

di *Force Measurements*

228.1 Overview

Use force measurements to analyze tip-sample interactions and determine optimal setpoints for atomic force microscopy (AFM). These NanoScope™ routines highlight short- and long-range attraction to and repulsion from a surface, characterize sample hardness and quantify the force applied to a surface by a tip as a function of probe deflection.

This support note is organized to answer in a natural order the basic questions that arise about Force Mode. The tools are placed at your disposal in the first two sections for those who are eager to explore. Subsequent sections step through motivation and applications and may be read first for a more thorough introduction before operating your SPM in Force Mode. The sections, in order, are, first: when to make force measurements (section 3). Second: what options are provided and where to find them (section 4). Third: why force plots are of interest and look the way they do (section 5). Fourth: how to proceed in making force measurements in contact and tapping imaging modes (sections 6 and 7). Fifth: related force measurements, Force Modulation and Force Volume, which are detailed in other support notes, and are here introduced briefly (sections 8 and 9).

Document Revision History: Support Note 228

Rev.	Date	Sections	Ref. DCR	Approval
Rev. F	23-May-01	All sections affected	0385	Alan Rice
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This support note reflects NanoScope software version 5.12 and is limited in scope to the Force Mode features of NanoScope software. For more information about the operation of your scanning probe microscope (SPM), refer to its *Instruction Manual*. For the full range of software options, refer to the *Command Reference Manual*. For a list of documentation available and further questions, contact Digital Instruments/Veeco Metrology Group.

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




Three font styles distinguish among contexts. For example:

Window or Menu Item / **BUTTON OR PARAMETER NAME** is set to **VALUE**.

228.2 Safety Precautions

This section highlights cautions to observe using a scanning probe microscope to perform force measurements.

Figure 228.2a Safety Symbols Key

Symbol	Definition
	This symbol identifies conditions or practices that could result in damage to the equipment or other property, and in extreme cases, possible personal injury.
	Ce symbole indique des conditions d'emploi ou des actions pouvant endommager les équipements ou accessoires, et qui, dans les cas extrêmes, peuvent conduire à des dommages corporels.
	Dieses Symbol beschreibt Zustände oder Handlungen die das Gerät oder andere Gegenstände beschädigen können und in Extremfällen zu Verletzungen führen können.
	This symbol identifies conditions or practices that involve potential electric shock hazard.
	Ce symbole indique des conditions d'emploi ou des actions comportant un risque de choc électrique.
	Dieses Symbol beschreibt Zustände oder Handlungen die einen elektrischen Schock verursachen können.
	This symbol identifies a laser hazard. Exposure could result in eye damage.
	Ce symbole indique un risque lié à un laser. Une exposition à ce laser peut entraîner des blessures aux yeux.
	Dieses Symbol bedeutet "Gefährliche Laserstrahlung". Laserstrahlung kann zu Beschädigung der Augen führen.

CAUTION: Only qualified personnel aware of the hazards involved may perform service and adjustments.

ATTENTION: Toute réparation ou étalonnage doit être effectué par des personnes qualifiées et conscientes des dangers potentiels.

WARNHINWEIS: Service- und Einstellarbeiten sollten nur von qualifizierten Personen, die sich der auftretenden Gefahren bewusst sind, durchgeführt werden.





CAUTION: Follow company and government safety regulations. Keep unauthorized personnel out of the area when working on equipment.

ATTENTION: Il est impératif de suivre les prérogatives imposées tant au niveau gouvernemental qu'au niveau des entreprises. Les personnes non autorisées ne peuvent rester près du système lorsque celui-ci fonctionne.

WARNUNG: Befolgen Sie die gesetzlichen Sicherheitsbestimmungen Ihres Landes. Halten Sie nicht autorisierte Personen während des Betriebs fern vom Gerät.



CAUTION: Voltages supplied to and within certain areas of the system are potentially dangerous and can cause injury to personnel. Power-down everything and unplug from sources of power before doing ANY electrical servicing. (Digital Instruments/Veeco Metrology Group personnel, only.)

ATTENTION: Les tensions utilisées dans le système sont potentiellement dangereuses et peuvent blesser les utilisateurs. Avant toute intervention électrique, ne pas oublier de débrancher le système. (Réservé au personnel de Digital Instruments/Veeco Metrology Group seulement.)

WARNHINWEIS: Die elektrischen Spannungen, die dem System zugeführt werden, sowie Spannungen im System selbst sind potentiell gefährlich und können zu Verletzungen von Personen führen. Bevor elektrische Servicearbeiten irgendwelcher Art durchgeführt werden ist das System auszuschalten und vom Netz zu trennen. (Nur Digital Instruments/Veeco Metrology Group Personal.)



CAUTION: Use of controls or adjustments or performance of procedures other than those specified herein may result in hazardous laser light exposure. The use of optical instruments with this product increases eye hazard.

ATTENTION: Toute utilisation, ou étalonnage ou essai de modification, autre que ci-dessous décrits, peut entraîner une exposition

dangereuse à la lumière du laser. L'utilisation de systèmes optiques avec ce produit peut entraîner un danger pour les yeux.

WARNUNG: Die falsche Verwendung dieses Gerätes mit nicht in diesem Handbuch beschriebenen Vorgehensweisen kann gefährliche Laserstrahlung freisetzen. Optische Instrumente, die zusammen mit diesem Produkt verwendet werden, können evtl. Augenschäden hervorrufen und verstärken.

228.3 *When to Make Force Measurements*

Force Measurement refers to three related metrics for tip-sample interaction: the basic *Force Plot*, a useful preliminary to most SPM imaging, *Force Volume* (an array of force plots at different sites on a sample) and *Force Modulation*, a measure of sample hardness. This support note (#228F) introduces force measurements generally and details creating force plots. Refer to *Support Note #240B, Force Volume* and *Support Note #310A, Force Modulation* for detailed procedures for making the other two types of force measurement.

Note: Force Measurements apply to Contact mode and TappingMode™ imaging. Force Mode is not relevant to scanning tunneling microscopy (STM) so is not an option when **MICROSCOPE MODE** is set to **STM**.

Ideally, you would like to know the conditions the tip will encounter upon engaging a sample before reaching its surface. However, especially at nanoscale distances, such an ideal is a logical impossibility: you must interact with the surface to characterize it. By that reasoning, one would begin with a force plot to optimize parameter settings which are then applied to scanning the sample of interest. While this is generally true, even a force plot requires an initial assessment of the location of the sample - and that search process is best achieved in standard tip/sample engagement! In summary, the natural sequence of operations to commence optimized imaging is:

1) Engage tip to sample with best-guess parameter settings; begin scanning.

Note: Standard engage procedures are detailed in the *Instruction Manual* that accompanies each SPM.

2) Switch to Force Mode, make a force plot and optimize parameter settings.

Note: Upon entering Force Mode, the probe is automatically retracted a short distance from the sample and scanning stops. A force plot is a record of the controlled approach and interaction of the tip and sample.

3) Return to Image Mode, re-engage (automatically): image is optimized.

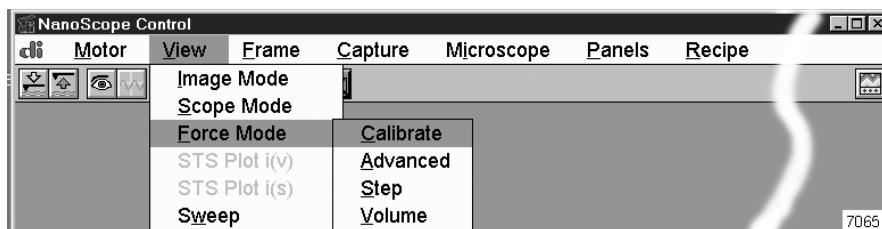
228.4 Force Measurement Control Panels

The various **Force** control panels allow the operator to precisely control the probe tip 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.

228.4.1 Entering Force Mode

Enter Force Mode from the **Real-time** menu (see [Figure 4a](#)).

Figure 228.4a Entering Force Mode in the Real-time Menu



Each of the four Force Mode options, **CALIBRATE**, **ADVANCED**, **STEP** and **VOLUME**, open a set of control panels in the Force Mode window (see [Table 4a](#)).

Table 228.4a: Control Panels Open in Force Mode

Control Panel in Force Mode	Force Option in Contact Mode or TappingMode			
	Calibrate	Advanced	Step	Volume
Main	✓	✓	✓	
Z Scan (Main)				✓
Scan		✓	✓	
Image Scan				✓
Feedback		✓	✓	✓
Auto		✓	✓	
Channel	✓	✓	✓	
Image Channel				✓
Force Channel				✓
FV Channel				✓

Note: A subset of Force Mode parameters also influence the **Real-time** operation of the microscope. For instance, gain parameter settings in **Feedback Controls** affect both Force and Image modes.

Force Volume control panels are detailed in *Support Note 240B, Force Volume*. The other panels listed in [Table 4a](#) are described next.

228.4.2 The Main Controls Panel

The **Main Controls** panel parameters govern moving the probe towards and away from the sample by applying a triangle wave to (i.e., *ramping*) the Z-axis piezoelectric actuator (see [Figure 4b](#)). **Z SCAN START** and **RAMP SIZE**, in particular, determine the appearance of force plots. **Main Controls** parameters are next introduced grouped by the Force Mode option(s) in which they appear.

