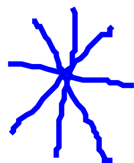

Support Note No. 231, Rev. C

Electric Force Microscopy (EFM)

Applicable to MultiMode™ Systems

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231.1 Electric Force Microscopy Overview

This support note describes how to perform electric force microscopy (EFM) imaging on a MultiMode™ scanning probe microscopy (SPM) system.[1] Similar to magnetic force microscopy (MFM)—and sharing many of its procedural techniques—this mode utilizes the Interleave™ and LiftMode™ procedures discussed in the product instruction manual. (The MFM techniques can be found in Support Note #229. Contact Digital Instruments for a copy of this document.).

All standard MultiMode SPMs are capable of electric field gradient imaging using amplitude detection techniques. By adding an Extender™ electronics module (see Figure 231-1), frequency modulation or phase detection can be used, giving improved results. Amplitude detection has largely been superseded by frequency modulation and phase detection. The Extender is required for surface potential imaging, and is strongly recommended for electric field gradient imaging.

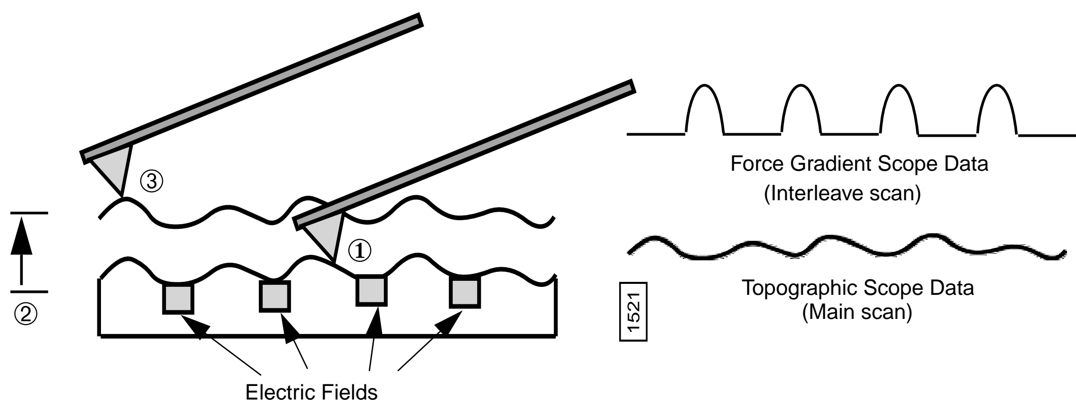
A special EFM cantilever holder is needed for applying a voltage to the cantilever. This is required hardware for surface potential imaging. Depending on the sample of interest, electric field gradient imaging may or may not require the EFM cantilever holder.

Figure 231-1 Extender™ Electronics Module—required for frequency phase detection MFM and EFM.

Two types of electric force microscopy are available using the MultiMode SPM system: 1) **electric field gradient imaging** and, 2) **surface potential imaging**.

1. Support Note 230 provides information on EFM for Dimension™ series systems.

Each of the electric field measurement techniques is based on a two-pass LiftMode measurement. LiftMode allows the imaging of relatively weak but long-range magnetic and electric interactions while minimizing the influence of topography (see Figure 231-2). 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 above the surface (to the lift scan height), and a second trace and retrace are performed while maintaining a constant separation between the tip and local surface topography. Both methods of electric force measurement are explained in this support note.



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 231-2 EFM LiftMode Principles

231.1.1 Electric Field Gradient Imaging Overview

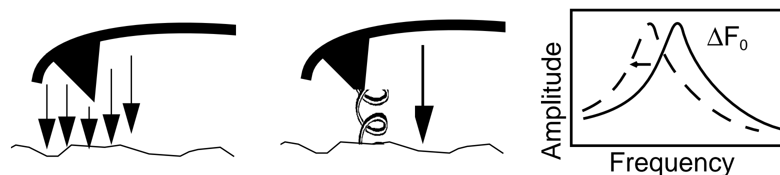
Electric field gradient imaging is a technique which measures variations in the electric field gradient above a sample. The sample may be conducting, nonconducting, or mixed. The electric field gradient can be generated by applied voltages or trapped charges. Since the electric field gradient is also shaped by the surface topography (e.g., sharp points on the surface concentrate the field gradient), large differences in topography can influence electric field variations. In general, the best samples for electric field gradient imaging have fairly smooth topography.

231.1.2 Surface Potential Imaging Overview

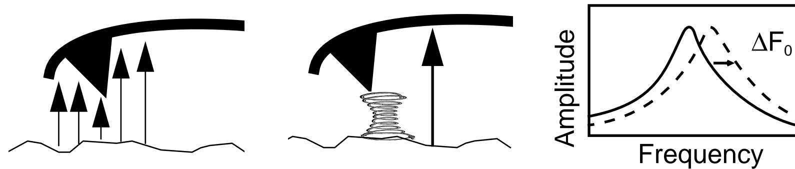
Surface potential imaging measures the effective surface voltage of the sample by adjusting the voltage on the tip so that it feels a minimum electric force from the sample. (In this state, the voltage on the tip and sample is the same.) Samples for surface potential measurements should have an equivalent surface voltage between ±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 also show contrast due to contact potential differences. Quantitative measurements can be made of the relative voltages within a single image. Immurements of absolute voltage can be challenging due to DC shifts and software limitations. Smooth samples work best in surface potential imaging, though topography artifacts tend to be smaller than in electric field gradient imaging. *This method requires the Extender Electronics Module and version 3.1 or later of the NanoScope III software*.

231.2 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 near its resonant frequency by a small piezoelectric element. 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.



Attractive gradient equivalent to additional spring in tension attached to tip. This counteracts the spring force of the cantilever, reducing the effective resonant frequency of the cantilever.



Repulsive gradient equivalent to additional spring in compression attached to tip. This adds to the spring force of the cantilever, increasing the effective resonant frequency of the cantilever.

Figure 231-3 Comparison of Attractive and Repulsive Forces to Action of a Taught Spring Attached to the Tip

Changes in cantilever resonant frequency can be detected in 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 231-4 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 V or more). For samples having rough surface topography or small voltage variations, this technique may be undesirable because topographic features appear in the LiftMode image.

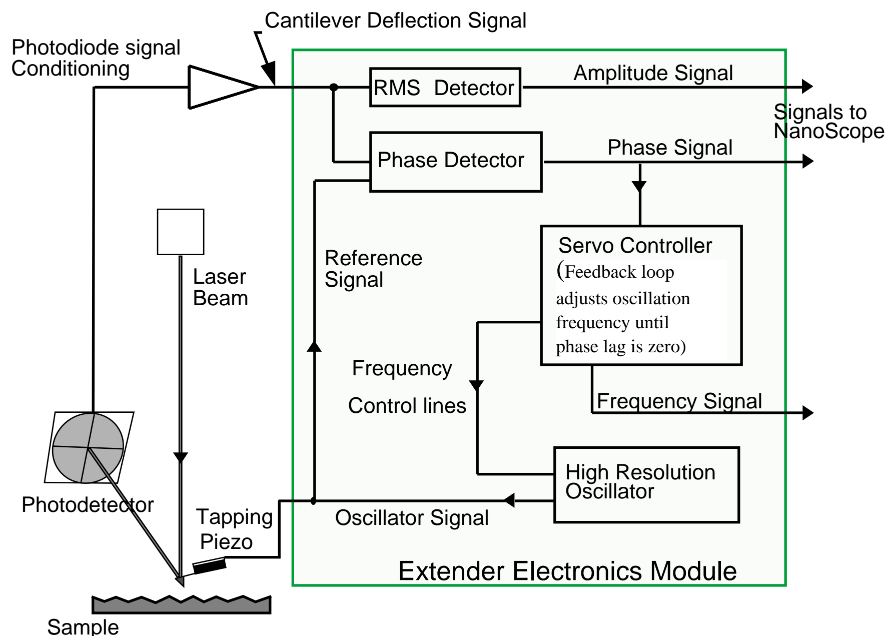


Figure 231-4 Diagram of Extender Electronics Module in Phase and Frequency Measurement Mode

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 also varies in much the same way as does the surface topography. Thus, electric field gradient LiftMode images measured by amplitude, phase or frequency detection often show contrast that is very similar to the surface topography. Samples with rough surface may be more successfully imaged by the surface potential measurement method described below.

