volume** generating a "map" of many individual force plots. To produce high-quality force plots, it is necessary to very precisely control the tip's position relative to the surface.

11.3.1. Force Plots

When performing **Force Plot** in TappingMode, the piezo moves to the center of the current XY scan, then turns off the XY scan motion. Next, a triangular waveform is applied to the Z electrodes of the piezo tube. As a result, the oscillating tip is moved up and down relative to the sample. This is exactly the same Z-axis piezo motion as for contact AFM. In TappingMode, however, the force plot is a graph of the piezo's extension versus the oscillating tip's amplitude, phase, or deflection.

Figure 11.9 represents a tip-sample-piezo relationship on a MultiMode system. The piezo positions the sample just below the tip (at the **Z scan start**), then extends a known distance closer to the tip (the **Z scan size**). If the oscillating tip encounters any surface forces, it will respond (its amplitude may decrease and/or its phase may change.

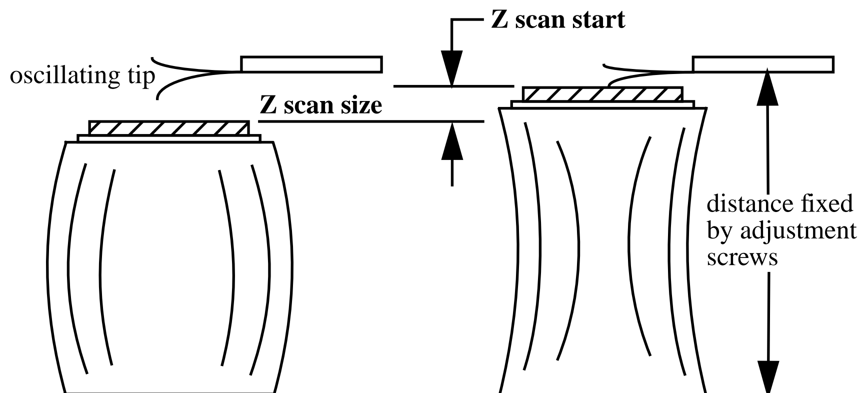


Figure 11.9. Relationship of *Z scan start* and *Z scan size*.

Uses of **Force Plot** in TappingMode include characterizing forces on the cantilever tip, diagnosing SPM performance, and calibrating the RMS amplitude, deflection or phase of the cantilever as a function of tip-sample distance. For example, as the oscillating tip is brought closer to the surface, the tip's motion is dampened, which

shows as an immediate drop in amplitude. When plotted, the graph resembles Figure 11.10.

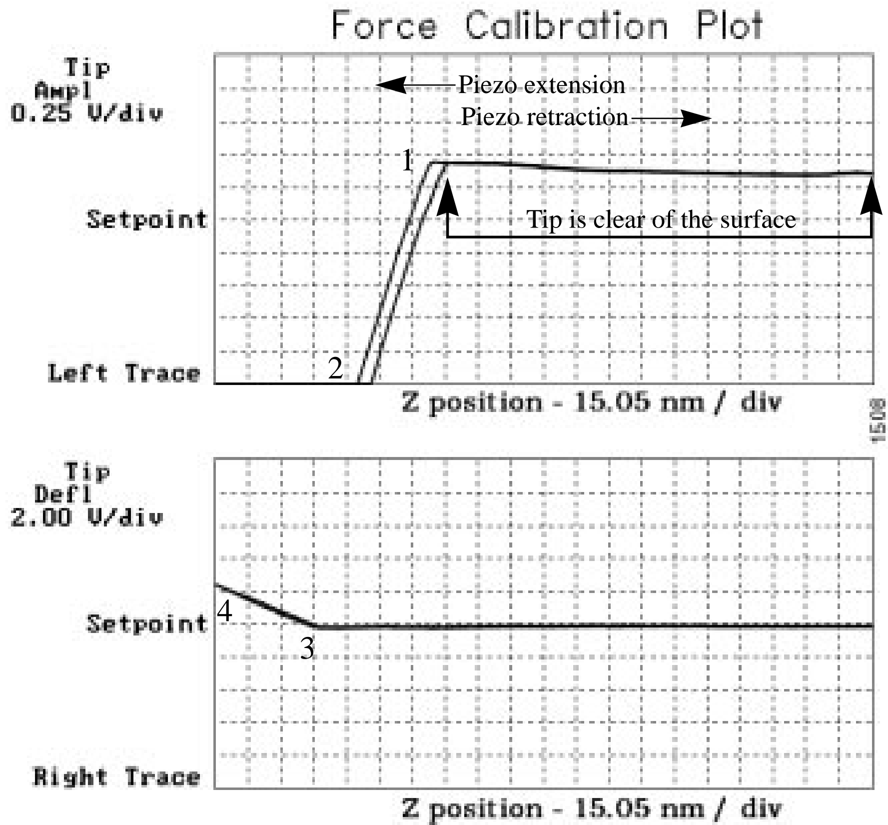


Figure 11.10. Force plot of piezo extension versus RMS amplitude (top) and deflection (bottom).

Figure 11.10 shows a two-channel TappingMode force plot. The vertical axes of the graphs represent the RMS amplitude (top) and deflection signal (bottom) of the cantilever. The position of the Z piezo is plotted along the horizontal axis. **Channel 1** (top) demonstrates how the cantilever's RMS amplitude decreases as the tip is positioned closer to the sample. The plot represents the RMS amplitude for one complete extension-retraction cycle of the piezo. The **Z scan rate** parameter in the **Z Scan Controls** panel defines the rate at which the piezo completes an extension-retraction cycle; therefore, the rate at which new curves are displayed. At point 1, the tip begins its interaction with the sample. The tip's oscillation amplitude begins to recede, dropping off as the tip moves closer to the sample surface. Between points 1 and 2, voltage to the piezo is increased, bringing the tip about 30 nm closer

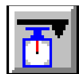
to the surface. Over the same interval, the cantilever's amplitude is diminished about 1.75 volts due to dampening effects.

Dividing the change in amplitude by the change in Z piezo position gives the responsiveness of the tip-sample interaction, displayed as a **Sensitivity** value in the **Z Scan Controls** menu. This value may be found directly by using the mouse to draw a line parallel to the plot's slope in the region between points 1 and 2 where the tip's amplitude is dampened. Tip dampening occurs as a result of mechanical-acoustic coupling between the tip and sample. As the tip descends closer and closer to the sample, oscillation eventually ceases and the amplitude drops to zero.

For TappingMode in this sloping region, each nanometer decrease in the cantilever position decreases the vibrational amplitude of the cantilever by two nanometers.

Channel 2 (bottom) in Figure 11.12 plots the average cantilever's deflection versus piezo extension. The deflection signal is low-pass filtered to eliminate the high-frequency TappingMode oscillation. Even as the tip's RMS amplitude is dampened during its encounters with the sample surface, the average deflection is unchanged. This condition changes, however, once the tip has been positioned so close to the sample that all oscillation ceases. Pressing the tip still further causes the average deflection to increase, applying a constant force to the sample.

At point 3 in Figure 11.10, the cantilever has begun to deflect. The region between points 3 and 4 may be hazardous to the tip, since the tip is pressed tightly against the sample surface. Most single crystal silicon TappingMode tips fail in this region, depending upon the hardness of the sample.



11.3.2. Obtaining a Force Plot of a Calibration Reference in TappingMode

CAUTION: Because TappingMode cantilevers are relatively stiff, Force Mode can potentially damage the tip and/or surface. DI recommends using Force Plot only with extreme caution.

ATTENTION: Le pointes Tapping étant relativement raides, l'utilisation du mode "Force" peut endommager la pointe ou l'échantillon. Digital Instruments recommande d'utiliser le mode "Force Plot" avec une extrême précaution.

WARNHINWEIS: Da TappingMode-Meßspitzen relativ steif sind, kann im Force Mode unter Umständen die Meßspitze oder die Oberfläche beschädigt werden. DI empfiehlt, den Force Plot nur unter größter Vorsicht anzuwenden.

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 ampli-

tude). If the amplitude is reduced to zero, the tip has almost certainly been blunted or broken, and/or the sample has sustained damage.

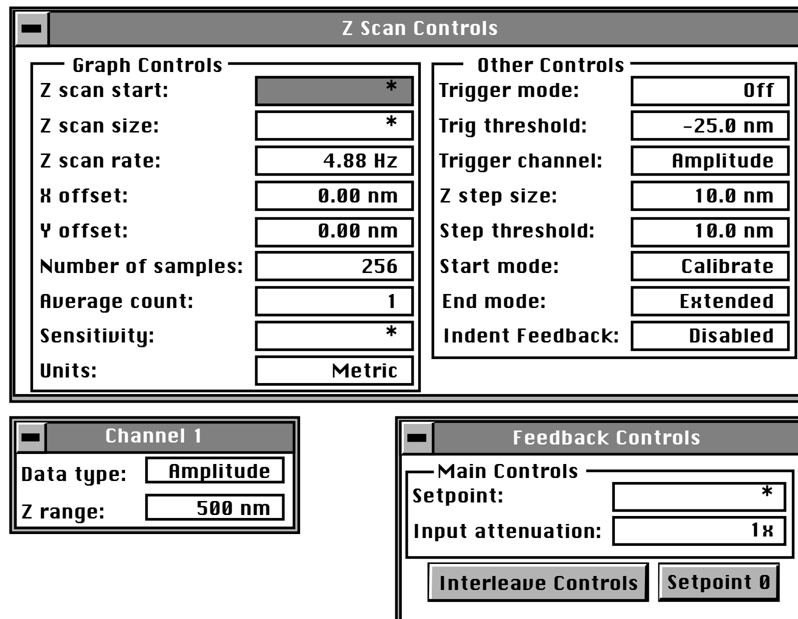
To make a TappingMode force plot of a silicon calibration reference, try the following:

1. Verify the tipholder is fitted with a TappingMode tip. Set the **AFM mode** parameter to **Tapping** and obtain a TappingMode image. You are now in **Image mode**.

2. To switch to **Force Mode**, click on the **Real Time / View / Force Mode / Plot** option. At least three panels should be visible: **Z Scan Controls**, **Feedback Controls**, plus one channel panel (e.g., **Channel 1**). Collectively, these panels allow the user complete control over tip-sample interactions. If any panels are not visible, pull down the **Panels** menu to select them.

The top menu bar offers a number of tip approach options detailed in the *Command Reference Manual*, Chapter 10. These buttons are not generally used for TappingMode and may be ignored.

3. Set the **Z Scan Controls**, **Feedback Controls** and **Channel** panel parameters to the settings shown below. (Note that the **Sensitivity** value shown here may differ from yours.)



The **Channel 1** panel should have its **Data type** parameter set to **Amplitude**.

4. To obtain a satisfactory force plot, it will be necessary to adjust the **Z scan start** parameter more positive. This may be easily done using the right arrow key.

11.3.3. Very High Contact Force

Figure 11.11 shows a curve produced when the tip is pushed too far into the sample. This is a very dangerous condition because the cantilevers used in TappingMode are stiff and brittle. If they are driven into the sample surface so that oscillation amplitude changes to zero, they will break.

The flat portion on the left side of the amplitude curve in Figure 11.11 occurs because the tip is so close to the surface that it no longer vibrates. As the piezo extends the tip further and 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 cantilever may become cracked or the tip shattered if deflection continues.

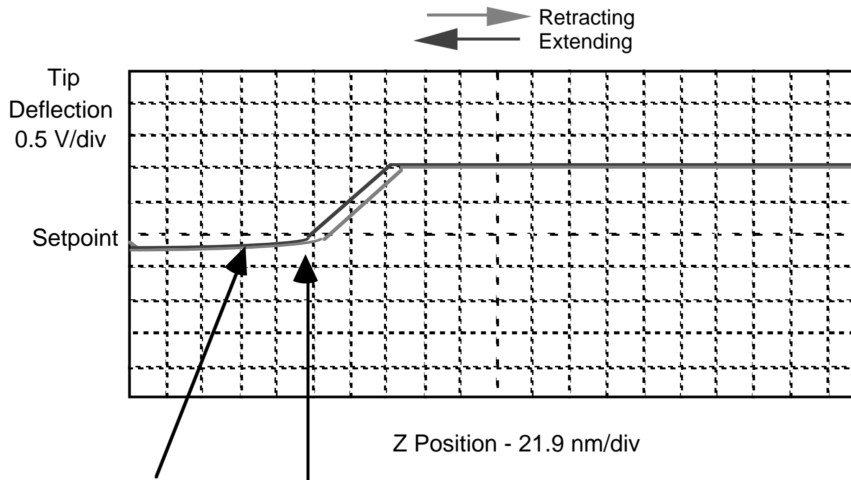


Figure 11.11. Amplitude force plot with high contact force.

This situation can be avoided by using triggers (see next section) and/or by reducing the value of **Z scan start** so there is no flat portion on the left side of the curve. Rapidly increasing the value of **Z scan start** is very dangerous since the total vibra-

tional amplitude of the cantilever is small relative to the total Z travel of the long-range scanner.

Plotting phase-versus-frequency in TappingMode force plots

Interest has increased in plotting the tip's phase-versus-frequency while encountering sample surfaces in TappingMode. This is available only for SPMs having an Extender™ Electronics Module. Just as imaging magnetic-electric forces in phase gives better resolution than amplitude, plotting force in terms of phase-versus-frequency allows improved observation of long-range attractive and repulsive forces between the tip and sample. Precise interpretation of force plots using phase-versus-frequency remains under investigation. Contact Digital Instruments for more information.

Regarding the selection of tips...

Almost any TappingMode tip may be used to obtain TappingMode force plots; however, the ultimate choice should depend upon the delicacy of the sample and/or the magnitude of the forces to be gauged. Recall that longer tips have lower spring constants (i.e., they are more pliant) and therefore offer greater sensitivity for most samples. Shorter tips, on the other hand, may afford better control when gauging strong attractive forces and are less prone to entrapment by surface tension forces. The user must ultimately experiment to determine the best tip.

11.3.4. Triggering

The **Other Controls** panel within the **Z Scan Controls** allows the use of various triggers when obtaining **Force Plot** and **Force Volume** plots. The idea of a trigger is simple: it limits the total amount of force exerted by the tip upon the sample. Depending upon which trigger is used and how it is set, it is possible to operate the trigger independent of drift (**Relative**) or at some arbitrarily fixed point (**Absolute**).

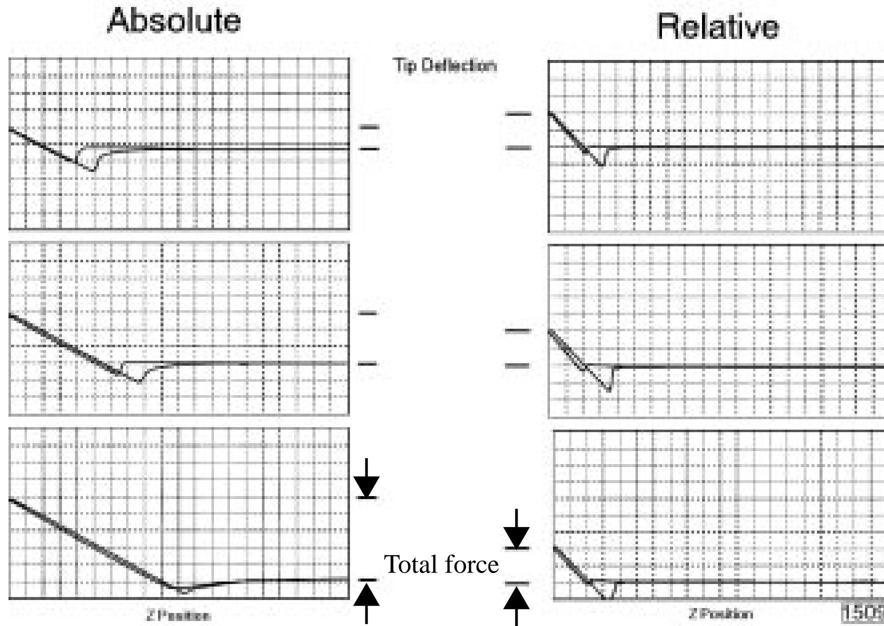


Figure 11.12. Reaction of absolute and relative triggers to drift.

The plots in Figure 11.12 above show the effect of drift on each of the two trigger types. The plot series shown on the right side of Figure 11.12 utilizes a **Relative** trigger and maintains force at a constant level defined by the **Trig threshold** parameter. As shown, the total force is maintained, even as the system drifts.

The plot series on the left side of Figure 11.12 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.

11.4. Force Plot Control Panels

The control panel window (Figure 11.13) is used to manipulate the microscope in **Force Plot** mode and is displayed on the control monitor. Among other things, the parameters control the start position and amplitude of the triangle wave applied to the Z piezo. The setpoint value of the cantilever deflection voltage used in the feedback loop can also be adjusted. With the **Motor / Tip Up** and **Tip Down** buttons (Figure 11.14), the stepper-motor can be used to adjust the position of the cantilever tip relative to the sample. The **Capture** button stores the force plot for Off-line viewing. It should be noted that some of the parameters influence the Real Time operation of the microscope, while most of the menu parameters affect only the Force Plot mode.

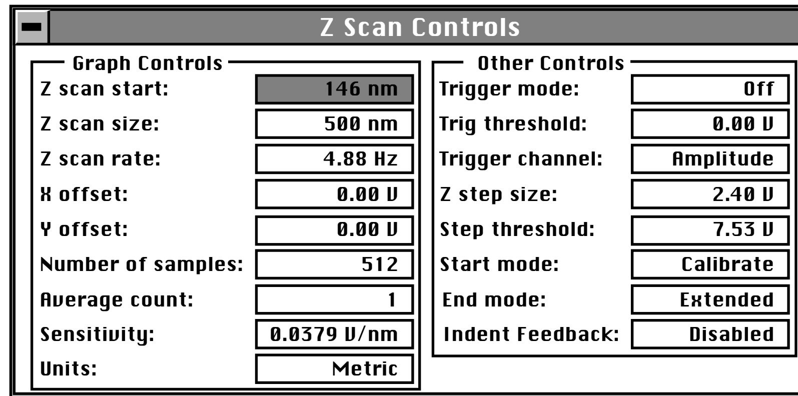


Figure 11.13. ***Z Scan Controls*** **panel.**

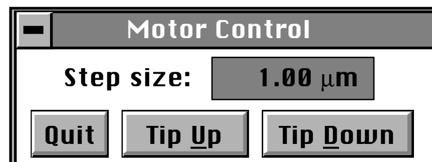


Figure 11.14. ***Motor Control*** **panel.**

The input sensitivity of the SPM head must be defined properly to keep the values on the vertical axis of the force plot accurate. The parameter **In sensitivity** should be set to **0.125** by using the **Real Time / Microscope / Calibrate / Detector** command for each scanner used with the MultiMode microscope.

11.4.1. Force Plot Control Panel Parameters

The various **Force Plot** control panels allow the operator to precisely control the probe tip's interaction with the sample surface. This is especially useful during contact AFM procedures, as it directly affects image quality and the degree to which the sample is influenced by forces from the tip. Items in the various **Force Plot** control panels are discussed below.

<u>**Z Scan Controls panel**</u>

Z scan start— Sets the maximum voltage applied to the Z electrodes of the piezo during the force plot operation. The triangular waveform is offset up and down in relation to the value of this parameter. Increasing the value of the **Z scan start** parameter moves the sample closer to the tip 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 **Force Plot** mode. *Decreasing* the value of this parameter shifts the force curve on the display to the left, while *increasing* the parameter shifts the curve to the right.

Z scan size— Fixes 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 has the effect of compressing the curve on the display.

Number of samples— Defines the number of data points captured during each extension-retraction cycle of the Z piezo. This parameter does not affect the number of samples used in **Image Mode**.

Average count— Sets the number of scans taken to average the display of the force plot. May be set between **1** and **1024**. Usually it is set to **1** unless the user needs to reduce noise.

Sensitivity (as applied to <u>contact AFM</u>)— This parameter relates the cantilever deflection signal to the Z travel of the piezo. It equals the slope of the deflection versus Z voltage line when the tip is in contact with the sample (e.g., between 3 and 4 in Figure 11.5). The NanoScope system automatically calculates and enters the value from the graph after the operator uses the mouse to fit a line to the graph. This item must be properly calculated and entered before reliable deflection data in nanometers can be displayed in Real Time mode. For a proper force curve, the line has a negative slope with typical values of **10-50 mV/nm**; however, by convention, values are shown as positive in the menu.

Sensitivity (as applied to <u>TappingMode</u>)**— Sensitivity** relates the vibrational amplitude of the cantilever to the Z travel of the piezo. It is calculated by measuring

the slope of the RMS amplitude versus the Z voltage when the tip interacts with the sample as shown in Figure 11.10. The NanoScope system automatically calculates and enters the value from the graph after the operator uses the mouse to fit a line to the graph. This parameter must be accurately calibrated before amplitude data obtained in LiftMode will be accurate.

Units— This item selects the units, either **Metric** lengths or **Volts**, used to define the parameters and the horizontal axis of the graph. Changing this item in **Force Plot** also changes it in the Real Time imaging mode. Metric units are obtained from the **Z sensitivity** for the particular scanner. under **microscope/calibrate/scanner**.

Feedback Controls panel

Setpoint (as applied to contact AFM)— The cantilever deflection voltage maintained by the feedback loop in Real Time mode can be adjusted by changing this parameter. In Force Plot, this parameter defines the centerline of the vertical, “Cantilever Deflection Voltage” axis of the force plot curve shown on the display monitor. Changing the **Setpoint** shifts the force plot curve on the graph, because the centerline value is always equal to the setpoint. For example, if the **Setpoint** is set to -3.0 volts, the cantilever deflection axis of the graph will be centered around -3.0 volts. Raising the **Setpoint** to -2.0 volts will shift the force plot curve down by one volt so the graph will be centered at -2.0 volts. Changing the value of this parameter in the Force Plot mode also changes the Real Time setpoint in the Real Time control panel.

Setpoint (as applied to TappingMode)— The RMS value of the cantilever deflection voltage maintained by the feedback loop in the Real Time mode can be adjusted by changing **Setpoint**. In the TappingMode force plot mode, **Setpoint** defines the centerline of the vertical, “Tip Amplitude” axis of the amplitude calibration plot shown on the display monitor. Changing the **Setpoint** shifts the amplitude curve on the graph, because the centerline value is always equal to the **Setpoint**. For example, if the **Setpoint** is at 3.0 volts, the RMS amplitude axis of the graph will be centered around 3.0 volts; raising the setpoint to 4.0 volts will shift the amplitude curve down by one volt, so the graph will be centered at 4.0 volts. Changing the value of this parameter in the **Amplitude Calibration** mode also changes the **Setpoint** parameter in the Real Time control panel.

Input attenuation—Changes the gain applied to the input signal. The input signal can be reduced by a factor of eight to see more of the Force Plot plot's signal amplitude. This parameter is also changed in the Real Time menu and affects the resolution of the system; therefore, it should be changed to **1X** before returning to imaging.

Drive frequency (<u>TappingMode</u> only)— This parameter defines the frequency at which the cantilever is oscillated. This frequency should be very close to the resonant frequency of the cantilever. Changing the value of this parameter in the **Force Plot** menu also changes the **Drive frequency** parameter in the Real Time control panel.

Drive amplitude (<u>TappingMode</u> only)— This parameter defines the amplitude of the voltage applied to the piezo system which drives the cantilever vibration. Changing the value of this parameter in **Force Plot** mode also changes the **Drive amplitude** parameter in the Real Time control panel. It is possible to fracture the cantilever by using too high of a drive amplitude; therefore, it is safer to start with a small value and increase incrementally. If the amplitude calibration plot consists of a flat line all the way across, changing the value of this parameter should shift the level of the curve. If it does not, the tip is probably fully extended into the surface.

<u>Channel 1, 2, 3 panels</u>

Data type— Amplitude (<u>TappingMode</u> only), **Deflection**, **Friction**, **Aux B—D**.

Z range— Vertically scales the cantilever deflection signal versus Z voltage graph shown 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, this parameter should be set to its maximum value, then gradually reduced.

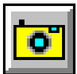
<u>Motor Control panel</u>

A separate **Motor Control** panel allows operators to move the probe at will. This control is used during the **Force Plot** procedure to adjust tip height above the sample surface. Movement is limited to a maximum of **1.0 µm** per step.

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.

<u>Menu Bar Commands</u>



Capture— The **Capture** button stores the force plot for Off-line viewing.

Probe menu commands— These commands allow for highly controlled probe movements to prevent damage to the tip. Each of these commands has an icon which can be accessed directly from the menu bar. **Probe** menu commands are especially helpful when performing force plots in TappingMode and are described in the *Command Reference Manual*.

11.5. Imaging Local Elasticity with Force Modulation

11.5.1. Introduction

This section describes the operation of force modulation (**Forcemod**) mode, which can be used to image local sample stiffness or elasticity. This method may be especially useful for imaging composite materials or soft samples on hard substrates where it is possible to obtain contrast between regions of different elasticity.

This section assumes knowledge of operation of contact mode AFM in air, as described in Chapter 7. It is useful, but not essential also to have experience operating with TappingMode.

Force modulation measures local sample elasticity by oscillating a probe such that its tip indents slightly into a sample. The tip will be able to indent a soft material more easily than a harder material. The amount of cantilever deflection is inversely related to the amount of indentation. For example, an extremely soft sample will allow the tip to indent deeply into the surface, resulting in a very small deflection of the cantilever. A very hard sample allows less indentation, with the cantilever deflected by a larger amount. The relative elasticity of the sample is measured by recording the amplitude of the tip deflection versus position over the sample as depicted in Figure 17. Contrast is generated as the cantilever scans the surface from left-to-right. When it encounters a hard site (dark area) embedded in the softer medium, the harder material absorbs less of the cantilever's energy, causing an increase in the cantilever's response and signal amplitude.

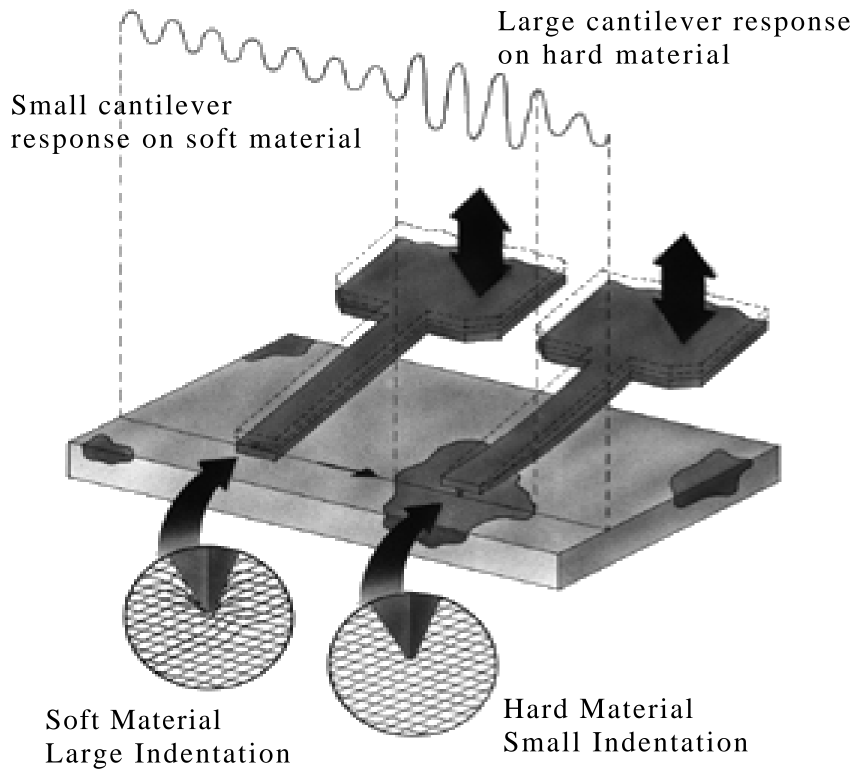


Figure 11.15. How contrast is generated in force modulation mode.

Force modulation requires the use of a special optional tipholder, shown in Figure 11.18. This tipholder uses a piezoelectric bimorph to oscillate the cantilever against a sample surface. The force modulation tipholder is similar to the standard tipholder; however, its piezo stack is much larger and lower frequency than the standard tipholder used for TappingMode operation.

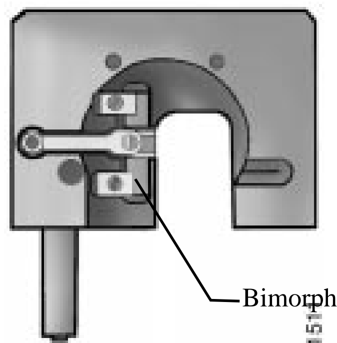


Figure 11.16. MultiMode force modulation tipholder.

To obtain good contrast while using force modulation, choose a cantilever that has a similar spring constant to the sample material. If the cantilever is much softer than the sample material, the cantilever will not indent sufficiently into the surface to generate an image. It may take some experimentation to find a cantilever that matches the sample's requirements. For rubber and plastic samples DI recommends trying, for example, 225 µm long force modulation (Model # FESP) silicon cantilevers. For more delicate, biological samples, try 450 µm long silicon cantilevers or silicon nitride cantilevers.

11.5.2. Selecting a Force Modulation Tip

The key consideration when selecting a force modulation cantilever is its spring constant. Ideally, the cantilever must have a spring constant which compliments the pliancy of the two materials being contrasted. (Or close to the pliancy of one, but not the other.) This way, the tip will indent 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 no contrast (elasticity) will be seen in the force modulation image. Instead, the image will consist primarily of edge and frictional artifacts. If a force modulation tip proves inadequate, the user may switch to a probe having a lower ("softer") spring constant (e.g., silicon nitride or a 450 µm, contact AFM tip). Extremely hard materials may require stiffer ("harder") tips such as 225 µm, single crystal silicon TappingMode tips.

Digital Instruments offers cantilevers having a wide range of spring constants which are listed below. The choice of tip depends upon how hard the sample is. Generally, it is preferable to use the stiffest cantilever giving the best elasticity contrast for the sample; try starting with a stiffer cantilever then working toward softer tips. For samples of unknown hardness, start with a force modulation cantilever (Model No. FESP) to determine whether the tip is sufficiently stiff, then adjust accordingly.

TABLE 7. Force Modulation Tips

Cantilever	Model No.	Cantilever Length	Spring Constant
Standard Silicon Nitride	NP, DNP	100—200 µm	0.01—0.6 N/m
Oxide-sharpened Silicon Nitride	NP-S, DNP-S	100—200 µm	0.01—0.6 N/m
Contact AFM Etched Silicon	ESP	450 µm	0.02—0.1 N/m
Force Modulation Etched Silicon	FESP	225 µm	1—5 N/m
TappingMode Etched Silicon	LTESP	225 µm	20—70 N/m
TappingMode Etched Silicon	TESP	125 µm	20—100 N/m

SOFTER
HARDER

11.5.3. Force modulation—Operating Principle

Force modulation mode is very similar to contact AFM mode. The NanoScope III system scans the cantilever over the sample surface while attempting to keep cantilever deflection constant. A setpoint is entered to tell the system how much the cantilever should be kept deflected during operation. In addition, the cantilever will be oscillated up and down by a piezoelectric bimorph in the tipholder so that the tip indents slightly into the sample surface as it is scanned across the surface. The NanoScope III system records the amplitude of the cantilever motion, which indicates the relative indentation of the tip into the surface.

Harder sites on the sample surface cause increased cantilever response and amplitudes and are rendered as darker areas of the image. Softer sites absorb more of the cantilever's energy, causing a reduced cantilever response and lower amplitudes; these sites are rendered as lighter areas of the image.

11.5.4. Force modulation—Procedure

This section gives instructions for operating in force modulation mode.

1. Choose the Force Modulation operating mode.

Go to the **Other Controls** panel. Switch the **AFM mode** parameter to **Forcemod**.

2. Adjust the setpoint

Enter a **Setpoint**. It is usually convenient to enter a **Setpoint** of **0.0 V**. Recall that higher setpoints cause the cantilever to push harder on the surface during scanning.

3. Set the MultiMode switch to TappingMode.

When using Forcemod, the **Vertical Deflection/Setpoint** is shown in the center of the lower meter.

4. Adjust the detector offsets

Turn the detector adjustment screws to center the laser spot on the laser detector. Typically, the **Vertical Deflection** is adjusted to approximately **-1.0 V**. The force that the cantilever will apply to the surface is related to the difference between the **Vertical Deflection** and the **Setpoint**. If the sample is very delicate, it may be preferable to set the **Vertical Deflection** value closer to the chosen **Setpoint**.