Figure 228.4b Main Controls Panel

Main Controls	
Ramp Controls	
Ramp channel:	Z
Ramp size:	500.0 nm
Z scan start:	500.0 nm
Scan rate:	1.38 Hz
Forward velocity:	173 V/s
Reverse velocity:	173 V/s
X offset:	-90.6 nm
Y offset:	-272 nm
Number of samples:	256
Average count:	1
Display	
Spring constant:	0.3000 N/m
Display mode:	Both
Units:	Metric
X Rotate:	0.00 °
<input type="button" value="Pause"/> <input type="button" value="Resume"/> 7066	

Parameters Available in the Calibrate Force Mode Option Only

Ramp Controls Subpanel:

- RAMP SIZE** - is the size of the total ramp along the Z-axis. The maximum range of voltages that may be applied to the Z actuator, **440V**, limits **RAMP SIZE**, which may alternatively be entered as a distance if **UNITS** is set to **METRIC**.
- Z SCAN START** - sets the maximum voltage applied to the Z-axis piezoelectric actuator during the force plot operation. The triangular waveform is referenced to **Z SCAN START**. Increasing **Z SCAN START** moves the sample closer to the tip. The units are volts or nanometers, depending on the setting of the **UNITS** parameter. **Z SCAN START** is constrained such that **Z SCAN START** minus **RAMP SIZE** is greater than -220V, the minimum voltage that may be applied to the Z-actuator. The initial value of **Z SCAN START** is equal to the average Z-center voltage just prior to entering Force Mode, when the tip is engaged on the sample. *Decreasing* the value of this parameter shifts the force plot on the display to the left, while *increasing* the parameter shifts the curve to the right.

Note: **Z SCAN START** is disabled if a trigger is enabled or if **RAMP CHANNEL** is not set to **Z**.

- **SCAN RATE** - is the frequency of Z ramp cycling in Hz. The acceptable range of values is roughly 0.01 to 30Hz (refer to the *Command Reference Manual* for more detail).
- **NUMBER OF SAMPLES** - defines the number (between **16** and **64,000**) of data points captured during each extension-retraction cycle of the Z-axis piezoelectric actuator. This parameter does not affect the number of samples used in Image Mode.

Display Subpanel:

- **UNITS** - either **METRIC** or **VOLTS** - quantify Z-axis travel, shared by Force Mode and Image Mode.
- (TappingMode only) **AMPLITUDE SETPOINT** - defines the tapping amplitude target of the AFM feedback control loop and therefore the tip-sample force. The acceptable range of settings is **±1.0V**.
- (Contact Mode only) **DEFLECTION SETPOINT** - defines the cantilever deflection target of the AFM feedback control loop and therefore the tip-sample force. The acceptable range of settings is **±1.0V**.

Parameters Available in Calibrate, Advanced and Step Force Options

Ramp Controls Subpanel:

- **RAMP CHANNEL** - identifies the output signal to ramp. Select **Z** for Force Mode.
- (You must enable this parameter under *-/Show-all-items* in the upper left corner of the Main Controls panel) **FORWARD VELOCITY** - is the speed of the ramp in volts/s or nm/s in the extend direction.

CAUTION: **FORWARD VELOCITY** and **SCAN RATE** interact. **FORWARD VELOCITY** adds a level of control - and complexity - that is rarely needed.



- **REVERSE VELOCITY** - is the speed of the ramp in the retract direction. See **FORWARD VELOCITY**; the enable instructions and caution apply as well to **REVERSE VELOCITY**.
- **X OFFSET** - is the X-axis displacement from the prior scanned image center to where Z-axis ramping is performed. The acceptable range of values is $\pm 220\text{V}$; the metric distance equivalents are scanning-head dependent.
- **Y OFFSET** - is the Y-axis displacement from the prior scanned image center to where Z-axis ramping is performed. The acceptable range of values is $\pm 220\text{V}$; the metric distance equivalents are scanning-head dependent.
- **AVERAGE COUNT** - totals the number, between **1** and **1024**, of Z-axis ramps averaged to display a force plot. Use more than one in the presence of noise.

Display Subpanel:

- **SPRING CONSTANT** - of the cantilever in nN/nm is entered to be associated with the force plot captured, but does not affect Force Mode operation. The value may be changed in Offline Mode.
- **DISPLAY MODE** - may be **EXTEND**, **RETRACT** or **BOTH**, indicating which portion of probe tip Z-axis travel appears.

Buttons in the Main Controls Panel

- **PAUSE** - stops an active force ramp immediately.
- **RESUME** - continues ramping after a pause.
- (In Contact Mode combined with the Calibrate option of Force Mode only) **SETPOINT 0** - overrides **DEFLECTION SETPOINT** to zero the setpoint.

228.4.3 Channel Panel(s)

The **Channel** panel in Force Mode directs data presentation on the display monitor and records measurements made there (see example [Figure 4c](#)). Up to three, but no less than one channel panel may be active in Force Mode. Channel 1 is assumed to be the active channel in this support note.

Figure 228.4c Channel 1 Panel

Channel 1	
Data type:	TM Deflect.
Data scale:	294.7 nm
Data center:	0 nm
Deflection Sens.:	58.94 nm/V

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Parameters Available in Calibrate, Advanced and Step Force Options

- **DATA TYPE** - is assigned in Force Mode, based on **Other Controls/MICROSCOPE MODE**. Regardless of the selection of **Real-time/Channel 1/DATA TYPE**, when switching to Force Mode, **Force Mode/Channel 1/DATA TYPE** is assigned **DEFLECTION** for **CONTACT MICROSCOPE MODE**, with alternative options of **FRICITION**, **AUX B**, **AUX C** and **AUX D**. Similarly, **Force Mode/Channel 1/DATA TYPE** is assigned **AMPLITUDE** for **TAPPING MICROSCOPE MODE**, with alternative options of **TM DEFLECTION**, **AUX B**, **AUX C** and **AUX D**. **Force Mode/Channel 3/DATA TYPE** only allows for options **AUX B**, **AUX C** and **AUX D**. The selected signal of each active channel is displayed as a force plot (i.e., versus tip-sample separation) on the display monitor.
- **DATA SCALE** - quantifies the vertical axis displayed. The acceptable range of values is that of the parameter plotted.
- **DATA CENTER** - offsets the centerline of the display by up to $\pm 1/2$ of the **DATA SCALE**. Only the display, not the data set, is affected.
- **DEFLECTION SENS(itivity)** - is the calculated slope, in units of distance/voltage of the contact portion of a force plot, only appearing in the **Channel** panel for **CONTACT MICROSCOPE MODE**.

Note: See the Command Reference Manual to perform a Contact Mode sensitivity calculation.

- **TM DEFLECTION SENS(itivity)** - is the calculated slope, in units of distance/voltage of the contact portion of a force plot, only appearing in the **Channel** panel for **TAPPING MICROSCOPE MODE** and **TM DEFLECTION DATA TYPE**.

Note: See the Command Reference Manual to perform a TappingMode sensitivity calculation.

Note: No sensitivity parameter appears in the **Channel** panel for the **AMPLITUDE DATA TYPE** in **TAPPING MICROSCOPE MODE**.

228.4.4 The Scan Mode Panel

The transition into and out of force plots is controlled from the **Scan Mode** panel.

Parameters Available in Both Contact and Tapping Modes

- ¥ **TRIGGER MODE** - allows limiting the force applied by the tip to the sample. Valid values are **RELATIVE** for drift-independence, **ABSOLUTE** for a fixed trigger, or **OFF**.
- ¥ **TRIG(ger) CHANNEL** - specifies which of the **Force Mode/Channel 1/DATA TYPE** options is triggered.

Table 228.4b: Scan Mode Trigger Channel Options

Scanning Mode	Data Type	Available Trigger Channels
Contact	Deflection	all 5 available Data Types
"	Friction	Friction, Deflection
"	Aux B, C, D	Aux B, C, D or Deflection
Tapping	Amplitude	all 5 available Data Types
"	TM Deflection	TM Deflection, Amplitude
"	Aux B, C, D	Aux B, C, D or Amplitude

¥ **TRIG(ger) THRESHOLD** - defines the maximum force applied to a sample (i.e., the upper left-most point of a force plot). The range of acceptable values depends on other parameters:

Table 228.4c: Scan Mode Trigger Threshold Range Dependencies

Scanning Mode	Limit Type	Limit Value	Data Type	Trigger Threshold Range
Contact	Deflection	2.5V	Deflection	-1.25V
"	"	20V	"	-10V
"	"	2.5V or 20V	Friction	0-10V
Contact or Tapping	Deflection or Amplitude	2.5V or 20V	Aux B, C, D	-10V
Tapping	Amplitude	2.5V	Amplitude	-1.25V
"	"	20V	"	0-10V
"	"	2.5V or 20V	TM Deflection	-2.5V

- **TRIG(ger) DIRECTION** - specifies **POSITIVE**, **NEGATIVE** or **ABSOLUTE** signal slope where the trigger (may) take effect.
- **Start mode** - start mode allows the user to switch between the various force modes without returning to image mode.
- **END MODE** - specifies the location of the tip when returning to Image Mode: **EXTENDED**, **RETRACTED** or **SURFACE**.
- **Z STEP SIZE** - is the distance the tip travels along the Z-axis toward the surface between performing force plot ramps. The tip is no longer incremented once the tip encounters the surface during a force plot ramp.
- **AUTO START** - begins force plot ramping immediately upon entering Advanced Force Mode, if **ENABLED**. In Step Force Mode, or with **AUTO START DISABLED** in Advanced Force Mode, ramping only begins with selection of the **SINGLE** or **CONTINUOUS** icons (shown) or **PROBE** pull-down menu entries.
- **RAMP DELAY** - of **0** to **250 SECONDS** is applied upon reaching a trigger threshold or ramp end (at the closest point to the sample in a ramp cycle).