Note: In many 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.

231.3 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 231.4.

Note: 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 on the microscope's baseplate and to the toggle switches on the Extender Electronics Module (if equipped). Jumper configurations are dependent on the type of microscope being used and the measurements desired. Section 231.3.1 provides jumper configuration instructions for basic microscope models operating *without the Extender Electronics Module* (see Jumper Configurations, Section 231.3.1.1 through Section 231.3.1.4). Section 231.3.2 provides jumper configuration instructions for basic microscope models operating *with the Extender Electronics Module* (see Jumper Configurations, Section 231.3.2.1 through Section 231.3.2.4.)

The location and orientation of the jumpers in the baseplate of the MultiMode is shown in Figure 231-5. 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.

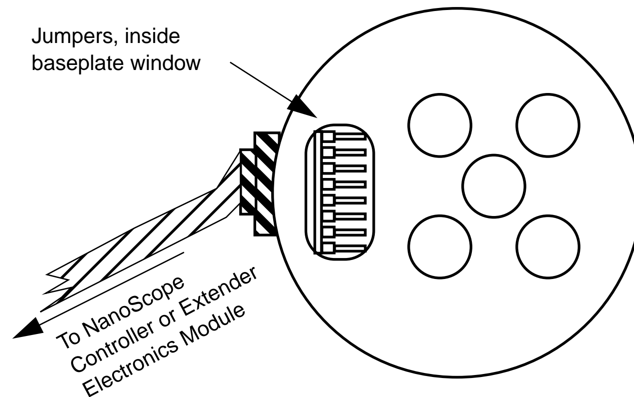


Figure 231-5 Diagram of MultiMode Baseplate Showing Location and Orientation of Jumpers

1. Carefully examine the following figures and identify which jumper configuration is appropriate for your application. The internal power supply, Analog 2, has a range of approximately ±12 V and can be connected to the tip or piezo cap. The piezo cap is the metal that covers the top of the scanner on which the sample puck is placed. External power supplies can be connected to the tip or piezo cap through the jumpers in the base of the MultiMode. An external power supply may also be attached directly to the sample. Make sure the sample is electrically isolated from the piezo cap.
2. Power down the NanoScope III controller. Unplug the cable from the base of the MultiMode.
3. Disconnect and remove the microscope head and the scanner.
4. Carefully tilt the MultiMode so that the baseplate is oriented as shown in Figure 231-5. Locate the jumpers. As shipped from the factory, jumpers on systems *without* the Extender Electronics Module should appear as shown in Figure 231-7, whereas jumpers on systems *with* the Extender Electronics Module should appear as in Figure 231-12.
5. Change the position of the jumpers to match the configuration you have selected.
6. After the jumpers are correctly configured, reconnect the cable to the base of the MultiMode. Replace and reconnect the scanner and head. Switch the Nanoscope controller power back on.

7. If the jumpers are configured to use the internal voltage source Analog 2, click on **di** / **Microscope Select** / **Edit** / **Advanced**. Set Analog 2 to **User defined**. Make sure to click OK to exit both dialog boxes. *Remember to reset Analog 2 to **Atten switch** when finished.* (For all other configurations Analog 2 should be left on **Atten switch**.)

   *For versions 4.23 and lower: If the jumpers are configured to use the internal voltage source Analog 2, click on the **Microscope / Calibrate / Detector** option to display the **Detectors Parameters** window. Switch the **Allow in attenuation** field to **Disallow**. Remember to set **Allow in attenuation** to **Allow** when finished. (For all other configurations it should be left on **Allow**.)*

8. If using the internal supply Analog 2 and the system has an Extender Electronics Module, flip the switch on the Extender box to Analog 2 as shown in Figure 231-6. *Remember to return this switch to the **Gnd/Surface Potential** position when finished.*

   Locate the two toggle switches on the backside of the Extender Electronics box (Figure 231-6), then verify that they are toggled as shown in Table 231-1.

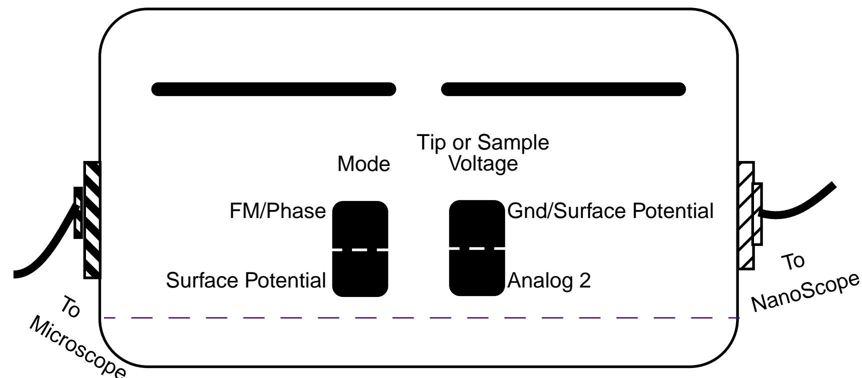


Figure 231-6 Toggle Switches on Back of Extender Electronics Module

	Mode		**Tip or Sample Voltage**	
	FM/Phase	**Surface Potential**[a]	**GND/ Surface Potential**	**Analog 2**
Electric field gradient imaging with Analog 2 connected to tip or sample	**X**			**X**
Electric field gradient imaging in all other configurations	**X**		**X**	

Table 231-1 Extender Electronics Module Toggle Switch Settings

a.**Note:** The toggle switch combination of Surface Potential = ON and Analog 2 = ON is **not recommended** and can produce erratic and undefined results.

9. Imaging can now be accomplished using the procedures in Section 231.4.

231.3.1 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 231-7.

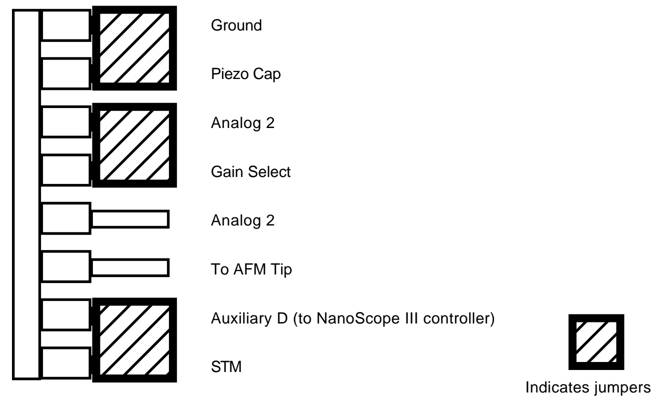


Figure 231-7 Normal Jumper Configuration for Systems *Without* the Extender Electronics Module as Shipped from Factory

231.3.1.1 Field Gradient Imaging—Voltage Applied to the Tip

The jumper configuration in Figure 231-8 connects the Analog 2 signal from the NanoScope III controller (± 12 V range) to the tip.

Note: In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

Remember to enable Analog 2 in the software.

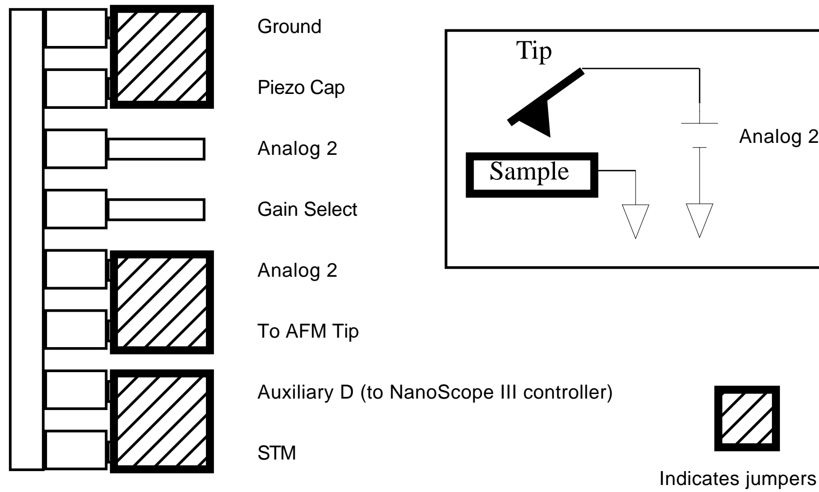


Figure 231-8 Jumper Configuration for Application of Voltage to Tip (For Systems *Without* the Extender Electronics Module)

231.3.1.2 Field Gradient Imaging—Voltage Applied to the Sample

The jumper configuration in Figure 231-9 connects the Analog 2 signal from the NanoScope III controller (± 12 V range) to the sample chuck.