5. Enter the Cantilever Tune menu

Regarding cantilever oscillation

The cantilever is oscillated by a small piezoelectric bimorph mounted in the tipholder. For most samples it is necessary to oscillate the bimorph at or near its resonant frequency. This ensures the cantilever moves with sufficient amplitude to produce elasticity contrast.

The bimorph's resonant frequency is the frequency at which it produces the greatest motion for a given **Drive Amplitude** (typically **5—30 kHz**). In free air, this resonant frequency will appear as one or two among several peaks on the **Cantilever Tune** screen; however, peaks due to cantilever resonance should be ignored. To distinguish between cantilever and bimorph peaks, the tip of the cantilever may be brought into light contact with a sample surface. This will dampen all but the bimorph peak(s), which will appear prominently on the **Cantilever Tune** screen. A procedure is described below that is familiar to users of TappingMode.

6. Set up and find the bimorph resonance.

Notes:

- It should be necessary to find the bimorph's resonant frequency only once. (Resonant frequencies are unique to each force modulation tipholder.) Once the resonant frequency is found, write it down for future use. The resonant **Drive frequency** may be used as a starting place each time that force modulation imaging is performed; however, the bimorph's resonant frequency in free air should be rechecked using **Cantilever Tune** each time a new tip is installed.

Ensure the tip is close to the surface (20-30 microns) before starting.

Select the **View / Cantilever Tune** menu, or click on the **Cantilever Tune** icon. The display monitor will plot the **Frequency Sweep**, showing the cantilever's oscillation amplitude vs. frequency. This plot is used to find the frequency where the bimorph oscillates with the largest amplitude.

Cantilever Tune

Auto Tune Controls
Start frequency: 0.000000 KHz
End frequency: 500.000 KHz
Auto Tune

Graph Controls
Sweep width: 40.0000 KHz
Sweep sample count: 256
Sweep graph range: 100 nm
Units: Metric

Main Controls
Drive frequency: 20.0000 KHz
Setpoint: -0.200 V
Drive amplitude: 867 mV
Input attenuation: 1x
Integral gain: 10.00
Proportional gain: 10.00

Quit Motor Interleave Controls

Start with a **Drive frequency** of approximately **20 kHz** and a **Sweep width** of approximately **40 kHz**. Enter a **Drive amplitude** of **1.5 V** to start (this will be

adjusted later). A series of peaks should be observed on the **Frequency Sweep** plot. A typical **Frequency Sweep** plot is shown in Figure 11.17.

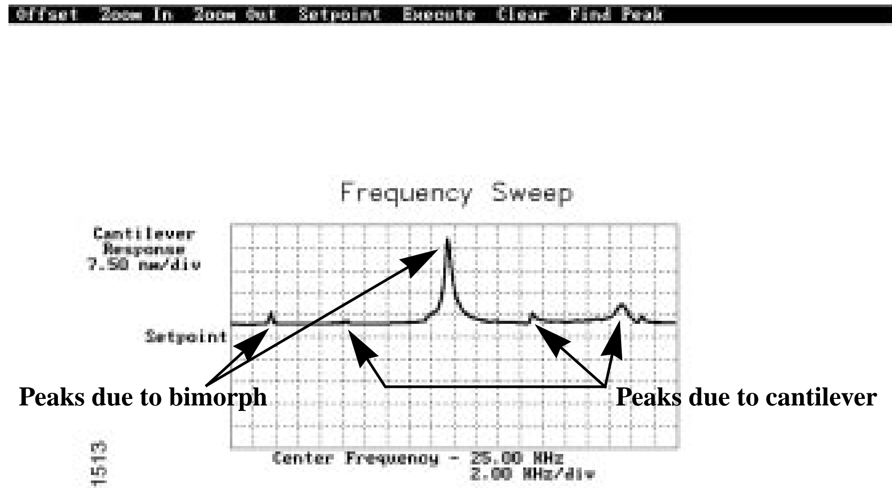


Figure 11.17. Typical frequency sweep plot. (NOTE: Several peaks may appear.)

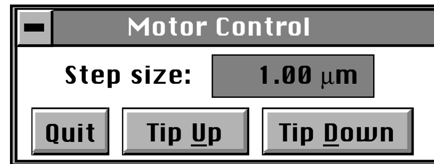
Adjust the **Sweep graph range** and/or the **Drive amplitude** until the peaks are clearly seen. Make certain, however, that the **Sweep graph range** and/or **Drive amplitude** is adjusted so that the top of each peak is seen. Note that while in **Force-mod** the **Setpoint** cannot be used to move peaks up and down.

7. Locate bimorph peaks; eliminate stray peaks.

In order to separate cantilever peaks from bimorph peaks, it will be necessary to lightly touch the tip to the sample surface. This will dampen all cantilever peaks, leaving behind those which are due solely to the bimorph.

To touch the tip to the sample surface, do the following:

Click on the **Motor** button located within the **Cantilever Tune** panel. The **Motor Control** panel will be displayed.



Set the **Step size** parameter at **1.00 µm**, then click on the **Tip Down** button repeatedly until the tip lightly contacts the sample surface. When the tip makes contact with the surface, a rapid change will occur in the **Frequency Sweep** plot: multiple peaks will give way to one or two larger peaks (e.g., at 8.00 and 20.00 KHz). An example is shown in Figure 11.18.

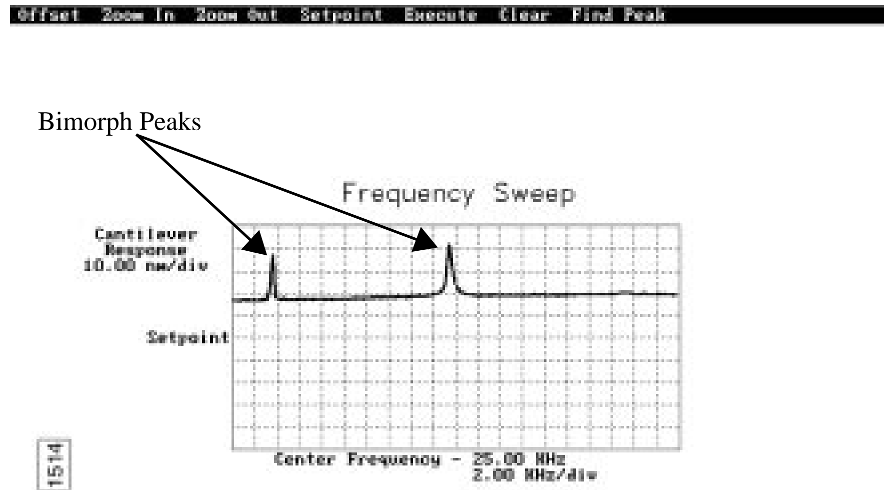


Figure 11.18. Typical frequency sweep plot showing bimorph peaks.

Identify the bimorph resonant peak(s). Bimorph resonances usually occur around 8 kHz and 20 kHz. Choose a peak, then click on the **Zoom In** command (top menu bar of display monitor) with the left mouse button. Two vertical lines will appear on the **Frequency Sweep** plot. Use the mouse to move the vertical lines back and forth until the selected peak is centered between the lines. Increase or decrease the zoom range by clicking on the left mouse button again. When the peak is centered and the zoom width has been adjusted, click twice on the right mouse button. This will automatically change the **Drive Frequency** and **Sweep width** and zoom in on the selected peak.

An alternative method is to use the **Offset** command on the display monitor to center the peak in the graph. Click on the **Offset** command with the left mouse button; a vertical green line will appear on the plot. Move the line until it is centered on the desired peak (usually the frequency with the highest amplitude), then click the left button again to lock it. Click on **Execute**. The computer will automatically change the **Drive frequency** so that the peak is centered. It may be necessary to click on this command more than once to get the peak properly centered.

Once the desired frequency is chosen, the **Frequency Sweep** plot should be recentered similarly to the plot shown below.

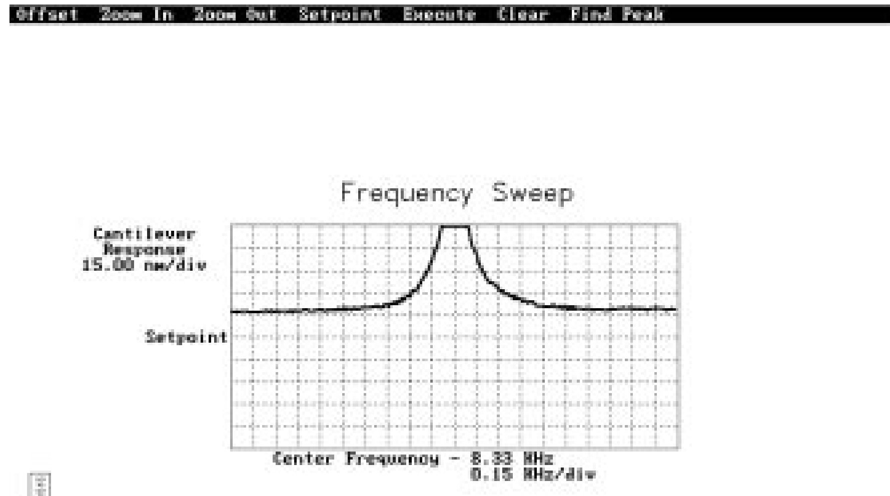


Figure 11.19. Correctly tuned forcemod frequency.

Drive frequency may also be changed by clicking on the **Drive frequency** parameter on the control monitor's **Feedback Controls** panel, then entering a new value.

REMEMBER: 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.

Notes

- If the bimorph resonant frequency was determined by placing the tip in contact with the surface, do not use that tip for force modulation imaging. Replace the cantilever with a new tip and double-check the resonant frequency without contacting the tip to the surface. From this point forward, cantilever tune should

be performed (without touching the surface) starting at or near the bimorph's resonant frequency each time a new tip is installed. Slight shifts in the frequency response may occur with different tips.

8. Check the scan parameters.

Check the various scan parameters, such as **Scan speed**, **Scan size**, **Integral gain**, etc. to verify they are reasonable for contact mode imaging. Also, it may be desirable to readjust the photo detector vertical adjustment screw if the Vertical Difference signal has drifted from the value originally set.

9. Select the proper Data Types.

The multi-image capability will be used to view both the topography data and the force modulation (elasticity) data at the same time. For the **Channel 1** image, select **Data Type: Height**. For the **Channel 2** image, select **Data Type: Amplitude**.

The **Data Type** chosen for the **Channel 1** image will be displayed when using the **Browse** command. If desired, the above choice can be reversed so that the surface topography (**Height**) is chosen for **Channel 2**.

10. Reduce the Drive Amplitude before engaging.

Reduce the **Drive Amplitude** to **0.00**.

11. Initiate the Engage command.

Select the **Motor / Engage** command. The motor will begin to move the SPM head down towards the sample and will stop once the cantilever is deflected to the chosen **Setpoint**. Under some conditions the system may "false engage" immediately after selecting the **Engage** command or before the cantilever actually reaches the surface. If this happens, reduce the **Drive Amplitude** and/or increase the **Setpoint** and try again. If other difficulties are encountered, see the "Troubleshooting" section of this manual.

12. Optimize scan parameters for Height Image.

First, adjust the **Integral Gain**, **Proportional Gain**, **Setpoint,** and **Scan Speed** to obtain a good topography (**Height**) image. This procedure will be very similar to optimizing the AFM for normal contact mode imaging, except that the gains will probably be higher. Typically, force modulation operation requires **Integral Gain** and **Proportional Gain** values of 10-20.

CAUTION: If employing stiffer cantilevers than those normally used, the sample may be damaged by the cantilever tip. To minimize this damage, make the **Setpoint** as low as possible.

ATTENTION! L'utilisation d'un levier plus rigide que ceux utilisés habituellement peut entraîner un endommagement de l'échantillon par la pointe. Afin de réduire les risques d'endommagement, ajuster la valeur cible du rétro-contrôle (**Setpoint**) à sa valeur la plus faible possible.

WARNHINWEIS! Falls Sie mit Cantilevern höherer Federkonstanten arbeiten, als normal üblich, kann die Probe durch die Spitze beschädigt werden.. Um eine evtl. Beschädigung zu minimieren bzw. zu vermeiden, benutzen Sie bitte einen Arbeitspunkt (Setpoint), der nur geringfügig über der freien Deflektion des Cantilevers liegt. Die Differenz zwischen Setpoint im „Feedback-Menü" und der freien Deflektion des Cantilevers (Photodetektorsignal A-B) sollte minimal sein.

Reduce the Setpoint using the cursor keys until the cantilever pulls off the surface. Note the value of the Setpoint when the cantilever pulled off the surface ("pull-off value"). Increase the **Setpoint** until the cantilever begins to touch the surface again and an image appears. Finally, readjust the setpoint so that it is slightly more positive than the pull-off value.

Be aware that there is a trade-off here! If the **Setpoint** is adjusted very close to the pull-off value, imaging will be performed with the smallest and least damaging force. However, imaging may be more unstable, as the cantilever may pull off the surface unexpectedly.

Also note that the setpoint value can affect the force modulation contrast as discussed below. It may be desirable to further adjust the setpoint after completing step 11.4.19.

13. Examine the contrast of the force modulation image.

The amplitude signal shows how much the cantilever can indent into the surface. Softer features will allow the cantilever to indent further into the sample, giving a small deflection amplitude, while hard features will allow little indentation and create large deflection amplitudes. The force modulation software is configured so that softer areas will appear lighter (higher) in the amplitude image. The relative elasticity of the sample is seen as contrast between dark and light areas in the amplitude image. Therefore, the amplitude image is really the inverse. This allows soft samples mounted on firm substrates to show up as positive shapes relative to the background.

14. Optimize Drive Amplitude for Force Modulation imaging.

Next, optimize the force modulation signal, which is the amplitude data. The amount of contrast and the quality of both **Height** and **Amplitude** images will depend on the **Drive amplitude**. In general, increasing the **Drive amplitude** will give greater contrast in the force modulation image.

It is possible, however, to set the **Drive amplitude** too high. In this case, as the **Drive amplitude** is increased, contrast will remain unchanged in the **Amplitude** image. Instead, the overall contrast in the force modulation image will remain roughly constant, but more artifacts will be observed in the image.

For example, if the drive amplitude is too high, the force modulation image will become contaminated by "edge effects" or "friction effects."

The best procedure is to operate at the lowest drive amplitude that gives sufficient contrast to examine the sample. Low drive amplitudes will also help extend the life of the cantilever tip and reduce sample damage.

Here is a suggested procedure for optimizing the drive amplitude:

- Reduce the drive amplitude to a very small value (say 100 mV).
- Increase the **Drive amplitude** with the arrow keys or by typing in new values. The contrast of the **Amplitude** (force modulation) image should increase.
- Continue increasing the **Drive amplitude** until sufficient contrast appears in the **Amplitude** image.

  If the **Drive amplitude** is increased to the point where contrast is no longer improving, reduce the **Drive amplitude** slightly from this value.

As mentioned above, the force modulation contrast will also depend on the **Setpoint**. In general, if using a larger **Setpoint** (larger tracking force) it will be necessary to 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 sometimes increasing) the **Setpoint** and then optimizing the **Drive Amplitude** again.

15. Readjust gains (if necessary).

The value of the **Drive amplitude** may also affect the contact mode image, making the system more or less likely to go into unwanted oscillations. If the **Drive amplitude** is changed by a large amount it may also be necessary to readjust the **Integral gain** and **Proportional gain.** The gains should be set as high as possible to track the sample topography, but not so high that they show the oscillation due to the bimorph oscillation. If the gains are set too high, parallel diagonal lines will be observed on the **Height** image. If an oscillation is seen, the feedback system is trying to cancel the cantilever oscillation by moving the Z-axis piezo in the opposite direction. If there is evidence of an oscillation in the Height data, turn the gains down until the oscillation stops.

Notes

- Oscillations not visible in height **Image Mode** data may be seen more easily using **Scope Mode**.

Sometimes an oscillation that appears in the data will be due to "aliasing" as described in the next section. If gains cannot be adjusted to eliminate unwanted oscillations without compromising the height image's quality, see the next section below.

16. Changing the Drive Frequency to eliminate "aliasing."

Under some conditions, unwanted oscillations will appear in the data due to aliasing of the **Drive Frequency** with the image pixel rate. This problem can be eliminated by changing the **Drive frequency** by small increments. Use the arrow keys to change the **Drive frequency** up or down very slightly until the oscillation disappears. On some materials, shifting the **Drive frequency** slightly (1-3 Hz) below or above a resonant peak value may improve image contrast. Operators are encouraged to experiment.

Notes

It is possible to see artifacts in force modulation images that are not due to differences in elasticity. Some artifacts to watch for are outlined below:

Edge effects

Sometimes force modulation images will show changes in amplitude at the edge of topographic features like steps or bumps. These artifacts may look like the derivative of the sample topography.

To see if a feature is an edge effect, try reversing the **Scan direction** from **Trace** to **Retrace,** for example. If the contrast reverses or the amplitude change now appears on the other side of the topographic feature, then the amplitude change is likely due to the topographic edge, not differences in elasticity. To minimize edge effects, reduce **Drive amplitude, Setpoint** or **Scan speed.** Also 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 is pushed into the sample.

This is shown schematically in Figure 11.20. The amplitude of cantilever motion can then be affected by differences in friction between two different materials. For this reason, force modulation images may also contain information about differences in local frictional forces. To reduce the influence of frictional effects, use a smaller **Setpoint** and/or **Drive amplitude**.

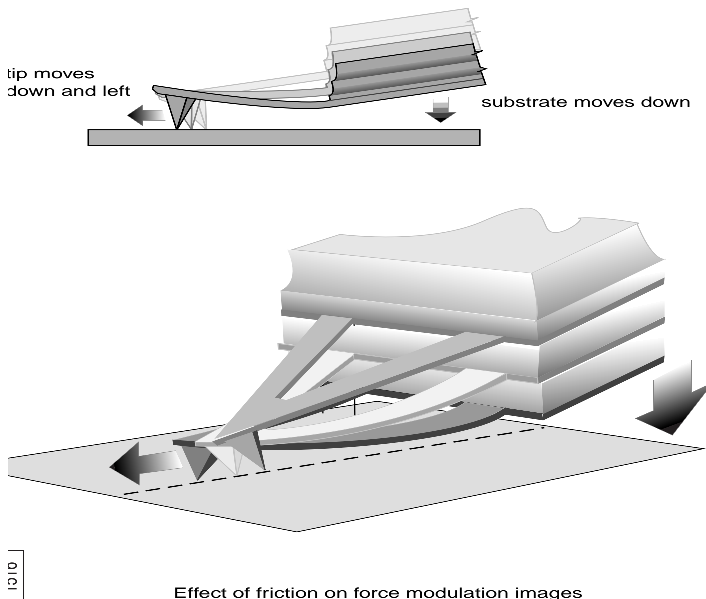


Figure 11.20. Effect of friction on force modulation images.

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 will indent deeper than a dull tip. Since it is possible for the tip to dull during imaging, consider replacing 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 having excellent elasticity contrast may not show up in force modulation if their absolute elasticity is out of instrumental range. A new method, “negative LiftMode,” is described below which may prove useful for imaging otherwise difficult materials.

11.6. Force Modulation with 'Negative LiftMode'

A new form of force modulation imaging utilizing TappingMode and LiftMode operation has recently been introduced. This method, known as "negative Lift-Mode," allows imaging of certain materials previously not visible with contact mode AFM force modulation. The method is especially suited for some softer materials, yielding higher resolution. A general procedure is described below.

Notes

- Force modulation contrast is very sensitive to the spring constant of the tip, which varies according to the length and thickness of the cantilever. TappingMode may be easily performed with either force modulation or TappingMode cantilevers, but may prove difficult with contact AFM silicon and silicon nitride cantilevers. (See table in Section 11.5.2 for guidance.)

11.6.1. Find Resonance of the Bimorph

The force modulation tipholder features a bimorph, which excites the probe and cantilever mount. Refer to Section 11.5.9 for guidance on finding bimorph resonance.

1. Ensure the probe is withdrawn from the sample surface. Switch the **AFM mode** parameter to **Tapping**.

2. Set **Drive amplitude** on the **Interleave Controls** panel to **200—400 mV**.

3. Go to the **Real Time / View / Cantilever Tune** dialog box. (For version 4.10 or later software, also click on the **Manual** button within this box.)

4. Next, select the **Interleave Controls** button to access tuning controls for interleaved scans.

5. The bimorph resonance may be found using manual controls. Bimorph resonance is usually about 8-12 KHz. Start with a **Sweep width** of **10 KHz** and a **Drive frequency** of **10 kHz**. Determine the **Drive frequency,** using Section 11.5.9 above as a guide.

6. Verify that the **Drive frequency** parameter in the **Interleave Controls** panels is set to the bimorph's resonant frequency. Click on the enable button next to the **Drive frequency** parameter; the enable button should appear green. Quit the **Cantilever Tune** dialog box.

11.6.2. Set Interleave Controls

1. Transfer to the **Real Time / Interleave Controls** panel.

2. Set the **Interleave scan** parameter to **Lift**. Set **Lift start height** and **Lift scan height** to **0.00 nm**.

3. For now, set the **Interleave mode** parameter to **Disabled**.

11.6.3. Obtain a TappingMode Image

While negative LiftMode force modulation data will be imaged using **Channel 2**, height data will be obtained using TappingMode on **Channel 1**. The user must therefore obtain a satisfactory TappingMode image.

1. Verify that the **Interleave mode** parameter on the **Interleave Controls** panel is **Disabled**. Verify that the **AFM mode** parameter in the **Other Controls** panel is set to **Tapping**.

2. Select **Real Time / View / Cantilever Tune**. On version 4.XX software, the cantilever may be tuned using **Auto Tune**. Enter a main controls **Start frequency** value of **5 KHz** and **End frequency** value of **400 KHz**. On version 3.XX software, use manual controls. Set the **Drive frequency** value in the **Main Controls** box of the **Cantilever Tune** dialog box to the cantilever's resonant frequency. Exit the **Cantilever Tune** dialog box.

3. Verify that all **Scan controls** and **Feedback Controls** parameters are set for obtaining a TappingMode image.

4. Set the **Channel 1** panel's **Data type** parameter to **Height**. Set the **Line direction** to **Retrace** and enter an appropriate **Z range** value for your sample.

5. Engage the sample surface and obtain a good TappingMode image.

11.6.4. Obtain a Negative LiftMode Force Modulation Image

1. Set the **Data type** parameter in the **Channel 2** panel to **Amplitude**. The **Line direction** parameter should be set to **Retrace**.

2. Switch the **Interleave mode** parameter in the **Interleave Controls** panel to Enabled. Negative LiftMode imaging is now in effect.

3. Using the left arrow key, optimize the negative LiftMode image by slowly decreasing the **Lift scan height** parameter in the **Interleave Controls** panel from zero until the surface is reached. In most cases, it should not be necessary to go below **-60.0 nm**. (This is sometimes best performed while in **Real Time / View / Scope Mode**.)

Notes

- Use of a negative **Lift scan height** value here gives rise to the term "negative LiftMode." When the interleaved scan is enabled, the tip's TappingMode height above the sample will be reduced by the **Lift scan height** amount. This places the oscillating tip in contact with the sample as surface features are profiled (from TappingMode data). 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.

4. Adjust the interleaved **Drive amplitude** and **Lift scan height** until the force modulation image is optimized. This may require some experimentation.

Notes

- If you are seeing a lot of contrast in the amplitude image before reaching the surface, try reducing the **Integral** and **Proportional gains**. It may be necessary to adjust gains slightly lower than when performing normal TappingMode imagery. A good way to check this is to enter a **Lift scan height** of **100 nm**, then adjust gains (and possibly **Drive amplitude**) until you see the minimum amount of contrast in the amplitude image. (This may be easily monitored in **Scope Mode**.) Once contrast is minimized, enter a **Lift scan height** of **0.0 nm** and begin approaching the surface.

11.6.5. Final Comments

Best results using negative LiftMode have been obtained on relatively smooth samples (< 500 nm vertical features); however, DI encourages experimenting with this technique on rougher surfaces as well.

11.7. Force Volume Imaging

11.7.1. Description

The usefulness of force plots has been shown in the preceding sections of this chapter. Force plots may be made even more useful by plotting across the surface of a sample at regular intervals, then analyzing the result. This is known as *force volume* imaging. The word *volume* is used because for each X-Y position there is a force curve in Z for some selected range. Each curve gives a cumulative force for that area of the sample. By plotting these values along X and Y coordinates, a force volume image is rendered which permits the viewing of stratified layers of force at various Z-axis heights above the sample surface.

Force volume images require preliminary use of the **Force Plot** function. The microscopist must have a basic working knowledge of this function before attempting **Force Volume** (see Section 11.2 above). Before obtaining a force volume image, it is also important to determine exactly what is sought after and what is available for a given mode of imaging. The matrix below summarizes each of the major imaging types.

Signal Type	Contact AFM	TappingMode
Amplitude		4
Deflection	4	4
Friction[a]	4	
Phase[b]		4
Frequency[b]		4
Potential[b]		4

a. The usefulness of force imaging with friction has not yet been determined.

b. Phase, frequency and potential measurements are available only on SPMs equipped with the Extender™ Electronics Module.

The type of force image captured from a surface will depend upon how the SPM is set up. For example:

- If an MFM image is being captured, force volume imaging (phase) allows the detection of long-range magnetic forces otherwise difficult to detect.

- For ordinary contact AFM, the use of force volume imaging (deflection) allows the user to see otherwise invisible electrostatic forces.

Notes

- It is incumbent upon the microscopist to carefully define all force volume experiments. At this writing, there are at least ten types of force volume imaging, including numerous combinations of triggers, drive frequency and amplitude, Z-axis direction, offset, etc. The utility of each image is subject to the interpretations and controls of the experimenter. This section of the *Instruction Manual* cannot cover all possible force volume types; therefore, discussion is limited to a general description of controls and an example in contact AFM. Experimentation is encouraged. Interpretations are subject to experimental controls.

11.7.2. Setting Up for *Force Volume*

To summarize, force volume imaging executes the following tip motions:

- The tip is lowered and pressed against the sample surface until the cantilever is deflected to the **Trig[ger] threshold** value. If the surface is not contacted within one **Z scan size** distance, the tip is extended one additional **Z scan size** (for a total distance of two times the **Z scan size)**, then retracted one **Z scan size**. This means that the tip is incrementally lowered (or "racheted") one **Z scan size** for each extension-retraction cycle until the surface is contacted, or the Z piezo reaches its total maximum of 220 volts.
- Once the tip contacts the surface and deflection attains the **Trig[ger] threshold** value, sample height is recorded for that Z-axis threshold. A force curve is recorded for the X-Y coordinate after the piezo retracts. The tip is lifted clear of the surface and translated in X-Y to the next coordinate. The entire process is then repeated.

The general sequence for obtaining a force volume image consists of the following:

1. Obtain a height, deflection, amplitude or phase image of the sample in **Image Mode**. On the **Channel** panel, select the same **Data type** intended for force plotting and for triggering.

2. Obtain a **Force Plot** of the sample surface (any portion of the surface will suffice). Using the force plot, determine the sample's general force characteristics and set the following parameters: **Setpoint**, **Z range**, **Z scan size**, **Z scan start**, and **Trigger mode**.

Notes

- A trigger will be required to obtain a height image during force volume imaging.

3. Return to **Image Mode**.

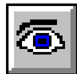
4. Obtain a **Force Volume** image by selecting **View / Force / Volume**. Set the following parameters in the **Z Scan Controls** panel: **Z scan rate**, **FV scan rate**, **Number of samples**, **Force per line**, **Samples per line**, **Display mode**, **Sample period**. Fine tune **Image Channel** parameters to obtain a height image. Based upon the experimental design, set parameters in the **FV** (**Force Volume**) **Channel** and **Force Channel** panels.

5. **Capture** the **Force Volume** image. Note that this will take longer than a normal image capture—the force volume image may consist of up to 512 samples taken 512 times per line at the **Z scan rate** per pixel.

6. Interpret the **Force Volume** image using **Off-line** controls.

11.7.3. Obtaining Force Volume Images Using Contact AFM

Contact AFM offers perhaps the most intuitive introduction to **Force Volume** imaging. The forces described here will be limited to simple attractive forces (e.g., electrostatic and surface tension) and repulsive forces (e.g., electrostatic, and hard contact or coulombic). The reader should already know how to obtain a basic contact AFM force plot. If not, review sections 11.1—2 in this chapter before proceeding further.



1. Mount sample and obtain a contact AFM image.

Initially, the sample should be familiar enough to obtain an image easily (e.g., silicon calibration reference). Make certain the **AFM mode** parameter in the **Other Controls** panel is set to **Contact**. After engaging the surface, use a moderate **Setpoint** value. Optimize the **Channel 1** parameters for a good image: set **Data type** to **Height** and **Real Time Planefit** to **Line**.

2. Obtain a force plot of the surface.

To obtain a force plot of the surface, select **Real Time / View / Force Mode / Calibrate**. Set the **Feedback Controls** panel **Input attenuation** parameter to **1x**. On the **Z Scan Controls** panel a recommended default for **Z scan rate** is **40 Hz**. To set

the **Sensitivity** parameter, use the mouse to draw a line on the force plots parallel to their slope. Click the left button to drop each end of the line; click on the right button to enter the **Sensitivity** value and erase the line.

The **Start mode** parameter should be set to **Calibrate**. The **Trigger mode** may be set to **Relative**.

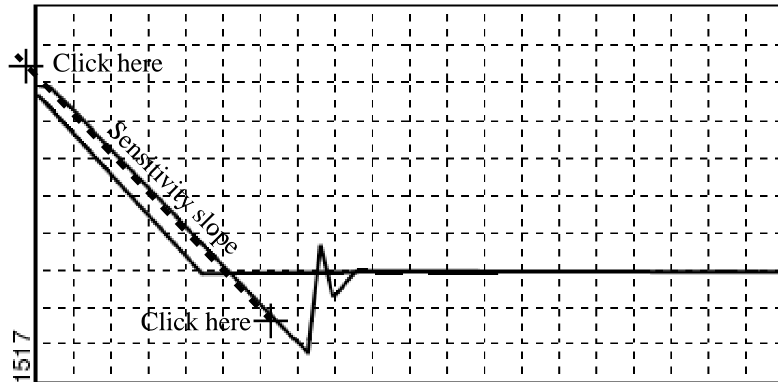
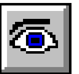


Figure 11.21. Obtaining a good force plot.

3. Return to *Image mode*.



When a good force plot is obtained, return to **Image mode**.

4. Obtain a *Force Volume* image of the surface.

To obtain a force volume image, click on the **Real Time / View / Force Mode / Volume** option. The display monitor will appear similar to the one shown below, except that most of the image windows will be blank at first. (A minimum of several minutes are required to obtain complete images.)

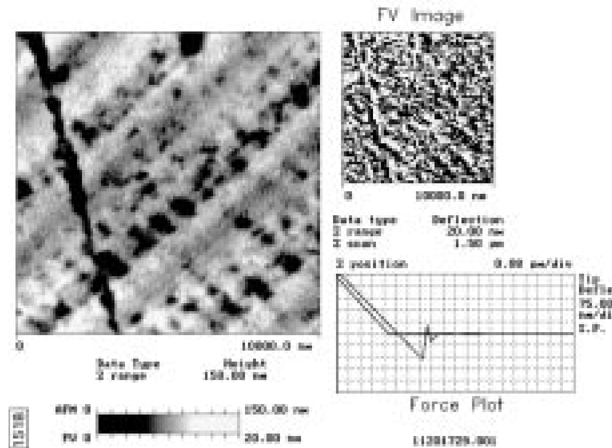


Figure 11.22. Force volume image.

The large image on the left side of the screen is a height image similar to the one shown in **Real Time / Image mode**; however, it may appear more pixelated. The smaller image at upper-right plots force as a function of Z distance. The resolution of the force plot will vary, depending upon various parameter settings. Finally, the force plot at the bottom-right corner of the screen displays a series of superimposed force plots for each scan line.

5. Set *Force Volume* parameters.

There are six **Force Volume** panels which control sampling and scanning for the force volume image and force curve. They are organized by function:

- **Image Scan Controls**— Real Time parameters for the height image shown in the upper-left corner of the display monitor, such as **Scan size**, **X**- and **Y offset**, **Scan angle**, and **Scan rate**.