- **REVERSE DELAY** - of **0** to **250 SECONDS** is applied at the start of a ramp cycle (at the farthest point from the sample).
- **AUTO OFFSET** - when **ENABLED**, corrects for Z-axis drift periodically while in Force Mode, by using Image Mode control feedback to locate the sample surface. Otherwise, **AUTO OFFSET** is turned **OFF**.

228.4.5 The Feedback Controls Panel

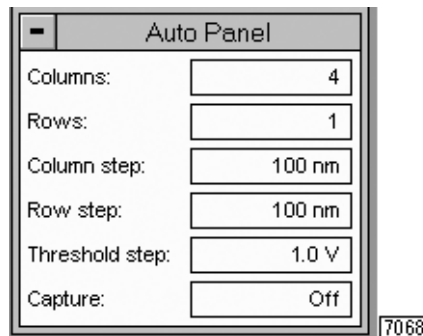
The **Feedback Controls** panel appears with identical parameters in both Real-time and Force Modes. See the *Command Reference Manual* or your *SPM Instruction Manual* for details of parameter settings.

One button is added to the **Feedback Controls** panel in Force Mode with Contact Mode imaging: **SETPOINT 0**, for overriding **DEFLECTION SETPOINT** to zero the setpoint (i.e., no deflection and no applied force). This is convenient for verifying a force plot is being generated when the **Channel 1/ DATA SCALE** is set too low for the plot to appear within the display.

228.4.6 The Auto Panel

The Auto panel specifies a matrix of sample sites for performing sequential force plots (see [Figure 4d](#)).

Figure 228.4d The Auto Panel



Auto Panel Parameters

- **COLUMNS** - is the number of distinct site Y-coordinate values.
- **ROWS** - is the number of distinct site X-coordinate values.

Note: The number of sample sites in the matrix is **ROWS**×**COLUMNS**.

- **COLUMN STEP** - is the Y-axis distance between sites.
- **ROW STEP** - is the X-axis distance between sites.
- **THRESHOLD STEP** - defines the maximum force applied to a sample at a site before moving to the next site (i.e., an analog of **Scan mode/TRIG(ger) THRESHOLD**).
- **CAPTURE** - stores a record of the force plot at each site when **ENABLED**. Otherwise, it is **DISABLED**.

Menu Bar Commands

The Force Mode window includes a menu bar and an icon bar in addition to control panels (see [Figure 4e](#)).

Figure 228.4e The Force Mode Window: Menu and Icons.



The pull-down menu options are listed in [Figure 4f](#). They apply both to Contact and Tapping imaging modes.

Figure 228.4f Force Mode Menu Options

Motor	View	Capture	(Probe)	Panels
Withdraw	Image Mode	(Capture)	(Run Continuous)	Ramp
Step motor		(Abort)	(Run Single)	Feedback
		(Continuous)	(Stop)	Mode
		Capture Filename	(Retract)	Auto
			(Approach Continuous)	Channels
			(Approach Single)	1
			(Auto Ramp)	2
				3

Legend:	Menu Item
	Parameter available in Calibrate, Advanced and Step Force Mode
	Parameter available in Advanced and Step Force Mode
	(Parameter grayed-out unless tip was engaged entering Force Mode)

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Each menu option is described next.

- **MOTOR** - controls tip-sample separation. Selections:

WITHDRAW - disengage from sample (see icon).

STEP MOTOR - opens the **Motor Control** panel (see [Section 4.7](#), next).



- **VIEW** - reverts to **Image Mode** (see “eye-con”).
- **CAPTURE** - stores the force plot for Off-line Mode viewing (see icon, and **ABORT** capture icon directly below it).
- **PROBE** - (shown grayed-out when probe withdrawn, as in [Figure 4e](#)) offers programmed probe motions. Selections:



RUN CONTINUOUS - performs the nominal **Force Calibrate** Z-axis cycling of amplitude **Z SCAN SIZE** (see icon).



RUN SINGLE - like **RUN CONTINUOUS**, but for a single cycle (see icon).



STOP - ends all probe motion (see icon).



RETRACT - lifts the tip away from the sample as far as possible using the Z-axis piezoelectric actuator, but not the Z-axis motor.



APPROACH CONTINUOUS - lowers the probe a distance **Main Controls/RAMP SIZE** toward the sample then raises it the same amount, moves the probe **Scan**

Mode/Z STEP SIZE closer to the sample and repeats the cycle, stopping at a tip deflection of **Scan Mode/TRIG THRESHOLD** and displaying the force plot (see icon).

APPROACH SINGLE - like **APPROACH CONTINUOUS** but ending after a single cycle or a tip deflection of **TRIG THRESHOLD** (see icon).



AUTO RAMP - begins sampling the matrix of sites as defined in the **Auto** panel.

- **PANELS** - specifies the active panels in a Force Mode option, allowing panels to be re-opened that were shut by clicking in their upper left corners.

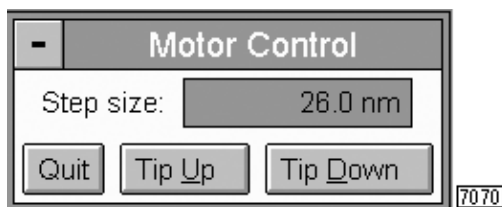
228.4.7 Motor Control Panel

Use the **Motor Control** panel (see [Figure 4g](#)) to move the tip on the Z-axis.



CAUTION: Do not step the motor when the tip is engaged with the sample; the tip and/or sample can be damaged.

Figure 228.4g Motor Control Panel



Fields and Buttons in the Motor Control Panel

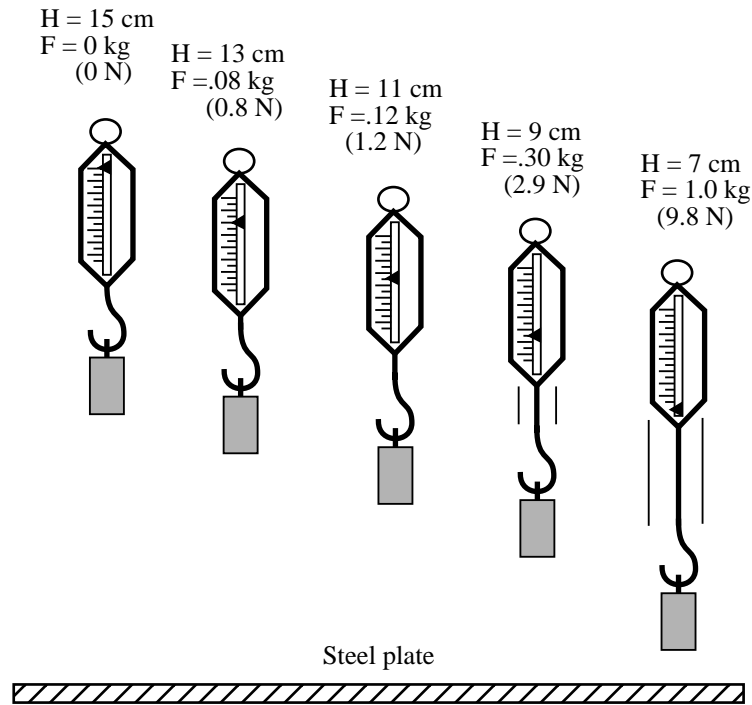
- **STEP SIZE** - is the Z-axis motion increment in microns. The accepted values range from **26 nm** to **1.04 μm**.
- **TIP UP** - moves the probe away from the sample by the **STEP SIZE** distance.
- **TIP DOWN** - moves the probe toward the sample by the **STEP SIZE** distance.
- **QUIT** - returns to Force Mode.

228.5 Force Plot Theory

A force plot is an observation of tip-sample interactions which yields information regarding the sample and/or tip. Not only do force plots insure against doing unintended damage while imaging on the nanoscale, but they provide guidance to obtaining optimized images.

The characteristics of a nanoscale force plot can be illustrated in a larger scale example. By way of analogy, suppose a materials researcher wants to compare the power of two different types of magnet. One magnet is made of ferrite; the other is stronger: a rare earth (e.g. SmCo, samarium cobalt) magnet.

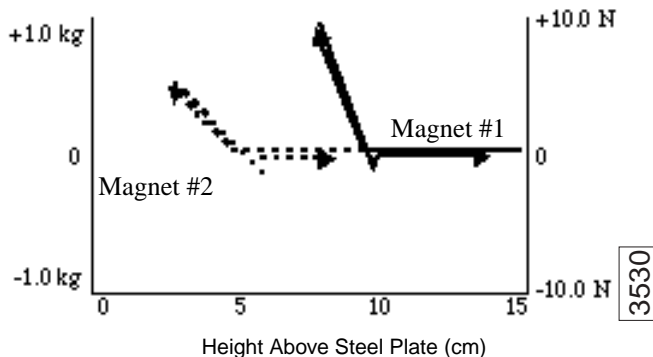
A simple way of measuring each magnet's strength would be to determine its pull on a steel plate. For example, the researcher could hang each magnet from a simple spring scale, "zero" the scale, then begin lowering the magnet toward a heavy steel plate. At regular distances from the plate, the amount of pull shown on the scale is recorded. At some unique height above the plate each magnet would be attracted strongly enough to attach itself to the plate. A plot of height, H , versus magnetic pull would detail the comparison of each magnet's power. A representation of this setup during a lowering cycle of one magnet is shown in [Figure 5a](#).