Remember to enable Analog 2 in the software.

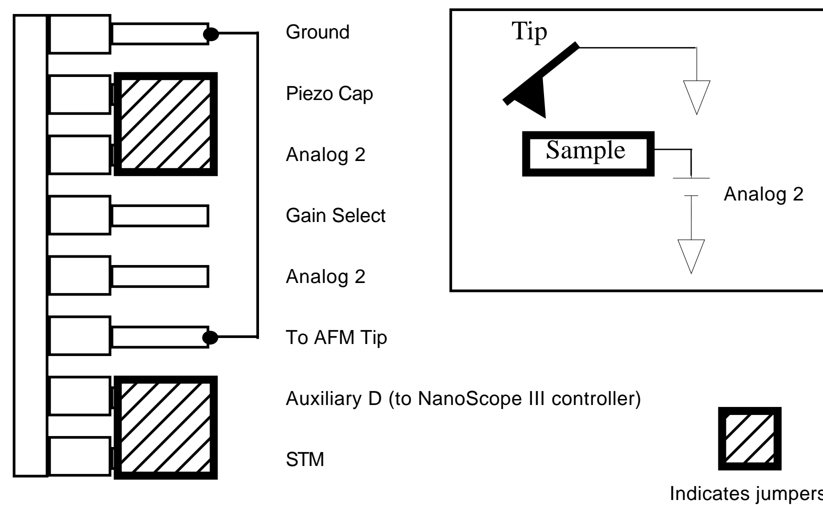


Figure 231-9 Jumper Configuration for Application of Voltage to Sample (For Systems *Without* the Extender Electronics Module)

231.3.1.3 Field Gradient Imaging—External Voltage Source Applied to the Tip

In some cases, it may be advantageous to use voltages greater than 12 V, or to use a pulsed power supply. If an external source of voltage is to be applied to the tip, configure jumpers as shown in Figure 231-10.

Note: In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

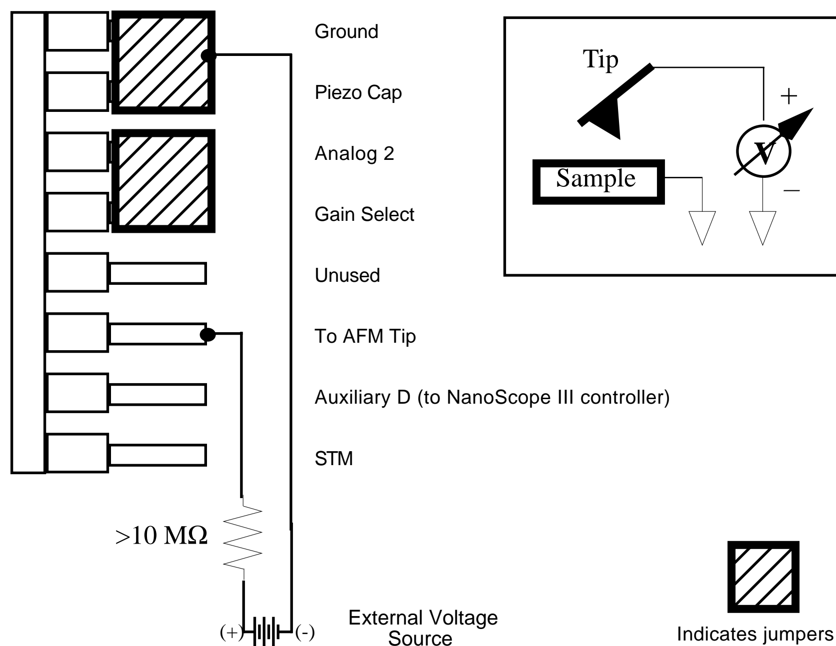


Figure 231-10 Jumper Configuration for Applying External Voltage to Tip (For Systems *Without* the Extender Electronics Module)

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 the jumper pins using soldered, push-on connectors. *Do not* solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult. The connection to ground can also be made to the AFM chassis or externally.

231.3.1.4 Field Gradient Imaging—External Voltage Source Applied to the Sample

In some cases, it may be advantageous to use voltages greater than 12 V, or to use a pulsed power supply. If an external source of voltage is to be applied to the sample, configure jumpers as shown in Figure 231-11.

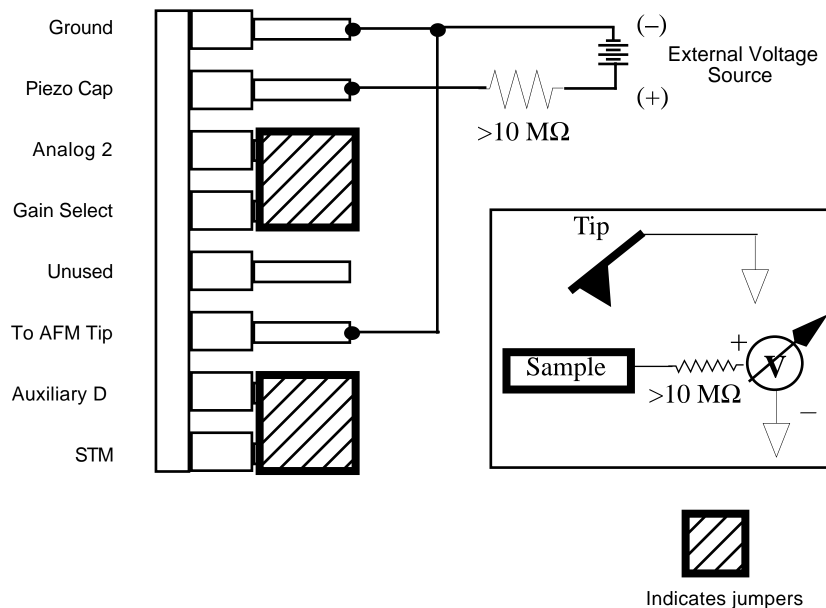


Figure 231-11 Jumper Configuration for Applying External Voltage to Sample (For Systems *Without* the Extender Electronics Module)

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 the jumper pins using soldered, push-on connectors. *Do not* solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

231.3.2 Jumper Configurations For Microscopes *With* the Extender Electronics Module

Reminder: Power down the controller and unplug the cable from the base of the MultiMode.

As shipped from the factory, systems *with* the Extender Electronics option should have an original jumper configuration as shown in Figure 231-12.

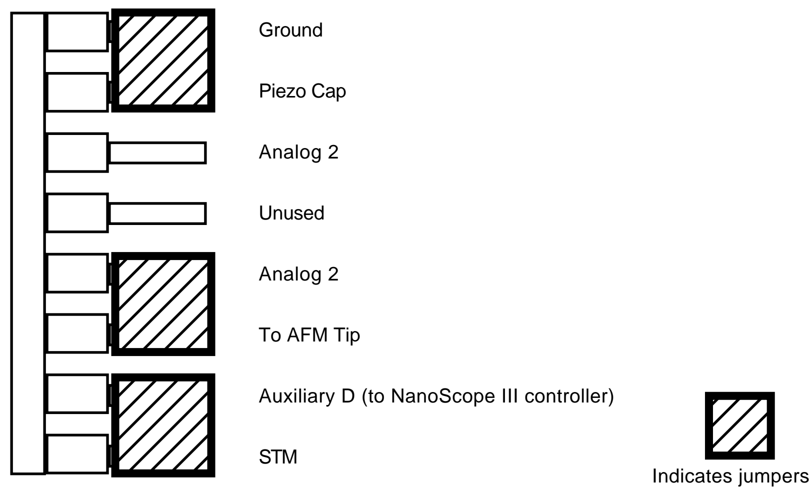


Figure 231-12 Normal Jumper Configuration as Shipped from Factory for MultiMode SPM *With* Extender Electronics Module Installed.