- **Image Channel**— More Real Time parameters affecting the height image.
- **Z Scan Controls**— Parameters affecting the tip's vertical movements, including **Z scan start**, -**size** and -**rate**.
- **Feedback Controls**— Parameters controlling **Setpoint**, and where the tip is to be turned around (triggered): **Trigger mode**, **-threshold** and **-channel**.
- **FV Channel**— Force Volume channel controls affecting the force volume image in the upper-right corner of the display monitor, including **Volume scale**, **Force per line**, **Z direction** and -**display**, and **Volume offset**.
- **Force Channel**— More parameters affecting the Force Volume image.

Remember: you are constructing a "map" of many separate force plots across the sample surface; therefore, there may be a great deal of data to sort through. Depending upon settings, capturing a detailed force volume image can require hours.

Notes

The total amount of data for any given force volume image file is limited to a maximum 1 Mb by the relation

$$\text{Force per line}^2 \times \text{Number of samples} \times 4 \leq 1 \text{ Mb}$$

The following parameters are carried over from **Force Plot** settings: **Z scan start**, **Z scan size**, and **Z scan rate**. Although these parameters may be altered during force volume imaging, they should obtain their initial settings from the **Force Plot** window.

Enter the **Number of samples** parameter to set the resolution (or number of "shells") of the force volume image and number of points plotted on the force curve at the bottom of the display screen. The value of this parameter will not affect the speed of the scan, but will affect the file size. A good default setting is **32**.

Next, enter the **Force per line** value. This parameter determines the number of force curves taken along each line of the sample and significantly affects capturing speed. Enter a number which allows sufficient resolving of force-defined features, but minimizes capturing time. A good default value is **64**.

Finally, enter the **Samples per line** value. This parameter sets the number of samples to be displayed in the height image only and does not affect the force volume image in any way. Increasing this parameter increases the capture time. A setting of **128** provides sufficient detail to resolve many small features while helping to reduce capture time.

The **Display mode** parameter may be set to show the **Extend** portion of the plot, the **Retract** portion, or **Both**.

For most imaging, the **Sample period** parameter should be held at **30 µs**. (Sample periods less than **30 µs** may be too fast and cause errors.) This parameter determines the sampling rate of data at the SPM's digital signal processor, which some experimenters may wish to increase. This parameter is coupled with two other parameters: the **Z scan rate** and **FV scan rate**; that is, changes in the **Sample period** will change both **Z scan rate** and **FV scan rate**.

Keeping the **Sample period** minimized at **30 µs** will yield optimal data averaging between Z-axis sampling points. As this parameter is increased, data averaging is lessened and data becomes discrete.

6. Adjust Feedback Controls parameters.

Feedback Controls panel parameters carry over from the **Force Plot** settings; however, changes may be required to protect the tip and optimize the force volume image.

For silicon nitride tips, the **Setpoint** may be adjusted to within plus or minus several volts of the microscope's vertical deflection signal value. For crystal silicon TappingMode tips, the **Setpoint** should normally be increased no more than 1-3 volts below the RMS amplitude voltage; this will help protect the tip.

In **Force Volume** imaging, triggers are used to set the turnaround point of the Z-axis piezo. The **Trigger mode** determines the type of trigger to be employed. Two types are offered: **Relative** and **Absolute**, or the trigger may be turned **Off**.

Notes

- If the **Trigger mode** is turned **Off**, no height image will be displayed.

The **Trigger channel** determines which data channel is to act as the trigger. (Normally, this will be the same as the **Data type** located on the **Force Channel** panel.)

A **Relative** trigger measures the trigger threshold relative to the free-air voltage deflection value and accommodates drift. An **Absolute** trigger sets the threshold relative to the **Setpoint**. Normally, the **Relative** trigger is the preferable default, as it offers better protection to the tip and sample, keeping total force on the surface independent of setpoint.

Notes

- When using a **Relative** trigger threshold, be certain a sufficiently large **Z scan size** is in effect to deflect the cantilever to the **Trig**[ger] **threshold** value AND lift the tip clear of the surface. This will ensure the tip is not "racheted" into the surface and dragged laterally through surface material during X-Y indexing. (Pre-version 4.23 software releases.)

To limit forces on the sample and tip, the **Z scan size** may be clipped to within some **Trig**[ger] **threshold** value. For example, a tip which is being oscillated along the Z-axis with a **Z scan size** of **500 nm** may have its **Trig threshold** set to **-25.0 nm**. When using a **Relative** type of **Trigger mode**, if the tip encounters the sample surface after extending 300 nm, it will halt its Z-axis extension at 325 nm, then turn around (retract). Thus, tip-sample forces are restrained and the force curve is defined for a controlled interval of tip-sample interaction.

Notes

- This example assumes the **Sensitivity** parameter has been properly set (see step #2 above) and that the detector's range has not been exceeded.

7. Adjust Force Channel parameters.

The **Force Channel** panel features parameters for the force plot at the bottom of the display monitor. In most ways it is exactly like a **Force Plot** graph, except that it superimposes up to eight multiple force plots (depending on **Samples per line**) simultaneously for each scan line. When the next line is scanned, previously shown plots are cleared.

The available settings for the **Data type** parameter will depend upon the type of imaging being done. For example, **Phase** is available only on microscopes in TappingMode which have an Extender™ Electronics Module. Also available are **Amplitude**, **Deflection**, **Phase**, **Potential**, and **Thermal**. Set the **Data type** accordingly.

Begin with a large **Z range** to locate the force plots. (At full size, the force plots will probably resemble a thin line.) Slowly decrease the **Z range** value until the force plots fill most of the graph.

The **Offset** parameter determines where force plots are to be graphed relative to the current **Setpoint**. When the **Offset** is **Off**, the graph's center horizontal line is positioned at the tip's **Setpoint** value. When **Offset** is **Enabled**, the center horizontal line is positioned at the tip's free-air voltage (i.e., voltage when the tip is clear of the surface).

8. Adjust FV [Force Volume] Channel parameters.

Parameters on the **FV** (Force Volume) **Channel** panel control the type and range of forces viewed in the force volume image on the display monitor. In addition, the **Volume scale** parameter also affects the viewable range of data captured during force volume imaging.

The **Z direction** parameter determines which portion of the Real Time force curve cycle (**Extend** or **Retract**) is shown on the display monitor's force volume image. For example, if the force of interest is material elasticity, the **Extend** portion of the curve is usually selected. If the force of interest is electrostatic attraction, the **Retract** portion of the curve is generally used.

The **Volume scale** parameter sets the range of values represented by the force volume image. Because force volume data is displayed line-by-line at periodic intervals, optimizing this parameter may require several adjustments. The results of changing this parameter are not seen until the next line of force volume data is displayed.

The **Z display** and **Volume offset** parameters work in unison to define which portion of the superimposed force curves is to be plotted in the display monitor's force volume image. These parameters are also simultaneously represented as a green cursor on the force plot graph. Both **Z display** and **Volume offset** may be changed by positioning the cursor cross with the mouse, or vice versa. (The cursor is repositioned automatically whenever these parameters are changed.) Each parameter's relationship to the cursor is shown in Figure 11.23.

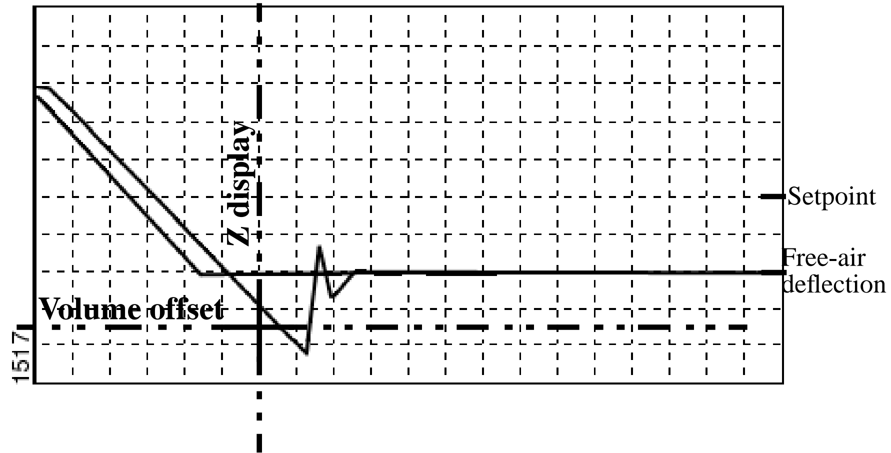


Figure 11.23. Z Display and volume offset in relation to cursor.

Changing the **Z display** parameter determines where along the Z-axis the force curve is to be plotted. For example, a **Z display** value of **25.0 volts** would plot that portion of each force curve's Z-axis having a minimum 25.0 volts tip-sample separation. The **Z display** parameter may be thought of as defining bands of force at fixed distances from the sample surface.

The **Volume offset** parameter adds or subtracts a constant value to the data signal to be plotted. For most applications, **Volume offset** should coincide with a value on the force curve itself. This is most easily accomplished by positioning the cursor on the force curve. This centers the force volume image within the color bar of the specified **Z range**.

Chapter 12 Interleave Scanning and LiftMode

Preface: Interleave Scanning & LiftMode™

Interleave is an advanced feature of Nanoscope version III 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 13 provides detailed instructions for obtaining MFM images of a standard magnetic sample. Enabling Interleave with the mode set to **Lift** enacts **LiftMode**™ (patent pending). 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. 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. 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 13. **LiftMode** was developed to isolate purely MFM and EFM data from topographic data.

Interleave can also be used in **Normal** mode. In this mode, the feedback is kept on while additional topography, 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 13 provides 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.

12.1. 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 12.1. This is the standard scan pattern of the NanoScope III.

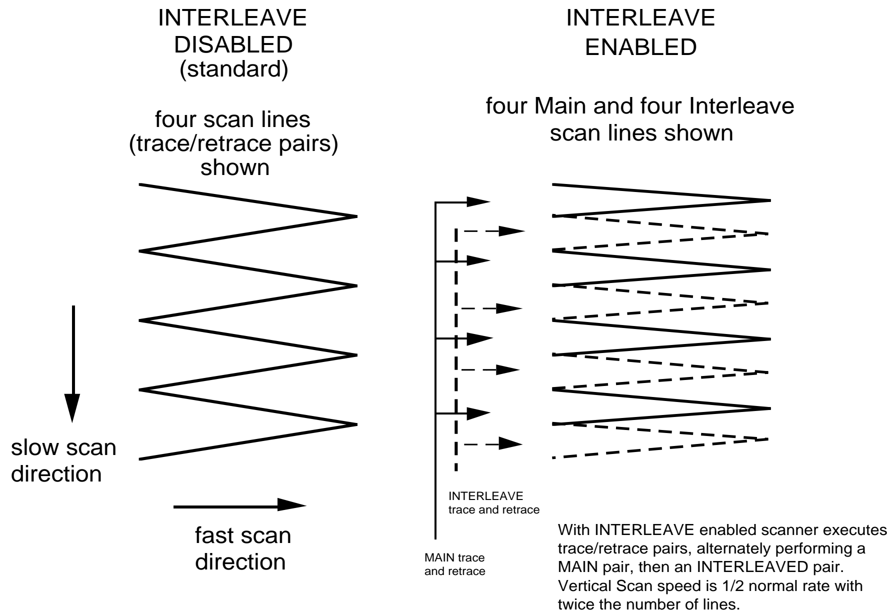


Figure 12.1. X-Y scan pattern.

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 12.1, 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**: **Normal** and **Lift**. With **Normal** 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; see below.

12.2. Lift Mode 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 12.2.

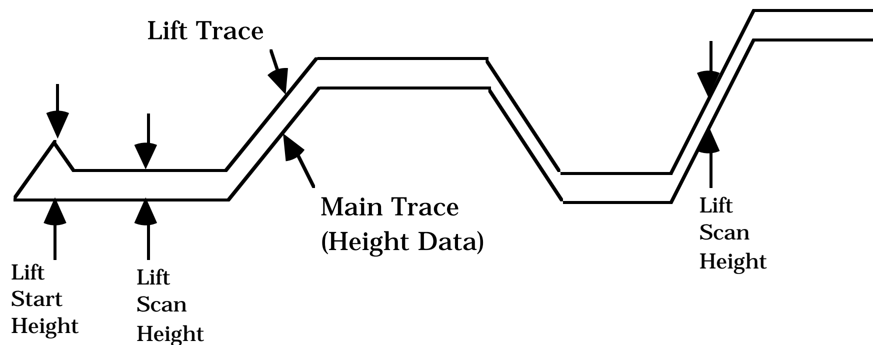


Figure 12.2. LiftMode profiles.

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.

12.3. Operation of Interleave Scanning / Lift Mode

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 13 for specific examples using MFM.

1) Obtain a topography scan using the appropriate (usually Contact or Tapping-Mode) 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** (**Normal** 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 **Setpoint** can be set differently in the **Interleave Controls** panel than in the main **Feedback Controls** panel; see section 12.4. However, it is often convenient to begin with the main and interleave values set equal; this can be done by toggling the bullets to the left 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 **Normal** mode, set the **Gains** and **Setpoint**.

Note that certain constraints are imposed: scan sizes, offsets, angles, and rates and numbers of samples per scan line are the same for the main and interleave data, and the imaging context (contact, TappingMode, or force modulation) must also match.

3) Enable the Interleave Scan.

Toggle the **Interleave mode** switch under Interleave Controls to **Enabled**. This begins Interleave scanning; note that the scan rate in the slow direction is halved.

4) Choose the Interleave Data Type

This can also be done before enabling the Interleave. In **Normal** mode, the choices of Data Type are the same as for the main scan. Depending on the type of microscope, **Lift** mode allows the options of amplitude, phase, or frequency data types for doing far-field (MFM or EFM) imaging; see Chapter 13 for detailed examples. Auxiliary channels are also available for some applications. Interleave data is typically displayed as the Second (right) Image.

5) Display the interleave data by switching **Scan line** (in the **Channel** panels) to **Interleave**.

Notes

- Lift scan height

  The lateral and vertical resolutions of the Lift data depend on the distance between tip and sample: the lower the tip, the higher the resolution. However, the **Lift scan height** must be high enough that the tip does not contact the sample during the Lift trace and retrace.

- Tip Shape

  As shown in figure 12.2, 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 12.2.

- Scan Line Direction

  The **Line direction** should be set to **Retrace** for both the main and interleaved scans. If it is set instead to Trace, a band may appear along the left side of the images due to the ramp between the surface and the **Lift scan height**.

12.4. Use of LiftMode with TappingMode

There are additional considerations when using LiftMode with TappingMode.

12.4.1. 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 13. (However, certain parameters can be set independently for the Interleave scan; see below.)

12.4.2. Setpoint Selection

When the main and interleave **Drive amplitudes** and **Drive frequencies** are equal (bullets under **Interleave Controls** disabled), 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**.

12.4.3. 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 toggling the flag on the left of the corresponding Interleave Control to "on" (green) and adjusting the value. This allows the tuning of a measurement in the Lift scan lines without disturbing the topography data acquired during the Main scan lines. The Interleave **Drive amplitude** must be set low enough that the tip does not strike the surface during the Lift pass.

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 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.

WARNHINWEIS: 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 13.

Notes

- The cantilever drive circuit features a filter capacitor to limit the rate at which the drive amplitude is changed between Main and Interleave scanning. If using scan rates above a few hertz, it may be advantageous to remove or disable the filter. For more information on how to disable the filter, contact Digital Instruments technical support.

12.4.4. Amplitude Data Interpretation

When monitoring amplitude data in LiftMode, brighter regions correspond to smaller amplitude, and darker regions to larger amplitude.

12.4.5. 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 13.

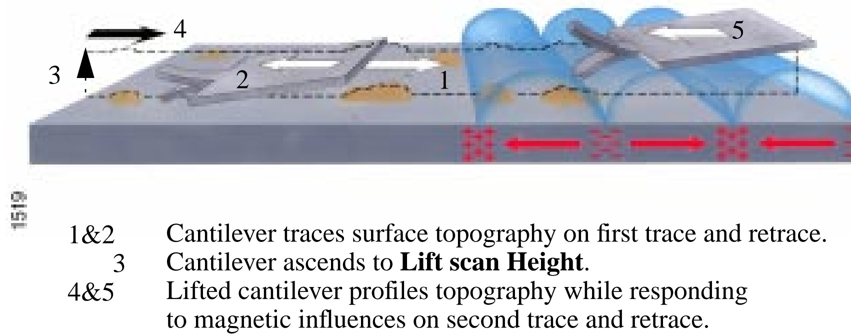
Small amplitudes must be used to sense force gradients, such as Van der Waals forces, which occur over short distances (typically a few nm). As much of the cantilever travel as possible should be within the range of the force gradient.

Chapter 13 Magnetic Force (MFM) Imaging

13.1. Magnetic Force Imaging Overview

MFM imaging utilizes the Interleave and LiftMode procedures discussed in Chapter 12; users are advised to read appropriate sections prior to attempting MFM imaging. Best results will be obtained with Digital Instruments' Extender™ Electronics Module. This hardware unit allows phase detection and frequency modulation for optimal MFM imaging. Instructions for software installation of the Extender Electronics Module can be found at the end of this chapter; complete instructions for hardware and software installation are included when you purchase your Extender Electronics Module.

In MFM, a tapping cantilever equipped with a special tip is first scanned over the surface of the sample to obtain topographic information. Using **LiftMode** as shown in Figure 13.1, the tip is then raised just above the sample surface. The surface topography is scanned while being monitored for the influence of magnetic forces. These influences are measured using the principle of *force gradient detection*. In the absence of magnetic forces, the cantilever has a resonant frequency f_0. This frequency is shifted by an amount Δf proportional to vertical gradients in the magnetic forces on the tip. The shifts in resonant frequency tend to be very small, typically in the range 1-50 Hz for cantilevers having a resonant frequency f_0 ~100 kHz. These frequency shifts can be detected three ways: **phase detection**, which measures the cantilever's phase of oscillation relative to the piezo drive; **amplitude detection**, which tracks variations in oscillation amplitude; and **frequency modulation**, which directly tracks shifts in resonant frequency. **Phase detection** and **frequency modulation** produce results that are generally superior to **amplitude detection**.



1&2	Cantilever traces surface topography on first trace and retrace.
3	Cantilever ascends to **Lift scan Height**.
4&5	Lifted cantilever profiles topography while responding to magnetic influences on second trace and retrace.

Figure 13.1. MFM LiftMode principles.

All standard Dimension-series SPMs and MultiModes are capable of MFM imaging using amplitude detection techniques. By adding an Extender™ Electronics module (see Figure 13-2), either system may also be used for frequency modulation or phase detection, giving improved results. Amplitude detection has largely been superseded by frequency modulation and phase detection. A more extensive discussion of force gradient detection and MFM imaging is given in the reprint *Magnetic Force Microscopy: Recent Advances and Applications*. Contact Digital Instruments to obtain a copy.

Notes

- In the instructions below, steps specific to phase and amplitude imaging are described independently. Use the icons in the margin to locate steps specific to either frequency modulation and phase detection, or amplitude detection.

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Figure 13.2. Extender™ Electronics Module (required for MFM phase detection and frequency modulation).

13.2. MFM Using Interleave Scanning and LiftMode

This section provides instructions for using the **LiftMode** of Interleave scanning to obtain MFM images. These guidelines will help in obtaining an MFM image of a standard magnetic sample (metal-evaporated video tape). Standard tape samples are provided with purchase of MFM probes, and can be obtained free of charge from Digital Instruments. 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 of interest. Many of the principles discussed here also apply to Electric Force Microscopy (EFM), described in Chapter 14.

For MFM procedures, NanoProbe™ magnetic coated tips are required. Various kinds of MFM probes are available for specific applications; contact Digital Instruments for more information. The remainder of this chapter assumes that the reader is familiar with the operation of TappingMode to obtain topographical images of a sample surface and has read the description of Interleave scanning in their manual.

LiftMode allows the imaging of relatively weak but long-range magnetic interactions while minimizing the influence of topography (Figure 13.1). Measurements are taken in two passes across each scanline; each pass consists of one trace and one retrace. In the first pass, topographical data is taken in TappingMode on one trace and retrace. 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. Using LiftMode, topographical features are virtually absent from the MFM image (see Figure 13.7).

The procedure below gives suggested parameter values that should work well for most applications. Further adjustment will, in some cases, improve the quality of MFM scans, and some experimentation may be needed to optimize the imaging of specific samples. See the suggestions at the end of this section.

As mentioned above, the NanoScope III uses *force gradient detection* for MFM imaging. Within this general technique, there are three possible schemes, known as *frequency modulation*, *phase detection*, and *amplitude detection*. All three are discussed here. Prior to 1994, amplitude detection had been the standard method for magnetic force imaging on the NanoScope III. However, hardware that allows phase detection and frequency modulation is now available for all TappingMode-capable microscopes in the form of Digital Instruments' Extender™ Electronics Module. (Microscopes without the Extender addition cannot utilize phase detection; for more information, contact Digital Instruments.) Phase detection and fre-

quency modulation detection are superior methods for magnetic force imaging, offering greater ease of use, better signal-to-noise ratios, and reduced artifact content as compared to amplitude detection. If extensive MFM imaging is planned, the Extender Electronics module is strongly recommended.

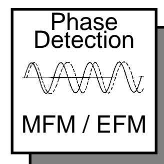
13.2.1. Procedure

1. Mount a NanoProbe magnetic probe on the scanner or tip holder. The tip should be magnetized with a strong permanent magnet before installing the tip holder on the AFM head. Tips are usually magnetized with the field aligned along the tip axis (perpendicular to the sample surface). The MFM then senses force gradients due to the perpendicular component of the samples's stray field. Tip magnetizers are provided with MFM probes purchased from Digital Instruments.

2. Set up the AFM as usual for TappingMode operation. In all **Channel** panels, the **Highpass** and **Lowpass** filters should be **Off**. Set the **Rounding** parameter in the **Microscope / Calibrate / Scanner** window to zero (**0.00**).

3. The procedure to tune the cantilever drive frequency and 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 8 of the product instruction manual. MFM cantilevers have resonant frequencies between 50 and 100 kHz. If using the **AutoTune** feature, these values can be used as bounds for the frequency sweep. With the Extender option, two curves appear in the **Cantilever Tune** box: the *amplitude* curve in white, and the *phase* curve in yellow (Fig. 229-3). Microscopes without the Extender Electronics Module display only the amplitude curve.

- **Setting a Drive Frequency for Phase Detection**
  The **Drive frequency** should be set to the center of the cantilever resonance, as shown in Figure 13.3. This will be done automatically if using **AutoTune.**



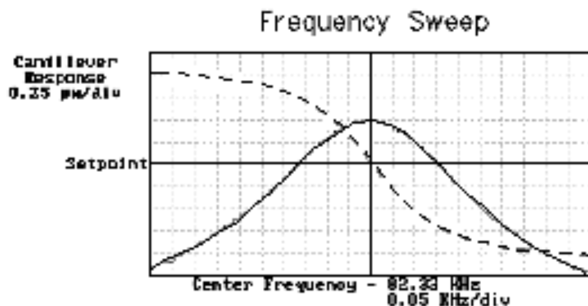
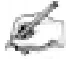


Figure 13.3. ***Cantilever Tune*** **for phase detection and frequency modulation.**

To correctly track the cantilever phase, the **Phase offset** parameter must be adjusted. This is automatically done in **AutoTune**; alternatively, **Zero Phase** can be selected from the menu bar above the **Cantilever Tune** frequency sweep window. The phase curve should appear as in Figure 13.3, 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 13.4.



Notes

The Extender electronics give a measure of the phase lag of the cantilever oscillation relative to the piezo drive. This measurement is monotonic versus frequency as is the true phase lag in degrees. The Extender measurement; however, has slightly different nonlinear characteristics *vs.* frequency. The measurement technique allows optimal signal-to-noise ratios; however, absolute values of phase data should be taken as approximate. Users requiring quantitative measures of force gradient are advised to use frequency modulation (See Section 13.2.2). Proceed to Step #4 below.

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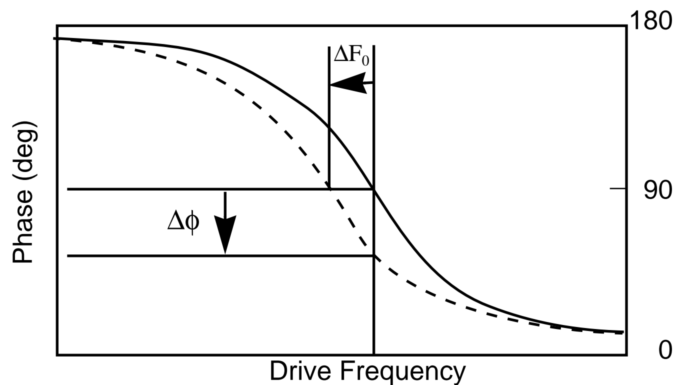
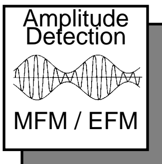


Figure 13.4. Shift in phase at fixed *Drive frequency*.



Setting a Drive Frequency for Amplitude Detection

Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 13.5. This can be done by using the **AutoTune** feature to first find the resonance peak, then using **Offset** under the **Cantilever Tune** menu bar to manually move the drive frequency to the side of the resonance. For

maximum sensitivity, set the **Drive frequency** to the steepest part of the resonance curve. As the tip oscillates above the sample, a gradient in the magnetic force will shift the resonance frequency f_0; (see Figure 13.6). Tracking the variations in oscillation amplitude while in LiftMode yields an image of the magnetic force gradients.

Proceed to Step #4 below.

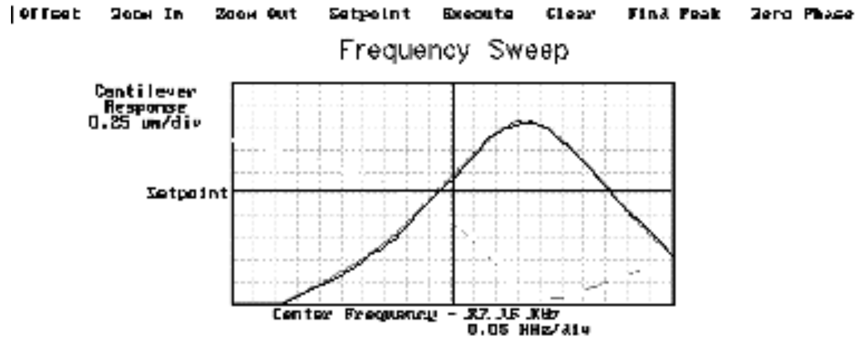


Figure 13.5. ***Cantilever Tune*** **for amplitude detection.**

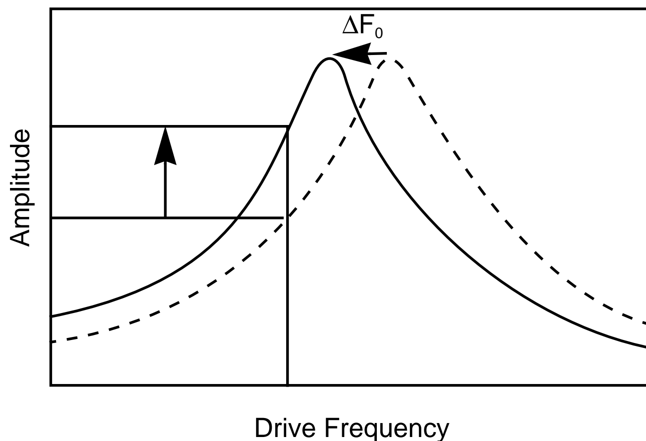


Figure 13.6. Shift in amplitude at fixed drive frequency.

4. Adjust the **Drive Amplitude** so that the RMS voltage response of the photodetector is approximately 2 volts. (Somewhat larger values may be beneficial if using amplitude detection.) This can be done with **Auto Tun**e by selecting an appropriate

Target Amplitude (in this case, 2V) before tuning, or by exiting **Cantilever Tune** and manually adjusting the **Drive Amplitude** parameter under **Feedback Controls**.

Quit Cantilever Tune and return to **Image Mode**. Under **Interleave Controls** set the **Lift start height** to **0 nm**, and **Lift scan height** to **100 nm**. (The lift height can be optimized later.) Set the remaining Interleave parameters (**Setpoint**, **Drive Amplitude, Drive frequency**, and gains) to the **Main Controls** values. This can be done by setting the flags left of the **Interleave Control** column to “off” (grayed bullets).

5. Under **Scan Controls**, set the **Scan size** to **5 µm** and **Scan rate** to **1–2 Hz**. For the left image, set the **Z range** to **75 nm** and the **Line direction** to **Retrace**. Engage the AFM and make the necessary adjustments to obtain a good topographical image while displaying height data. Use the maximum possible **Setpoint** to ensure that the tip is contacting the surface only lightly. The image should be similar to the topographic image shown on the left of Figure 13.7. The surface is fairly flat with lubrication nodules of various sizes. A good image of the nodules provides a check that the tip is sharp.

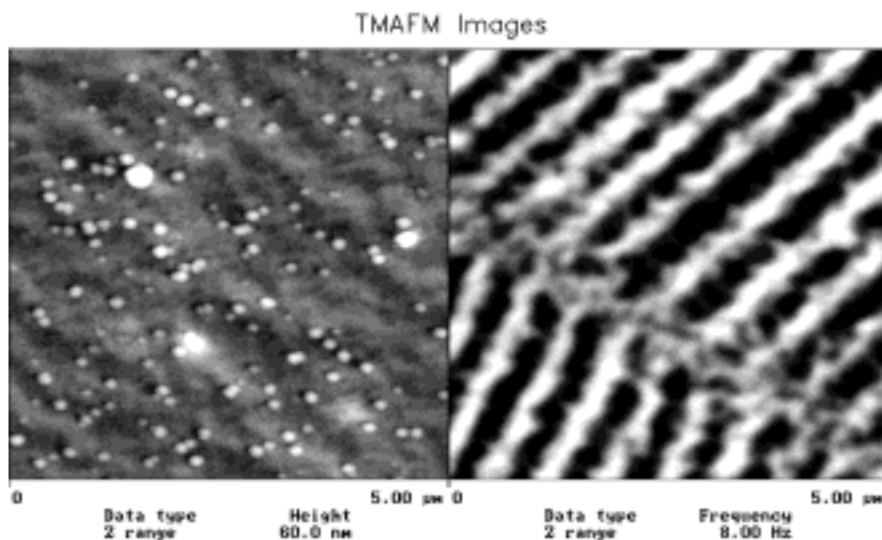
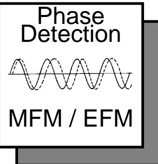


Figure 13.7. Topographic (left) and magnetic force gradient image (right) of metal evaporated tape at 100 nm *Lift scan height.*

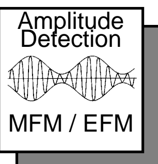
6. The MFM data can be shown on Channel 2; however, the parameter settings are different depending on whether Phase Detection or Amplitude Detection is being used



- **Phase Detection**
  Set the Channel 2 image **Data type** to **Phase**, **Z range** to **3 degrees**, and **Line direction** to **Retrace**.

Notes

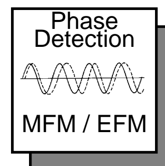
It is important that the **Scan direction** be set to **Retrace** for both the main and interleave scans. If instead it is set instead to **Trace**, a band may appear along the left side of the images due to the time taken for the tip to move between the surface and the lift scan height.



- **Amplitude Detection**
  Set the Channel 2 image **Data type** to **Amplitude**, **Z range** to **1 nm**, and **Line direction** to **Retrace**.

7. Change **Interleave mode** to **Enable** to invoke **LiftMode**. Set the Channel 2 **Scan line** to **Interleave** to display the interleaved data. (This can only be done after **Interleave mode** is **Enabled**.) A magnetic force gradient image similar to that shown on the right of Figure 13.7 should appear as the Channel 2 image. The alternating dark and light stripes represent the recorded magnetic information, and signify a varying resonant frequency and hence magnetic force gradient on the tip. As usual, keep the **Setpoint** as large as possible while consistent with a good image. Wider scans (> 25 µm) will reveal separate tracks in which the magnetic stripes are at different angles.

13.2.2. Frequency modulation.



With the Extender Electronics Module, it may be desirable to use frequency modulation. This activates a feedback loop which modulates the **Drive Frequency** to keep the cantilever's phase lag at 90 degrees relative to the drive, corresponding to resonance. The frequency **Data Type** displays the resulting shift in **Drive Frequency** in *Hz*, and gives the most direct, quantitative image of force gradients.

To enable frequency modulation, follow the procedure above for obtaining an MFM image with phase detection, but switch the Channel 2 image **Data type** to **Frequency**. Try a **Z range** (frequency shift) of approximately **10 Hz**. Select **Other Controls**, then adjust the frequency modulation gains. Setting both frequency modulation **Integral gain** and **Proportional gain** to **100** is a good starting point. As with the topography gains, the scan can be optimized by increasing the gains to maximize feedback response, but not so high that oscillation sets in. With 225 µm MFM cantilevers, gains are usually in the range of **100–250**.