Figure 228.5a Pulling Force at Regular Intervals During Lowering Cycle

Similarly, the researcher could pull each magnet away from the plate—measuring the pulling force at regular intervals—until the magnet breaks free. The pull-off point of each magnet would give an additional index of its holding power.

Note: For the sake of simplicity, force is represented here by mass in kilograms. Force, with unit N, or newton = kg-m/s², is mass multiplied by acceleration (the latter measured here in meters per second squared, due to gravity), so 1 kg ~ 9.8 N.

The scale and magnet are lowered and lifted in a controlled cycle and the pulling force measured at 1 cm height intervals. A plot of this experiment using two magnets might appear as shown in [Figure 5b](#).

Figure 228.5b Graphical Representation of Pulling Forces.

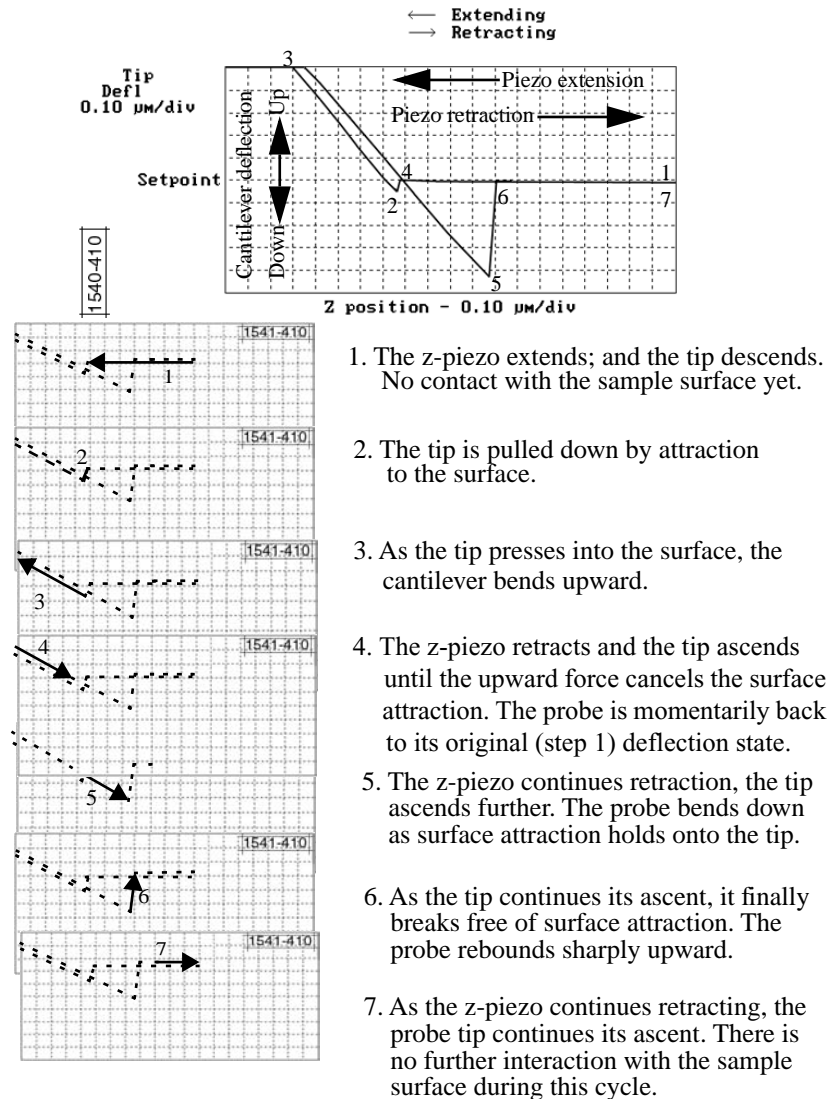
The plot shows each magnet's attraction as it approaches the plate, and its tenacity as it is pulled off the plate. Assuming both magnets are the same size, this reveals at least two things about each magnet's power. First, magnet #1 is stronger, attaching to the steel plate with 1.0 kg of pulling force at 8 cm, and magnet #2 is weaker, attaching at 3 cm with only 0.6 kg. Secondly, the nonzero slope of the attracting portions of the graphs reveals something about the range and intensity of each magnet's field. Whereas magnet #1 exhibits a quick attachment and pull-off from the plate with a steep curve, magnet #2 exhibits more sluggish attachment and pull-off with its less-sloped curve, due to a weaker magnetic field.

This simple model illustrates the dynamics of force plots using SPM tips on sample materials. SPM force plots at regular intervals across a sample can map electrical properties, elastic modulus or chemical bonding strengths.

228.6 Contact Mode Force Measurements

The simplest SPM force plot is made in Contact Mode using a silicon nitride probe tip. Low spring constant (pliant), V-shaped silicon nitride probes are sensitive to attractive and repulsive forces (see [Figure 6a](#) and [Figure 6c](#)).

Figure 228.6a Dissection of a Force Plot



In analogy to the graphed introductory illustrative example (see [Figure 5b](#)), the horizontal axis measures the z-piezo-controlled probe tip-to-sample distance and the vertical axis documents both positive and negative probe tip deflection in the Contact Mode force plot ([Figure 6a](#)).

The force gage/magnet example correlated features of a force plot with peak strength, as well as with the gradient, or rate of change, of a magnetic field. Similarly, the SPM graphic highlights the onset of sample-tip interaction (step 2, which depends on the strength of surface attraction), and sensitivity (steps 3, or 4, or 5, where the slope converts force - in this case applied deliberately by the z-piezoelectric actuator - into a measurable deflection of the pliant probe). Once the SPM is calibrated using the known forces induced by z-piezo motion of the probe, measured cantilever deflection becomes an indicator of the balance between forces at the probe tip, that applied by the cantilever versus resistance to it from the sample.

Sample-tip Attraction

The SPM probe tip “jump-to-contact” point (step 2, [Figure 6a](#)) is due to electrostatic attraction and/or surface tension (capillary) forces.

Attraction is also evident in step 5 as the cantilever is pulled away from the sample. Eventually, the sample “lets go” and the probe tip rebounds sharply upward (step 6 in [Figure 6a](#)).

Knowing the spring constant of the probe as a cantilever, the sample-tip attractive force is measured precisely.

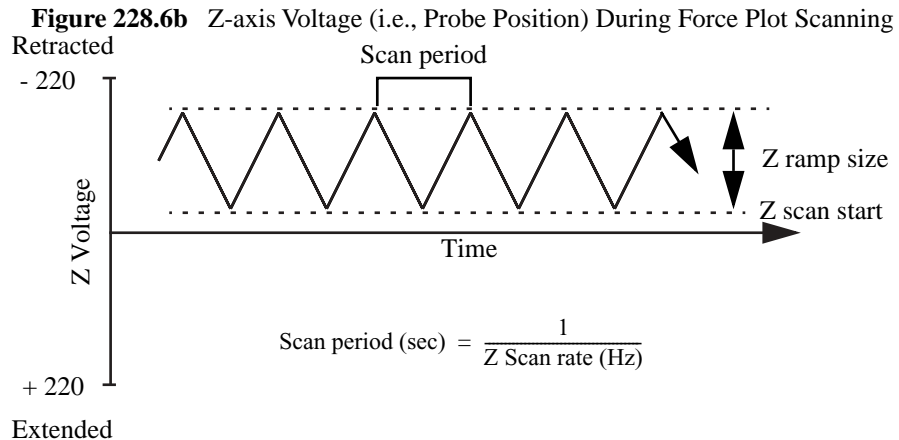
Note: Although attractive forces appear small, the tip is extremely sharp. Since only a few nanometers of the tip interact significantly with the sample, even minute forces add up, being concentrated in the small tip volume. Many materials are easily dented by the tip under such conditions. Force plots facilitate adjusting the feedback setpoint to apply minimal force to the sample during contact AFM.

Material Surface Properties

The force plot procedure highlights sample surface strength. In [Figure 6a](#), the tip is in constant contact with the sample during steps 3 through 5. The amount of cantilever flexion per unit of probe displacement downward indicates material deformability. For example, if the material is very hard, pressing the probe downward results in a relatively large amount of cantilever flexion. The cantilever flexes less during its descent into soft material. A force plot can also provide a quantitative measure of sample elasticity (refer to the example: Radmacher, *et. al.*, 1994, *Science*, Vol. 265: 1577-1579).

228.6.1 Force Plots in Contact Mode

Real-time/View/Force Mode/CALIBRATE) allows the user to quickly check the interaction between probe and sample. With this option selected, the X- and Y-piezo rastering voltages are held at zero while a triangular waveform is applied to the Z-piezoelectric actuator of the scanner tube (see [Figure 6b](#)).



