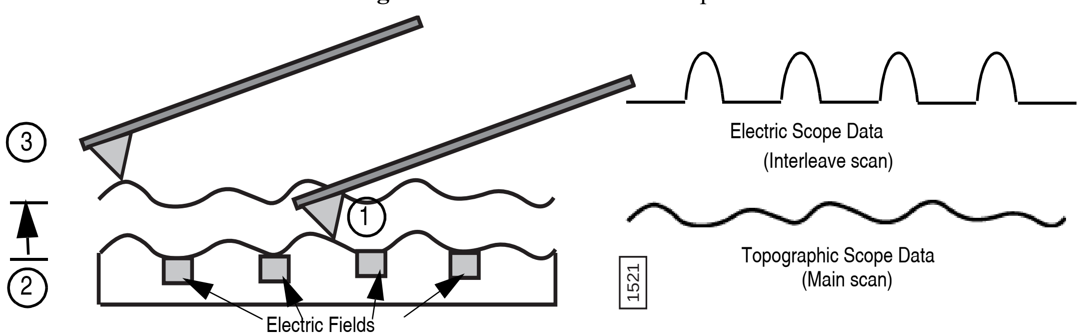
Electric Techniques on MultiMode™ Systems

231.1 Electric Techniques Overview

This support note describes how to perform two electric techniques on a MultiMode™ scanning probe microscopy (SPM) system: **Electric Force Microscopy** (EFM) and **Surface Potential Detection**.

Electric techniques are similar to magnetic force microscopy (MFM) and share many of the same procedural techniques. Electric techniques and MFM both use the **Interleave** and **LiftMode** procedures. The two-pass LiftMode measurement allows the imaging of relatively weak but long-range magnetic and electrostatic interactions while minimizing the influence of topography (See Figure 231.1a). LiftMode records measurements in two passes, each consisting of one trace and one retrace, across each scanline. First, LiftMode records topographical data in Tapping Mode on one trace and retrace. Then, the tip raises to the lift scan height, and performs a second trace and retrace while maintaining a constant separation between the tip and local surface topography.

Figure 231.1a LiftMode Principles



1. Cantilever measures surface topography on first (main) scan.
2. Cantilever ascends to lift scan height.
3. Cantilever follows stored surface topography at the lift height above sample while responding to electric influences on second (interleave) scan.

Revision History

Revision	Date	Section(s) Affected	Ref. DCR	Approval
F	01-20-2011	Re-branded		Ruth Wishengrad
E	03DEC01	Reformat	________	Mark Hardin
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This support note includes the following:

231.1.1 Electric Force Microscopy

Electric Force Microscopy measures variations in the electric field gradient above a sample. The sample may be conducting, nonconducting, or mixed. Because the surface topography shapes the electric field gradient (e.g. sharp points on the surface concentrate the field gradient), large differences in topography make it difficult to distinguish electric field variations due to topography or due to a true variation in the field source. The best samples for EFM are samples with fairly smooth topography. The field source can include trapped charges, applied voltage, etc.

All standard MultiMode SPMs are capable of EFM imaging using amplitude detection techniques. By adding an Extender™ electronics module, you can use the MultiMode system for frequency modulation or phase detection with improved results (See Figure 231.1b). Frequency modulation and phase detection have largely superseded amplitude detection, so the Extender™ electronics module is strongly recommended for electric force microscopy.

Note: Applying a voltage to the tip requires a special electric cantilever holder (model MMEFCH).

Figure 231.1b Extender™ Electronics Module

231.1.2 Surface Potential Detection

Surface potential detection 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 DC voltage on the tip and sample is the same.) Samples for surface potential measurements should have an equivalent surface voltage of less than ±10 volts, and operation is easiest for voltage ranges of ±5 volts. The noise level of this technique is typically 10 mV. Samples may consist of conducting and nonconducting regions, but the conducting regions should not be passivated. Samples with regions of different materials will also show contrast due to contact potential differences. Quantitative voltage measurements can be made of the relative voltages within a single image.

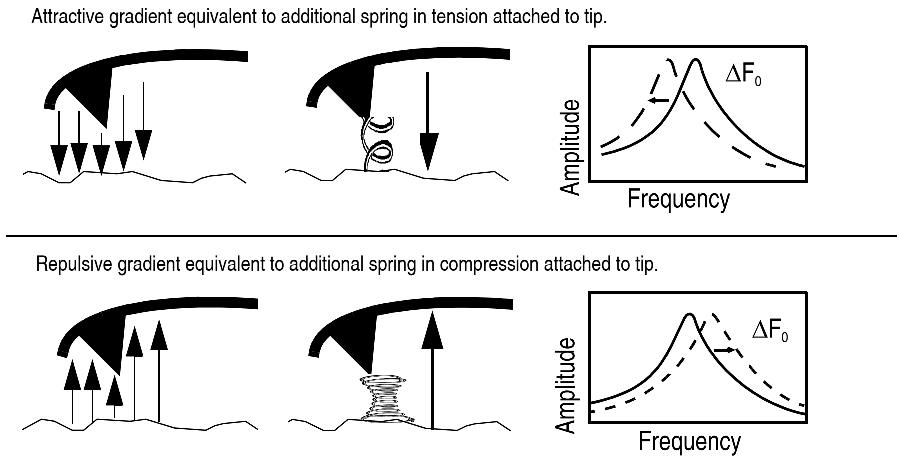
Note: This method requires the Extender Electronics Module and version 3.1 or later of the NanoScope software, and the special electric cantilever holder for the MultiMode (model MMEFCH).