231.3.2.1 Field Gradient Imaging—Voltage Applied to the Tip

Notice that the jumper configuration in Figure 231-13 connects the Analog 2 signal from the NanoScope III controller (± 12 V range) to the tip, and is **exactly the same** as the jumper configuration shown in Figure 231-12, the standard configuration as shipped from the factory.

Note: In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

Remember to enable Analog 2 in the software and to set the Extender box switch to Analog 2.

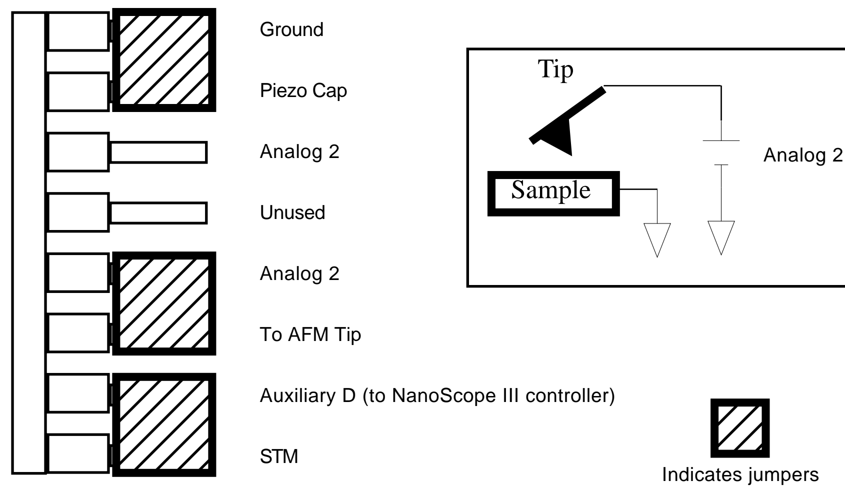


Figure 231-13 Jumper Configuration for Application of Voltage to Tip—Standard Configuration of Systems *With* the Extender Electronics Module as Shipped from Factory

231.3.2.2 Field Gradient Imaging—Voltage Applied to the Sample

The jumper configuration in Figure 231-14 connects the Analog 2 signal from the NanoScope III controller (± 12 V range) to the sample.

Remember to enable Analog 2 in the software and to set the Extender box switch to Analog 2.

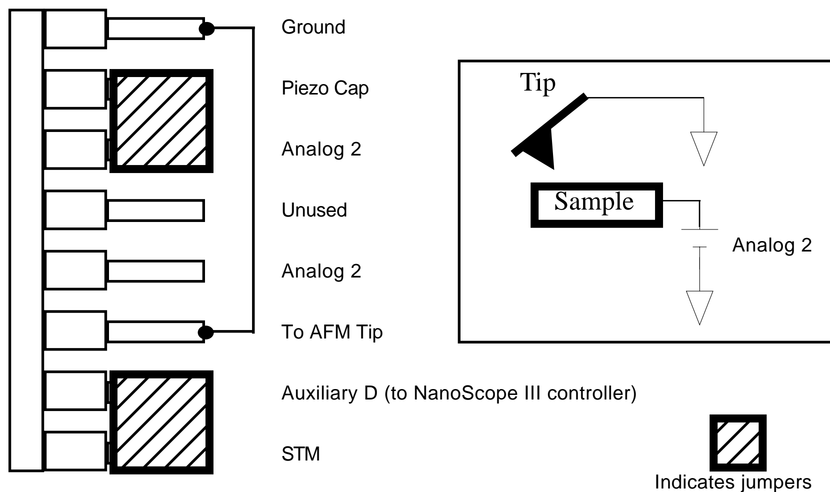


Figure 231-14 Jumper Configuration for Application of Voltage to Sample (For Systems *With* the Extender Electronics Module)

231.3.2.3 Field Gradient Imaging—External Voltage Source Applied to the Tip

In some cases, it may be advantageous to use voltages greater than 12 V, or to use a pulsed power supply. If an external source of voltage is to be applied to the tip, configure jumpers as shown in Figure 231-15.

Note: In all configurations which apply voltage to the tip, an E-field cantilever holder is required. Contact Digital Instruments for more information.

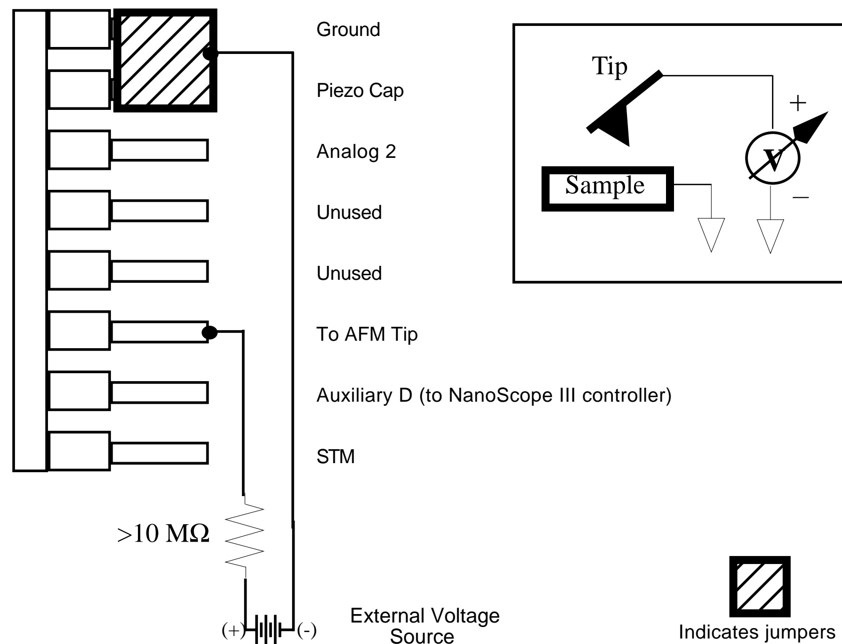


Figure 231-15 Jumper Configuration for Applying External Voltage to Tip (For Systems *With* the Extender Electronics Module)

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 the jumper pins using soldered, push-on connectors. *Do not* solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

231.3.2.4 Field Gradient Imaging—External Voltage Source Applied to the Sample

In some cases, it may be advantageous to utilize voltages greater than 12 V, or to utilize a pulsed power supply. If an external source of voltage is to be applied to the sample, configure jumpers as shown in Figure 231-16.

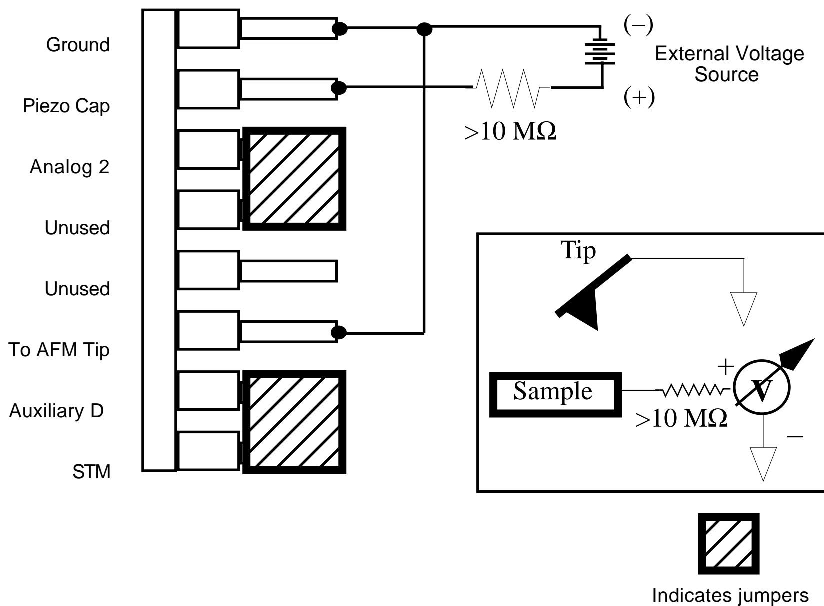


Figure 231-16 Jumper Configuration for Applying External Voltage to Sample (For Systems *With* the Extender Electronics Module)

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 the jumper pins using soldered, push-on connectors. *Do not* solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

231.4 Electric Field Gradient Detection— Procedures

Note: Amplitude detection is the only procedure described here that can be done without the Extender Electronics Module; however, this method is no longer recommended (see Section 231.4.3).