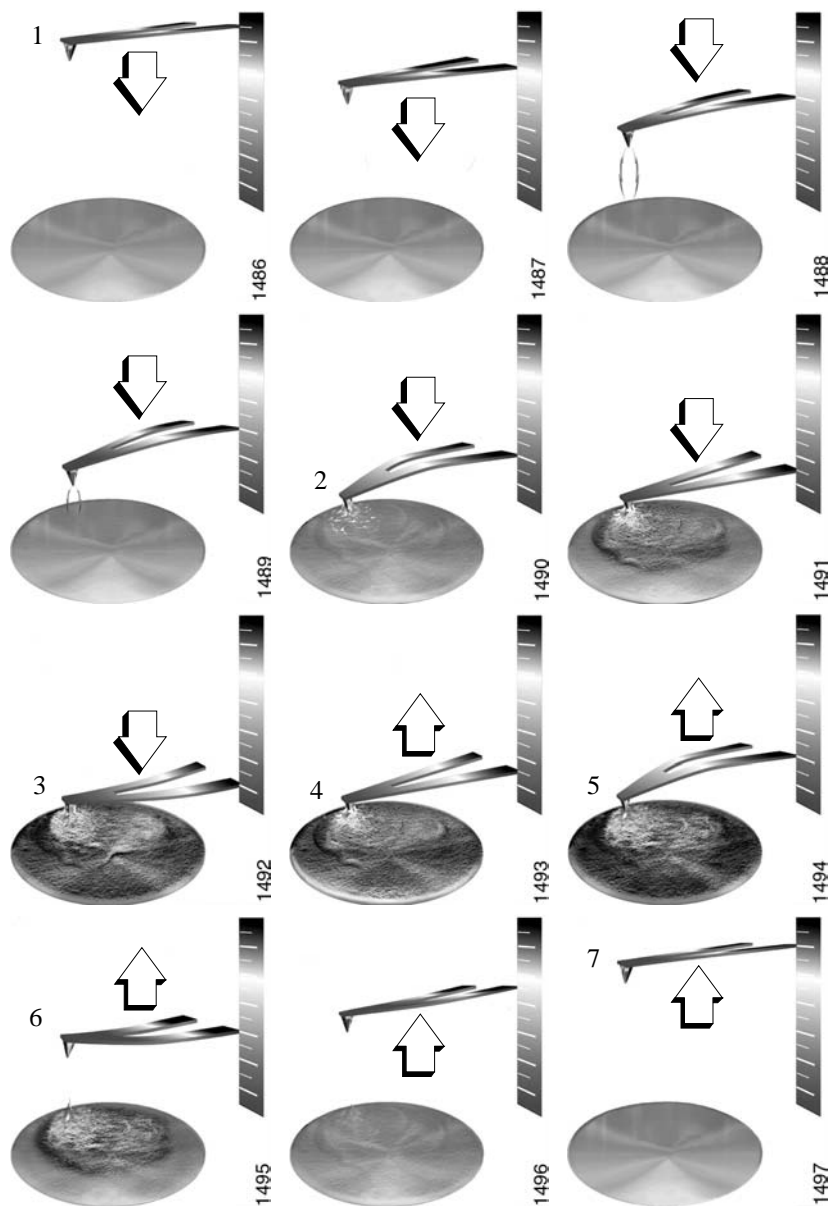
Note: The graph shown in [Figure 6b](#) is not generated in NanoScope software and is only included here for illustration.

As a result of the applied voltage, the probe tip moves up and down relative to the sample as shown in [Figure 6c](#). The **Z SCAN START** parameter records the Z-piezo position when the tip was last engaged with the sample, while the **RAMP SIZE** parameter programs the total travel distance of the piezo. Therefore, the maximum travel distance is obtained by setting the **Z SCAN START** to **+220** volts (i.e., fully extended), with the (**Z**) **RAMP SIZE** set to

440 volts (i.e., full range). Both parameters appear in the **Main Controls** panel.

Note: In the **ADVANCED** option of Force Mode, keep **RAMP CHANNEL** set to **Z** (instead of **ANALOG 1**) or **Z SCAN START** is replaced with **RAMP END**.

As the Z-piezoelectric actuator moves the probe up and down, the cantilever-deflection signal from the photodiode detector is monitored. The force plot, a graph of the cantilever deflection signal as a function of voltage applied to the scanner tube, shows on the display monitor. The control monitor displays various control panels used to control the microscope in Force Mode.

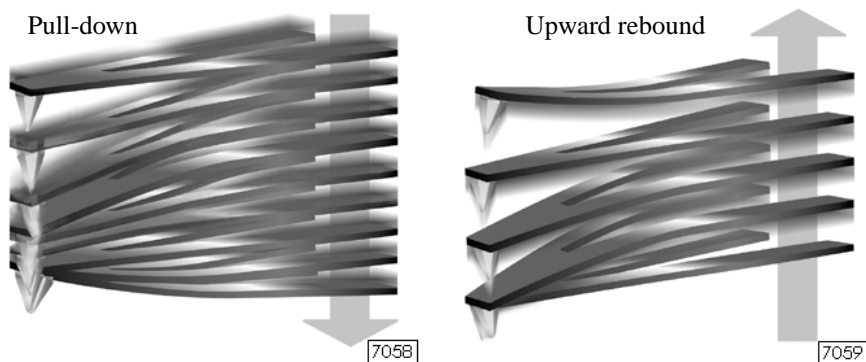
Figure 228.6c Tip-Sample Interaction during Force Calibrate

228.6.2 The Force Plot and Piezo Extension-Retraction Cycle

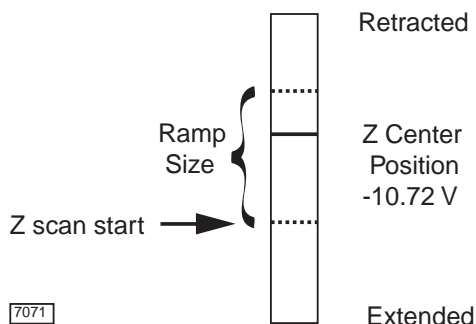
Figure 6c highlights portions of the 7-step force plot shown in **Figure 6a** with the corresponding relative positions of the tip and sample. The force plot depicts the deflection signal for each complete extension-retraction cycle of the Z-piezo actuator (see **Figure 6b**). The **Z SCAN RATE** parameter in the **Force Mode/Main Controls** panel defines the rate at which the Z-piezo completes an extension-retraction cycle (and therefore the rate at which a force plot is refreshed).

Figure 6d below represents the effects of tip pull-down due to attraction forces as the tip nears the surface (left), and the sudden, sharp rebound that results as the tip is pulled free of those forces (right).

Figure 228.6d Comparison of Pull-down and Upward Rebound.



The display monitor presents the Z piezo voltage on a linear scale from +220 volts (extended) to -220 volts (retracted). The range of piezo travel is represented by two white lines on the scale. The lower white line corresponds to **Z SCAN START**, while the spacing between the lines corresponds to **(Z) RAMP SIZE**. The average voltage applied to the Z-piezo tube just prior to entering **Force Mode** is represented by **Z Center Position** on the display monitor scale (see **Figure 6e**).

Figure 228.6e Z Scan Indicator on the Display Monitor

Note: In NanoScope software version 4.23 and later, the tip is retracted by the **SCAN SIZE** above the surface when first entering the force modes of **CALIBRATE, ADVANCED, INDENT, SCRATCH** or **VOLUME**. If using **STEP**, the tip will retract to the limit of **-220V**. 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.

228.6.3 Troubleshooting Contact AFM Force Measurements

To minimize or calculate the contact force between the tip and sample, it is important to obtain a good force plot. This procedure is described in detail in your product *Instruction Manual*.

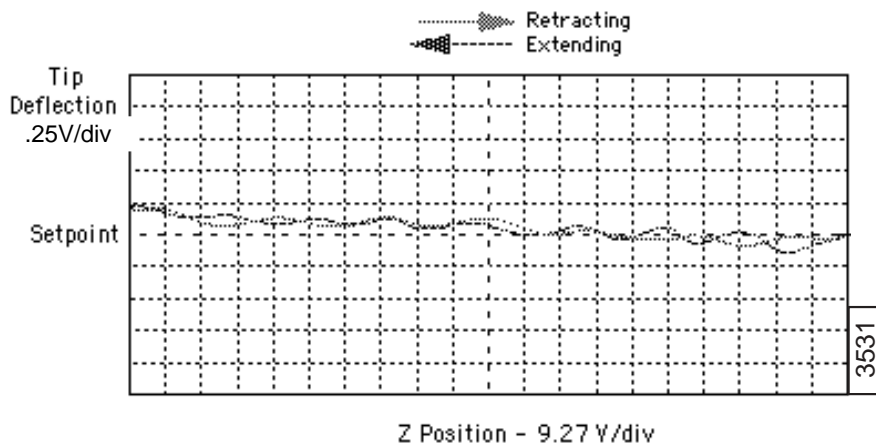
False Engagement

Figure 6f illustrates a force plot 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 **Force Mode/Motor/TIP DOWN** button can be used to move the tip down to the surface, there are other ways to correct a false engagement: 1) increase the setpoint and engage again; or 2) adjust the photodetector positioner to make the top/bottom differential voltage more negative, then engage the tip again. These have the effect of pushing the cantilever farther up before the setpoint is reached. Refer to your product *Instruction Manual* for troubleshooting tips on false engagement.

Figure 228.6f False Engagement (“J” Scanner)



Helpful Suggestions for Working with Force Measurements

The **TIP UP** and **TIP DOWN** buttons of the **Motor Controls** panel, which is opened by selecting **Force Mode/Motor/Step Motor**, provide coarse adjustment of the **Z SCAN START** voltage. With these buttons the SPM head, including probe and scanner, are moved vertically (in much the same way that probe position is adjusted with the **Z SCAN START** parameter).