13.3. Troubleshooting Suggestions

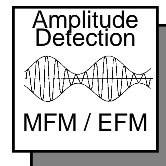
13.3.1. MFM image verification.

The procedure described above should produce a good magnetic force gradient image of the videotape sample. If there is a problem, check that the **Interleave Mode** is set to **Lift**, that **Interleave** is **Enabled** and that the **Scan Line** is set to **Interleave**. Check also that the **Interleave** values of **Drive Amplitude** and **Drive Frequency** are initially set equal to the main **Scan Controls** values.

13.3.2. 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 that the **Interleave Setpoint** has no physical effect in **Lift-Mode** since there is no surface feedback during the lift pass.

13.3.3. Optical interference.



When using Amplitude Detection, optical interference may sometimes appear in the Lift (magnetic force gradient) image when imaging highly reflective samples. Optical interference appears as evenly spaced, sometimes wavy lines with ~1–2 µm spacing superimposed on the lift image. This occurs when ambient laser light (i.e., light passing around or through the cantilever, then reflecting off the sample) interferes with laser light reflecting from the cantilever. Interference can be alleviated by moving the beam spot up the cantilever away from the tip; about 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.

Notes

- Optical interference is essentially eliminated by using phase detection or frequency modulation.)

13.4. Advanced Topics

13.4.1. *Lift scan height* and magnetic imaging resolution.

The most important parameter affecting imaging resolution is the **Lift scan height**. The range of 10–200 nm is most useful. In general, MFM resolution is roughly equal to the lift height: smaller **Lift Scan heights** give better resolution; conversely, magnetic features smaller than the **Lift Scan height** may not be resolved. The tip also experiences stronger fields close to the surface, giving improved signal-to-noise ratios.

For example, the image of metal-evaporated tape in Figure 13.7 has a resolution limited by the 100 nm **Lift Scan height**. To improve the resolution, try reducing the **Lift scan height** to ~ **25 nm**. Ensure that the tip does not strike the surface on the low point of its swing in the Lift image. Tip strikes appear as black or white spots, or even noisy, high-contrast streaks crossing the image. If the tip begins to strike the surface, reduce the **Interleave Drive Amplitude**. (In general, MFM tips are not damaged by intermittent tip strikes, except in extreme cases of very large amplitude and small lift heights.) An example of an image of the metal-evaporated tape taken with a **Lift scan height** of **30 nm** is shown in Figure 13.8. Note the fine magnetic structure that is not visible in Figure 13.7. When imaging a sample for the first time, begin with moderate **Lift scan heights** (**50 nm or greater**), then adjust downward. On relatively smooth samples (e.g., hard disks), lift heights down to 0 nm can be used, as long as the drive amplitude is adjusted accordingly. (Lift scan heights of 0 nm still correspond to a non-zero mean tip-sample distance. See the section on **Set-point** below.) It is usually not beneficial to use **Lift scan heights** much smaller than the surface roughness. Users are encouraged to experiment for the best images on their samples.

The ultimate resolution of MFM with the NanoScope III is near 20 nm. Resolution is affected by properties of the tip, including mechanical sharpness and magnetic

structure. When in good condition, NanoProbe magnetically coated tips routinely give 50 nm resolution, and many achieve 30 nm or better.

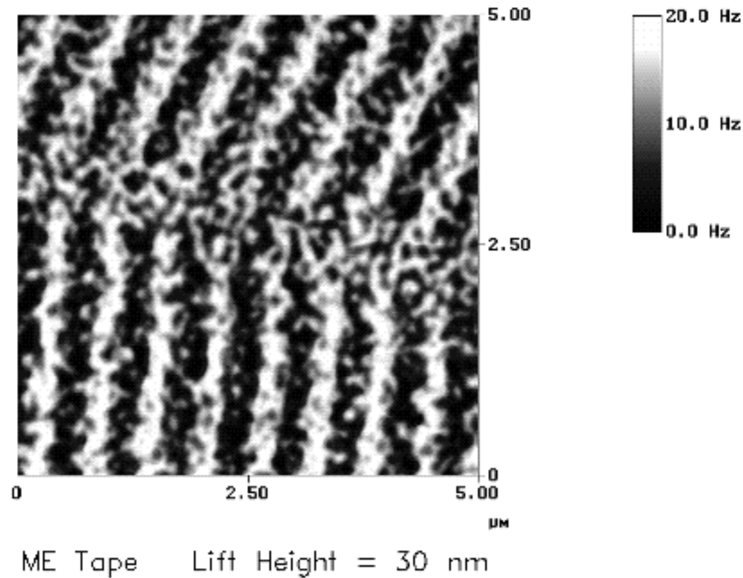


Figure 13.8. High-resolution magnetic force gradient image of metal evaporated tape at 30 nm *Lift scan height.*

13.4.2. Fine Tuning Interleave Controls

Certain scanning parameters found under **Interleave Controls** can be set to values in the Interleave (Lift) scan that differ from the values in the main scan. These parameters are enabled by clicking on the circular flag to the immediate left of the desired Interleave control, toggling its state from “off” (gray bullet) to “on” (green bullet). When the flag is set to “off”, the main scan control parameter setting takes precedence. When the flag is set to “on,” the displayed Interleave scan value is active, overriding the main scan value.

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

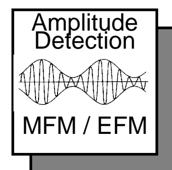
illustration, try setting the Interleave **Drive Amplitude** to **0**; the resulting phase image will be pure noise because measuring the phase of a non-oscillating cantilever is meaningless.

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.

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 en mode intercalé (**Interleave Mode**), vérifier que la valeur de tension appliquée à l'oscillateur piézo-électrique est inférieure à celle appliquée à l'oscillateur en mode imagerie (**Main Drive Amplitude).** Le non respect de cette procédure peut entraîner la destruction de la pointe.

WARNHINWEIS: 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.



- 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 13.6. With phase detection and frequency modulation, changes in amplitude produce no change in contrast, and results are thus more reproducible than with amplitude detection.

Notes

- On some microscope models, there is a lowpass filter in the scanning electronics that prevents fast switching of the **Drive Amplitude** between main and Interleave scanning. This can interfere with very fast rates (> a few Hz).

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 13.9). In

TappingMode, the average tip-sample distance h_T is equal to the oscillation amplitude, which is determined by the setpoint. Large variations in setpoint can thus change the total tip-sample distance in **Liftmode**, sometimes with visible results in the magnetic image. For this reason, reproducible results are most easily obtained by using consistent setpoints. Note that a lift scan height of 0 nm still gives a mean tip-sample distance of h_T in **LiftMode**.

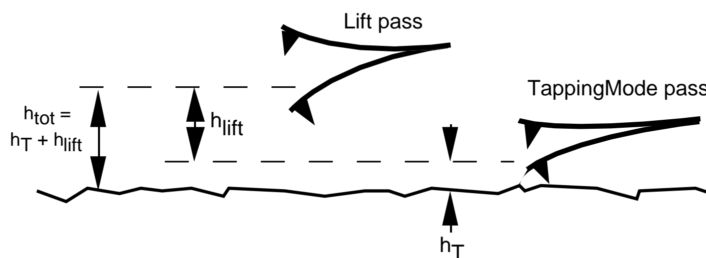


Figure 13.9. Tip heights and oscillation amplitudes in TappingMode and LiftMode.

The relationship between setpoint voltage and oscillation amplitude is known as the *sensitivity.* Its value can be determined with **Force Calibration**; see Chapter 11 in the product instruction manual. For 225 µm MFM cantilevers, the sensitivity is typically in the range 15-20 nm/V. A 1V **Setpoint** typically corresponds to h_T ~15-20 nm.

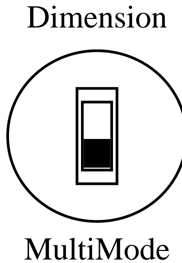
13.5. Installation of Extender Electronics Module

In the Spring of 1994, Digital Instruments began making available its Extender Electronics Module (sometimes referred to as “phase box” or “phase extender”) for customers wanting phase detection and frequency modulation MFM. Hardware changes, different for each system type, are required to retrofit Dimension Series systems or MultiMode systems originally shipped without the Extender Electronics module.

For MultiMode systems, the Extender is installed using a 37-to-37 pin ribbon cable between the NanoScope III or IIIA SPM controller and the Extender Electronics Module, and a 37-to-25 pin ribbon cable from the Extender to the microscope. The hardware change consists of replacing a circuit board in the base of the microscope (this may be done by either the customer or a factory representative). Detailed installation instructions are provided with the new hardware when shipped. For more information, contact Digital Instruments.

13.5.1. Setting the Extender Electronics for Dimension or MultiMode

The Extender Electronics box is equipped with a slider switch for switching internal electronics between Dimension-series and MultiMode SPM signals. This switch may be accessed through a hole on the underside of the box. In the drawing below, the switch is set to “**MultiMode**”.



For MultiMode SPMs, it should always be set to “**MultiMode**”. Use a pencil to access the switch through the hole.

WARNING: Do not insert a conducting object (e.g., screwdriver) into the Phase Extender box while it is engergized.

ATTENTION: Ne pas insérer d’ objet conducteur (par exemple: un tournevis) dans le boîtier d’extension de phase (Phase Extender Box) quand celui-ci est sous tension.

WARNUNG: Stecken Sie keine leitfähigen Teile (zum Beispiel Schraubenzieher) in die Phase Extender Box, während diese eingeschaltet ist.

13.5.2. Microscope parameter files.

There are two parameter (par) file contexts that are used with the Extender Box: Extended AFM and Extended STM. The former of these two contexts is used for the following operational modes: contact AFM, TappingMode AFM, LFM, MFM, force modulation, electric field measurement and others. The latter context is for use only with STM.

For each scanner intended to be used with the Extender, there must be a PAR file for the context required. For example, if there are three scanners, all intended for both STM and AFM use, a total of six PAR files are required.

Notes

- Due to the many Digital Instruments' microscope systems available, the variety of scanners, when the system was purchased, and the system's history of software updates, not all systems will have PAR files with the same name or naming scheme. You can determine the exact name of the PAR file to be modified by using the DOS EDIT file editor to list the contents of the file /SPM/SYSTEM.PAR. The name of the most recent PAR file in use will be on the line that starts with the words \Microscope file:.

CAUTION: When closing the System.par file after viewing it, if you are asked to SAVE the file or to SAVE CHANGES, be sure to say **No**.

ATTENTION: A la fermeture du fichier System.par, lorsque l'ordinateur demande SAUVEGARDER (SAVE) ou SAUVEGARDER LES CHANGEMENTS (SAVE CHANGES), répondre toujours **NON**.

WARNHINWEIS: Falls Sie sich mit einem Editor die Datei „system.par" anschauen und beim Verlassen des Editors gefragt werden, ob Sie die Datei speichern („SAVE") bzw. die Änderungen speichern wollen („SAVE CHANGES"), antworten Sie bitte mit **„Nein"** (**„No"**).

To modify the PAR files on a MultiMode or Dimension system, locate the proper PAR file in the /SPM/EQUIP directory on your computer. Use a text editor to modify the file. The text must be saved as plain ASCII (standard DOS text). DI recommends using the DOS EDIT file editor. (Microsoft Word® or other similar desktop-editors normally save files with embedded formatting commands causing the par files to be unreadable by the NanoScope software.) Using the EDIT program, modify the file to include a new line at the bottom of the file. For example:

If the line

```
\Is FM: No
```

is present in the file, then change the line to read:

```
\Is FM: Yes
```

If the line

```
\Is FM: No
```

is not present, add the line:

```
\Is FM: Yes
```

Then locate the line:

```
\In Polarity: Reverse
```

and change it to read:

```
\In Polarity: Forward
```

Then locate the line:

```
\In Polarity: AUX D: Reverse
```

Change it to read:

```
\In Polarity AUX D: Forward
```

Repeat the above procedure for each scanner (i.e. PAR file) in use. After the cables are connected, power up the NanoScope controller and start the microscope control program.

13.5.3. Important points.

1. Extender-compatible microscope electronics are required to permit operation of the phase detection extender option. Standard electronics on these microscopes require hardware upgrades. Consult your Digital Instruments sales representative for details.

2. Turn off the power to the NanoScope controller whenever connecting or disconnecting the Extender.

3. In **LiftMode**, the best performance is obtained if the RMS amplitude is kept below 7.0 volts, the limit of the RMS output's linear operation.

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Chapter 14 Electric Force (EFM) Imaging

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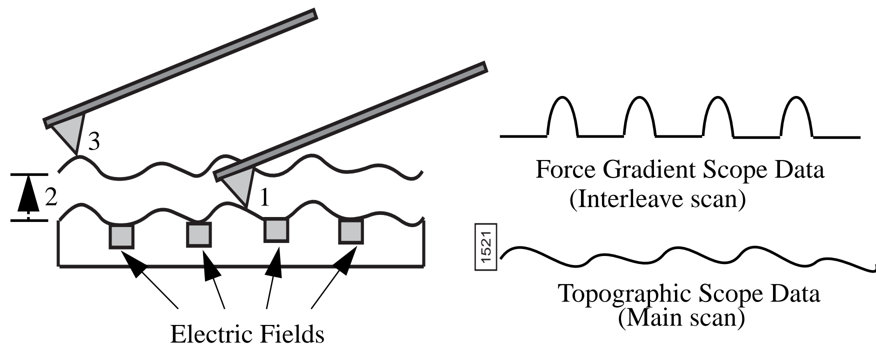
14.1. Electric Force Microscopy Overview

This chapter describes how to perform electric force microscopy (EFM) imaging on a MultiMode SPM system. Similar to magnetic force microscopy (MFM)—and sharing many of it's procedural techniques—this mode utilizes the Interleave and LiftMode procedures discussed in previous chapters. Please read those chapters prior to attempting electric force measurements.

All standard MultiMode SPMs are capable of EFM imaging using amplitude detection techniques. By adding an Extender™ electronics module (see Figure 14.2), the MultiMode SPM may also be used for frequency modulation or phase detection, giving improved results. Amplitude detection has largely been superseded by frequency modulation and phase detection. This hardware unit is required for surface potential imaging, and is strongly recommended for electric field gradient imaging.

Two types of electric force microscopy are available using the MultiMode SPM system: 1) **electric field gradient imaging**; and, 2) **surface potential imaging**. Each of the electric field measurement techniques are based on a two-pass **LiftMode** measurement. **LiftMode** allows the imaging of relatively weak but long-range magnetic and electrostatic interactions while minimizing the influence of topography (see Figure 14.1). Measurements are taken in two passes (each consisting of one trace and one retrace) across each scanline. First, topographical data is taken in TappingMode on one trace and retrace. The tip is then raised to the final scan height, and a second trace and retrace performed while maintaining a constant separation between the tip and local surface topography. Both methods of electric force measurement are explained in this chapter.



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.

Figure 14.1. EFM LiftMode principles.

Figure 14.2. Extender™ Electronics Module—required for frequency phase detection MFM and EFM.

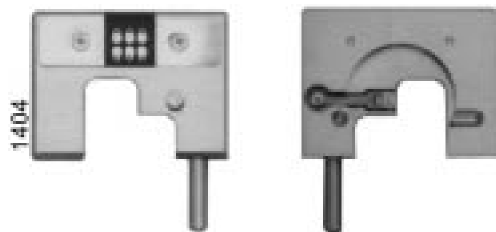


Figure 14.3. EFM probe tip holder, top and bottom view (left to right).

14.1.1. Electric field gradient imaging overview.

Electric field gradient imaging is a technique which measures variations in the electric field gradient above a sample. The sample may be conducting, nonconducting, or mixed. Since the electric field gradient is also shaped by the surface topography (e.g. sharp points on the surface concentrate the field gradient), large differences in topography can make it difficult to distinguish electric field variations. In general, the best samples for electric field gradient imaging are samples that have applied voltages of roughly 1 volt or more, samples with fairly smooth topography, or samples with trapped charge. Samples with insulating layers (passivation) on top of conducting regions may also be good candidates for electric field gradient imaging.

14.1.2. 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 ±10 volts, and operation is easiest for voltage ranges of ±5 volts. The noise level of this technique can be as low as a few mV. Samples may consist of conducting and nonconducting regions, but the conducting regions should not be passivated. Samples with regions of different materials will also show contrast due to contact potential differences. Semi-quantitative voltage measurements can be made on samples if the system is carefully calibrated on a sample at a known voltage. *This method requires the Extender Electronics Module and version 3.1 or later of the NanoScope software*.

14.2. Electric Field Gradient Detection—Theory

Electric field gradient imaging 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 14.3.

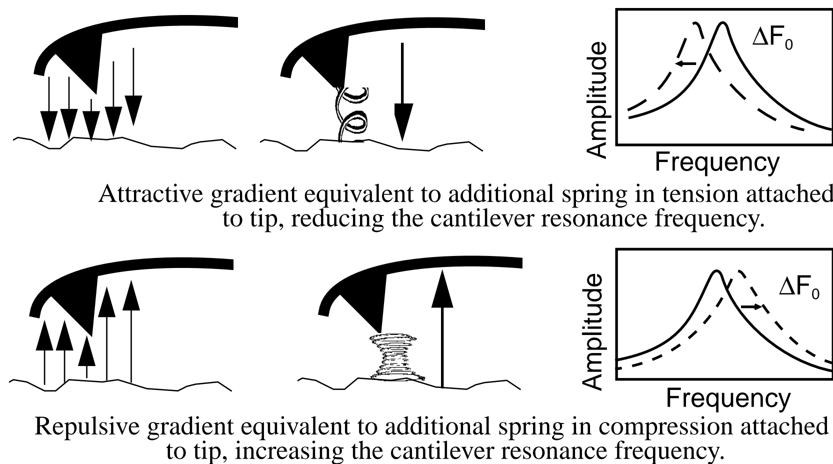


Figure 14.4. Comparison of attractive and repulsive forces to action of a taut spring attached to the tip.

Changes in cantilever resonant frequency can be detected in one of the following ways:

- Phase detection—*with Extender Electronics Module only.*
- Frequency modulation (FM)—*with Extender Electronics Module only.*
- Amplitude detection—**not recommended due to artifacts**.

All of the above methods rely on the change in resonant frequency of the cantilever due to vertical force gradients from the sample. Figure 14.4 shows a diagram of how the Extender Electronics module provides the 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 (voltage differences of 1.0 or more). For other samples having rough surface topography or small voltage variations, this technique may be undesirable because topographic features will appear in the **LiftMode** image.

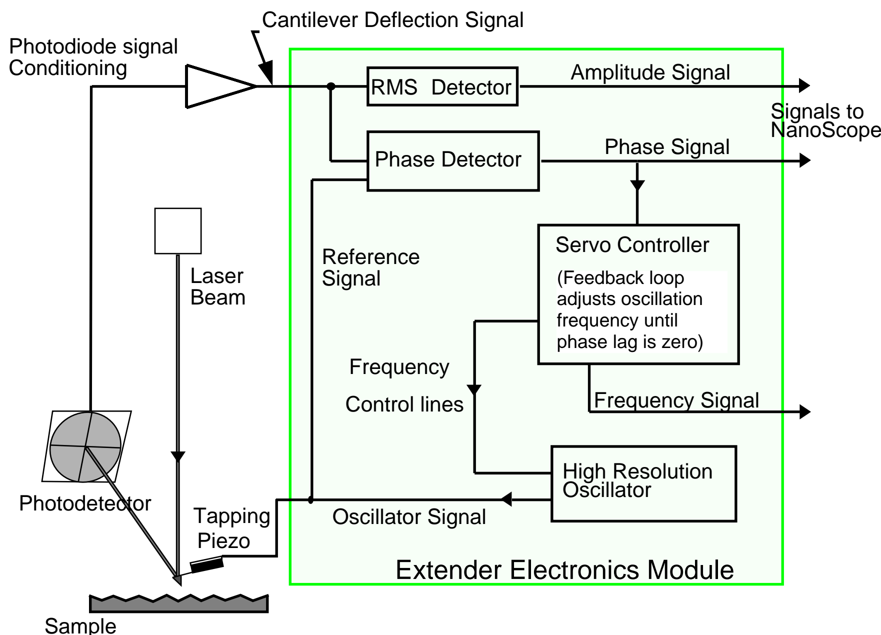


Figure 14.5. Diagram of Extender Electronics Module in phase and frequency measurement mode.

Topographic features appear in the **LiftMode** image because local force gradients are heavily influenced by surface structure. That is, sharp features on sample surfaces concentrate the local force gradient. This happens on all roughness scales, so the local force gradient will also vary in much the same way as does the surface topography. Thus, electric field **LiftMode** images measured by amplitude, phase or frequency detection often show contrast that is very similar to the surface topography. Samples with rough surface topography or with smaller potential variations are more successfully imaged by the surface potential measurement method described below.

Notes

In most cases, it is necessary to apply a voltage across 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 the application of voltage.

14.3. Electric Field Gradient Detection—Preparation

This section explains how to conduct electric field gradient imaging by applying a voltage to the tip or sample to generate electric fields. If the sample being imaged 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 14.4.

Before attempting to reconfigure the jumpers, carefully read the following Jumper Configuration sections.

When it is necessary to apply voltage to the tip or sample, minor changes must be made to the jumpers in the microscope's baseplate and the toggle switches on the Extender Electronics Module (if equipped). Original jumper configurations and jumper changes are dependent on the microscope being used and the measurements desired. Section 14.3.1 provides jumper configuration instructions for basic microscope models operating *without* the Extender Electronics Module (*See:* **Jumper Configuration Sections A, B, C, D.**). Section 14.3.2 provides jumper configuration instructions for basic microscope models operating *with* the Extender Electronics Module (*See:* **Jumper Configuration Sections E, F, G, H.**)

The location and orientation of the jumpers in the baseplate of the MultiMode is shown below in Figure 14.5. To change the jumpers, it should not be necessary to remove the baseplate; they can easily be changed through the rectangular opening in the bottom of the baseplate using a pair of non-conducting tweezers. For non-

EFM applications and surface potential operation, jumpers are usually left in their original positions or returned to their original positions.

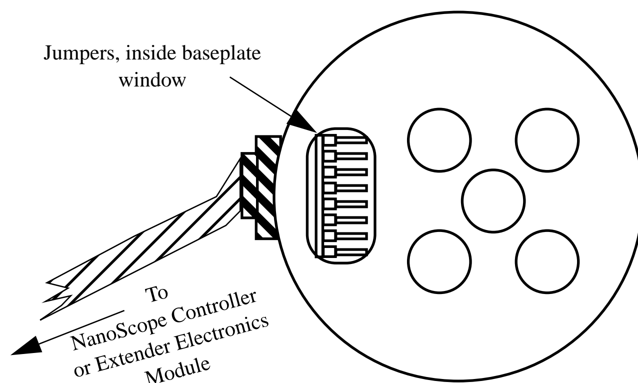


Figure 14.6. Diagram of MultiMode baseplate showing location and orientation of jumpers.

1. Carefully examine the following figures and identify which jumper configuration, if any, is appropriate for your application.

2. Power down the NanoScope III controller and turn off all peripherals. Unplug the NanoScope III power cable from the microscope's controller electronics box.

3.Ensure the tip is not engaged on the sample. Disconnect and remove the microscope head and the scanner.

4. Carefully tilt the MultiMode so that the baseplate is oriented as shown in Figure 14.5. Locate header and jumpers. As shipped from the factory, jumpers on systems *without* the Extender Electronics Module should appear as shown in Figure 14.6, whereas jumper systems *with* the Extender Electronics option should appear as in Figure 14.11.If the microscope is to be used for EFM imaging in cases where voltage is applied to the tip or sample, it is necessary to change the jumpers.

5. Depending upon whether voltage is to be applied to the tip or sample, and the amount of voltage to be used, reconfigure jumpers on the baseplate header using the jumper configuration as shown in the appropriate sections below.

6. After the jumpers are correctly configured, apply power to the microscope and all peripherals. Boot the computer and start the NanoScope software.

7. If the jumpers are configured to use an external voltage source, click on the **Microscope / Calibrate / Detector** option to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Allow**.

8. Imaging can now be accomplished using the procedures in Section 14.4.

14.3.1. Jumper configurations for systems *without* the Extender Electronics Module

As shipped from the factory, the jumper configuration on a MultiMode SPM without the Extender Electronics Module should appear as shown in Figure 14.6 below.

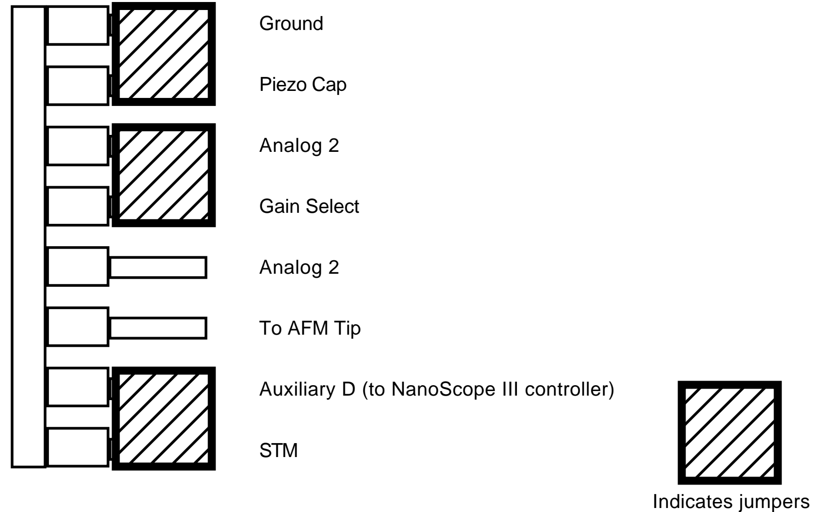


Figure 14.7. Normal jumper configuration for systems *without* the Extender Electronics Module as shipped from factory.

A: Field Gradient Imaging—Voltage Applied to the Tip

The jumper configuration in Figure 14.7 connects the Analog 2 signal from the NanoScope III controller (± 12 VDC range) to the tip.

Notes

In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

Enabling the Analog 2 Voltage Line:

The "Analog 2" voltage line is normally used by the NanoScope to control the attenuation (1x or 8x) of the main feedback signal. This application of EFM imaging uses the Analog 2 signal for EFM data. Therefore, input attenuation must be disabled for the duration of the EFM experiments. To do this, click on the **Microscope / Calibrate / Detector** option to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Disallow**. Return to the main **Feedback Controls** panel; the **Analog 2** field should now be enabled. This signifies that voltage is now being supplied via the "Analog 2" pin located on the baseplate header.

Remember to restore (**Allow in attenuation**) upon completion of EFM imaging.

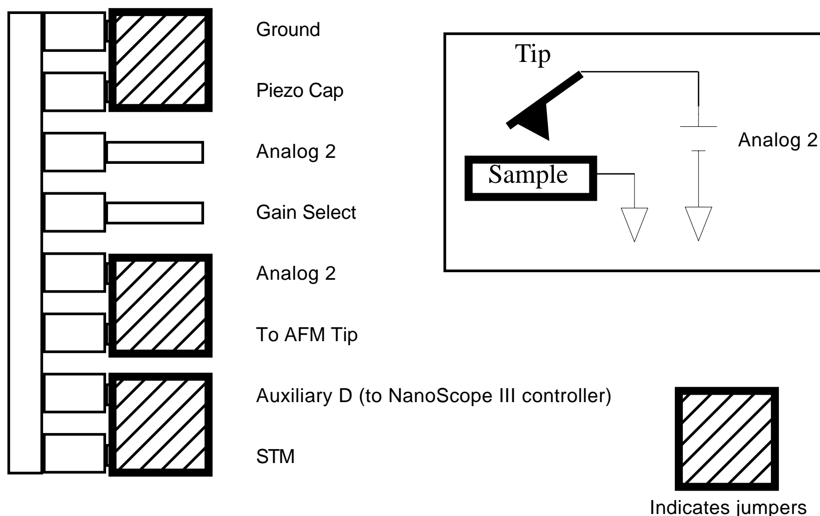


Figure 14.8. Jumper configuration for application of voltage to tip (for systems *without* the Extender Electronics Module).

B: Field Gradient Imaging—Voltage Applied to the Sample

The jumper configuration in Figure 14.8 connects the Analog 2 signal from the NanoScope III controller (± 12 VDC range) to the sample chuck.

Enabling the Analog 2 Voltage Line

The “Analog 2” voltage line is normally used by the NanoScope to control the attenuation (1x or 8x) of the main feedback signal. This application of EFM imaging uses the Analog 2 signal for EFM data. Therefore, input attenuation must be disabled for the duration of the EFM experiments. To do this, click on the **Microscope / Calibrate / Detector** option to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Disallow**. Return to the main **Feedback Controls** panel; the **Analog 2** field should now be enabled. This signifies that voltage is now being supplied via the “Analog 2” pin located on the MultiMode baseplate header.

Remember to restore (**Allow in attenuation**) upon completion of EFM imaging.

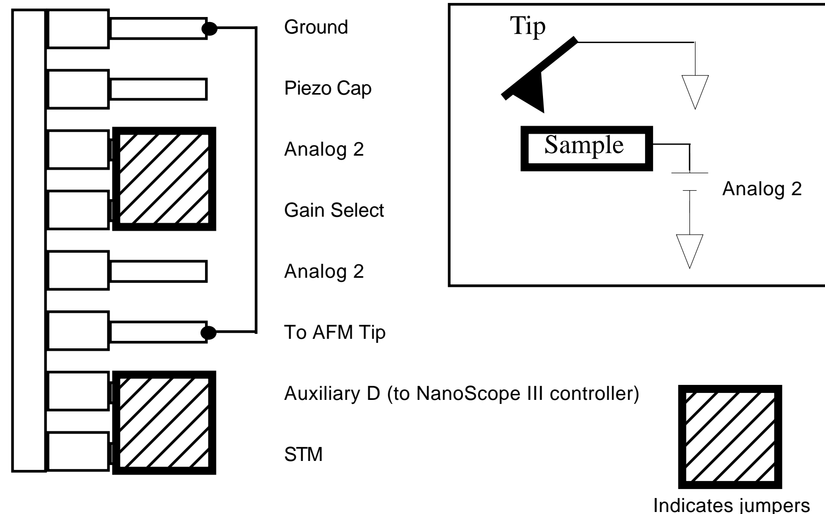


Figure 14.9. Jumper configuration for application of voltage to sample (for systems without the Extender Electronics Module).

C: Field Gradient Imaging—External Voltage Source Applied to the Tip

In some cases, it may be advantageous to use voltages greater than 12 VDC, or to use a pulsed power supply. If an external source of voltage is to be applied to the tip, configure jumpers as shown in Figure 14.9.

Notes

- In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

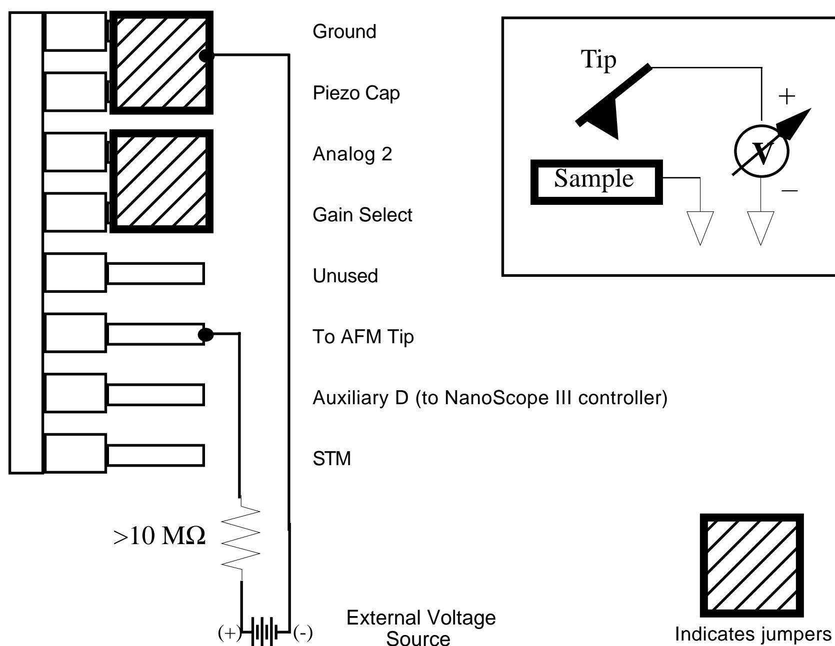


Figure 14.10. Jumper configuration for applying external voltage to tip (for systems *without* the Extender Electronics Module).