1. 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 introduces noise.

   The only exception is if power is connected directly to the sample (e.g., a circuit with ground and power leads).

2. Mount a metal-coated NanoProbe cantilever into the electric field cantilever holder. MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz, model MESP) 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.

3. 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).

4. Select **View / Cantilever Tune.**

5. Follow the instructions below for the type of electric force imaging desired, **Phase Detection** or **Amplitude Detection**.

231.4.1 Phase Detection

Phase Detection is only available when the Extender Electronics Module has been correctly configured into the system.

1. In the **Auto Tune Controls** window, set **Start frequency** and **End frequency** to appropriate values for your cantilever (e.g., for 225 µm MFM cantilevers, set **Start frequency** to 40 kHz and **End frequency** to 100 kHz). Select **Auto Tune**.

   Two curves appear on the Cantilever Tune graph (see Figure 231-17): the *amplitude* curve in white (the solid line in Figure 231-17), and the *phase* curve in yellow (the dashed line).

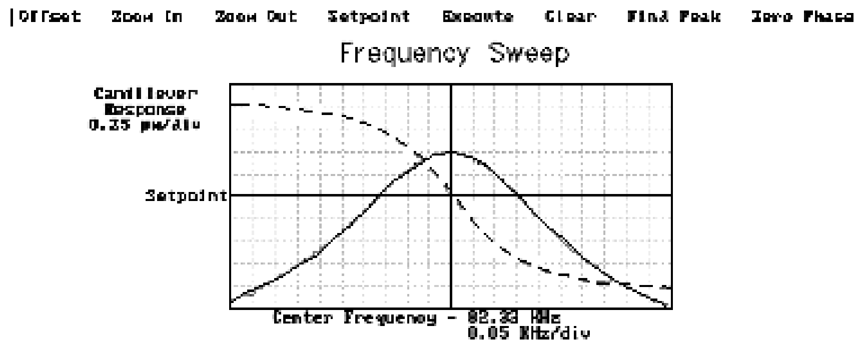


Figure 231-17 Phase Detection Cantilever Tune (Extender Electronics Module Installed)

The phase should decrease with increasing frequency and cross the center line (0° point) at the peak frequency. The phase curve then correctly reflects the phase lag between the drive voltage and the cantilever response. Again, gradients in the electric force cause a shift ΔF_0 in the resonance frequency. Resonance shifts also give rise to phase shifts $\Delta\phi$ which can then give an image of the electric force gradients; see Figure 231-18.

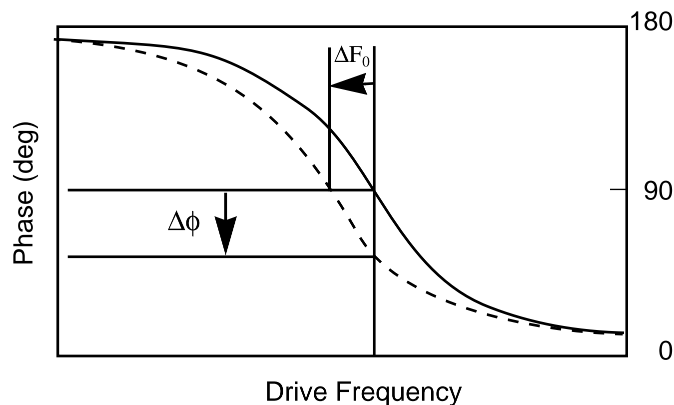


Figure 231-18 Shift In Phase at Fixed Drive Frequency

2. Quit **Auto Tune** and return to **Image Mode**.
3. Engage the AFM and make the necessary adjustments to obtain a good topography (**Height**) image on Channel 1.

4. In the Interleave Controls panel set the **Lift start height** to 0 nm, and **Lift scan height** to 100 nm. (The lift height can later be optimized.) Set the remaining Interleave parameters (**Setpoint**, **Drive amplitude**, **Drive frequency**, gains, etc.) to the main Feedback Controls values. This can be done by setting the flags to the left of each parameter to “off” (grayed bullets).

5. Set the Channel 2 **Data type** to **Phase** and choose **Retrace** for the scan **Line direction** on both Channel 1 and 2 images.

6. In the Interleave Controls panel set **Interleave mode** to **Lift**. Set the Channel 2 **Scan line** to **Interleave** to display interleave data. This screen should now display the cantilever phase change due to electrical force gradients from the sample in the right image and topography in the left image.

   *Versions 4.23 and lower: Set **Interleave scan** to **Lift**. Switch **Interleave mode** to **Enable**. Set the Channel 2 **Scan line** to **Interleave** to display interleave data.*

7. If Analog 2 is being used to apply voltage to the tip or sample, it is recommended to apply it only during the **Interleave line** if feasible. Set **Analog 2** to the desired voltage in the interleave Controls panel. In the Feedback Controls panel set **Analog 2** to 0 V (this sets the applied voltage to 0 during the main scanning line). When a voltage is applied during the main line, it can make TappingMode operation more difficult.

   Versions 4.23 and lower: There is no separate Analog 2 setting available in the Interleave Control panel, so Analog 2 must be set in the Feedback Controls panel. Please read the advice below.

   If a voltage is applied during the main TappingMode line (as well as the interleave line) either through Analog 2 or an external source, extra care must be taken to ensure proper operation. It is possible to enter a [[??]] where the tip is never actually touching the surface even though the setpoint requirement is being met. As the feedback moves the tip closer to the sample surface to try to meet the requested amplitude setpoint, the resonant frequency of the tip is shifting (as shown in Figure 231-19). The closer the tip is to the surface, the larger the force gradient and the more the resonant frequency shifts. It is possible for the resonant frequency to shift enough so that the amplitude setpoint is met without the tip touching the surface [[Illustration??]]. The tip can still be a significant distance away from the sample. The height image has comparatively poor resolution as does the corresponding electric field gradient image. Generally a lower amplitude setpoint is needed to really tap the surface. This phenomenon can be observed by executing a Force Calibration curve. [[illustration??]] The slow (non-linear) decrease in amplitude is due to the resonant frequency shift. The steep linear decrease in amplitude is from the tip encountering the sample surface. An amplitude setpoint below [[??]] should be used in this case to ensure a good image.

8. Optimize the **Lift scan 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 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.
9. 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.

231.4.2 Frequency Modulation

For more quantitative results, Frequency Modulation (FM) can be used. This technique provides a direct measure of the change in resonant frequency felt by the cantilever. In the Interleave Controls panel set the **Input feedback** to **Frequency**. Switch the **Data Type** for Channel 2 to **Frequency**. It may be necessary to optimize the FM gains in the Other Controls panel to properly track the shifts in resonant frequency (starting values: FM **igain** = 40 and FM **pgain** = 60).

*Versions 4.23 and lower: There is no **Input feedback** setting; just switch the **Data Type** for Channel 2 to **Frequency**.*

231.4.3 Amplitude Detection

Note: This imaging method, although described here, is **not recommended** due to the presence of artifacts.

Amplitude Detection, unlike Phase Detection, is available *with* or *without* the optional Extender Electronics Module. This section describes the differences in set up for EFM imaging for systems *without* the Extender module. When performing 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 231-19.

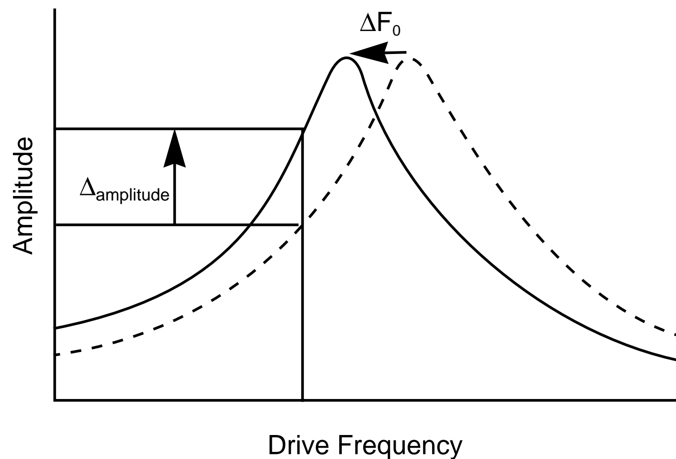


Figure 231-19 Shift In Amplitude at Fixed Drive Frequency (Extender Electronics Module *Not* Installed.)

1. Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 231-20.

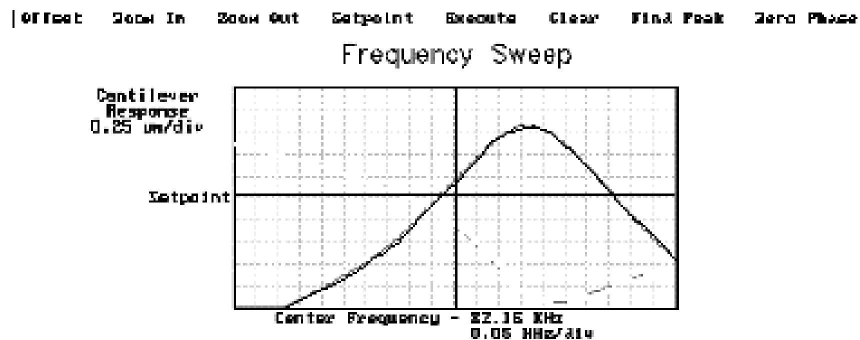


Figure 231-20 Amplitude Detection Cantilever Tune (Extender Electronics Module *Not* Installed)

2. For maximum sensitivity, set the **Drive frequency** to the steepest part of the resonance curve. As the tip oscillates above the sample, a gradient in the electric force shifts the resonance frequency F_0 (see Figure 231-19). 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 231-19.

3. When using Amplitude Detection, optical interference may sometimes appear in the lift (electric force) image when imaging highly reflective samples. Optical interference appears as evenly spaced, sometimes wavy lines with about 1–2µm spacing superimposed on the lift image. This occurs when ambient laser light (i.e., light passing around or through the cantilever, then reflecting off the sample) interferes with laser light reflecting from the cantilever. Interference can be alleviated by moving the beam spot up a little along the cantilever away from the tip; about one-third of the cantilever length from the tip usually works well. On the MultiMode head, the adjustment can be refined by carefully moving the beam spot laterally on the cantilever while scanning until interference fringes are minimized.

   Note: Optical interference is essentially eliminated by using Phase Detection or Frequency Modulation, available only with the Extender Electronics Module.

231.5 Surface Potential Detection—Theory

Note: Surface potential detection EFM is only possible using the Extender Electronics Module and an EFM cantilever holder. 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 231-21. 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

$$V_{ac} \sin \omega t$$

is applied directly to the cantilever tip. This creates an oscillating electric force at the frequency ω on the cantilever. The oscillating force has the following amplitude:

$$F = \frac{dC}{dz} \Delta V_{dc} V_{ac}$$

where $\frac{dC}{dz}$ is the vertical derivative of the tip-sample capacitance.

$\Delta V_{dc} = V_{tip} - V_{sample}$, 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.

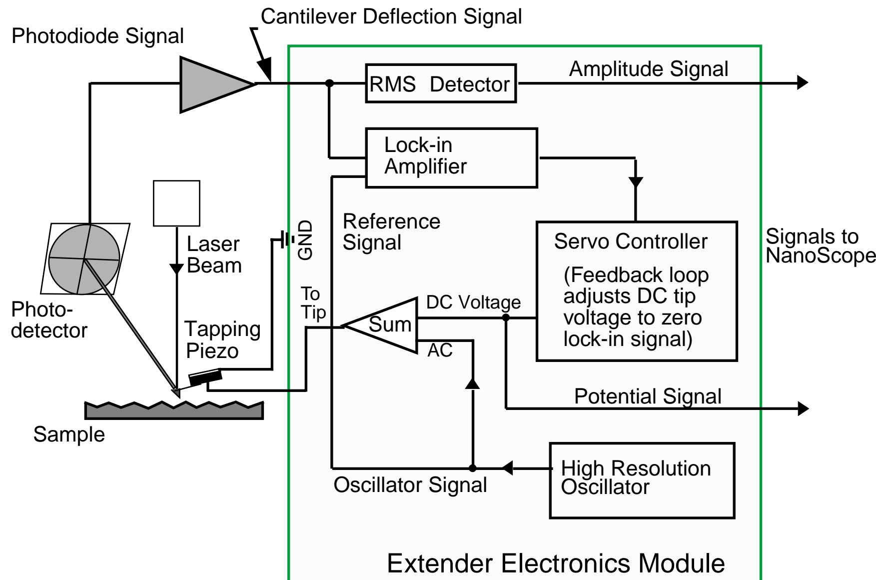


Figure 231-21 Simplified Block Diagram of Extender Electronics Module in Surface Potential Mode

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. Additionally, when the tip and sample are at the same DC voltage ($\Delta V_{dc}=0$), the cantilever feels 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 is 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.

231.6 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 through the piezo cap, or by making direct electrical contact to the sample. In both cases, jumper configurations in the bottom of the microscope must be set to match the environment desired.

Note: 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. Unplug the cable from the base of the MultiMode.
2. Locate the jumpers on the electronic board, visible in a window on the bottom of the MultiMode as shown in Figure 231-5. As shipped from the factory, the baseplate jumpers should appear as shown in Figure 231-22.

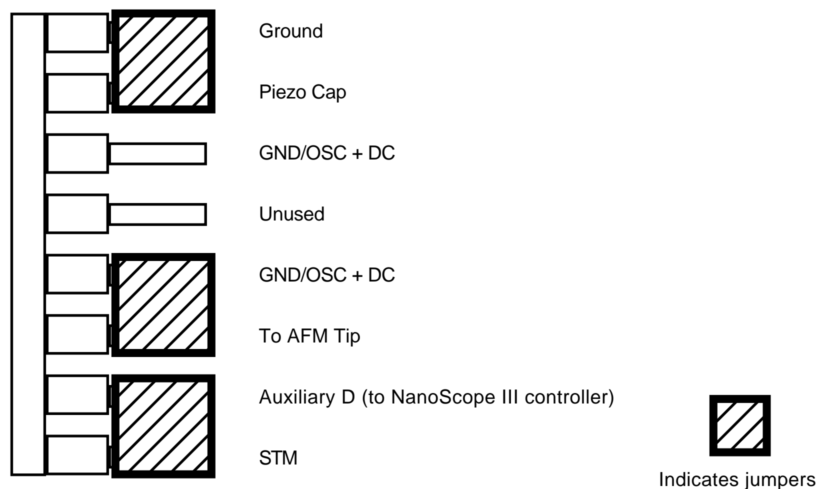


Figure 231-22 Normal Jumper Configuration as Shipped from Factory for MultiMode SPM *With* Extender Electronics Module Installed. Sample Is Held at Ground.

3. Depending upon whether voltage is to be applied to the sample directly or indirectly, reconfigure jumpers if indicated

231.6.1 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 231-22), and the sample should be electrically insulated from the piezo cap (see Figure 231-23).

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 piezo cap is held at ground; be certain that the sample is electrically insulated from the piezo cap. A piece of Kapton tape covering the bottom of a steel sample puck works well.

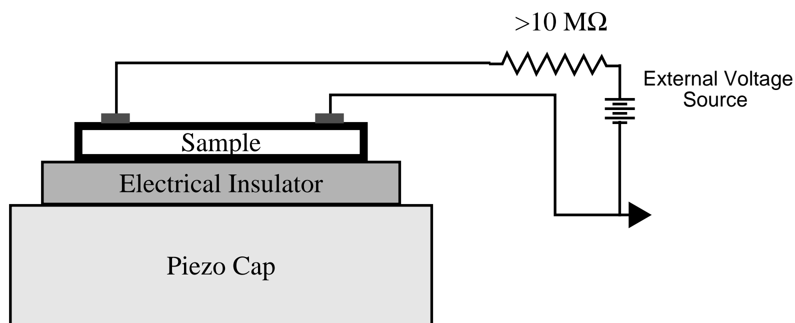


Figure 231-23 Insulating Sample from Piezo Cap

231.6.2 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 231-24.