Note: Use the **Step Motor** function 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

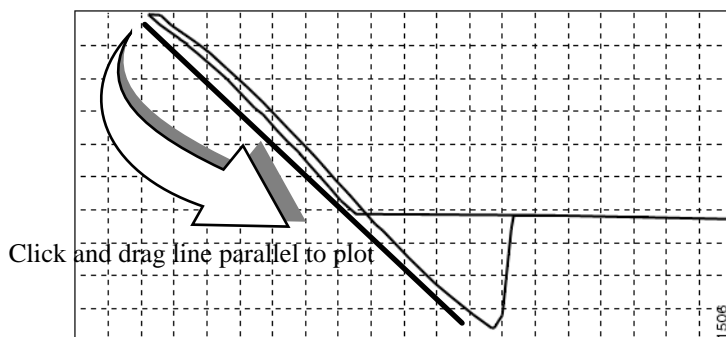
the Z-axis leadscrew has some backlash, it may be necessary to rotate the screw several turns by clicking on the **TIP UP** or **TIP DOWN** buttons before movement is obtained.

The photodiode positioner can be used as a coarse adjustment for **Setpoint** voltage. Changing the beam position on the photodiode by rotating the mirror adjustment knob shifts the force plot on the graph. Moving the photodiode down by rotating the positioner counter-clockwise 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.

228.6.4 Sensitivity Determination

“Sensitivity” describes the ratio of cantilever response to Z-axis actuation. Initially this is a ratio of voltages: the cantilever deflection signal versus the voltage applied to the Z-piezo. To convert these voltages to travel distances, the **SENSITIVITY** parameter is set in **Force Mode**. Once sensitivity is thus calibrated, tip deflection can be read in nanometers. Sensitivity is equal to *the slope of the force plot while the cantilever is in contact with the sample surface*. Complete the following steps to set the **SENSITIVITY**:

- Obtain a good force plot on the display monitor.
- Position the cursor in the vicinity of one end of the contact portion of the curve.
- Click on the left mouse button to fix one endpoint of a line segment.
- Drag the mouse to position the line segment parallel to the contact portion of the force plot (see [Figure 6g](#)).

Figure 228.6g Use the Mouse to Set the Sensitivity Parameter

- The second click of the left mouse button causes the system to calculate the slope of the line segment and enter the calculated value as the Sensitivity.
- A click of the right mouse button removes the line segment from the screen.

Note: **SENSITIVITY** is expressed either as the photodiode voltage versus the distance traveled by the piezo, or as the photodiode voltage versus the voltage applied to the piezo, depending on the setting of the **UNITS** parameter (**VOLTS** or **METRIC**). Typical values of -10 to -50mV/nm for the negative slope are reported in absolute value, by convention.

If **SENSITIVITY** is calibrated on a material much stiffer than the cantilever, it measures the AFM optical lever sensitivity (i.e., how many volts of deflection signal are produced by a given deflection of the cantilever tip). The sensitivity 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.

CAUTION: It is important to calibrate the **SENSITIVITY** parameter on a hard substrate as described here **BEFORE** using the force plot vertical scale for quantitative measurements.



Note: To ensure accuracy, **SENSITIVITY** should be recalculated each time the laser spot position on the undeflected cantilever or its resulting reflected light position on the photodiode changes.

228.6.5 Force Minimization

Force Calibrate allows the contact force of the probe on the sample surface to be minimized. **SETPOINT** determines the nominal deflection of the cantilever and, therefore, the nominal force applied by the cantilever during data collection.

Operating the SPM below the point of zero deflection of the cantilever minimizes the contact force of the probe on the sample. Negative deflection results from the probe tip being attracted to the surface. However, the engagement process requires a setpoint which is greater than the voltage at zero deflection. Nonetheless, the operating point can be changed after the probe has been engaged.

The setpoint can be adjusted while viewing the force plot. The setpoint is usually made more negative so that it lies between the horizontal segment of the force plot, which corresponds to the zero deflection point, and the minimum of the retraction scan where the probe pulls off the sample surface, V_{CSmin} . The contact force is minimized when V_{CSmin} is on the centerline of the deflection-signal axis. In practice, V_{CSmin} is preferred a little below the centerline since V_{CSmin} is the point where the cantilever pulls off the surface and operation at this deflection is unstable.

Changes to **Feedback Controls/SETPOINT**, whether in Force Mode or Image Mode, are preserved in switching to the other mode. After exiting Force Mode, if the image looks good, the tip force can be decreased further by lowering the **SETPOINT** in small increments until the probe pulls off. Resetting the **SETPOINT** to a value larger than the full-range of the display recaptures the probe. (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 probe retracts provides a low contact force.

If a high initial contact force will adversely affect the sample, engage the probe with a very small scan size. Then minimize the force while the tip is confined to a small area of the sample (which *will* experience the relatively high initial engagement force). Once the force is minimized, increase the **SCAN SIZE** and/or offset the scan to a different area of the sample.

Note: 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.

Note: In Contact, **Force Mode**, **AMPLITUDE SETPOINT** defines the centerline of the deflection (vertical) axis of force plots.

228.6.6 Calculating Contact Force

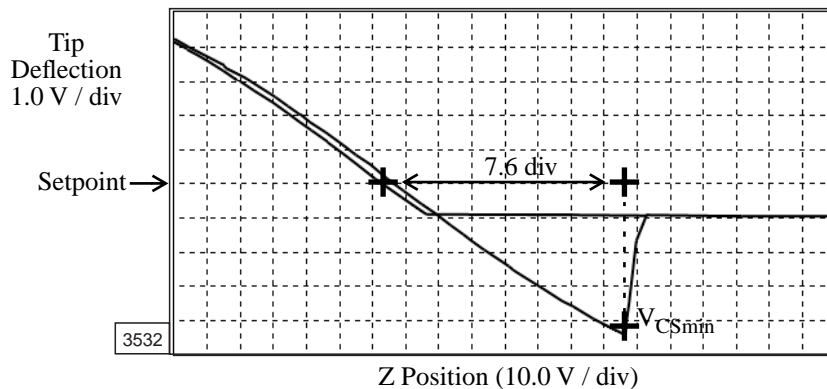
Given the spring constant, k , of the cantilever, contact force is calculated at the setpoint - the value of the deflection signal maintained by the feedback control loop. Contact force is defined by the cantilever spring equation:

$$F = k(\Delta Z)$$

where ΔZ is the Z distance from the control point to V_{CSmin} in nanometers.

An example of how to compute contact force from a **Force Calibrate** graph is shown in [Figure 6h](#).

Figure 228.6h Computing Contact Force



Assume **SENSITIVITY** has been calibrated (see [Section 6.4](#)) and has the value of 12 nm/V. The 7.6 horizontal divisions spanned by the z-piezo actuator between the setpoint and the rebound of the tip from the sample at deflection voltage V_{CSmin} are converted into travel distance, ΔZ , using the horizontal scale and the sensitivity of the force plot:

$$\Delta Z = (7.6 \text{ div})(10.0\text{V/div})(12\text{nm/V}) = 912 \text{ nm}$$

Given a cantilever spring constant of $k = 0.6\text{N/m}$, the tip contact force is calculated from the cantilever spring equation:

$$F = (0.6\text{N/m})(912\text{nm}) = 547.2 \text{ nN}.$$

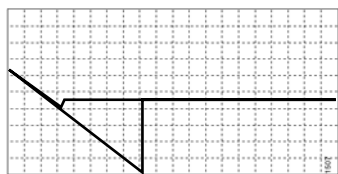
When the **DATA TYPE** is set to **HEIGHT** with the feedback gains set high, the tip tracks the sample surface with nearly constant cantilever deflection, so similarly constant contact force over the entire scan area.

Contact force determination is not as straightforward on images captured with the **DATA TYPE** set to **DEFLECTION**. When collecting deflection data, the feedback gains are set low so the sample stays at a constant Z-axis separation from the probe 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 as before from the cantilever spring equation. The force applied at other points on the sample is calculated relative to the nominal force by using deflection data and the spring-constant of the cantilever.

Note: **SENSITIVITY** must have been previously determined for both the nominal and deflected contact force calculations to be accurate.

228.6.7 Interpreting Force Plots

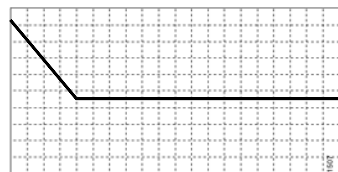
Force plot features correlate with surface characteristics such as adhesion, hardness and boundary layer forces as illustrated in [Figure 6i](#). For this reason, user interpretation enters into determining **SENSITIVITY** (see [Figure 6g](#) in [Section 6.4](#)).