A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to pins on the header using soldered, push-on connectors. *Do not* solder leads directly to the header pins, as the heat may cause damage and/or make jumpering the pins difficult. The connection to ground can be made to the AFM chassis or externally.

D: Field Gradient Imaging—<u>External Voltage Source</u> Applied to the <u>Sample</u>

In some cases, it may be advantageous to use voltages greater than 12 VDC, or to use a pulsed power supply. If an external source of voltage is to be applied to the sample, configure jumpers as shown in Figure 14.10.

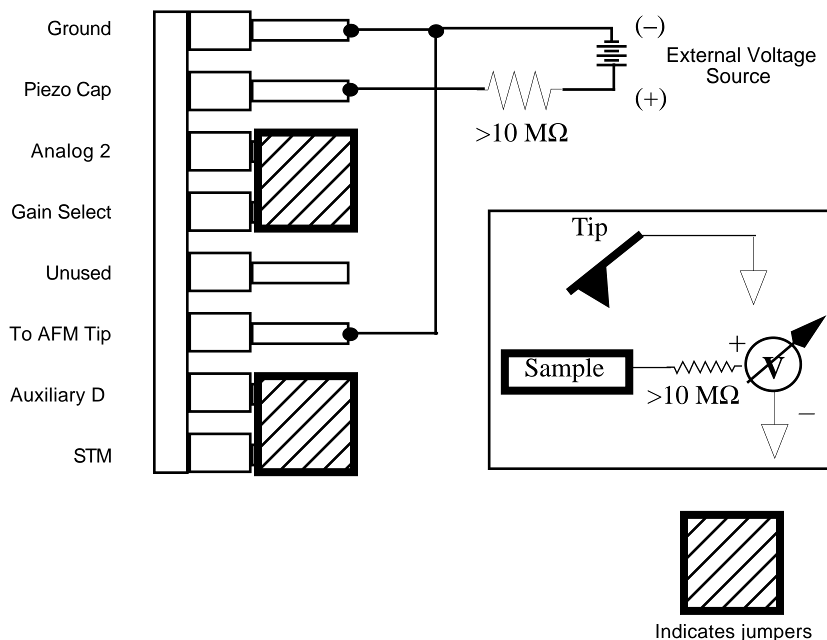


Figure 14.11. Jumper configuration for applying external voltage to sample (for systems *without* the Extender Electronics Module).

A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to pins on the header using soldered, push-on connectors. *Do not* solder leads directly to the header pins, as the heat may cause damage and/or make jumpering the pins difficult.

14.3.2. Jumper Configurations For Microscopes *With* the Extender Electronics Module

REMINDER: Power down the microscope and turn off all peripherals. Unplug the NanoScope III control and power cables from the system before attempting to adjust jumper configurations.

As shipped from the factory, systems *with* the Extender Electronics option, should have an original baseplate jumper configuration as shown in Figure 14.11.

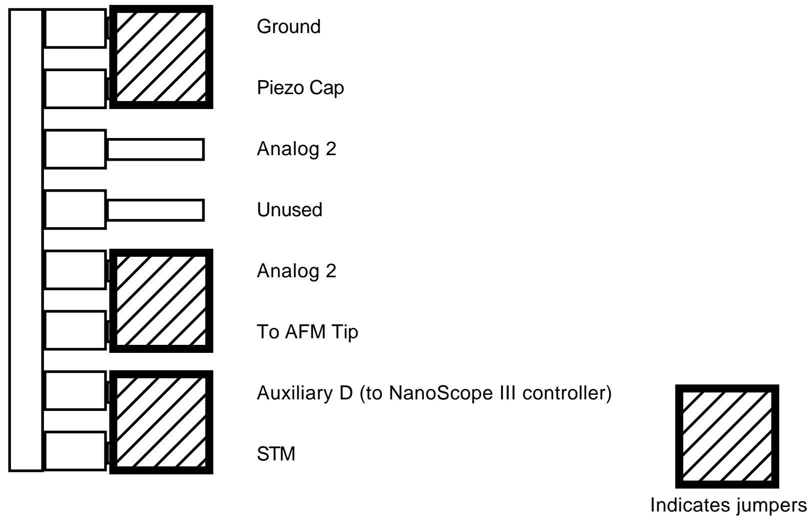


Figure 14.12. Normal jumper configuration as shipped from factory for MultiMode SPM *with* Extender Electronics Module installed.

E: Field Gradient Imaging—Voltage Applied to the Tip

Notice that the jumper configuration in Figure 14.12 connects the Analog 2 signal from the NanoScope III controller (± 12 VDC range) to the tip, and is **exactly the same** as the jumper configuration shown in Figure 14.11, the standard configuration as shipped from the factory.

Notes

- In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

Enabling the Analog 2 Voltage Line

The "Analog 2" voltage line is normally used by the NanoScope to control the attenuation (1x or 8x) of the main feedback signal. This application of EFM imaging uses the Analog 2 signal for EFM data. Therefore, input attenuation must be disabled for the duration of the EFM experiments. To do this, click on the **Microscope / Calibrate / Detector** option to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Disallow**. Return to the main **Feedback Controls** panel; the **Analog 2** field should now be enabled. This signifies that voltage is now being supplied via the "Analog 2" pin located on the MultiMode baseplate header.

Remember to restore (**Allow in attenuation**) upon completion of EFM imaging.

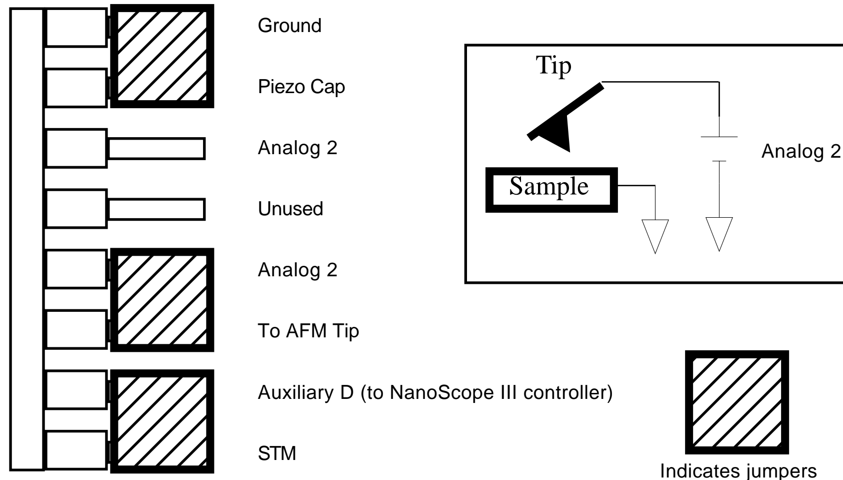


Figure 14.13. Jumper configuration for application of voltage to tip—standard configuration of systems *with* the Extender Electronics Module as shipped from factory.

F: Field Gradient Imaging—Voltage Applied to the Sample

The jumper configuration in Figure 14.13 connects the Analog 2 signal from the NanoScope III controller (± 12 VDC range) to the sample.

Enabling the Analog 2 Voltage Line

The “Analog 2” voltage line is normally used by the NanoScope to control the attenuation (1x or 8x) of the main feedback signal. This application of EFM imaging uses the Analog 2 signal for EFM data. Therefore, input attenuation must be disabled for the duration of the EFM experiments. To do this, click on the **Microscope / Calibrate / Detector** option to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Disallow**. Return to the main **Feedback Controls** panel; the **Analog 2** field should now be enabled. This signifies that voltage is now being supplied via the “Analog 2” pin located on the baseplate header.

Remember to restore (**Allow in attenuation**) upon completion of EFM imaging.

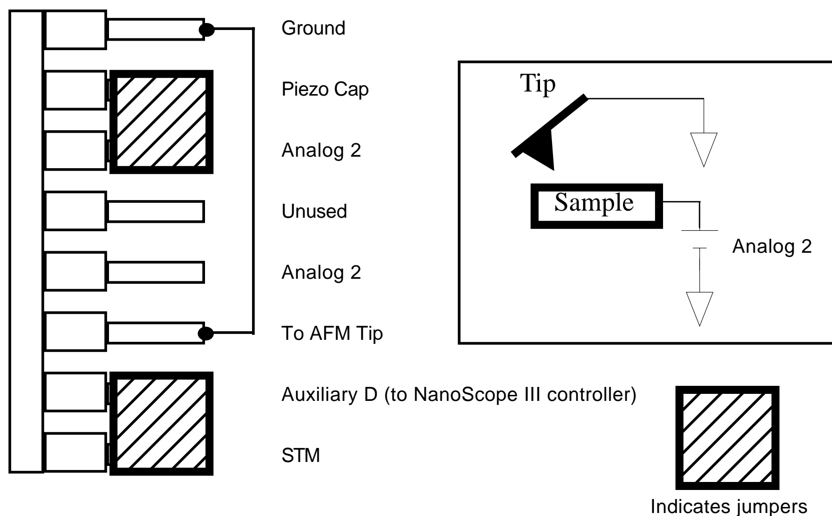


Figure 14.14. Jumper configuration for application of voltage to sample (for systems *with* the Extender Electronics Module).

G: Field Gradient Imaging—External Voltage Source Applied to the Tip

In some cases, it may be advantageous to use voltages greater than 12 VDC, or to use a pulsed power supply. If an external source of voltage is to be applied to the tip, configure jumpers as shown in Figure 14.14.

Notes

- In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

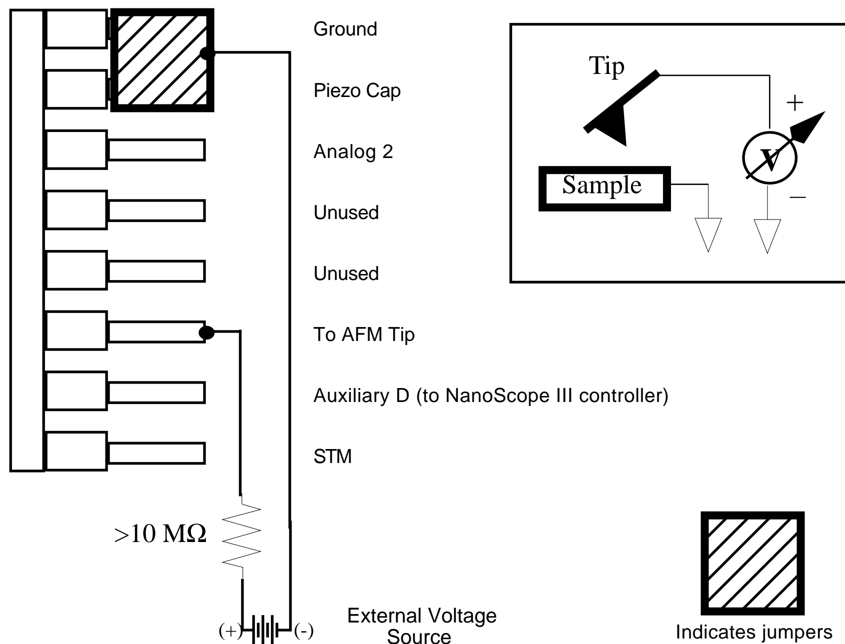


Figure 14.15. Jumper configuration for applying external voltage to tip (for systems *with* the Extender Electronics Module).

A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to pins on the header using soldered, push-on connectors. *Do not* solder leads directly to the header pins, as the heat may cause damage and/or make jumpering the pins difficult.

H: Field Gradient Imaging—External Voltage Source Applied to the Sample

In some cases, it may be advantageous to utilize voltages greater than 12 VDC, or to utilize a pulsed power supply. If an external source of voltage is to be applied to the sample, configure jumpers as shown in Figure 14.15.

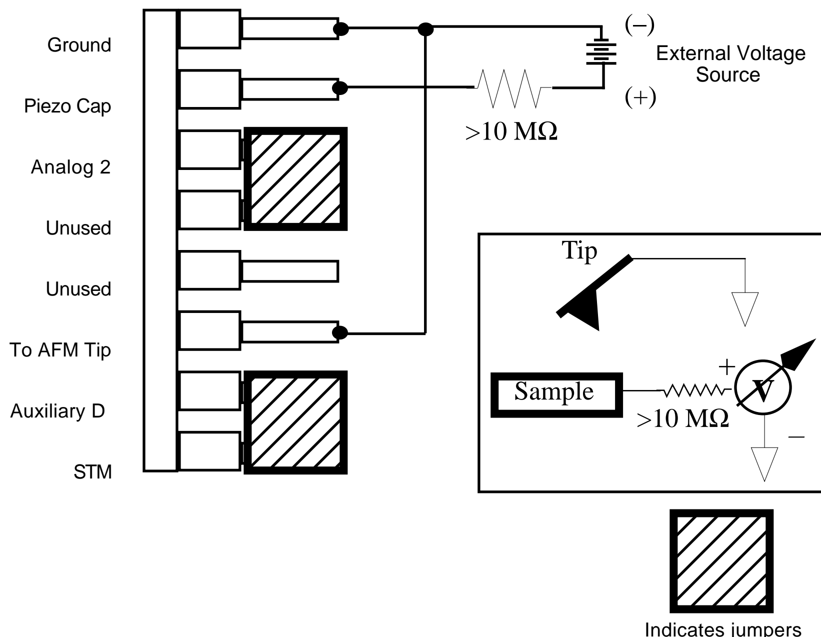
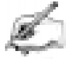


Figure 14.16. Jumper configuration for applying external voltage to sample (for systems *with* the Extender Electronics Module).

A current-limiting resistor (e.g., 10-100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to pins on the header using soldered, push-on connectors. *Do not* solder leads directly to the header pins, as the heat may cause damage and/or make jumpering the pins difficult.

14.4. Electric Field Gradient Detection—Procedures



Notes

- Amplitude detection is the only procedure described here that can be done without the Extender Electronics Module; however, this method is no longer recommended (see Section 14.4.2).

1. Locate the two toggle switches on the backside of the Extender Electronics box (Figure 14.16), then verify that they are toggled as shown in Table 14.1.

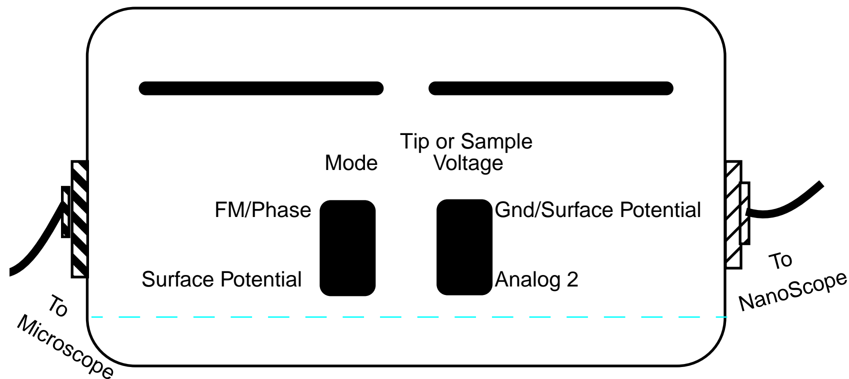


Figure 14.17. Toggle switches on back of Extender Electronics Module.

	Mode		Tip or Sample Voltage	
	FM/Phase	Surface Potential	GND/Surface Potential	Analog 2
TappingMode Contact AFM MFM	√		√	
Surface Potential		√	√	
Apply voltage to tip or sample (Use for electric field gradient imaging; tunneling AFM)	√			√

Table 1.1. Extender Electronics Module toggle switch settings.

Notes

- The toggle switch combination of Surface Potential = ON and Analog2 = ON is **not recommended** and can produce erratic, undefined results.

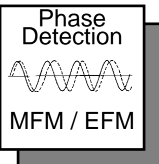
2. Electrically connect the sample by mounting it to a standard sample disk or stage using conducting epoxy or silver paint. Ensure the connection is good; a poor connection will introduce noise.

3. Mount a metal-coated NanoProbe cantilever into the electric field cantilever holder. MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz) usually work well. It is also possible to deposit custom coatings on model FESP silicon TappingMode cantilevers. Make sure that any deposited metal you use adheres strongly to the silicon cantilever.

4. Set up the AFM as usual for TappingMode operation. In the **Channel** panels, be certain all **Highpass** and **Lowpass** filters are **Off**. Set the **Rounding** parameter in the **Microscope / Calibrate / Scanner** window to zero (0.00).

5. Select **View / Cantilever Tune.**

6. Follow the instructions below for the type of electric force imaging desired, **Phase Detection** or **Amplitude Detection**.



Phase Detection

Phase Detection is only available when the Extender Electronics Module has been correctly configured into the system.

- In the **Cantilever Tune** window, set **Start frequency** and **End frequency** to appropriate values for your cantilever (e.g., for 225 µm MFM cantilevers, set **Start frequency** to 40 kHz and **End frequency** to 100 kHz). Select **Autotune**.

- Two curves appear on the Cantilever Tune graph: the *amplitude* curve in white, and the *phase* curve in yellow. (In Figure 14. 17, the *phase* curve is the dashed line and the *amplitude* curve is the solid line.).

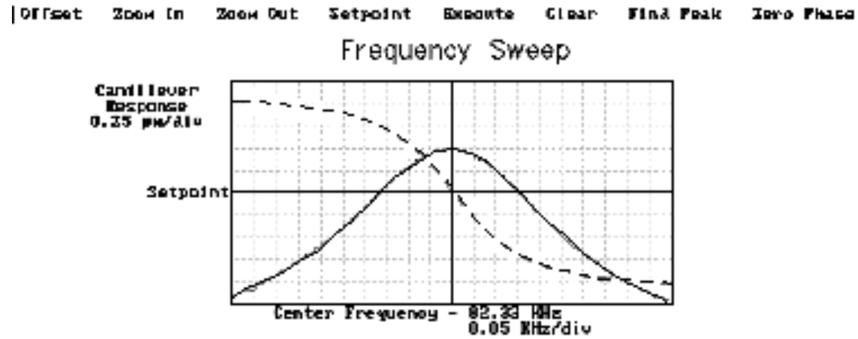


Figure 14.18. Phase detection *Cantilever Tune* (Extender Electronics Module installed).

The phase should decrease with increasing frequency and cross the center line (90° point) at the peak frequency. The phase curve then correctly reflects the phase lag between the drive voltage and the cantilever response. Again, gradients in the electric force will cause a shift ΔF_0 in the resonance frequency. In this case, resonance shifts give rise to phase shifts $\Delta\phi$ which can then give an image of the electric force gradients; see Figure 14.18.

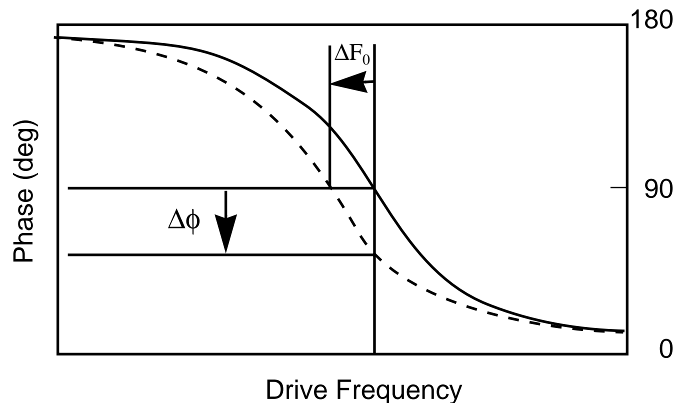
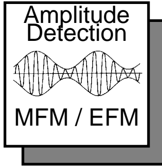


Figure 14.19. Shift in phase at fixed *Drive frequency.*

- Under Interleave Controls set the **Lift start height** to **0 nm**, and **Lift scan height** to **100 nm**. (The lift height can later be optimized.) Set the remaining Interleave parameters (**Setpoint**, **Drive amplitude, Drive frequency**, and gains) to the main Feedback Controls values. This can be done by setting the flags (left of the Interleave Control column) to “off” (grayed bullets).
- **Quit Auto Tune** and return to **Image Mode**
- Engage the AFM and make the necessary adjustments to obtain a good topography (**Height**) image on Channel 2.
- Select the **Interleave Controls** panel. Set the **Setpoint**, **Drive amplitude**, **Drive frequency** to Main (“off” grayed bullets). Verify that **Interleave scan** is set to **Lift**.
- Choose a **Lift scan height** of **100 nm**. This will be optimized later. Set the Channel 1 image **Data type** to **Phase** and choose **Retrace** for the scan **Line direction** on both Channel 1 and 2 images.
- Switch **Interleave mode** to **Enable** to start **LiftMode**. Set the Channel 1 **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 left image and topography in the right image.
- Optimize the **Lift height**. For high-resolution, make the **Lift scan height** as small as possible without crashing the tip into the surface. If the tip crashes into the surface it creates bright or dark streaks across the image. Also, if the **Lift scan height** is set extremely low, the tip may continuously “tap” on the surface during the **LiftMode** scan. Check this by toggling between the **Interleave** and main scan lines for the phase image. The two images will look very similar if the tip is continuously tapping on the surface during the **LiftMode** scan. In this case, increase the **Lift scan height** until the **Interleave** scan image abruptly changes, indicating that it is now oscillating above the surface and not continuously tapping.
- Adjust the sample or tip voltage to confirm that contrast is due to electrical force gradients. On very rough samples, contrast in **LiftMode** images may be from air damping between the tip and surface. It is often useful to look at the phase data in **Scope Mode** while adjusting the tip or sample voltage up and down. Contrast due to electrical force gradients should increase or decrease as the tip-sample voltage is changed.
- For more quantitative results, switch the to the frequency **Data Type** for Channel 1. This technique provides a direct measure of the change in resonant frequency felt by the cantilever. It may be necessary to optimize the FM (frequency modulation) gain to properly track the shifts in resonant frequency. This is described in detail in Chapter 13 of the appropriate product instruction manual.

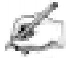
14.4.1. Amplitude Detection



14.4.1.1. With Extender Electronics Module.

To set up for **Amplitude Detection** field gradient imaging on systems *with* the Extender module installed, follow the instructions in *Section 14.4.1, Phase Detection*, with the exception that the Channel 1 **Data Type** should be set to **Amplitude**.)

14.4.1.2. Without Extender Electronics Module.



Notes

- This imaging method, although described here, is not recommended *without* the Extender Electronics module due to the presence of artifacts.

Amplitude Detection, unlike **Phase Detection**, is available *with* or *without* the optional Extender Electronics Module. This section describes the differences in software set up and imaging for EFM systems *without* the Extender module. When EFM imaging *without* the Extender module, changes in the cantilever amplitude provide an *indirect* measure of shifts in the cantilever resonance frequency as shown in Figure 14.19.

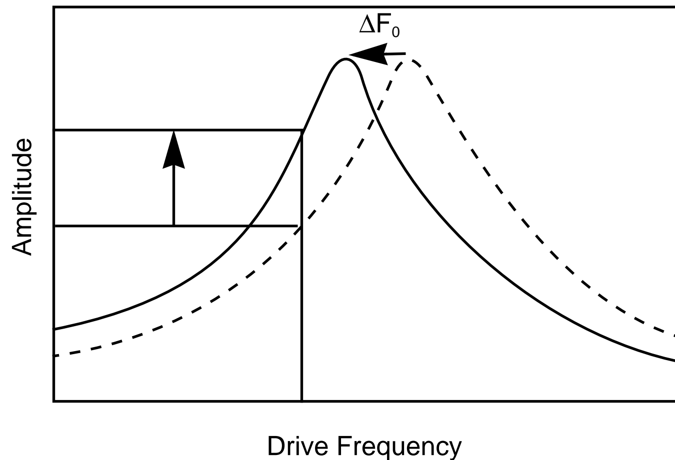


Figure 14.20. Shift in amplitude at fixed *Drive Frequency* (Extender Electronics Module not installed).

- Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 14.20 below.

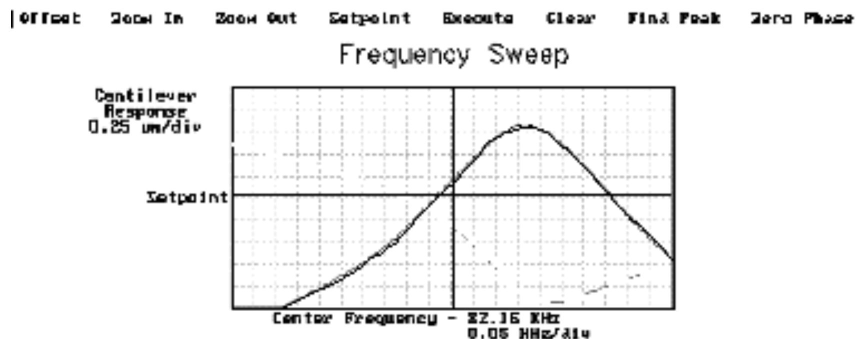


Figure 14.21. Amplitude detection *Cantilever Tune* (Extender Electronics Module *not* Installed).

- For maximum sensitivity, set the **Drive frequency** to the steepest part of the resonance curve. As the tip oscillates above the sample, a gradient in the magnetic force will shift the resonance frequency F_0; (see Figure 14.19).

  Tracking the variations in oscillation amplitude while in **LiftMode** yields an image of the electric force gradients. Either side of the resonance may be used, though we have obtained slightly better results on the low side, as shown in Figure 14.19.
- When using **Amplitude Detection**, optical interference may sometimes appear in the lift (magnetic 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 the cantilever away from the tip; about one-third of the cantilever length from the tip usually works well. On the MultiMode head, the adjustment can be refined by carefully moving the beam spot laterally on the cantilever while scanning until interference fringes are minimized.

Notes

- Optical interference is essentially eliminated by using phase detection or frequency modulation, available only with the Extender Electronics Module.

14.5. Surface Potential Detection—Theory

Notes

- Surface potential detection EFM is only possible using the Extender Electronics Module. This section does not apply to microscopes which are not equipped with the Extender Electronics Module.

The Extender Electronics Module allows measurement of local sample surface potential. This is similar to techniques called Scanning Maxwell Stress Microscopy and Kelvin Probe Microscopy. Surface potential detection is a two-pass system where the surface topography is obtained in the first pass and the surface potential is measured on the second pass. 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 14.21. On the first pass, the sample topography is measured by standard TappingMode. In TappingMode the cantilever is physically vibrated near its resonant frequency by a small piezoelectric element. On the second pass, the piezo that normally vibrates the cantilever is turned off. Instead, to measure the surface potential, an oscillating voltage $V_{ac}\cos\omega t$ is applied directly to the cantilever tip. This creates an oscillating electrostatic force at the frequency ω on the cantilever. The oscillating force has the following amplitude:

$$F = \frac{dC}{dz}V_{dc}V_{ac}$$

where $\frac{dC}{dz}$ is the vertical derivative of the tip/sample capacitance.

$V_{dc} = V_{tip} - V_{sample}$, the dc voltage difference between the tip and the sample,

and V_{ac} is the amplitude of the oscillating voltage applied to the cantilever tip.

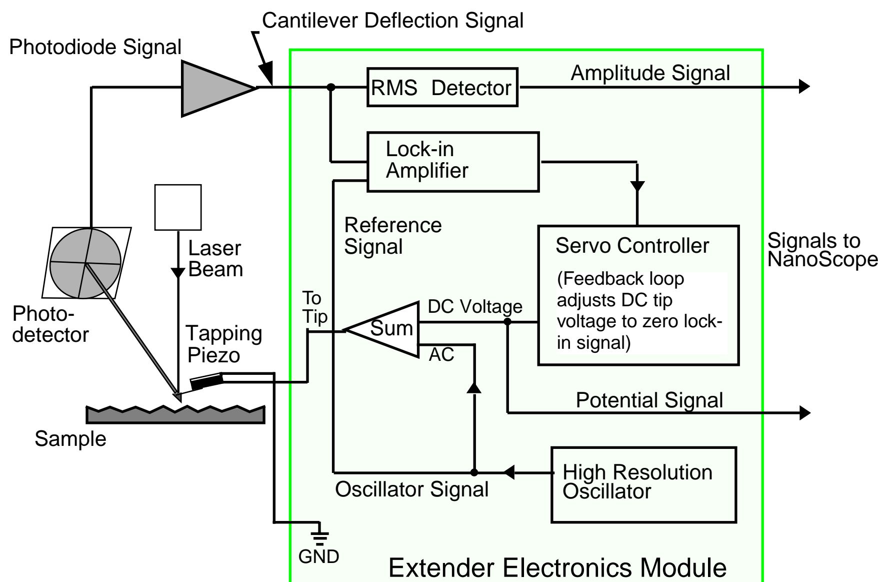


Figure 14.22. Simplified block diagram of extender electronics module in surface potential mode.

The key here is that the force on the cantilever depends on the product of the ac drive voltage and the dc voltage difference between the tip and the sample. And, when the tip and sample are at the same dc voltage (V_{dc}=0), the cantilever will feel no oscillating force. The Extender Electronics Module uses this fact to determine the effective surface potential on the sample, V_{sample}. The Extender determines the local surface potential by adjusting the dc voltage on the tip, V_{tip}, until the oscillation amplitude becomes zero. At this point the tip voltage will be the same as the unknown surface potential. The voltage applied to the cantilever tip V_{tip} is recorded by the NanoScope III to construct a voltage map of the surface.

14.6. Surface Potential Detection—Preparation

It is often desirable to apply a voltage to one or more areas of a sample. This may be done in two ways: by connecting a voltage to the sample mounting chuck, or by making direct contact to the sample. In both cases, jumper configurations in the bottom of the microscope must be changed to match the environment desired.

Notes

- In addition to any reconfigured jumpers, remember to connect the common or negative terminal of an external power supply to the MultiMode ground or the AFM chassis.

1. Power down the NanoScope controller and turn off all peripherals. Unplug the NanoScope power cable from the microscope's electronic box.

2. Locate the jumpers on the electronic board, visible in a "window" on the bottom of the MultiMode per Figure 14.5. As shipped from the factory, the baseplate jumpers should appear as shown in Figure 14.22.

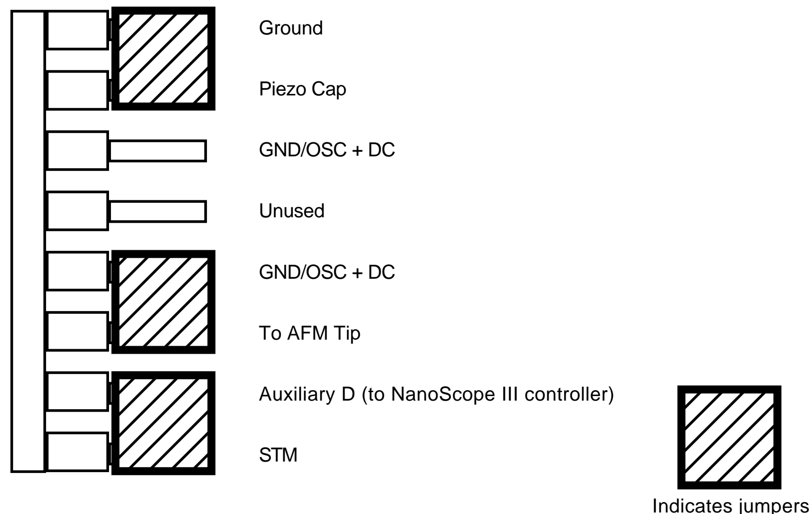


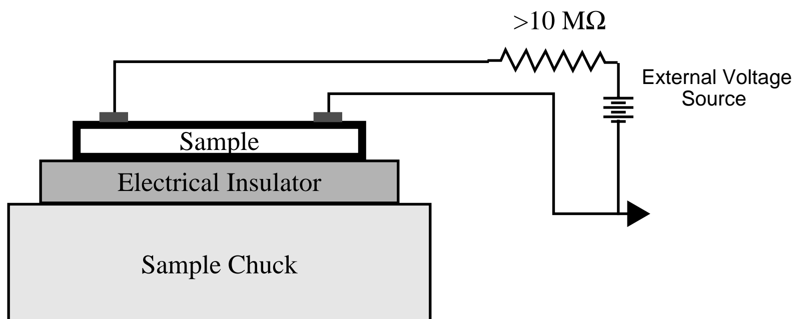
Figure 14.23. Normal jumper configuration as shipped from factory for MultiMode SPM with Extender Electronics Module installed. Sample is held at ground.

3. Depending upon whether voltage is to be applied to the sample directly or indirectly, reconfigure jumpers if indicated.

14.6.1. Applying Voltage to the Sample Directly

When voltage is applied directly to the sample, there is no need to reconfigure the jumpers. They should remain jumpered as shipped from the factory (Figure 14.22), and the sample should be electrically insulated from the chuck.