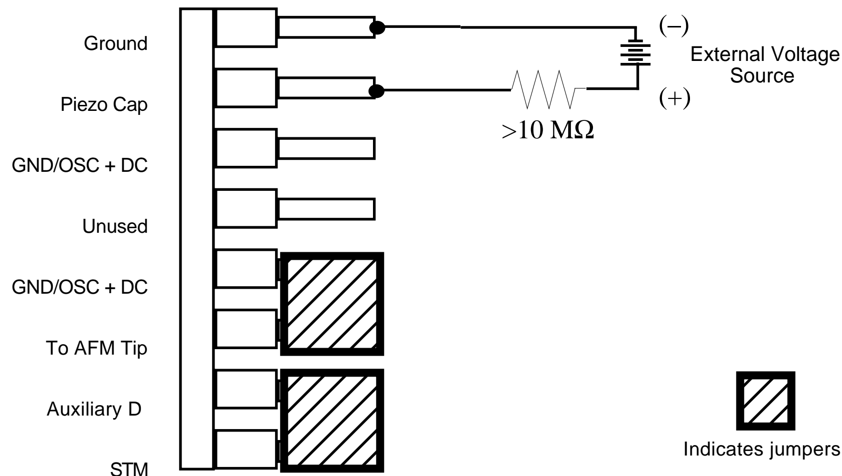


Figure 231-24 Jumper Configuration for Application of Voltage to Sample via Sample Chuck

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 the jumper pins using soldered, push-on connectors. Do **not** solder leads directly to the 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 in Figure 231-25.

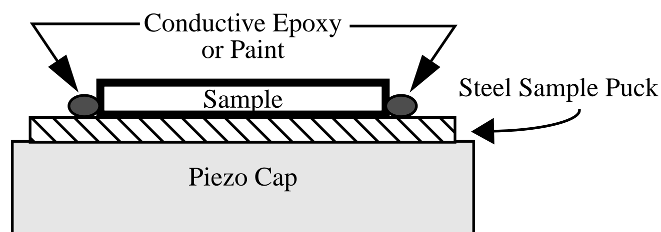


Figure 231-25 Separating Sample from Piezo Cap Using Steel Sample Puck

231.7 Surface Potential Imaging—Procedure

1. Locate the two toggle switches on the backside of the Extender Electronics box (Figure 231-26), and verify that they are toggled as shown in Table 231-2.

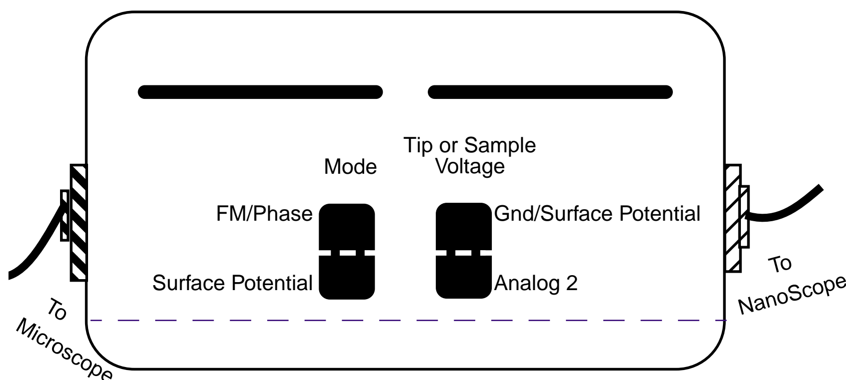


Figure 231-26 Toggle Switches on Back of Extender Electronics Module

	Mode		Tip or Sample Voltage	
	FM/Phase	**Surface Potential**[a]	**GND/ Surface Potential**	**Analog 2**
Surface Potential		X	X	

Table 231-2 Extender Electronics Module Toggle Switch Settings for Surface Potential Imaging

a. **Note**: 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. This is a special cantilever holder used for both EFM and Surface Potential that can be recognized by the white Teflon washer beneath the screw at the base of the cantilever clip. MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz, model MESP) 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.

It is also possible in some cases to use uncoated tips. The metallic cobalt-chrome coating and low spring constant/resonance frequency of the standard MESP tips make them well suited for sensitive electrical measurements. However, the presence of this coating also produces a larger tip radius, and the wearing of the coating can cause significant changes in the detection of the electric field in the immediate vicinity of the tip. It has been suggested (Jacobs, et al.) that these changes in tip shape account for many of the DC shifts observed in Surface Potential images. Recently, uncoated silicon probes have been successfully used for making Surface Potential measurements. The standard TappingMode probes (Models TESP) and the Force Modulation probes (Model FESP) are highly n-doped silicon probes that are conductive enough for Surface Potential imaging. The advantage to using the uncoated silicon tips is the sharp tip radius for the topographic imaging and the absence of changes in the metallic coating during surface potential imaging. However, there may be a difference in sensitivity between the coated and uncoated probes. FESP tips have a lower spring constant and should be more sensitive to smaller forces than TESP tips.

4. Set up the AFM as usual for TappingMode operation.
5. Use **Cantilever Tune** and **AutoTune** (as described in Section 231.4.1) to locate the cantilever’s resonant peak. Two curves should appear in the Cantilever Tune box—the amplitude curve in white and the lock-in curve in yellow. In Surface Potential it is more important than usual that the resonant peak is symmetric. If the peak is unsatisfactory, its shape can often be changed by readjusting the position of the cantilever chip in the holder. The laser and photodiode may require readjustment after the cantilever is moved. 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 now affect the lock-in signal instead (see Figure 231-21).
6. Engage the AFM and make the necessary adjustments for a good TappingMode image while displaying height data in Channel 1. It is essential to have a good height image before proceeding. Any measurement using LiftMode, including Surface Potential, is dependent on accurate height information. Go into Scope Mode (**View** / **Scope Mode**) and check to see that the Trace (white) and Retrace (yellow) match well. There may be an offset between them, but the shape and slopes of features should be the same.
7. Under the Panels menu select **Interleave Controls**. 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 parameters. When the bullet next to a parameter is green,

the value shown is used during the interleave scan. To fix any parameter so that it is the same on the main and interleave scans, click on the green bullets to the left of that parameter. The green bullet changes to “off” (gray) and the main Feedback Controls value for that parameter is used.

Set the interleave **Drive frequency**, **Amplitude setpoint**, and **SPM Feedback** to the main feedback values (set buttons gray).

8. The Interleave Controls **Drive amplitude** is the AC voltage that is applied to the AFM tip. Higher **Drive amplitudes** produce larger electric forces 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 potentials to be measured are very large, it is necessary to choose a small **Drive amplitude** (it is not recommended to use less than 2 V), while small surface potentials can be imaged more successfully with large **Drive amplitudes**. To start choose a **Drive amplitude** of 6 V.

9. Set the **Drive phase** in the Interleave Control panel. For MESP or FESP cantilevers (resonant frequency ~60-80khz) enter a **Drive phase** of –90 degrees. For TESP cantilevers (with resonant frequency ~300khz), enter a **Drive phase** of –160 degrees. Make sure the corresponding button is green. (In regular TappingMode the **Drive phase** compensates for phase lag in the mechanical response of the tapping piezo in the cantilever holder. This piezo is not used in the Surface Potential measurement, so a different **Drive phase** setting is required.)

   *Versions 4.23 and lower: The above **Drive phase** must be entered in the Feedback Controls panel, because there is not a separate setting available in the Interleave Controls panel.*

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.

    In the Interleave Controls set the **Input feedback** to **Potential**. Set **Interleave mode** to **Lift**. Set the Channel 2 image **Data type** to **Potential** and select **Interleave** for the **Scan line**. For both data channels (height and potential) set the **Scan line** direction to **Retrace**. The retrace direction should be chosen because the lift step occurs on the trace scan.