Figure 228.6i Force Plot Examples

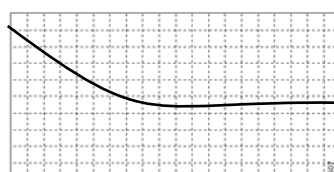
Large adhesion



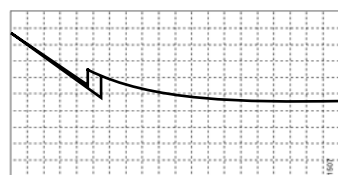
Small adhesion



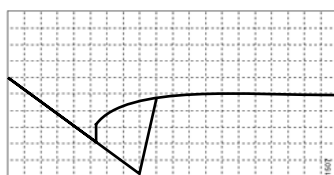
Hard sample



Soft sample



Long-range repulsion



Long-range attraction

228.7 TappingMode Force Measurements



CAUTION:

Because TappingMode cantilevers are relatively stiff, **Force Calibrate** can potentially damage the tip and/or surface. *Before using **Force Calibrate**, the user should read and understand this section.*

Force plots, basic or advanced, provide a representation 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 tip deflection, phase, or root-mean-square (RMS) amplitude. Force plots are available in two forms: **Force Plot** and **Force Volume**. **Force Volume** consists of an array of force plots. To produce high-quality force plots, it is necessary to very precisely control the tip's position relative to the surface.

228.7.1 Force Measurements

Commencing **Force Plot** in TappingMode, the probe moves to the center of the current XY scan, then XY scan motion is turned off. Next, a triangular waveform is applied to the Z-axis piezoelectric actuator (see [Figure 6b](#)), just as in a contact AFM force plot. In TappingMode, however, the force plot is a graph of the oscillating tip amplitude, phase, or deflection versus the probe extension along the Z-axis.

[Figure 7a](#) represents tip-sample dynamics for a MultiMode™, scanned sample SPM, system and [Figure 7b](#) is a similar representation for Dimension™ Series systems, which are scanned tip SPMs. The Z-axis piezoelectric actuator positions the sample just below the tip (at the **Z SCAN START**), then extends a known distance farther from the tip (the **Z SCAN SIZE**), whether by moving the sample (e.g., MultiMode) or the probe (e.g., Dimension). If the oscillating tip encounters any surface forces, the oscillation amplitude or phase are altered.

Figure 228.7a Relationship of Z scan start and Z scan size for MultiMode SPMs

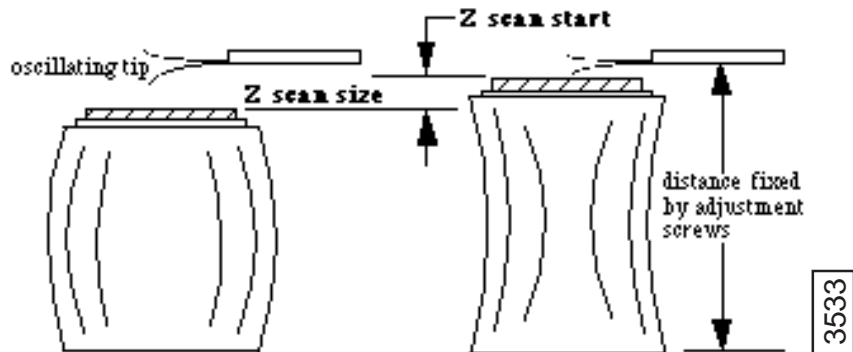
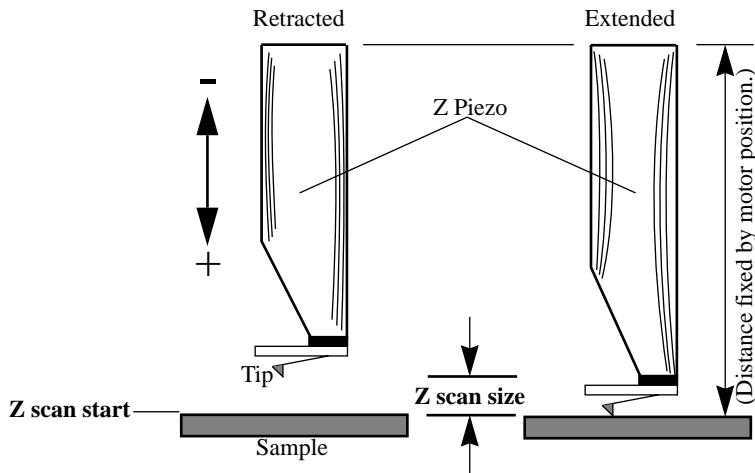
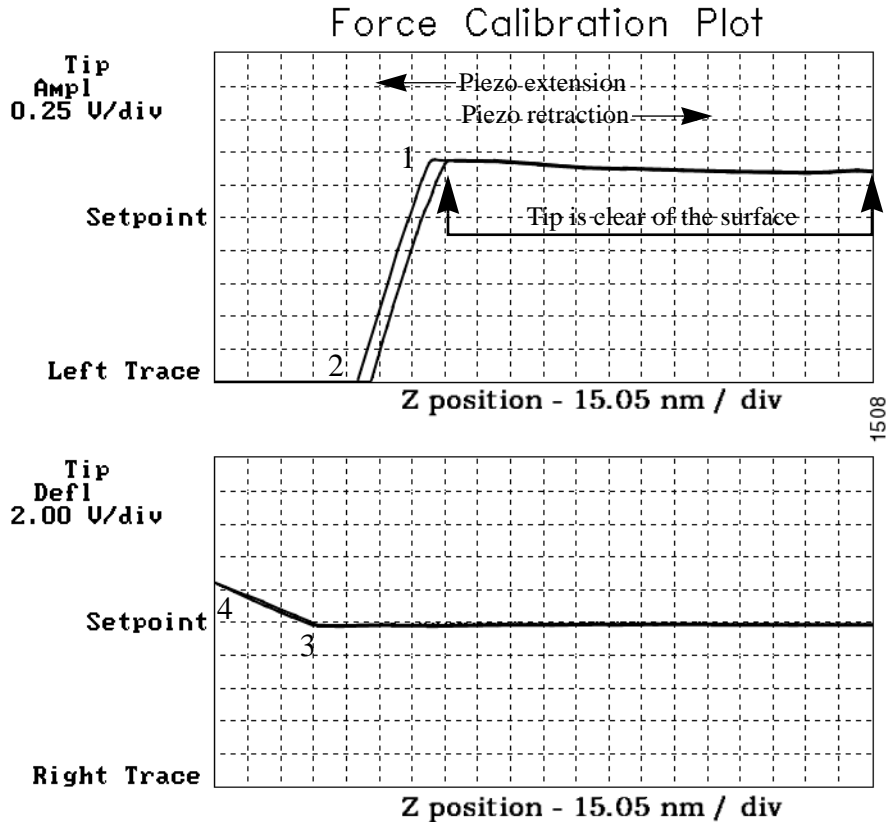


Figure 228.7b Relationship of Z scan start and Z scan size for Dimension SPMs

A TappingMode **Force Plot** is useful in characterizing boundary layer forces on the probe tip, optimizing SPM performance, and calibrating the phase, deflection or RMS amplitude of cantilever oscillation as a function of tip-sample distance (see [Figure 7c](#)). Cantilever RMS amplitude decreases as the tip approaches the sample. The plot represents the RMS amplitude for one complete Z-axis extension-retraction cycle. **Z SCAN RATE** defines the rate at which the piezo completes an extension-retraction cycle, therefore, the rate at which new curves are displayed. Click **Real-time/View/Force Calibrate** to open the **Main Controls** and find **Z SCAN RATE**.

Figure 228.7c Force Plot of RMS Amplitude (top) and Deflection (bottom) versus Z-axis Extension



Between points 1 and 2 in [Figure 7c](#), cantilever oscillation amplitude is damped by approximately 1.75 volts when the tip advances approximately 30 nm closer to the surface. TappingMode **SENSITIVITY** (in the **Main Controls** panel) is computed by dividing the change in RMS amplitude by the change in probe Z-position. Calibrate **SENSITIVITY** by drawing a line parallel to the RMS amplitude plot slope between points 1 and 2 in the same way as was done for contact AFM sensitivity (see [Figure 6g](#)).

The deflection signal ([Figure 7c](#), bottom) has been low-pass filtered to eliminate the high frequency TappingMode oscillation. The average cantilever position, or deflection, is unchanged as the RMS amplitude declines. The cantilever deflects (between points 3 and 4 in [Figure 7c](#)) once the RMS amplitude has been damped to zero by mechanical-acoustic coupling, and finally contact, with the sample surface. A single crystal silicon TappingMode tip is easily broken if deflected against a hard surface.

228.7.2 Obtaining a Force Plot of a Calibration Reference in TappingMode

**CAUTION:**

It is easy to blunt a stiff TappingMode probe tip with excess contact force during a **Force Plot** measurement. If the RMS amplitude is reduced to zero, the tip has almost certainly been blunted or broken, and/or the sample has sustained damage. Damage can be avoided by using triggers ([Section 7.3](#)) and/or by reducing the value of **Z SCAN START** so that RMS amplitude never declines to zero. Rapidly increasing the value of **Z SCAN START** is dangerous since the total vibrational amplitude of the cantilever is small relative to total Z travel. If scanner extension continues farther than point 2 in [Figure 7c](#) and produces a horizontal trace (i.e., no change in oscillation amplitude), then the probe is likely to have broken.

**CAUTION:**

Too high a **DRIVE AMPLITUDE** can fracture a cantilever. Start with a small value and increase incrementally. If RMS amplitude does not increase in response, the tip is probably pressed into the sample surface and should be withdrawn before proceeding.

When obtaining force plots in TappingMode, set up scan parameters so that the reduction of amplitude is approximately 25 percent of the free air amplitude. The amplitude **SETPOINT** defines the centerline of the vertical Tip Amplitude axis of a TappingMode force plot (as well as being the feedback control parameter).