Connect the external voltage source directly to the sample by attaching fine gauge wire to appropriate contacts (e.g., on integrated circuits connect electrical leads directly to pads). For normal operation, the sample chuck is held at ground; be certain to carefully any electrical connections from the sample chuck.



14.6.2. Applying Voltage to the Sample Through Piezo Cap

When voltage is to be applied to the sample via the sample piezo cap for indirect surface potential imaging, configure the jumpers as shown in Figure 14.23.

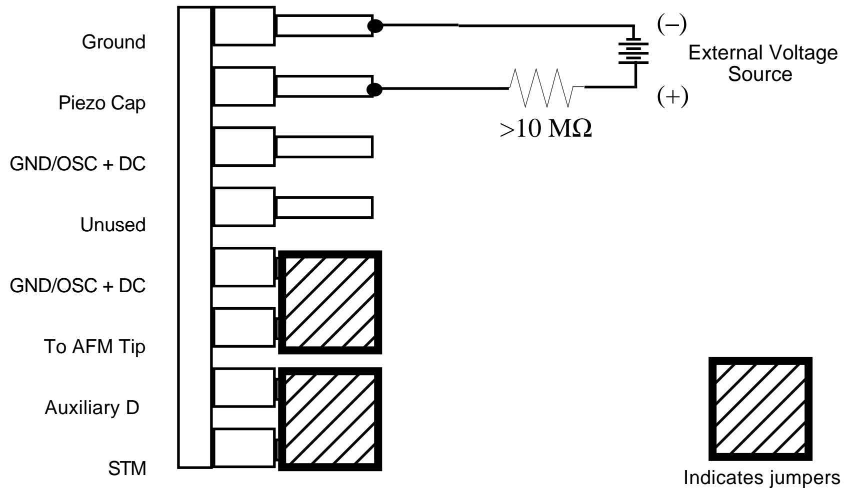
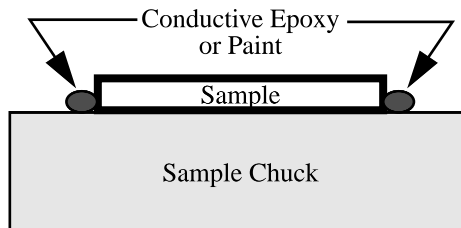


Figure 14.24. Jumper configuration for application of voltage to sample via sample chuck.

A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to pins on the header using soldered, push-on connectors. ***Do not solder leads directly*** to the header pins. Heat may cause damage and/or make jumpering the pins difficult.

The sample should be electrically connected directly to the chuck or a standard sample puck using conductive epoxy or silver paint as shown below:



14.7. Surface Potential Imaging—Procedure

1. Locate the two toggle switches on the backside of the Extender Electronics box (Figure 14.24), then verify that they are toggled as shown in Table 14.2.

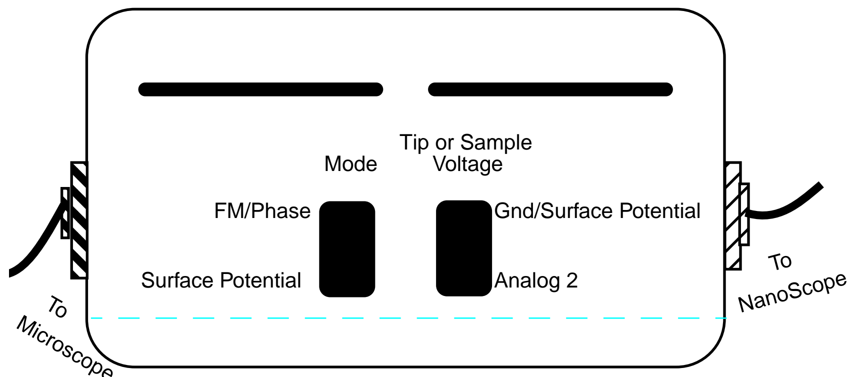


Figure 14.25. Toggle switches on back of Extender Electronics Module.

	Mode		Tip or Sample Voltage	
	FM/Phase	Surface Potential	GND/Surface Potential	Analog 2
TappingMode Contact AFM MFM	√		√	
Surface Potential		√	√	
Apply voltage to tip or sample (Use for electric field gradient imaging; tunneling AFM)	√			√

Table 1.2.Extender Electronics Module toggle switch settings for surface potential imaging.

Notes

- The toggle switch combination of Surface Potential = ON and Analog2 = ON is **not recommended** and can produce erratic and undefined results.

2. Mount a sample onto the sample holder. Make any external electrical connections that are necessary for the sample.

3. Mount a metal-coated NanoProbe cantilever into the electric field cantilever holder. MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz) 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.

4. Set up the AFM as usual for TappingMode operation.

5. Use **Cantilever Tune: AutoTune** (as described in Section 14.4.1) to locate the cantilever's resonant peak. Remember, however, that the Extender box has been reconfigured so that the phase detection circuitry now acts as a lock-in amplifier. Any procedures that are normally used to view or adjust the phase signal will now affect the lock-in signal instead (see Figure 14.21).

In this case, two curves should appear in the **Cantilever Tune** box: the *amplitude* curve in white and the *lock-in* curve in yellow. 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 signal 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.

6. Engage the AFM and make the necessary adjustments for a good TappingMode image while displaying height data.

7. Select the **Interleave Controls** command. This brings up a new set of scan parameters that are used for the interleaved scan where surface potential is measured. Different values from those on the main scan may be entered for any of the interleaved scan parameter. To fix any of the parameters so they are the same on the main and interleave scans, click on the green bullets to the left of particular parameter. The green bullet changes to "off" (gray) and the parameter value changes to the main Feedback Controls value. Set the interleave **Drive frequency** to the main feedback value. Enter an interleaved **Setpoint** of **0 V**. Set **Interleave scan** to **Lift**.

8. Enter an Interleave Controls **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 ±10 V. 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 **5 V**.

9. Set the Channel 2 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 data collection occurs on the retrace.

10. Choose a **Lift start height** of **0 nm** and a **Lift scan height** of **100 nm**. The **Lift scan height** can be readjusted later.

11. Switch **Interleave mode** to **Enable** to start LiftMode. Now, when the microscope completes a topographic scan line (trace and retrace) the system turns off the TappingMode piezo and switches the oscillator signal to the cantilever. The cantilever is driven electrostatically according to the interleave **Drive amplitude** that has been selected. Also, when **Potential** is selected as the **Data type** for the Channel 2 image, a feedback circuit is enabled in the Extender box which adjusts the dc voltage on the tip to maintain the cantilever oscillation amplitude at zero. To do this, the feedback circuit uses the lock-in signal of the cantilever oscillation and tries to keep this value at zero volts. As detailed in the Section 14.5 above, when the cantilever oscillation amplitude has returned to zero, the dc voltage on the tip and sample are the same. The NanoScope records the dc voltage applied to the tip and this signal is displayed in the **Potential** data type.

12. Adjust the FM gains. The feedback loop that is used by the Extender Electronics for surface potential measurements is the same as the one used in Frequency Modulation (FM) for magnetic and electric force gradient detection, as described previously. The feedback loop should be tuned in a similar manner to the FM setup. Select **Other Controls** and adjust the FM gains. Setting both FM **Integral gain** and FM **Proportional gain** to **15.0** is a good starting point. 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. The gains often need to be much lower for potential measurements than for standard FM measurements. More information on tuning the feedback loop is given in the Troubleshooting section below.

13. 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.

14. For large sample voltages or qualitative work, select **Data type** = **Phase** instead of **Potential**. When the Extender box 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 ±10 V (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 Extender box, select the **Data type = Phase**.

14.7.1. 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 +10 V or -10 V. Here are some tips to see if the feedback loop is working properly with no oscillation:

- Go into **Scope Mode** and look at the Potential signal. If oscillation noise is evident in the signal, reduce the FM gains. If oscillations persist even at very low FM gains, 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 FM gains may be increased for improved performance.

In **Scope Mode**, if the Potential signal is perfectly flat and shows no noise even with a small Z-range, the feedback loop is probably stuck at ±10 V. (You can verify this by changing the value of **Real-time planefit** to **None** in the **Channel 1** panel.) Reduce the **Scan rate** and watch the display monitor 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 on the display monitor 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**. It may also be helpful to momentarily turn the **Interleave mode** to **Disabled**, then back to **Enabled**. Also try reducing any external voltage that is being applied to the sample to stabilize the feedback loop, then turn the voltage back up.

Chapter 15 Calibration Procedures, Troubleshooting and Maintenance

This chapter provides detailed instructions for the fine calibration of Digital Instruments MultiMode SPMs. Additionally, the latter part of the chapter focuses on problems commonly encountered during operation of the microscope and then concludes with maintenance procedures for the MultiMode SPM adjustment screws.

Operators should establish a three-month service schedule for mainentance and calibration. 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.

15.1. SPM Calibration Theory—Overview

Digital Instruments employs a software-guided calibration procedure for all its microscopes. The procedural particulars of how calibration is executed using NanoScope software are beyond the scope of this document and include proprietary methods exclusive to Digital Instruments. The theory, however, is straight forward and consists of the steps listed below.

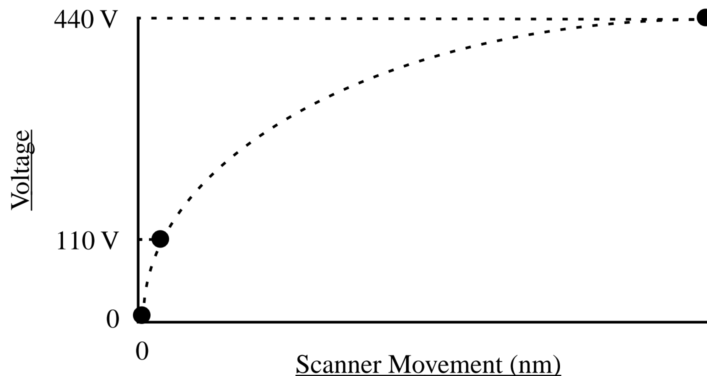
- Scan a calibration reference having surface features of precisely known dimensions.
- Compare known dimensions on the calibration reference's surface with those estimated by the SPM software.
- Adjust calibration parameters until the SPM's dimensions accord with the true dimensions of the reference.

The **Capture Calibration** and **Autocalibration** routines are designed to optimize measuring accuracy over the entire measuring range of the scanner. In order to obtain the finest accuracy possible for a given scan size, the scanner calibration parameters can be manually fine-tuned. This may prove useful in applications where measuring accuracy must be better than 1 percent.

15.1.1. Theory behind calibration

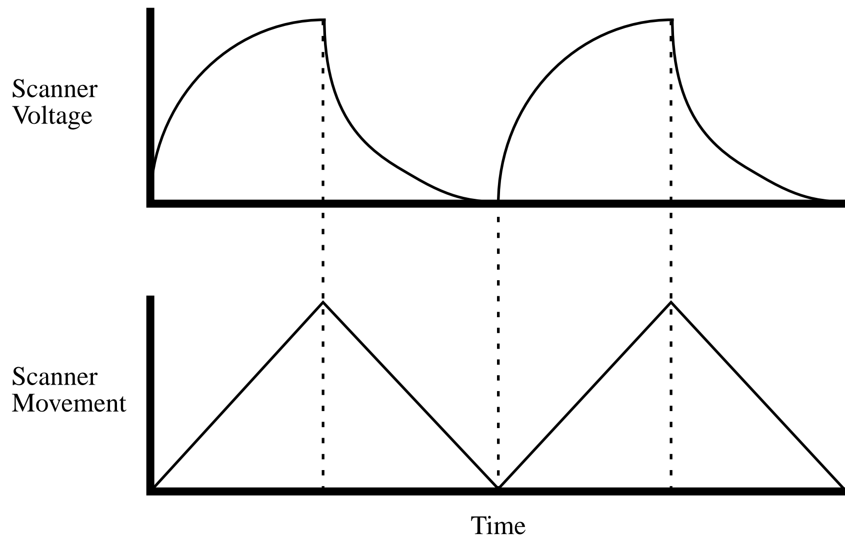
For purposes of calibration, the NanoScope software employs various derating and coupling parameters to model scanners' nonlinear characteristics. By precisely determining points along the scanner's sensitivity curve, then applying a rigorous mathematical model, full-range measuring capabilities can be made accurate to better than 1 percent.

Consider the sensitivity curve represented here:



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.

Unfortunately, things are more complicated. 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.



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 above. 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).

Through rigorous quality control of its scanner piezos, Digital Instruments has achieved excellent modeling of scanner characteristics. Two calibration points are typically used for fine-tuning: at 150 and 440 volts. (A third point is assumed at 0 nm/volts.) These three points yield a second-order sensitivity curve to ensure accurate measurements throughout a broad range of scanner movements.

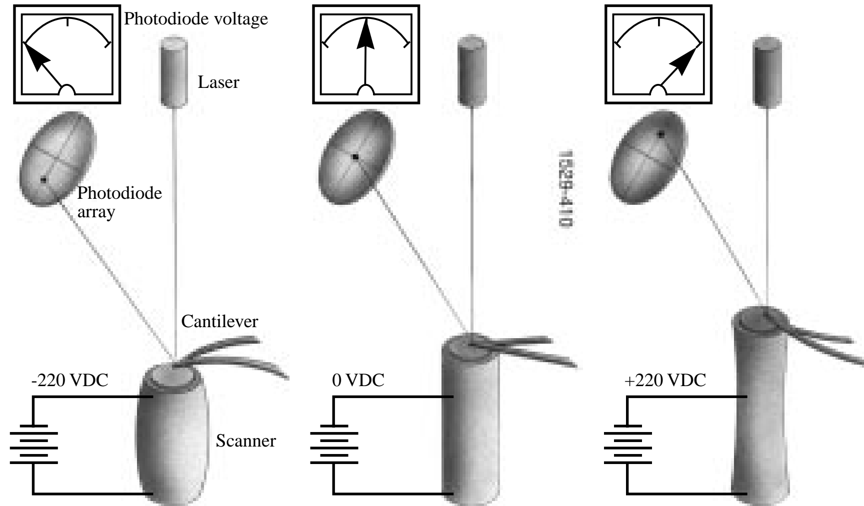
Because scanner sensitivities vary according to how much voltage is applied to them, the reference must be thoroughly scanned at a variety of sizes and angles. The user then tells the software the distance between known features on the reference's surface, and a parameter is recorded to compensate the scanner's movements. The X, Y and Z axes may be calibrated in any sequential order; however, the linearization adjustments (Section 15.4) must be performed before any calibrations are attempted (otherwise, they will be undone by the linearity adjustments).

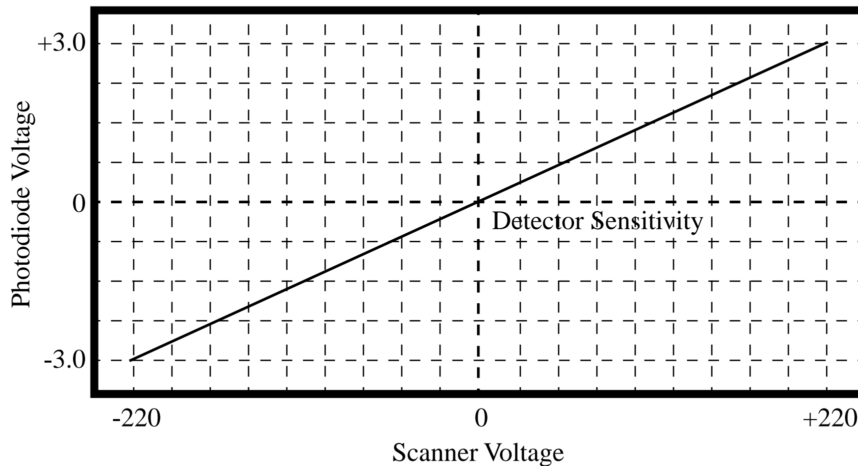
15.2. Sensitivity and Scanner Calibration —Background Information

Scanners serve as the "movers and shakers" of every scanning probe microscope. Typically, they 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 very precisely manipulate sample-tip movement in order to scan the sample surface. In some models (e.g., Small Sample MultiMode) the scanner tube moves the sample relative to a stationary tip. In other models (e.g., Dimension SPMs) the sample is stationary while the scanner moves the tip.

Not all scanners react exactly the same to a voltage. Because of slight variations in the orientation and size of the piezoelectric granular structure (polarity), material thickness, etc., each scanner has a unique "personality." This personality is conveniently measured in terms of *sensitivity*, a ratio of piezo voltage-to-piezo movement. Sensitivity is not a linear relationship, however. Because piezo scanners exhibit more sensitivity (i.e., more movement per volt) at higher voltages than they do at lower voltages, the sensitivity curve is just that—curved. This non-linear relationship is determined for each scanner crystal and follows it for the life of the scanner. As the scanner ages, its sensitivity will decrease somewhat, necessitating periodic recalibration.

The diagram below represents scanner crystal voltage versus photodiode voltage. In this instance, detector sensitivity is given as volt per volt, a parameter provided in the **Force Calibration** screen.





The **Microscope / Calibrate / Scanner** function displays the **Scanner Calibration** dialog box, allowing users to enter the sensitivity of their scanner's X-Y axes. Here, sensitivity is measured in terms of lateral displacement for a given voltage (nm/volt). In addition, other parameters measure the scanner's X-Y coupling and other effects.

15.3. Calibration References

As described above, each scanner exhibits its own unique sensitivities; therefore, it is the task of the user and software to precisely measure these sensitivities, then establish software parameters for controlling the scanner. This task is accomplished with the use of a calibration reference.

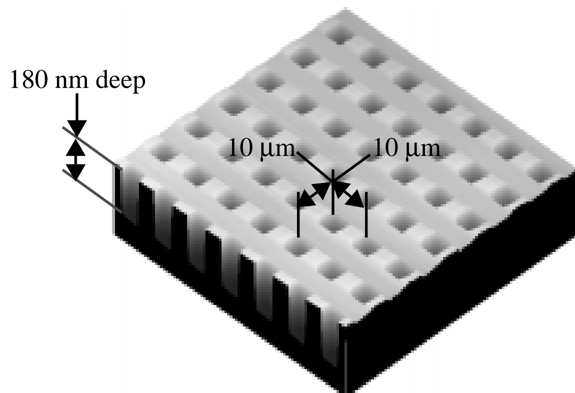


Figure 15.1. Section of Digital Instruments platinum-coated, silicon calibration reference.

One such calibration reference is shown in Figure 15.1 above. This calibration reference consists of a silicon substrate having a regular series of pits, each 180 nm deep, which is plated with platinum. Pits are spaced apart on 10 µm centers. Other, similar surfaces are available having 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. By using fine calibration techniques, it is possible to calibrate the SPM to much better than this.

15.4. Linearity Correction Procedure

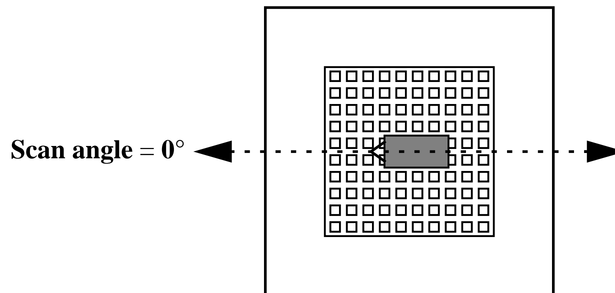
For applications which demand good linearity, the following procedure can be used to optimize the linearity correction parameters for individual scanners. As discussed previously, linearity correction is especially important for long-range scanners (such as the “J” and “K”).

15.4.1. Check scanner parameter values

If the system's original scanner parameters have been deleted, the scanner parameters can be copied from the software backup disk which is sent with every system. Individually purchased scanners are shipped with a head/scanner disk which contains the backup files, or a hard copy of the scanner parameters. In the event that files are not found, fax or call Digital Instruments for scanner calibration records.

15.4.2. Align calibration reference

Load the silicon calibration reference into the SPM. The reference will need to be aligned with the microscope scanner so that the tip scans parallel to the reference’s features with the **Scan angle** set at **0** degrees.



For “D” and “E” scanners, use a 1-micron pitch reference. For “G,” “J,” and “K” scanners, use a 10 micron pitch height reference. Align the reference within about 2 degrees of perpendicularity to the scan axes.

15.4.3. Set *Real Time* parameters

Set parameters in the control panels to the following values:

Panel	Parameter	Setting
Scan Controls	Scan Size	440 V
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	2.44 Hz
	Number of samples	256
	Slow scan axis	Enabled
	Z limit	440 V
Other Controls	Units	Volts
Channel 1	Data type	Height
	Z range	~ 20 V[a]

a. Adjust the **Z range** parameter to obtain the best contrast.

15.4.4. Set up SPM for contact AFM.

Set up the microscope for contact AFM imaging: set the **AFM mode** to **Contact**. (The microscope can also be calibrated using STM; however, this example will utilize contact AFM.) Set the **Scan angle** to **0** degrees. Adjust Real-time parameters to obtain a good-quality, maximum **Scan size** image. The **Scan rate** should be set to a value of **2.44 Hz** and the **Number of samples** parameter should be set at **256**.

With the sample engaged, check the scanning line relative to the reference's features; the scan should be orthogonal to the pits on the reference. If the reference requires rotation, **Withdraw** and rotate the sample to improve orthogonality between sample and scan line. Repeat until features are oriented orthogonally with the scan frame.

15.4.5. 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. To measure a captured image's orthogonality, view it using the **Off-line / View / Top View** function. On the display monitor, click on the **Angle** command, then use the mouse to draw a cursor between the edges or centers of widely spaced pits (see left figure

below). The angle should be measured with the vertex near the center of the image and the vertices in the upper-right or lower-left quadrant. The angle will be displayed on the white status bar at the bottom of the display monitor. Use the mouse to drag the cursor until it is oriented correctly,

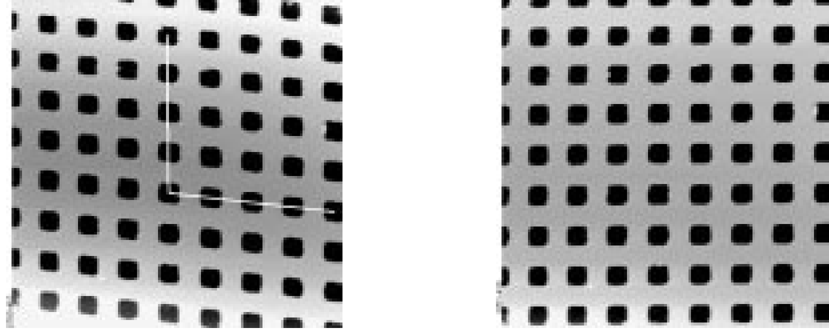


Figure 15.2. Left image: non-orthogonal image. Notice how pits are aligned with the vertical (slow) axis but skewed with the horizontal (fast) axis. Right image: a corrected, orthogonal scan.

then read the angle off the status bar. If the angle differs by more than a half degree, a correction will be required; otherwise, skip ahead to the step below.

15.4.6. Adjust sample orthogonality.

To adjust the orthogonality of the scan, click on **Real Time / Microscope / Calibrate / Scanner** to access the **Scanner Calibration** panel. The **Orthogonality** parameter is displayed on the bottom-right corner of the panel. Enter the difference between 90 degrees and the angle measured in the Top View image. (For example, if the angle measured in the Top View image was 92.5 degrees, enter a value of **-2.5** degrees into the **Orthogonality** parameter.) Click the **Ok** button to exit the **Scanner Calibration** panel, then capture another image and re-measure the angle. Repeat correction of **Orthogonality** until the scanned image shows less than 0.5 degrees of error. After a major change to the orthogonality parameter, it may be necessary to physically realign the calibration standard to the image frame.

15.4.7. Adjust *mag0* and *arg0*

Select **Microscope / Calibrate / Scanner** to display the **Scanner Calibration** dialog box. Set **mag0** and **arg** as follows:

When adjusting linearity, it is essential to keep in mind which part of the scan is the beginning and which is the ending. When the **Line direction** is set to **Trace**, the beginning of the fast scan is on the left, as indicated by the arrow base. When viewing up or down scans, be careful to check the image against itself, not the residual

image from the previous scan. Also, be careful to compare only the part of the scan drawn since the last parameter change.

Because the display monitor screen itself is not linear, the best way to check **mag0** and **arg** is with the Real-time **Zoom** box.

A. Adjusting **Fast mag0**

After engaging, click on **Microscope / Calibrate / Scanner**. This will invoke the **Scanner Calibration** window. As parameters values are changed, the effects will be seen on the display monitor. Now move the mouse cursor to the display monitor and select **Zoom Out**. This will produce a box whose size and position can be changed by alternate clicks on the left mouse button. Adjust the box until it is about one-third the size of the scan. Click *once* on the right mouse button to park the box and free the cursor, which will jump to Execute. (Clicking twice will execute a **Zoom**, which we don't want to do.)

Select (highlight) the scanner parameter to be modified. The first one to do is **Fast mag0**. Now move the zoom box to the start of the fast scan (on the left if the Line direction is set to Trace). Move and size the zoom box until the beginning third of the scan's features are exactly aligned with the zoom box. *The beginning third of the scan will be the standard for judging almost all of the linearity values.* It is a good technique to ignore the set of features near the edges of the scan since these may be distorted slightly. Now move the zoom box to the ending third of the scan. Align one side of the zoom box with some features and observe how well the other side aligns with the features under it.

Compared to the beginning third of the scan, if the features in the ending third of the scan are too big to fit the same number of them inside the zoom box, type a smaller number for **Fast mag0**. If the features are too small, increase **Fast mag0**. Change **mag0**s by about 0.1 to 0.3 units at a time. Remember, every time a parameter is changed, the entire scan axis is affected, so after every change it is necessary to resize the zoom box at the beginning third of the scan and re-compare the ending.

B. Adjusting **Fast arg**

Now that the beginning third of the scan is equal to the ending third, the center may need adjusting. Just as before, if the center features are too big for the box, reduce the **Fast arg**. If the features are too small, increase the **Fast arg**. Change **arg**s by 0.2 to 0.5 units at a time. Remember, changes affect the whole scan, so keep resizing the zoom box after changes.

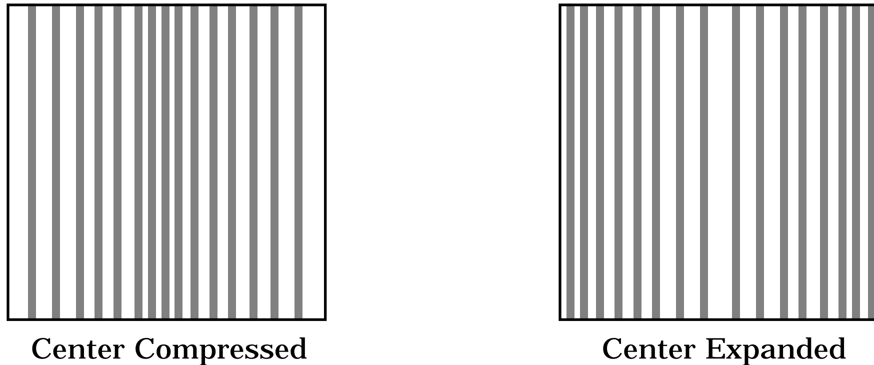


Figure 15.3. X scan linearization—compression vs. expansion.

Notes

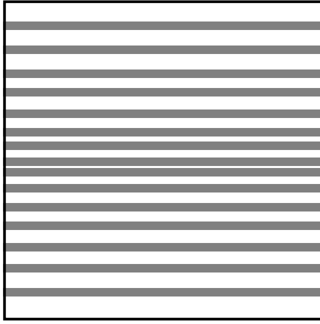
- After an adjustment in **Fast arg** is made, **Fast mag0** may need to be readjusted. Continue steps A and B until the rulings are evenly spaced across the Fast-axis. (If using a one-dimensional reference, it may be desirable to do step E {Adjusting **Fast mag1**} before proceeding; because adjusting the slow linearity will require the one-dimensional reference to be physically rotated 90 degrees. After finishing step E, return to here.)

After **Fast mag0** and **Fast arg** have been set, insert these values for **Slow mag0** and **Slow arg**. These will serve as close starting points before adjusting the slow linearities.

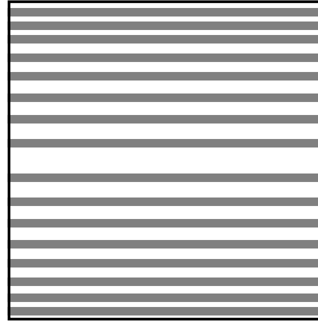
C. Adjusting **Slow mag0**

Follow the same instructions as for **Fast mag0**. Setting the slow linearities requires a lot of time because after making a parameter change it is necessary to wait until a fresh third of a scan has begun with the new parameters before the zoom box can be resized. Move the resized box to the ending of the scan and be prepared to measure the ending quickly. If the user is quick enough, a new parameter value can be typed

in before the scan starts again. Be careful not to confuse scan top and bottom with beginning and ending, since the scan direction alternates. Adjust the zoom box to fit the beginning third of the scan and check the ending third. If the features at the ending third are too big for the box, reduce the parameter. If the features are too small, increase. Be careful to compare only parts of the current scan, not the previous one.



Center Compressed



Center Expanded

Figure 15.4. Y scan linearization—compression vs. expansion.

D. Adjusting **Slow arg**

Follow the same instructions as for **Fast mag0**. Adjust the zoom box to fit the beginning and ending of the scan, then check the center. If the features in the center are too big, reduce the **Slow arg**. If the features are too small, increase.

After an adjustment of **Slow arg**, **Slow mag0** may need to be readjusted. Continue to follow steps C and D until the rulings are evenly spaced along the slow axis.

E. Adjusting **Fast mag1**

Initial Adjustment

Close the **Scanner Calibration** window by selecting **OK**. (Selecting **Restore** will reset the parameters to the values when the box was opened.) Change the **Scan size** to **150 volts**. Select **View / Scope Mode**. On the display monitor, select **Dual Trace**. If the two scope traces do not overlap, **Fast** $\mathbf{mag_1}$ needs to be adjusted. On the control panel, select **Slow scan axis**. When some tall features appear on the scope trace, tap the keyboard's right or left arrow key to toggle **Slow scan axis** to **Disabled**. Select **Microscope / Calibrate / Scanner** to open the **Scanner Calibration** box. Select **Fast mag1**. Use the left and right arrow keys to change the value until the two traces are aligned. (The yellow retrace line will shift in the same direction as the arrow.) When done, select **OK** to close the **Scanner Calibration** box. Set **Slow scan axi**s back to **Enabled**. Select **View / Image Mode**.

Fine Adjustment

Initial adjustment is usually adequate; however, if more precision is desired, adjust **Fast mag1** using the same procedure as in step A (see “Adjusting **Fast mag0**”). As before, set the **Zoom** box for the beginning of the scan and then check the ending. (Because the scan is small, it may be necessary to use a **Zoom** box up to one-half as large as the scan). If the ending of the scan is bigger than the beginning, reduce **Fast mag1**. If the ending is too small, increase the value.

F. Adjusting **Slow mag1**

With the **Scan size** still set to **150 volts**, select **Microscope / Calibrate / Scanner**. Select **Slow mag1** and input the value from **Fast mag1**. For medium-sized scanners (C to F), it is probably worth stopping here, because the **Slow mag1** value is usually 100-120 percent of the **Fast mag1** value. However, if more precision is desired, adjust **Slow mag1** using the same procedure as in step C (see “Adjusting **Slow mag0**”). As before, set the **Zoom** box for the beginning of the scan and then check the ending. (Because the scan is small, it may be necessary to use a **Zoom** box up to one-half as large as the scan). If the ending of the scan is bigger than the beginning, reduce **Slow mag1**. If the ending is too small, increase the value.

•Wait one complete frame with the new value before readjusting the **Slow mag1** value.

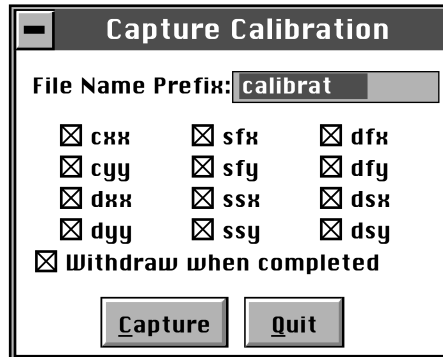
This completes the linearity adjustments. Check the final result by capturing an image and checking it with the **Off-line / Modify / Zoom** window. The scanner is now ready for calibration of the X and Y parameters.