    *Versions 4.23 and lower: Set **Interleave scan** to **Lift**. Set **Interleave mode** to **Enabled**. There is no **Input feedback** setting; just switch the **Data type** for Channel 2 to **Potential**.*

11. 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 electrically according to the interleave **Drive amplitude** that has been selected. Also, a feedback circuit is now 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 Section 231.5, 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 Module for Surface Potential measurements is the same as the one used in Frequency Modulation for magnetic and electric force gradient detection, as described previously. Select **Other Controls** and adjust the FM gains. Setting FM **igain** to 15 and FM **pgain** 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. More information on tuning the feedback loop is given in Section 231.7.1.

13. Optimize the Lift scan heights. The best resolution is achieved with the **Lift scan height** at the smallest value possible that does not make the tip 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. Because the tip is not oscillating during the Potential measurement (the feedback loop works to keep the amplitude zero), the lift height is generally smaller than with other LiftMode techniques. **Lift scan heights** down to –5nm are possible on smooth samples. This lower limit to the **Lift scan height** are affected by the sample roughness, scan speed, target amplitude used during tuning, etc.

14. **Open Loop Operation**: Sometimes it is useful to run Potential in the "open-loop" configuration. This means that the Potential feedback loop is disabled and the data is only qualitative. The AC voltage is applied to the cantilever as in the standard Potential operation (the tapping piezo used for mechanical driving of the tip is disabled). Because the feedback is disabled, there is no adjustment of the DC voltage on the tip, so the oscillating electrical forces drives the cantilever into motion. This motion can be monitored by observing the lock-in signal (the input to the feedback loop), which is called "phase" in the software. Set up the system as described above with the following changes: Set the FM **igain** and FM **pgain** to zero. Select **Phase** as the **Data type** for Channel 2. Note that turning the FM gains to zero stops further changes to the DC voltage on the tip but does not set the tip voltage back to zero.

    *Versions 4.23 and lower: Open loop must be run before the feedback loop is ever turned on. When **Potential** is selected as the data type for Channel 2, the feedback loop is enabled. The feedback loop remains on until the tip is withdrawn. Instead of selecting **Data type** to **Potential** in step 10, select **Data type** to **Phase**.*

231.7.1 Troubleshooting

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. Verify this by changing the value of **Realtime planefit** to **None** in the Channel 2 panel and increasing the **Data scale** to the maximum value. The scope trace is at one of the limits. Common reasons for this include:

  - A regular cantilever holder is being used instead of the EFM cantilever holder.

  - An inappropriate **Drive phase** is being used, For an MESP cantilever make sure the **Interleave Drive** phase is set to –90 degrees. *Versions 4.23 and lower: Feedback Control Drive phase is set to –90 degrees.*

  - The switches on the Extender Module are incorrectly set. Make sure they are set to Surface Potential and GND/Surface Potential.

  - An incorrect electrical connection is being made. Verify that the sample is connected properly to ground or a power supply. Verify that the jumpers in the base of the MultiMode are properly set for your configuration.

231.A Surface Potential Electronics

Two manual switches on the outside of the Extender box configure the electronics for the operation of Surface Potential. Each switch controls two internal switches that move in tandem. Figure 231-27 shows the connections when both manual switches are set to Surface Potential.

The selection of Surface Potential instead of FM/Phase enables an analog switch that toggles position with the main and interleave scan lines. The switch controls the electrical connections to the tip and the tapping piezo.

- During the main scan line the output of the oscillator, $V_{ac}\sin(\omega t + \varphi_1)$, is connected to the tapping piezo which mechanically drives the cantilever at or near its resonance, ω. Another part of the switch grounds the tip (or sample, depending on the jumper positions).
- During the interleave scan line the tapping piezo is grounded and the cantilever is no longer driven mechanically. The oscillator signal is added to the DC output of the Potential feedback loop, V_{tip}, and applied to the tip (or sample). The tip can now be driven electrically.

When FM/Phase is selected, as in EFM, the switch is always in the main position and the oscillator drives the tapping piezo only.

In Surface Potential the second manual switch is set to Gnd/Surface Potential to allow the tip to be grounded or connected to the voltage required for Surface Potential measurements (as discussed above). For EFM the switch is set to **Analog 2**, and the internal power supply Analog 2 is connected to the tip (or sample) and controlled through the software.

An analog multiplier and low pass filter are used as a lock-in amplifier to generate the phase signal. The oscillator has two outputs, both at the same frequency. One output, $V_{ac}\sin(\omega t + \varphi_1)$, has an adjustable amplitude set in the software by the **Drive Amplitude**. The other output is a reference signal with fixed amplitude, $\sin(\omega t + \varphi_1)$. This reference signal is multiplied with the photodiode signal, $A\sin(\omega t + \varphi_2)$, giving an output of:

$$\sin(\omega t + \varphi_1) \times A\sin(\omega t + \varphi_2) = (A/2)\left[\cos(\varphi_1 - \varphi_2) - \cos(2\omega t + \varphi_1 + \varphi_2)\right]$$

The resonant frequencies of tapping cantilevers are well above 1.5 kHz, so the low pass filter reduces the signal to $(A/2)\cos(\varphi_1 + \varphi_2)$. This is the Surface Potential phase signal which contains *both* phase and amplitude information. The feedback loop zeroes this signal, and thus the cantilever’s oscillation, by adjusting the applied

potential V_{tip}. In regular tapping mode operation (i.e., FM/Phase is selected) the photodiode signal has automatic gain control that normalizes out the contribution from the cantilever's amplitude (A), giving a phase signal proportional only to $\cos(\varphi_1 + \varphi_2)$.

231.A.1 Theory

A good way to understand the response of the cantilever to electric forces is to start with the energy in a capacitor, *U*.

$$U = \frac{1}{2}C(\Delta V)^2$$

C is the local capacitance between the AFM tip and the sample; ΔV is the voltage difference between the two. The force on the tip and sample is the rate of change of the energy with separation distance:

$$\mathrm{F} = -\frac{dU}{dZ} = -\frac{1}{2}\frac{dC}{dZ}(\Delta V)^2$$

In the operation of Surface Potential the voltage difference ΔV consists of both a DC and an AC component. The AC component is applied from the oscillator, $V_{ac}\sin(\omega t + \varphi)$; ω is the resonant frequency of the cantilever.

$$\Delta V = \Delta V_{dc} + V_{ac}\sin(\omega t + \varphi)$$

ΔV_{dc} includes applied DC voltages (from the feedback loop or externally applied), work function differences, surface charge effects, and any voltage losses within the electronics. Squaring ΔV and using the relation $2\sin^2 u = 1 - \cos(2u)$ produces:

$$F = \underbrace{-\frac{1}{2}\frac{dC}{dZ}\left(\Delta V_{dc}^2 + \frac{1}{2}V_{ac}^2\right)}_{\text{DC term}} \underbrace{- \frac{dC}{dZ}\Delta V_{dc}V_{ac}\sin(\omega t + \varphi)}_{\omega\text{ term}} \underbrace{+ \frac{1}{4}\frac{dC}{dZ}V_{ac}^2\cos(2\omega t + 2\varphi)}_{2\omega\text{ term}}$$

The oscillating electric force at ω acts as a sinusoidal driving force that can excite motion in the cantilever. The cantilever responds only to forces at or very near its resonance, so the DC and 2ω terms do not cause any significant motion of the cantilever. In regular TappingMode, the cantilever's response (RMS amplitude) is

directly proportional to the drive amplitude of the tapping piezo. Here the response is directly proportional to the amplitude of the F_ω drive term:

$$\textit{amplitude of } F_\omega = \frac{dC}{dZ}\Delta V_{dc} V_{ac}$$

If $\Delta V_{dc} = 0$, the electric drive force at ω is zero and the cantilever does not oscillate. The Surface Potential feedback loop adjusts the applied DC potential on the tip, V_{tip}, until the cantilever's response is zero. V_{tip} is the Potential data that is used to generate a voltage map of the surface. Note that the input to the feedback loop is actually the Surface Potential phase signal (the cantilever's amplitude multiplied by $\cos[\varphi_1 - \varphi_2]$). The phase term is used to determine the polarity of ΔV_{dc}. The sign of the voltage difference is needed so the feedback loop knows whether to adjust the tip voltage higher or lower.

Figure 231-27 Surface Potential Extender Box Electronics