To make a TappingMode force plot of a silicon calibration reference, complete the following

1. Verify the probe holder is fitted with a TappingMode probe. Mount the calibration reference on the SPM stage or sample puck. Set the **AFM MODE** parameter to **TAPPING** and obtain a TappingMode image. You are now in **Image mode**.
2. To switch to the **Force Calibrate** panel, click on **Real Time/View/Force Mode/ADVANCED**. At least three panels should be visible: **Main Controls**, **Feedback Controls**, plus at least one channel panel (e.g., **Channel 1**); (see [Figure 7d](#)).

Note: 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.

Note: Click on **Real Time/View/Force Mode/CALIBRATE** to open the **Main Controls** control panel only (see [Figure 7e](#)).

Figure 228.7d The Advanced Force Calibrate Window

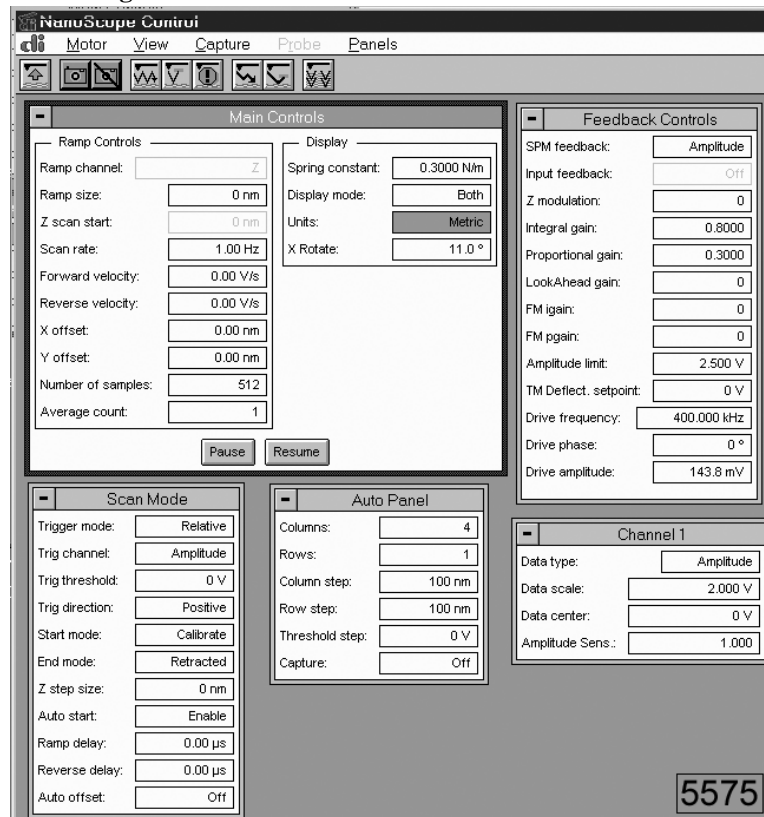


Figure 228.7e The Basic Force Calibrate Window

The screenshot shows a software window titled "Main Controls" and "Channel 1".

Main Controls

- Ramp Controls:** Ramp size: 0 V, Z scan start: 0 V, Scan rate: 1.00 Hz, Number of samples: 512.
- Display:** Units: Volts, TM Deflect. setpoint: 0 V.

Channel 1

- Data type:** Amplitude (selected).
- Data scale:** 2.000 V.
- Data center:** 0 V.
- Amplitude Sens.:** 1.000.

A digital display in the bottom right corner shows the value 5576.

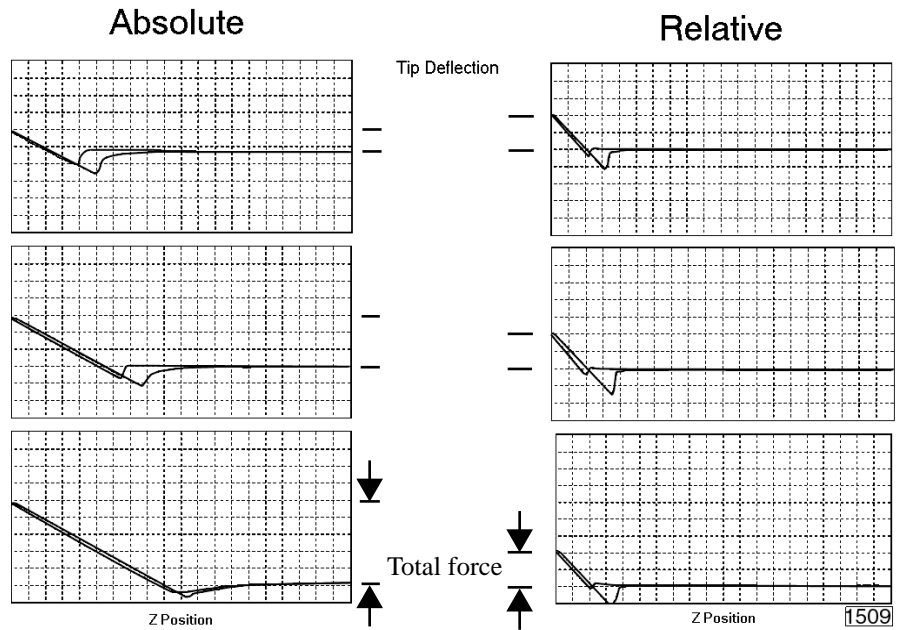
3. Set the **Main Controls**, **Feedback Controls** and **Channel** panel parameters.
4. Set the **Channel 1/DATA TYPE** parameter to **AMPLITUDE**.

Note: The **Z SCAN START** parameter may be adjusted using the left-right arrow keys.

228.7.3 Triggering

The **Scan Mode** panel within the **Force Calibrate Advanced** window provides triggers to limit the force exerted by the tip on the sample when making **Force Plot** measurements. A **Relative** trigger maintains force at a constant level defined by **TRIG THRESHOLD**. An **Absolute** trigger limits force using the **SETPOINT**. [Figure 7f](#) illustrates force build-up and limiting for each trigger type in the case of thermal or mechanical drift that decreases tip-sample separation.







Figure 228.7f Reaction of Absolute and Relative Triggers to Drift



228.8 Introduction to Force Volume

Force plots generated at regular intervals on a sample surface are characterized as force volume imaging. For each X-Y position in the sampling array, cantilever response is plotted as a function of Z-axis travel.

Force volume images require preliminary use of the **Force Measurement** function (see [Section 6](#) for Contact Mode force measurements, and [Section 7](#) for TappingMode force measurements). The matrix below summarizes the major force imaging types.

Signal Type	Contact AFM	TappingMode
Amplitude		
Deflection		
Phase ^a		
Frequency ^b		
Potential ^b		

- a. Phase, frequency and potential measurements are available only on SPMs equipped with the Extender™ Electronics Module or controlled by a NanoScope IV controller.

The type of force image captured from a surface will depend upon how the SPM is set up. For example:

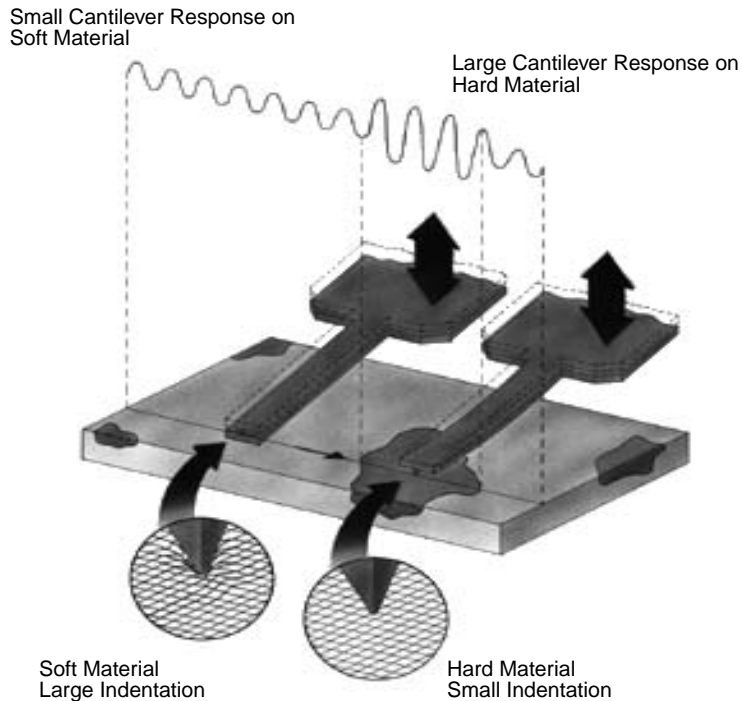
- If a MFM image is being captured, force volume imaging (phase) allows the detection of long-range magnetic forces otherwise difficult to observe.
- For ordinary contact AFM, force volume imaging (deflection) maps electrostatic forces.

For more detail on Force Volume functions, refer to Support Note 240, *Force Volume*.

228.9 Introduction to Force Modulation

Sample elasticity is measured by oscillating a probe such that its tip slightly indents a sample. Soft materials indent more easily than hard samples. Cantilever deflection is inversely related to indentation. Soft samples themselves bend instead of the cantilever; hard samples deflect the cantilever more than the sample. The elasticity of a sample is inferred from tip deflection (see [Figure 9a](#)). Force modulation results from the variation in tip deflection with elasticity while scanning a sample of variable hardness.

Figure 228.9a Contrast Generation in Force Modulation Mode



For more detail on Force Modulation functions, refer to Support Note 310, *Force Modulation*.