*15.5. X-Y Calibration using **Capture Calibration***

Scanners are calibrated by setting parameters in the NanoScope software using the **Capture Calibration** command. This basic step must be performed before fine calibration. The basic procedure using version 4.XX software and a 10-micron calibration reference (Figure 15.1) is described in this section. Earlier versions of NanoScope software are similar. Note that “D” and “E” scanners require a 1-micron cross-ruling.

1. With the **Scan rate** set at **2.44 Hz** and **Number of samples** parameter at **256**, a full **Capture Calibration** will require about 70 minutes. Increasing the **Number of samples**, or decreasing the **Scan rate** will significantly increase the required time.

2. Using the mouse, click on **Real-time / Capture / Calibration**. The **Capture Calibration** dialog box will be displayed:

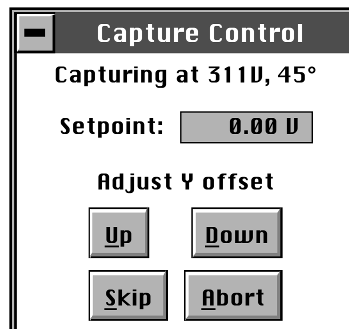


The **Capture Calibration** dialog box lists twelve parameters used in the calibration procedure. If this is a first-time calibration, or if the microscope's calibration has not been checked within the last three months, verify that all parameters are selected. Click on the **Capture** button to initiate the automatic calibration routine.

Notes

- The microscope will begin an automatic series of scans on the reference which require about one hour total 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 "look" like the normal reference. For example, pits may resemble trenches and features may be presented at various angles.

3. As each routine is executed, the user may need to adjust the scan slightly to optimize the calibration image. This is accomplished using the **Capture Control** dialog box, which is displayed on the control monitor throughout the calibration routines.



Note the status bar at the bottom of the control monitor. The “capture” status will start at skip 2—meaning that the program will skip the current scan and 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.) If the scan has not settled out by the time the capture status changes to “On,” click on the **Skip** button to increment the capture to skip 1, skip 2, or skip 3. Do not click on **Abort** unless you want to stop the entire **Capture Calibration** program.

Features should extend across as much of the displayed image as possible. If portions of features are missing, or if the image is blank, click repeatedly on the **Adjust Y offset / Up** or **Down** button to adjust the scan until more of the features are imaged. When the image looks optimized, allow the software to capture the image without disturbing it. The software will then automatically index to the next image.

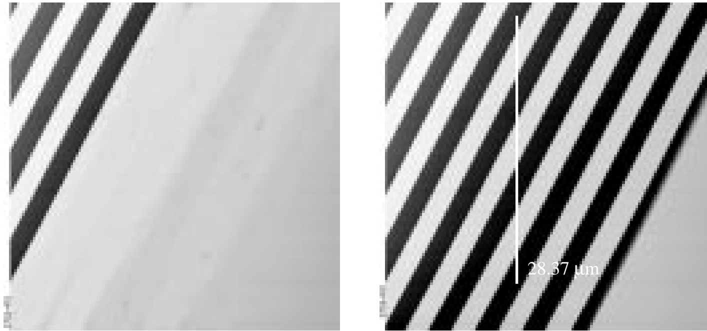


Figure 15.5. Use the *Adjust Y offset / Up* or / *Down* buttons to improve a partial calibration image (left). Calibration image at right is improved and may be used to measure distances between features.

After the first four images with the diagonal stripe pattern have been captured, the user can usually leave the system unattended while the program continues to completion. Some of the next images may seem stretched in one dimension; however, this is normal.

When all calibration images have been obtained, the software will prompt the user that it is finished.

4. Go to the **Capture** directory (!:) where all **Capture Calibration** files have been saved. Click on **Off-line / File / Browse** (or the **Browse** icon) to review all **Capture Calibration** files.

5. Verify that all calibration images contain features spanning the full width and height of the image frame. (See right Figure 15.5 above as an example.)

Notes

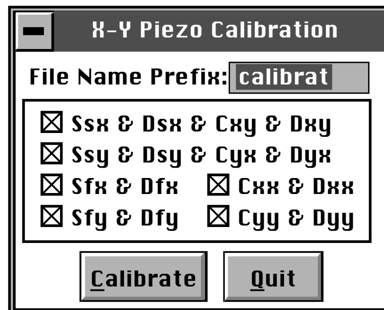
- All images unsuitable for calibration must be recaptured. Record the file name extensions of any unusable files (e.g., .cxy, .dyy), then delete them. Reengage the reference surface and select **Capture Calibration** again. Verify that the file name prefix is identical to that of the usable files, then deselect all usable file name extensions from the last capture (click on their box to remove the "X"). Finally, click on the **Capture** button to recapture the selected files.

15.6. Off-line / Utility / Autocalibration

After the **Capture Calibration** routine is completed, the user measures surface features contained within each image and enters their dimensions into the software. The software compares its estimates with the actual (user-entered) dimensions to make final corrections. This portion of the calibration is carried out using the **Off-line / Utility / Autocalibration** command.

To utilize the **Off-line / Utility / Autocalibration** command, do the following:

1. Select any of the captured calibration images in the Capture (!:) directory. Click on the **Off-line / Utility / Autocalibration** command. The control monitor will display the **X-Y Piezo Calibration** dialog box:



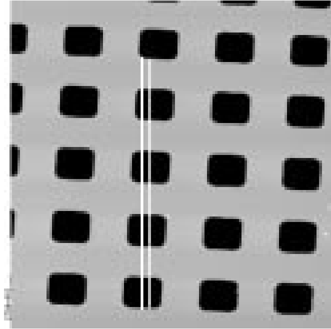
Verify that the file name prefix assigned to the captured files from the **Capture Calibration** routine described in Section 15.5 above is correct.

2. For normal calibration, verify that all parameters are selected in the dialog box, then click on **Calibrate** to execute the routine.

The software will sequentially present various calibration images on the display monitor while prompting the user to draw either a vertical line or a horizontal line. The control monitor will simultaneously display various dialog boxes—one for each image—requesting that a distance be entered.

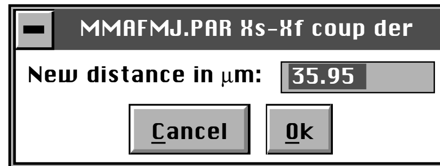
3. Use the mouse to draw a line on the image. The line should be drawn to span as many features as possible, preferably connecting like edges. For example, consider the following:

Autocalibration



Draw a vertical line

In this example, a line is drawn from the bottom edge of one feature to the bottom edge of another feature four rows away—a distance of 40 microns. The control monitor simultaneously displays a dialog box for entering the distance indicated by the white line:



The distance displayed in the box (in this example, 35.95 µm) is the software's estimate (based on current calibration values) of the length of the line drawn on the image. If the line length is different from the value shown, it will have to be changed. (In this example, the user would enter a value of **40**.)

4. Enter the distance covered by the white line drawn on the image. If a 10-micron reference is being employed, like portions of features are spaced 10 microns apart (e.g., between bottom edges, left sides, etc.).

Please Note: Features must be measured without regard to how they appear in calibration images. Remember, features may be represented as having stretched, distorted, or angled appearances due to the unusual movements employed during **Capture Calibration** scanning. Regardless, features are separated by the same (e.g., 10-micron) spacings.

5. Continue drawing lines and entering measured distances until all **Capture Calibration** images have been measured. When the software is finished, it will prompt the user that it is done.

If **Capture Calibration** and **Autocalibration** routines have been completed correctly, the SPM should be calibrated within 1-2 percent accuracy over most of the scanner's measuring range. Should it be necessary to obtain still better accuracy, the SPM can be fine-tuned to obtain maximum measuring accuracy. This is accomplished through the use of calibration parameters discussed in the next section.

15.7. Fine-tuning for X-Y Measuring Accuracy

Fine-tuning is usually performed at two **Scan size** settings: **150** and **440** volts. Both horizontal and vertical measurements of sample features are made, then compared with actual distances. Based upon this comparison, computer parameters are fine tuned. To fine tune your SPM for maximum X-Y measuring accuracy, review each of the steps below.

Notes

- If you are using an "A" scanner to image atomic-scale features, it will necessary to substitute graphite or mica for the silicon calibration reference. See Section 15.9 below for directions in atomic-scale calibration of the X- and Y-axes.

15.7.1. Prepare system for fine-tuning.

1. Set the **Scan size** parameter on the **Scan Controls** panel to the maximum value (440 volts). Verify that the **Scan angle** is set at **0.00** degrees. Mount a calibration reference into 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.). Optimize the image quality.

Please note: Your calibration and fine-tuning procedures are no better than the procedures and references used. **Choose both carefully!**

15.7.2. Measure horizontally at 440 V scan size.

1. Set the **Scan size** parameter on the **Scan Controls** panel to the maximum value (**440** volts). Verify that the **Scan angle** is set to **0.00** degrees. **Engage** the surface.

2. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a horizontal line between them. (For example, on a 10-micron, silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The screen will display the measured distance between pits next to the line.

Now check to see whether the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, skip to Section 15.7.3.

3. Based upon the results in step #2 above, perform the following calculation:

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

That is, divide the known distance by the distance displayed next to the line drawn in step #2 above. Record this quotient.

4. Click on the **Real time / Microscope / Calibrate / Scanner** option. The **Scanner Calibration** dialog box will be displayed:

Scanner Calibration

X fast sens:	230 nm/V	Y fast sens:	228 nm/V
X fast derate:	0.230 nm/V^2	Y fast derate:	0.218 nm/V^2
X slow sens:	267 nm/V	Y slow sens:	257 nm/V
X slow derate:	0.286 nm/V^2	Y slow derate:	0.270 nm/V^2
Xs–Xf coupling:	0.548 nm/V^2	Ys–Yf coupling:	0.569 nm/V^2
Xs–Xf coup der:	0.027 pm/V^3	Ys–Yf coup der:	0.222 pm/V^3
Xs–Yf coupling:	0.078 nm V^2	Ys–Yf coupling:	0.134 nm V^2
Xs–Yf coup der:	-0.04 pm/V^3	Ys–Yf coup der:	0.0668 pm/V^3
Fast mag0:	1.40	Slow mag0:	1.47
Fast mag1:	1.05	Slow mag1:	0.860
Fast arg:	3.00	Slow arg:	3.00
Fast cal freq:	2.44 Hz	Slow cal freq:	4.77 Hz
Piezo cal:	440 V	Rounding:	0.00
Allow rotation:	Allow	Orthogonality:	0.00 deg

Ok Cancel

Multiply the quotient obtained in step #3 above by the **X fast sens** value shown on the **Scanner Calibration** panel, then enter the new value. The new value will adjust the scanner's fast axis to more closely match calculated distances with actual feature distances. The new sensitivity setting will take effect as soon as it is entered. To save it to the computer's hard disk, click on the **Ok** button.This will also close the Scanner Calibration panel.

15.7.3. Measure vertically at 440 V scan size

1. Return to the image of the calibration reference. Clear the horizontal line drawn in Section 15.7.2 above (click the right mouse button, or click on **Clear**), then draw a vertical line between features. After waiting for at least three full scans (to allow the piezo to stabilize), select two widely spaced features and draw the line from like portions of features (top edge-to-top edge, etc.). The SPM will display the calculated distance between features.

2. Check to see whether the microscope's calculated distance agrees with the known vertical distance. If there is significant disagreement between the two, fine

tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, skip to Section 15.7.4 below.

3. Based upon the results in step #2 above, perform the following calculation:

$$\frac{\text{Known distance between features}}{\text{SPM-calculated distance between features}}$$

That is, divide the known distance by the distance displayed next to the line drawn in step #2 above. Write this value down.

4. Click on the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **Y slow sens** parameter.

5. Write down the **Y slow sens** value shown on the **Scanner Calibration** panel. Multiply the quotient obtained in step #3 above by the **Y slow sens** value shown on the **Scanner Calibration** panel, then enter the new value. This should adjust the scanner's slow axis to more closely match calculated distances with actual feature distances. To save the new parameter value, click on the **Ok** button.

15.7.4. Measure horizontally at 150 V scan size

1. Set the **Scan size** parameter on the **Scan Controls** panel to one-third the maximum (**150** volts). Verify that the **Scan angle** is set to **0.00** degrees, and that **Units** (**Other Controls** panel) is set to **Volts**. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a horizontal line between them. (For example, on a 10-micron, silicon reference, draw the line from the left side of one pit to the left side of another pit as far away as possible.) The microscope will display the measured distance next to the line.

Now check to see whether the microscope's measured distance agrees with the known horizontal distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, skip to Section 15.7.5.

3. Adjustments may be made in one of two ways. The first method uses to trial and error to "dial in" in the correct value. The second method calculates a precise correction.

- *Trial and error method*— Go to the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **X fast derate** parameter (or **Y fast derate** for Y-axis adjustment). If the measured distance is less than the actual distance, decrease the **X fast derate** parameter slightly (or **Y fast derate** for Y-axis adjustment) and re-measure image features. Adjust deratings up or down until measurements accord with known feature distances.
- *Calculation method*— Perform the following calculation:

$$\frac{s - \left(\frac{a}{m} \cdot [s - d(440 - v)]\right)}{440 - v}$$

Where s is the **Sens** value; a is the actual distance; d is the derating value; m is the measured distance; and, v is the **Scan size** in volts.

4. Click on the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **X fast derate** parameter.

5. Write down the **X fast derate** value shown on the **Scanner Calibration** panel. Multiply the value obtained in step #3 above by the **X fast derate** value shown on the **Scanner Calibration** panel, then reenter the new **X fast derate** value. This should adjust the scanner's fast axis to more closely match calculated distances with actual feature distances. To set the new parameter value, click on the **Ok** button.

15.7.5. Measure vertically at 150 V scan size

1. Select two widely-spaced features on the sample image of known separation, then use the mouse to draw a vertical line between them. (For example, on a 10-micron, silicon reference, draw the line from the top edge of one pit to the top edge of another pit as far away as possible.) The microscope will display the measured distance next to the line.

Now check to see whether the microscope's measured distance agrees with the known vertical distance. If there is significant disagreement between the two, fine tuning will be required; go to step #3 below. If the displayed distance agrees with the known distance, no further calibration is required.

2. If the displayed distance does not agree with the known distance, perform the calculation shown in Step #3 above to make a correction.

3. Click on the **Real time / Microscope / Calibrate / Scanner** function to display the **Scanner Calibration** dialog box. Select the **Y slow derate** parameter.

4. Write down the **Y slow derate** value shown on the **Scanner Calibration** panel. Multiply the value obtained in step #2 above by the **Y slow derate** value shown on the **Scanner Calibration** panel, then enter the new value. This should adjust the scanner's slow axis to more closely match calculated distances with actual feature distances. To set the new parameter value, click on the **Ok** button.

15.7.6. Change *Scan angle* and repeat calibration routines

As a final step, change the **Scan angle** on the **Scan Controls** panel to **90** degrees, then repeat steps 15.7.2—15.7.5 above for the following parameters: **Y fast sens**, **X slow sens**, **Y fast der**, and **X slow der**. This will ensure the scanner is calibrated along both the X- and Y-axis.

15.8. Calibrating Z

With respect to obtaining accurate Z-axis measurements, users should be aware that while it is generally not difficult to obtain accurate X-Y calibration references, it is much more difficult to obtain accurate Z-axis results. Users should be aware that Z-axis calibration is very sample-dependent. It is not easy to control Z piezo dynamics because the Z-axis does not move at a constant rate, as the X- and Y-axes do during scans. Furthermore, offsets can effect the piezo over a period of minutes. The platinum-coated, silicon calibration references distributed by Digital Instruments have 180 nm vertical features which are accurate to within ± 3 percent and are used throughout the examples provided within this section. If greater accuracy is desired, an appropriate calibration standard must be selected, and/or a metrology head employed using a Digital Instruments Dimension Series microscope.

15.8.1. Engage surface and begin imaging

Set up the microscope for contact AFM imaging. Engage the surface and adjust Real-time parameters to obtain a good-quality, maximum **Scan size** image. The **Scan rate** should be set to a value of **2.44 Hz** and the **Number of samples** parameter should be set at **256** or **128**.

Verify that the **Z Center Position** value shown next to the image display hovers around 0 volts. If the **Z Center Position** value is non-zero, use the **Real Time / Motor / Tip Up** and **Tip Down** buttons to adjust.

15.8.2. Adjust *Scan size*

Depending upon the type of scanner employed, adjust the **Scan size** parameter to include part of a pit, or up to 1 pit, within the image, along with portions of the surrounding flat area. The image should include Z-axis depths from the highest to the lowest elevations.

“A” scanners, when set at their maximum **Scan size** (440 volts), may image only a small portion of one pit. Adjust the sample and/or microscope stage until one side of a pit is visible, along with portions of the flat, “rim” area around the periphery of the pit.

Since it may be difficult and/or time consuming to locate a pit in the sample using an “A” scanner, an alternate method of locating a pit is to use the **Scope mode** option in real time imaging. After engaging on the surface of the sample, set both **Realtime Planefit** and **Offline Planefit** to **offset**. Go to **Scope mode**. Watching the Scope trace diagram, slowly turn the X-Y stage adjustment screws on the AFM microscope. (Do not turn more than a 1/2 turn in each direction.) When the scanner tip encounters a pit the Scope trace diagram will display a step in the normally flat line, see Figure 15.6. Be sure that the Z center position is fairly close to zero (±50 V).

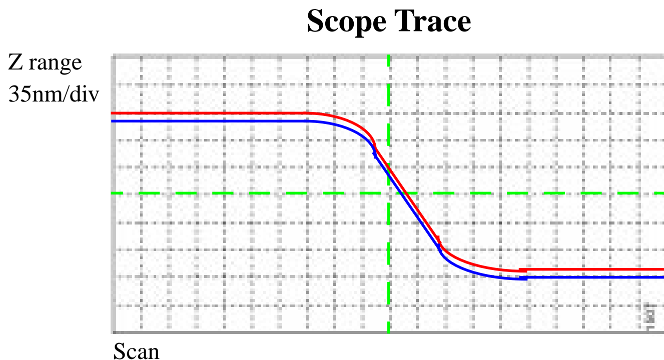


Figure 15.6. Scope Trace screen will display a step when the tip encounters a pit in the reference sample.

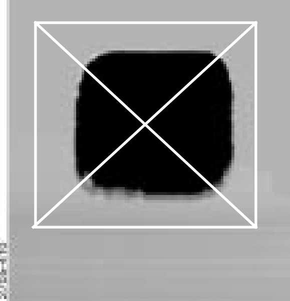
After finding the step in the Scope mode screen, click back to Image mode. The image should now encompass a portion of one pit.

Scanners larger than “A” can image an entire pit. It is not necessary to change the Realtime and offline planefits for these scanners.

15.8.3. Capture and correct an image

Capture an image by clicking on **Capture** in the **Real Time** screen, or use the **Capture** icon. When the image is captured, go to the Off-line screen.

It will be necessary to remove all tilt and scan line errata from the image before continuing. To remove scan line errata, select **Off-line / Modify / Flatten Manual**. The **Flatten order** parameter in the dialog box should be set to **1**. Go to the display screen, then draw a stopband over the pit as shown here:

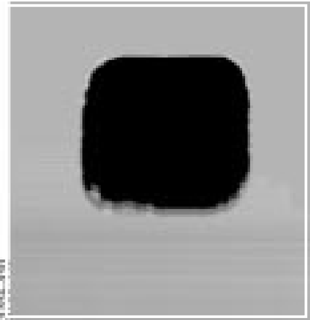


If the image encompasses one pit, or a portion of one pit (if you are using an “A” scanner), cover as much of the pit as possible with a stopband while leaving the remainder of the top surface exposed. Click on **Execute** to complete the flattening procedure, then **Quit** the dialog box.

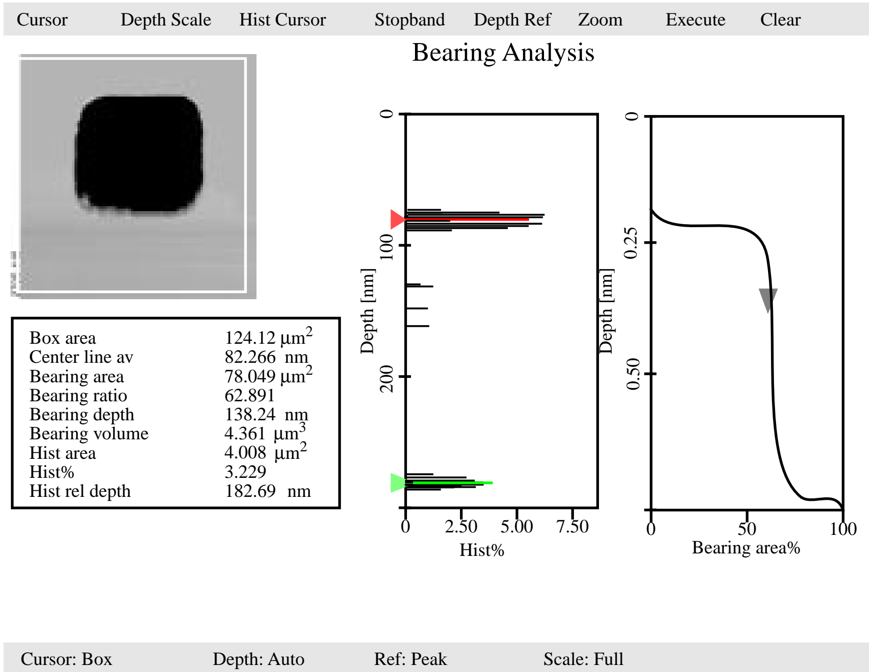
15.8.4. Measure vertical features

With the image corrected, its vertical features may now be measured. This is performed using **Bearing** analysis so that more data points are utilized.

Select the **Off-line / Analyze / Bearing** command. Go to the display screen and draw a cursor box over the entire image.



To commence **Bearing** analysis, click on **Execute** in the display monitor's top menu bar. Height data within the drawn cursor box will be graphed on the display monitor, and should show two, prominent spikes. These spikes correspond to two elevations on the surface: the bottom of each pit and the top surface.



Use the mouse to slide the top (red) line cursor on the **Hist%** graph to the centerline of the top data spike as shown in the example above. Select **Hist Cursor / On** to generate another (green) line cursor; position this one on the centerline of the bottom data spike. The distance between the two cursors is given at the **Hist rel depth** field inside the data box. (Depending upon whether the red cursor is positioned over the top or bottom data spike, the **Hist rel depth** value may be either positive or negative. It doesn't matter; take the absolute value.)

If a 10-micron, silicon calibration reference from Digital Instruments has been used, the **Hist rel depth** value should be very close to **180 nm**. Its variance from this value gives a good indication of the microscope's measuring error in the Z-axis. If the value is more than 2% from 180 nm, it will be necessary to make adjustments (see Section 11.8.5 below); write down the **Hist rel depth** value now.

To exit the **Bearing** dialog box, click on **Quit**.

15.8.5. Correct *Z sensitivity*

If the depth of the pit on the 10-micron silicon calibration reference deviates significantly from 180 nm, it will be necessary to correct the **Z sensitivity** parameter in the **Z Calibration** dialog box. Corrections here do not change the voltage applied to the scanner's Z-axis piezo crystal, but affect scaling applied throughout the Z-axis measuring range.

Transfer to the **Z Calibration** dialog box by clicking on **Real Time / Microscope / Calibrate / Z**. The **Z sensitivity** parameter is listed at the top-left and should be highlighted. Write this value down now.

Perform the following calculation:

$$\frac{180\text{ nm}}{\text{Hist rel depth value}}$$

That is, divide the actual depth of features (180 nm for the 10-micron calibration reference) by the measured depth (indicated in **Bearing** analysis by the **Hist rel depth** value). Multiply this quotient by the **Z sensitivity** value in the **Z Calibration** dialog box, then replace the value with the product. Click on the **Ok** button to enter the new **Z sensitivity** value.

Note

- The numerator value above (180 nm) is for Digital Instruments' 10-micron silicon reference. For other calibration references, set the numerator equal to the depth of features measured by **Bearing** analysis. Ideally, calibration references used should have features with heights comparable to those being imaged and measured on samples.

15.8.6. Recheck Z-axis measuring accuracy and correct

After steps 15.8.1—15.8.6 above have been completed, it will prove useful to recheck the Z-axis measuring accuracy of the SPM. Repeat steps 15.8.4—15.8.5 above until the required accuracy is obtained.

Note

- SPMs leaving Digital Instruments are normally calibrated to within 2 nm of accuracy using the 10-micron silicon reference. This corresponds to an accuracy of 1 percent. If greater accuracy is required, use a very high-precision calibration height standard with features comparable to those being imaged on samples.

15.8.7. Calculate *Retracted* and *Extended offset deratings*

Note

- It is not necessary to perform this procedure on "A" scanners.

Piezoelectric materials exhibit greater sensitivity at higher voltages. In steps 15.8.1–15.8.6 above, the Z-axis was calibrated while scanning near the middle of its voltage range (i.e., **Z Center Position ~ 0 V**). In this section, the Z-axis piezo will be calibrated while extended and retracted to offset the increased sensitivity.

1. Engage the surface as described in Sections 15.8.1—15.8.2.

2. Use the **Real Time / Motor / Tip Up** button until the **Z Center Position** reads **100 V**.

EXPLANATION: 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 steps 15.8.3—15.8.4 above to determine the measured depth of the calibration standard with a 100 volt **Z Center Position**.

Record the measured depth; it should read 180 nm on a Digital Instruments 10 μm silicon calibration reference (model STS-1800). If the depth measured by the extended piezo is in error by more than two percent, an adjustment will be required.

4. Click on the **Real Time / Microscope / Calibrate / Z** option to display the **Z Calibration** panel, then click on the **Extended offset der** parameter.

Perform the following calculation:

$$(1 + \text{current offset der}) \frac{180\text{nm}}{\text{meas. depth}} - 1$$

For example, if current offset = 4% and measured depth = 175nm, then:

$$(1 + .04) \frac{180\text{nm}}{175\text{nm}} - 1 = .0697$$

5. Enter 6.97 for the External offset derating % menu item.

6. The procedure for calculating and setting the **Retracted offset der** is exactly the same as for the Extended offset der; however, the piezo must be *retracted* by 100 V. Repeat steps #2-6 above in this section; however, start by using the **Motor Control** panel and the **Tip Down** button to retract the piezo to a **Z Center Position** of **-100 Volts**.

15.9. Calibration of “A” Scanners for Atomic-scale Measurement

The “A” scanner is the smallest scanner, with a total travel of approximately 0.4 µm along each axis. Its compact design lends excellent stability for atomic scans, and requires slightly modified X-Y calibration procedures. These are treated in this section. The procedure for X-Y calibration described below is essentially the same as those described in Sections 15.7.2–15.7.3 of this manual; however, they substitute graphite or mica atoms for the pits seen on silicon calibration references. A similar procedure is outlined in Chapter 9 of this manual for STM imaging of graphite. Please note that this procedure applies only to X-Y calibration of atomic-scale imaging. The Z-axis is calibrated in the normal way using a silicon calibration reference as described in Section 15.8 above.

15.9.1. Prepare sample.

The calibration may be conducted with either highly ordered pyrolytic graphite (HOPG) or mica. Mica should be used for contact AFM; HOPG for STM. Both mica and HOPG need to be cleaved to obtain a good flat surface. Cleaving is accomplished by adhering tape to the surface and pulling it off; this produces a fresh surface of atoms having a regular lattice.

Place the sample to be used on a puck, then attach to the scanner cap.

2. To obtain contact AFM atomic-scale images, try the following **Real Time** parameter settings:

Panel	Parameter	Setting
Scan Controls	Scan Size	12 nm
	X offset	0.00 nm
	Y offset	0.00 nm
	Scan angle	0.00 deg
	Scan rate	61.00 Hz
	Number of samples	512
	Slow scan axis	Enabled
	Z limit	440 V
Feedback Controls	Integral gain	12.00
	Proportional gain	4.00
	LookAhead gain	0.00
	Setpoint	0 V

Panel	Parameter	Setting
Other Controls	AFM mode	Contact
	Input attenuation	1x
Interleave Controls	Interleave mode	Disabled
Channel 1	Data type	Height
	Highpass filter	3–4
	Lowpass filter	1

3. Engage the surface and adjust the **Integral gain** and **Setpoint** to obtain a good image. The **Setpoint** should be kept low if possible, and the **Z Center Position** should be close to **0 V**. Notice that the **Scan rate** is set much higher (~ **61 Hz**) for atomic-scale images, this to defeat some of the noise due to thermal drift.

If difficulty is experienced obtaining an image, **Withdraw** and try a different site on the surface, then **Engage** again. Many users find it easiest to obtain good images measurements if the sample is rotated until atoms are oriented vertically.

4. Once a satisfactory image is obtained, **Capture** it. The image should appear similar to the image of graphite shown below. The captured image will be used for the calibration procedure described in the next several steps.

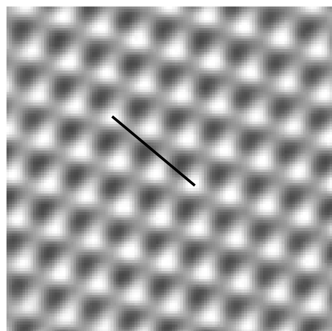
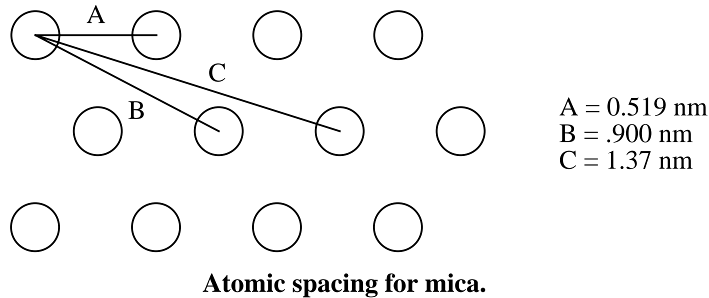
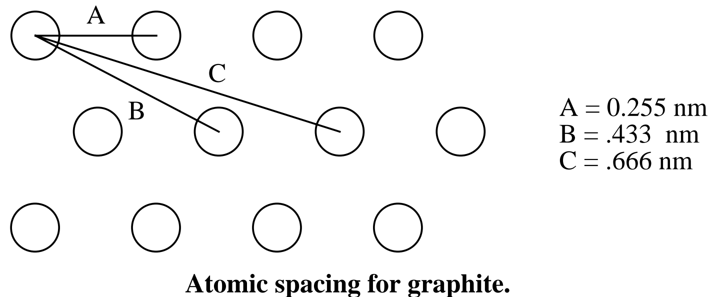


Figure 15.7. Typical atomic scan of graphite. Note the highly regular lattice of the atoms. The cursor line describes a distance of 6.66 Å.

5. Go to the **Off-line / View / Top View** option and measure the spacings between atoms using the mouse. Depending upon whether the sample is graphite or mica, the spacings should measure as shown below.



Atomic spacing for mica.



Atomic spacing for graphite.

Record the spacings between at least ten atoms observed in the captured image. It will prove useful to take several samples of each measurement and average them to ensure accurate results. (This can be done quickly by alternatively “walking” the cursor line from atom to atom; the average distance will be shown on the bottom-right corner of the display monitor’s status bar.) If the measurements vary by more than 2 percent from the dimensions shown above, a correction should be made.

6. Correction of the X- and Y-axis is essentially the same procedure as described in Sections 15-7.2—15-7.3 of this support note and will not be repeated here. The only significant difference is that the known distances must be adjusted for the smaller, atomic spacings of the atoms. Furthermore, only the sensitivity parameters are adjusted for atomic-scale imaging; namely:

- X fast sens at 0º **Scan angle**
- Y slow sens at 0º **Scan angle**
- Y fast sens at 90º **Scan angle**
- X slow sens at 90º **Scan angle**

The derating parameters are *not* adjusted for atomic-scale imaging, including:

- X fast derate
- X slow derate
- Y fast derate
- Y slow derate
- Retracted offset der
- Extended offset der

As referenced in Section 15.7.9, the sensitivity parameters must be calibrated with the **Scan angle** set at both **0** degrees and at **90** degrees.

7. Z-axis calibration is done in the normal way, using a silicon calibration reference. Refer to Section 15.8 for instructions on calibrating the Z-axis.

15.10. Troubleshooting the MultiMode SPM

Depending on the operating mode being used, the symptoms and subsequent resolution may vary. For this reason, problems listed in this section are divided into contact AFM and TappingMode. STM problems are described in Chapter 9 of this manual.

Some of the problems and cures associated with contact AFM are also relevant to TappingMode. If problems occur, it may be helpful to read through the Contact AFM problems and vice-versa. The most common problem while operating in TappingMode is due to unusual resonance peaks in the **Real Time / View / Cantilever Tune** command. Double peaks in the resonance curve can be caused by improper seating of the cantilever or a fracture in the cantilever. In general, bad cantilevers produce strange resonance curves, so the first thing to do when an unusual resonance curve occurs is to clean the groove on the cantilever mount and reseat the cantilever. If this does not improve the resonance curve, install a new cantilever.

15.10.1. Contact AFM: False engagement

The main cause of false engagement is optical interference on the photodiodes, which causes the vertical deflection (A-B) voltage to slowly move toward the setpoint voltage. Once the vertical deflection voltage reaches the setpoint voltage, the feedback loop assumes the tip has contacted the sample.

The source of the optical interference comes from either: i) the reflective gold coating on the cantilever, allowing some of the laser spot through and onto the sample surface; or, ii) stray laser light from the main beam interacting with the surface directly, causing optical path length related interference. The direct substrate reflection is more of an issue with reflective substrates that have large steps or topography. These effects can cause scattered light to be reflected onto the photodiode assembly along with the reflected beam from the back of the cantilever. The scattered light causes changes in the vertical deflection (A-B) voltage as the AFM head is lowered to the sample during engagement.

IMPORTANT! If a false engagement occurs, it can be detected easily by adjusting the **Setpoint** and observing the **Z Center Position** change. Increasing the **Setpoint** voltage by 1 volt should cause the **Z Center Position** to change by 1—5 volts. If the **Z Center Position** changes by a large amount (10—20 volts), the system is false engaged.

It is a good habit to watch the vertical deflection (A-B) differential voltage while the tip is engaging. The voltage should ideally jump (not drift slowly) from some negative value to the setpoint voltage. If it drifts slowly it means that reflected light is affecting the photodiode voltage. If this is the case, the engagement process can be aborted with the **Esc** key. The photodiode position should be adjusted to make

the Vertical Deflection voltage more negative than the setpoint voltage before trying the engage sequence again.

Sources of false engagement include:

- Incorrect optical alignment of laser spot on cantilever. This alignment will cause a greater amount of laser light to reflect off of the sample.
- The sample has a region on it that touches the cantilever before the tip does.
- Foreign material stuck on the cantilever beam that is lower than the tip.
- Check the cabling between the computer and the controller, and between the controller and the microscope. Any discontinuity in the microscope signals can cause an immediate engage.
- The **Setpoint** may be set more negative than the vertical deflection (A-B) voltage (this applies only to contact AFM modes). This false engagement is immediate and the computer will not show any motor travel or time delay after one gives the command. To correct for this condition, select **Withdraw** and check that the vertical deflection voltage reads a voltage more negative than the setpoint voltage.

15.10.2. Contact AFM: Head appears engaged but does not track surface features

Occasionally, the head appears to be engaged but surface features do not appear on the display monitor. This usually results from false engagement (see Section 15.1.1 above), poor laser alignment, or something other than the tip coming in contact with the sample surface.

An easy way to check for laser alignment problems is to check the laser beam using a magnifier or slip of paper. Click on the **Withdraw** command to remove the cantilever from the surface and recheck alignment with the magnifier or paper method (see Chapter 5). When satisfied with the alignment, readjust the top-bottom differential signal before re-attempting engagement.

Occasionally, when the optical head is not leveled properly, something other than the cantilever tip (usually the corner of the substrate or the cantilever clip) will contact the surface during engagement. The system may engage, but the image will not contain any features. Testing the SUM signal after withdrawing the tip several times is a good test for this failure mode. Generally, the SUM signal will be far from its original value if something other than the tip contacts the surface. The head should be leveled and the laser realigned if this has occurred.

15.10.3. Contact AFM: Head does not engage

The complementary problem to false engagement is not engaging at all. Here are some common causes of this problem:

- The tip may have started too far from the sample. Lower the tip's height above the sample surface by using the coarse alignment screws, then repeat the engagement procedure.
- The laser may be misaligned and/or the cantilever may be broken. When this occurs, replace the cantilever substrate, realign the laser, and repeat the engagement procedure.

15.10.4. Contact AFM: Head engages immediately

If the microscope engages immediately after the **Engage** icon is selected, the problem may be one the following: 1) The **Setpoint** may be lower than the feedback voltage. Select **Withdraw** a few times and verify that the upper DVM reads a negative voltage of -1.0 to -4.0 Volts. Adjust the **Setpoint** to zero or slightly above and try to engage again. If the microscope still engages immediately, check the cabling between the computer and the NanoScope controller and also between the controller and the microscope. Any discontinuity in the voltage feedback will cause an immediate engage.

15.10.5. Contact AFM: Displacement of material

This is generally caused by too much tracking force. Use the **Force Calibration** window after engagement for setting the correct tracking force.

The AFM can be tuned to operate in the attractive region of the force curve. This will take advantage of the fluid layer which captures and holds the tip to the sample. This effectively reduces the tracking force. Be aware of the fact that this means the tip is pulling away from the sample. This operating condition might cause the tip to pop off of the sample surface.

15.10.6. Contact AFM: Lines in the image

Lines can be caused by:

- AFM tip picking up contamination. The tip will effectively become longer and this will cause the feedback loop to raise the tip to keep the same tracking force. The contamination can come off of the tip which will cause another level shift in the image. This problem will show up as large bands in the captured image.
- Friction. Some samples have a stronger frictional interaction with the tip than others. The cantilever will bend and straighten due to the tip sticking and slipping as it is dragged across the surface of the sample. The result is a line-by-

line level shift in the captured image. The trace and retrace scan directions can actually be inverted from each other if the friction is high enough. A good practice is to use the scope image mode in dual trace display. The trace and retrace directions should be close to each other. Trace and retrace can invert if there is friction present between the tip and the sample surface.

15.10.7. Contact AFM: Problems with silicon nitride cantilevers

Sometimes nitride cantilevers can be a problem due to lateral warping or torquing of the nitride beams. This causes the reflected light to spill off to the side as the tip begins to engage the sample. Warping or torquing of the cantilever is associated with some older cantilever wafers. If engagement does not work the first time, try changing the actual lever used, i.e., go to the next lever on the same chip. Usually, the wide 200 µm-long lever has the most lateral warping. If this is the case, try the wide, short cantilever next to it. Generally, the narrow-legged silicon nitride cantilevers (both of which are on the same side of the chip) will have difficulties with the laser optics due to laser beam spillage over the side of the cantilever. This effect is more pronounced for samples which are highly reflective.

Using a microscope that has an interferometric objective lens, it is possible to observe five or more contour lines following the length of the legs of the cantilever on a warped cantilever probe. Cantilever probes which are not warped will have contour lines parallel to the substrate edge. Nikon® sells interferometric objective lenses for their Optophot and similar microscopes (such as the MPlan 40(x) DI 0.5 210/0). This is a recommended lens for observing contour lines on the cantilever for diagnostic purposes. It is beneficial to use a bandpass color filter with this lens. Please consult Nikon for further information.

15.10.8. Contact AFM: Image vertical dimensions are not correct

1. When invoking the **Highpass filter**, height information will not be accurate. The Highpass filter removes the DC component of scan information. This will invalidate the height information in the image.

2. The **Sensitivity** parameter must be calculated (as described in Chapter 6) or deflection data will be inaccurate.

Note

- The gains must be set low to reduce movement of the Z piezo when acquiring deflection data used in height measurements.

15.10.9. Contact AFM: *Z Center Position* goes out of range

The **Z Center Position** voltage is a measure of the average voltage to the Z electrode. The image will disappear when the Z center position reaches either the fully extended or the fully retracted ends of the Z center indicator. Possible causes include:

- Having the **Z limit** too low.
- Tilted sample. The sample should be as level as possible, particularly for larger scans, because tilt in the sample can cause the Z scan to run out of range.
- Mechanical drift will cause the sample-to-tip distance to change slowly bringing them apart completely, or too close together. If this is the case, the Z center will show either +220 (extended) or -220 (retracted), respectively.
- Drift in the optical path

Differentiating between optical path or mechanical drift is the first step in eliminating this problem. The force curve is a very useful tool for this purpose. Go to the force curve immediately after engaging. The force curve has two main regions: the sloping regions when the sample is in contact with the tip, and the flat region when the tip is free. Intersection of these two regions is what is important. Watch the force curve and determine whether the curve drifts vertically or horizontally; vertical drift is indicative of optical path drift, while a horizontal drift is due to a mechanical change in tip-to-sample separation.

Use the following as first steps in correcting drift:

- Verify that the sample, tip and stage are all stabilized. There should be no free movement between any of these components
- Recheck that the cantilever is firmly seated in its place and that there are no dirt particles wedged beneath it. Check that everything is tight and secure. Once this is checked, there may still be some drift. Using the **Tip Up** and **Tip Down** command can bring the sample back into range. (This may have to be done due to any number of factors.)
- Check for thermal stability. The SPM should not be located directly in the path of heating or air conditioning ducts; also, avoid locating the SPM near large windows which trap solar heat. Thermally caused drift due to thermal expansion of SPM components is the most common cause of mechanical drift. The MultiMode tends to heat up and drift when used with the acoustic isolation hood because it traps heat produced by electronics in the base and head.

15.10.10. Contact AFM: Poor image quality

If the tip is engaged with the upper DVM reading a stable, near-setpoint value and the Z center position is not overly sensitive to small changes in **Setpoint**, then laser alignment is probably good and the tip is scanning the sample. If image quality is poor with distorted shapes and low contrast, try adjusting the gains first, then optimize the scan direction to take advantage of the best tip shape to improve image quality. Vary the **Scan angle** parameter in the **Scan Controls** panel and see if this will help clean up the image. There is some variation in the shape of tip from substrate-to-substrate and the tip shape can affect the engagement process. (For example, a blunt tip may tend to engage falsely.) Therefore, changing the angle at which the tip scans some surfaces may have a significant effect.

Other scan parameters may also have a significant effect on the image quality. Varying the **Scan rate**, **X** and **Y offsets**, filtering, feedback gains and ranges all may improve the image (or make it worse!). Once engaged, adjust parameters in the **Scan controls** panel to improve the image quality. It is unlikely that adjusting the parameters will damage the tip unless the feedback gains are set too high, causing severe oscillations of the tip. In general, the **Scan rate** should be lower for large scans and for samples having tall features. The **Scan rate** can be increased on flat samples.

The offsets and zoom commands should be used to locate good clean regions on the sample. Filtering can improve atomic-scale images, but it is usually better to go without filtering on larger scans. Highpass filtering will distort the height information in all but atomic scans. For height data, the **Integral** and **Proportional gains** should be high but not high enough to cause oscillations. For deflection data, the feedback gains should all be low (close to zero). **LookAhead gain** should only be used for samples with regular features oriented along the slow axis. The user is encouraged to review the descriptions of all the parameters in the *Command Reference Manua*l and to experiment with parameters on a known sample.

Selecting a large **Scan size** and a high scan rate for a few scans can “sweep” an area clear. Decreasing the **Scan size** to image within the “swept” area can improve the quality of atomic images.

Finally, adjust the force exerted on the sample. Engagement requires a positive deflection of the cantilever, but the microscope will operate at much lower forces and lowering the force sometimes improves the image quality. Either use the force calibration to set the force or lower the **Setpoint** in small increments.

15.10.11. Contact AFM: *Force Calibration* command does not seem to work

Most often, the **Force Cal.** command will not provide a meaningful display, when first invoked. **Z scan start** and **Z scan size** as well as **Setpoint** affect the position and shape of the curve, and will probably require adjustment to achieve a useful display. The negatively sloped sensitivity line should, however, always be straight for a proper force curve.

15.10.12. Contact AFM: Image vertical dimensions are not correct

When collecting deflection data or invoking the **Off-line / Highpass** filter, height information may not be accurate. The **Highpass** filter removes low frequency information from the scan. This can distort the height information in the image. The **Sensitivity** parameter must be calculated properly or deflection data will be inaccurate. The gains must also be low to reduce Z-axis movement of the piezo. Even with the sensitivity properly calibrated and the gains as close to zero as possible, height data is more accurate than deflection data if the feedback gains are properly adjusted.

15.10.13. Contact AFM: *Z Center Position* goes out of range

The sample should be as level as possible, particularly for larger scans, because tilt in the sample can cause the Z scan to run out of vertical range.

15.10.14. Contact AFM: Image goes white

If, when using an “A” scanner, the image goes white after a couple of scans and the Z center voltage is still within range, check the **Real Time Planefit** parameter on the active **Channel** panel. When using an “A” scanner, it should usually be set to **Line** or **Offset**. In general, this will be a problem anytime there is a significant amount of drift in the Z center voltage, but drift is especially prevalent with “A” scanners.

15.10.15. TappingMode: Streaks on the trailing edge of surface features

Streaks are an indication of the tip not tracking the surface due to either: 1) insufficient tapping force; 2) an excessively fast scan rate; or, 3) gain values set too low. Streaking may also result from any combination of these factors.

Try the following procedures to eliminate this condition:

- Reduce the setpoint voltage. This increases the amount of tapping force on the surface. This is probably the thing that will be most effective. Be careful when doing this on soft samples. The sample surface can still be disturbed even though the forces are very small.
- Reduce the scan rate. The scan rate needs to be slower in TappingMode than in contact AFM. Typically it should be around 1-3Hz.
- Increase the integral and proportional gains. This will speed up the response time of the Z piezo transducer.

15.10.16. TappingMode: Lines across the image

Lines oriented in the fast scan direction can be caused by the tip sticking to the surface. This condition may be remedied by increasing the RMS voltage. Working with a larger RMS has the effect of giving the tip more energy to pull off of the surface. To correct this condition, try the following approach:

1. Use arrow keys (p) to increment the **Setpoint** voltage positively. Do this while monitoring the **Z Center Position** voltage on the display monitor. Increase the Setpoint voltage until the **Z Center Position** voltage jumps to the fully retracted position.

2. Note the current **Setpoint** voltage value. This value is just slightly greater than the RMS voltage currently used.

3. Increase the Setpoint voltage another 2 volts.

4. Use arrow keys to increase the **Drive amplitude** (press 2-3 times). This will increase the RMS voltage output from the microscope. (Increasing the RMS voltage means the cantilever is oscillated harder, making it less subject to capture by a sticky surface.)

5. Use arrow keys to reduce the Setpoint voltage until the **Z Center Position** voltage begins to move away from the retracted position. Continue to reduce the Setpoint voltage until the topographic image on the display monitor “pops” into clear view.

15.10.17. TappingMode: Rings around features on the surface.

This effect might also be described as the image looking as though it is half submerged beneath water. This is caused by operating with a drive frequency too close to cantilever resonance.

Use the arrow keys to increment the drive frequency a little lower. Do this while watching the Real Time scan. Be aware that the RMS voltage might also reduce.

Repeat the steps in the "lines across the image" troubleshooting description to adjust the RMS voltage.

15.10.18. TappingMode: Multiple or repeating patterns

The tip is probably chipped. This is usually caused by using too much tapping force on the surface, or because the tip encountered a feature too high to successfully traverse. Solution: change the tip.

Notes

- Operating with a smaller difference between the RMS voltage and the **Setpoint** voltage means that less tapping force is being used. This may damage the tip because the tip may not be following the surface, allowing it to crash into vertical features.

15.10.19. TappingMode: Image goes white or black

If the image goes white or black after a few scans and the **Z Center Position** voltage is still within range, check the Off-line **Planefit** sub-command on the control monitor. Off-line **Planefit** is normally set to **Full**. (See full description of the Off-line **Planefit** command in Digital Instrument's *Command Reference Manual*.

15.10.20. Fluid Imaging: Image drifts.

Occasionally, there may be some image drift due to the o-ring (used to contain fluid) sliding across the sample surface. To reduce movement of the o-ring, the fluid cell needs to be set up such that there is minimal lateral movement of the SPM head with respect to the sample once the o-ring is installed. This is best accomplished by keeping the head level and positioning the tip as closely as possible to the surface before installing the o-ring so that the lateral movement of the tip during engagement is minimized. Also, moving the positioning screws at the bottom of the optical head should also be minimized once the o-ring is installed. Further suggestions for solving this problem consist of the following:

- Use the fluid cell without the o-ring as described in Chapter 7 of this manual. This technique works best on hydrophobic surfaces. If you have a surface that does not allow a liquid drop meniscus to form or stabilize (e.g., mica), a water repellant marker may be used to outline a boundary for holding the drop. This type of marker, the "PAP PEN," can be purchased from the The Binding Site, 5889 Oberlin Drive #101, San Diego, CA 92121; 1-800-633-4484; Fax: 619-453-9189.

Notes

For applications where fluid flow is necessary, this option should not be used. Be certain to check for leaks and wipe up any spilled fluid as soon as possible to avoid damage to the scanner.

- The new vertical engage scanners allow the tip to approach the sample without the lateral movement caused by the stepper motor. Thus, lateral stress on the o-ring is absent. Vertical engage scanners are available in the 125-micron (“J”), and 10-micron (“E”) sizes. Contact Digital Instruments for more information.
- Lightly coating the area of the o-ring which contacts the sample surface with white petrolatum or vacuum grease will allow the o-ring to slide across the surface, minimizing lateral stress. This also forms a better fluid-tight seal between the o-ring and sample. Be aware that some solvents (e.g., non-polar organic solvents) may dissolve some lubricants into the fluid.
- Replace the o-ring with a slice of thin-walled glass, plastic, or stainless steel tubing. The diameter and thickness of the ring of tubing should be chosen to prevent contacting the inner or outer walls of the curcular groove in the glass cantilever holder. This gives the head more room to move laterally during engagement and for positioning the tip over the sample surface. The height of the ring of tubing must be chosen so that it is not too tall to prevent the tip from reaching the sample surface, or so short that the ring does not reach the bottom of the glass cantilever holder before engagement. Glue the ring of tubing to the steel sample puck or to the sample to prevent leaks.

Notes

When using the ring of tubing in the fluid cell, fluid cannot be circulated through the cell as when using the supplied o-ring.

- When positioning the o-ring on the surface, adjust the positioning knob at the base of the head to move it slightly forward. This will counter some of the lateral stress on the o-ring due to the head shifting rearward during engagement.

  To prevent leaks when using the supplied o-ring, it is best to place the o-ring on the sample *before* placing the head on the scanner.

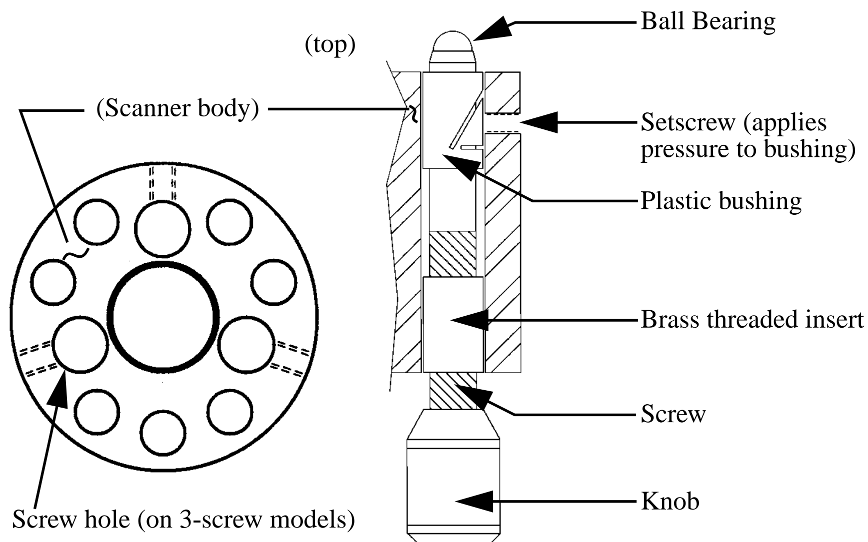
15.11. MultiMode SPM Adjustment Screw Maintenance Procedure

This section covers maintenance procedures for adjustment screws used on Digital Instruments Small Sample microscopes. These screws are used to support and position the head relative to the scanner, and must be cleaned periodically to ensure smooth operation. Instructions for the removal, cleaning, lubrication and replacement of adjustment screws are included in this section. A schedule of inspection and service is recommended at least **every three months**.

If adjustment screws are inspected and cleaned regularly, they should last the life of the scanner without replacement. Contamination of the screws with grit depends heavily upon the operating environment, types of material(s) being scanned, and operator handling.

15.11.1. Inspection

Small Sample SPMs utilize fine-pitch (1/4”-80) adjustment screws; in some scanners, two of them are manually turned, and a third (rear) screw is turned by a motor located in the microscope base. The Vertical Engagement “JV” scanner has just one adjustment screw. The diagram below illustrates a screw and adjacent hardware installed on later-model (post-August 1993) microscopes. Earlier models have their brass threaded inserts installed at the top of the scanner body (rather than the bottom) and feature a Teflon bushing. Inspection and service is similar for all types.



Adjustment screws are threaded into brass inserts, which are affixed to the scanner body with epoxy. Although screws are not heavily lubricated, a light film of oil is applied to them at the factory to prevent galling. This allows sufficient lubrication for fine adjustment, while minimizing drift (i.e., loosening) between the screws and scanner body due to the slow displacement of lubricant from screw threads.

Problems develop whenever screws become fouled with fine grit: screws may be difficult to turn and/or exhibit any of the following symptoms:

- Eccentric, rotational "limp" (i.e., alternatively easy, then difficult to turn).
- Faint, crunching or grinding noises when rotated.
- Microscope cannot engage sample surface, i.e., motor is unable to rotate rear adjustment screw.

If *any* of these conditions are noted, screws should be backed out and cleaned as described below.

Notes

- If screws are frozen (i.e., cannot be rotated), DO NOT attempt to force them! Return the scanner body and screws to Digital Instruments for repair.

The user should inspect screws *at least* every three months, more often if possible. It is a good practice to check screws whenever the scanner body is removed by turning manually and feeling for resistance. This is especially true of the rear, motor-actuated screw, which may be fouled without the user's notice.

15.11.2. Remove adjustment screws.

To remove adjustment screws, do the following:

1. Remove SPM head and disconnect the scanner body from the Small Sample base by pulling its cable connector straight up. Hold the scanner body firmly in your hand.

2. Gently turn each screw to check for resistance. Turn counterclockwise until backed out of its screw hole. If resistance is experienced in turning the screw, stop, rotate briefly in the opposite direction, then retry. If resistance is experienced on later-model scanners, loosen the setscrew which applies pressure to the plastic bushing (see diagram above), then try again.

3. If screws are frozen (i.e., cannot be rotated at all), DO NOT attempt to force them. Return the entire scanner body and screws to Digital Instruments for repair.

15.11.3. Inspect for physical damage.

Adjustment screws are made of hardened stainless steel; threaded inserts are made of a much softer brass. If a screw becomes fouled with hard grit it may bind against the threaded insert. If the bound adjustment screw is forcefully rotated, the screw will almost always destroy the brass insert: threaded inserts may become cross-threaded, or stripped entirely of threads. If this occurs, they will have to be pressed out and replaced; return to Digital Instruments for repair.

1. Once screws are removed from the scanner body, they may be washed using methanol. Do not use strong solvents such as methyl chloride, MEK, benzene, etc. Use a fine brush or swab to remove grit from between threads and shoulder. Observe caution around rubberized knob surfaces; certain solvents may dissolve them! Be sure to remove all grit from surfaces; air dry.

2. Use a swab stick (e.g., Q-tip, Puritan swab, etc.) to carefully clean grit from the threaded brass inserts. Be sure to remove all grit from threads; air dry.

CAUTION! Do NOT splash solvent on the scanner tube or wiring at the center of the scanner body—certain components (e.g., wiring insulation) may be dissolved, causing scanner failure!

ATTENTION! Ne pas éclabousser le tube en céramique piézo-électrique ou le montage à l'intérieur du tube avec un solvant. Certains composants pourraient être dissous, entraînant une défaillance du tube.

WARHINWEIS! Verwenden Sie keine Lösungsmittel auf dem Scanner-Röhrchen oder den Kabelanschlüssen am Piezo - manche Komponenten (z.B. Isolation der Anschlußdrähte) könnten sich auflösen und eine Fehlfunktion des Scanners nach sich ziehen.

3. Carefully inspect surfaces for signs of wear or damage. If small burrs are visible inside of the threaded brass inserts, they may removed by gently sanding with fine emory cloth, then recleaned using a swab and solvent. If threads are cross-threaded or stripped, the unit will have to returned to Digital Instruments for repair.

15.11.4. Clean guide bushings.

Plastic guide bushings are installed to stabilize screws and increase rigidity. Tolerances between screws and bushings are very tight, necessitating removal of all grit. Use a swab and methanol as described above to clean bushings thoroughly.

Notes

- It is not necessary to remove setscrews from the scanner base to clean bushings. (Setscrews are used only to apply pressure to the shoulder of each adjustment screw; see diagram above.) If setscrews are removed, they should be retorqued to approximately 4 in/oz., or until the adjustment screw can be turned snugly without binding.

15.11.5. Lubricate.

Apply a very fine layer of lubricant to each adjustment screw. Lubricant may consist of high-vacuum grease, optical coupling grease, or equivalent. Screws should exhibit a slight sheen and no more, indicating that they have been finely coated.

Notes

- Excessive use of oil and grease on screws can cause the head to drift slightly. This is due to a slow displacement of lubricant between the screw and threaded insert. As lubricant is slowly squeezed out between the screw and threaded insert, the screw settles, causing the head to lower itself (and the tip) toward the sample. This is especially apparent during atomic-resolution imaging.

15.11.6. Reinstall.

1. Verify that all screws, threaded inserts and plastic bushings are free of grit.

2. Replace adjustment screws in their threaded inserts. Carefully turn them down clockwise until the shoulder of the screw engages the plastic bushing, and the screw's ball bearing appears at the top of the scanner body.

Notes

- If screws bind during reinstallation, *stop immediately.* Back the screw out again, reclean as described above, then redo installation. Recheck that the setscrew is properly torqued at the plastic bushing. If screw still cannot be installed without binding, return to Digital Instruments for repair.

3. Verify that each screw turns freely in both directions, but feels snug. (Adjustment screws should turn freely while exhibiting sufficient rigidity to stabilize the head.)

15.12. Vertical Engagement Scanners-Installation, Use, and Maintenance

Digital Instruments now offers “E” and “J” scanners which permit vertical engagement without significant lateral movement. The vertical scanners feature the following:

- Completely motorized tip-sample engage.
- Fully vertical engage head does not tilt.
- Easy to locate tip on desired scan area.
- 0.5 µm lateral repeatability on subsequent engages.
- Accommodates samples up to 8.0 mm thick.
- Integrated fluid seal at top of scanner resists fluid spillage.
- Retaining springs mount permanently to scanner body.

Your vertical scanner should come shipped with the following:

- MMAFMJ.PAR (“J” scanners) or MMAFME.PAR (“E” scanners) parameter file on diskette[1].
- Stabilizing screw, P/N AFM-SCN-579
- Stabilizing screw, P/N AFM-SCN-580

In addition, you will require a calibration reference, such as a P/N STR10-180.

15.12.1. Hardware Installation

Installation of the vertical scanner is very similar to earlier models. To install the vertical scanner, do the following:

15.12.2. Remove old scanner.

If the SPM is engaged, disengage the sample by clicking on the **Withdraw** icon several times. Unplug and remove the SPM head from the microscope. Remove the sample, then unplug and remove the old scanner body.

1. Parameter file name may be labeled with scanner’s serial number (e.g., 1234JV.PAR).

15.12.3. Install vertical scanner.

Set the vertical scanner atop the MultiMode AFM or LFM support ring with the scanner's leadscrew at the back. The leadscrew should slip into the flexible coupling. If the leadscrew does not seat completely into the coupling, toggle the **Tip Up-Down** switch on the microscope base; this will rotate the coupling until the leadscrew seats.

Plug the vertical scanner cable into the receptacle on the left-rear edge of the support ring.

Secure the scanner to the ring using one of two screws supplied (see Figure 223-1 below).

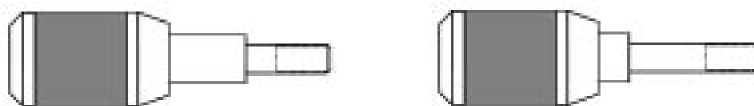


Figure 15.8. Stabilizing screw for securing the vertical scanner to the support ring. MultiMode AFM screw (left), and screw for other SPMs (right).

One screw is supplied with a longer shoulder (P/N AFM-SCN-580); this screw should be used with all MultiMode SPM bases. Use the short-shouldered screw (P/N AFM-SCN-579) for all other SPM models. The screw is inserted through a small hole on the front-underside of the support ring. Turn the screw until snug; do not overtighten.

IMPORTANT! The use of a fastening screw is necessary to ensure optimal system resolution and noise-free operation. DO NOT attempt to operate the scanner atop the support ring without a fastening screw!

15.12.4. Install parameter file.

The vertical scanner is shipped with a diskette containing its corresponding parameter file; this parameter file must be loaded into the computer's **EQUIP** directory. To load the file, exit the NanoScope software, then place the diskette into the floppy disk drive. Locate the EQUIP directory on the computer's hard drive, then transfer to it (e.g., **CD \EQUIP**).

NOTE:

There may be more than one \EQUIP directory, especially if the SPM uses more than one version of the NanoScope software. If so, copy the file to ALL directories and sub-directories labeled “EQUIP.”

The parameter file may be copied with any name, as long as it includes a .PAR extension. Make certain the vertical scanner’s parameter file name is not the same as a preexisting file; otherwise, it will overwrite the preexisting file. The parameter file is copied using a DOS command. For example, for a “J” scanner:

COPY B:\MMAFMJ.PAR C:\EQUIP\1234JV.PAR

Once the parameter file is copied, reboot the NanoScope software to resume.

NOTE:

The vertical scanner parameter files are identical to all other “J” and “E” scanner parameter files except for the **Motor sensitivity** parameter. On vertical scanners this parameter value is larger than older models. A summary of **Motor Sensitivity** parameter values is provided below.

	***Motor Sensitivity* parameter value (Tip travel per half step)**	
	AFM, LFM, TipView STM	**MultiMode™ AFM**
Vertical Scanners	**80**	**110**
3-screw Scanners	**19**	**26**

15.12.5. Select scanner.

Transfer to the **Real Time / Microscope / Select** panel. Click on the “**J”** or “**E**” button within the “Scanner” selection box, depending upon whether a “J” or “E” scanner is to be installed. If another “J” or “E” scanner parameter file already exists in the \EQUIP directory, both choices will be presented. Select the newly copied parameter file, then click on the panel’s **Ok** button to exit.

15.12.6. Inspect scanner.

Load a calibration reference (e.g., P/N STR10-180) on the MultiMode SPM, engage the surface and obtain an image. Verify that the scanner is working properly, and take a series of measurements on the calibration reference to ensure accuracy.

If the calibration reference is not measuring accurately, it may be necessary to calibrate software parameters to ensure measuring accuracy. This calibration procedure is detailed in the latest revision of *Support Note 217, Calibration Procedures*. Follow instructions provided in this document to properly recalibrate your vertical scanner.

15.13. Troubleshooting

15.13.1. Scanner is not properly calibrated.

Verify that the scanner's parameter file has been properly copied to each of the computer's **\EQUIP** directories. Check the parameter file name; it should include a .PAR extension (e.g., JVSCANR.PAR). Try to reselect the scanner using the **Real Time / Microscope / Select** panel.

If the parameter file is correctly selected but measures inaccurately, it may be necessary to calibrate the scanner. This calibration procedure is detailed in the latest revision of *Support Note 217, Calibration Procedures*.

15.13.2. Sample will not move (Tip "Up/Down" switch doesn't work).

If the vertical scanner is adjusted to its maximum tube height, the leadscrew may disengage from the motor coupling on the MultiMode base. (This is a built-in feature to prevent motor burn-out.) To reengage the leadscrew, remove the scanner from the support ring, then manually back the screw out several turns. Replace the scanner on the support ring and reattempt motorized movement.

If the leadscrew becomes fouled with grit, rotation may become difficult or impossible. To inspect, remove the scanner from the support ring and turn the leadscrew manually while feeling for resistance. If the leadscrew resists rotation, it will have to be backed out, cleaned with solvent and lubricated. (DO NOT attempt to clean the leadscrew while in the scanner body; solvents will ruin the scanner circuitry!) A leadscrew cleaning procedure is detailed in *Support Note 216* available from Digital Instruments.

NOTE:

If screw is frozen (i.e., cannot be rotated), DO NOT attempt to force it! Return the scanner body and screw to Digital Instruments for repair.

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