PART I: Electric Force Microscopy

231.2 EFM Theory

Electric Force Microscopy is analogous to standard MFM, except that gradients being sensed are due to electrostatic forces. In this method, the cantilever is vibrated by a small piezoelectric element near its resonant frequency. The cantilever resonant frequency changes in response to any additional force gradient. Attractive forces make the cantilever effectively “softer,” reducing the cantilever resonant frequency. Conversely, repulsive forces make the cantilever effectively “stiffer,” increasing the resonant frequency. A comparison of these effects is shown in Figure 231.2a.

Figure 231.2a Comparison of Attractive and Repulsive Forces

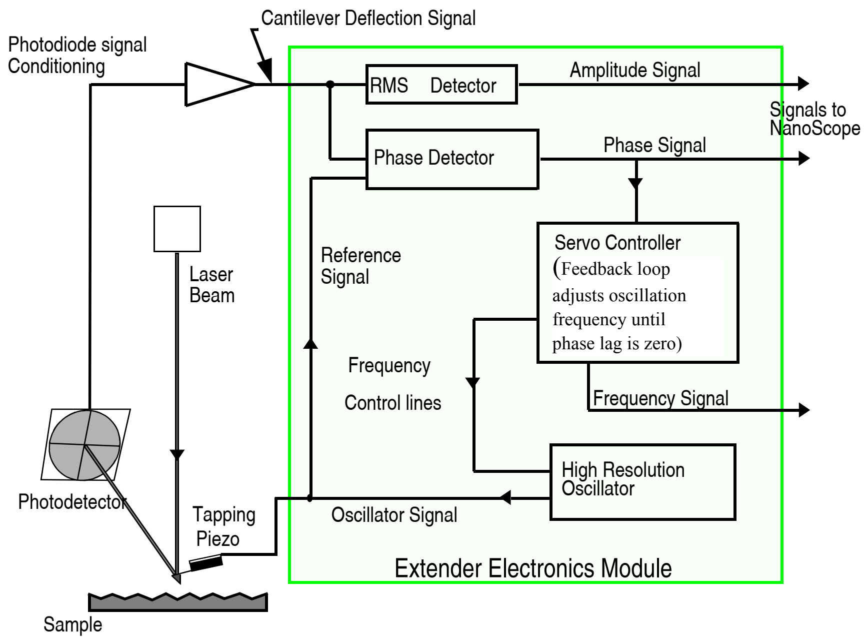


Changes in cantilever resonant frequency are detected in one of the following ways:

- Phase detection (with Extender Electronics Module only)
- Frequency modulation (with Extender Electronics Module only)
- Amplitude detection

All of the above methods rely on the change in resonant frequency of the cantilever due to vertical force gradients from the sample. Figure 231.2b shows a diagram of how the Extender Electronics Module provides signal enhancement and feedback allowing gradient detection. The best candidates for electric field gradient imaging are smooth samples that have large contrasts in the electric force gradient due to material differences or regions at substantially different potentials.

Figure 231.2b Diagram of Extender Electronics Module in Phase and Frequency Measurement Mode



In many cases, you must apply a voltage to the tip or sample to achieve a high-quality image. Various methods for applying voltages to the tip and sample are included in the sections that follow. Samples with permanent electric fields may not require voltage application.

231.3 EFM Preparation/Applying Voltage

This section explains how to conduct EFM imaging by applying a voltage to the tip or sample to generate electric fields. If the sample being imaged has a permanent electric field that does not require the external application of voltage, the steps below are not required and you can proceed to Section 231.4.

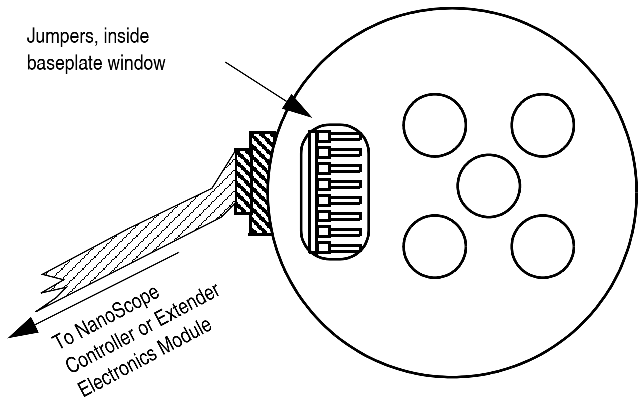
231.3.1 Jumper Configuration

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. This section provides jumper configuration instructions for basic microscope models operating *with* and *without* the Extender Electronics Module.

The location and orientation of the jumpers in the baseplate of the MultiMode is shown in Figure 231.3a. To change the jumpers, do not remove the baseplate; you can easily change the jumpers 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 in their original positions.

Figure 231.3a MultiMode Baseplate Showing Jumper Location and Orientation



1. Carefully examine the configurations in the following sections and identify which set-up is appropriate for your application.

   Note: 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 covering 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. To apply a voltage to the tip (Analog 2 or from external supply) you must have the electric cantilever holder (MMEFCH).

2. Power down the NanoScope controller.
3. Unplug the cable from the base of the MultiMode.
4. Disconnect and remove the microscope head and the scanner.
5. Carefully tilt the MultiMode so that the baseplate is oriented as shown in Figure 231.3a.
6. Locate the jumpers.

   Note: As shipped from the factory, jumpers on systems **without** the Extender Electronics Module should appear as shown in Figure 231.3c, whereas jumpers on systems *with* the Extender Electronics Module should appear as in Figure 231.3h.

7. Change the position of the jumpers to match the configuration you have selected.

8. After correctly configuring the jumpers, reconnect the cable to the base of the MultiMode. Replace and reconnect the scanner and head.

9. Power-up the Nanoscope controller.

10. Set Analog 2.

    Note: If the configuration you choose uses Analog 2, it must be enabled in both the software and the hardware.

Software

1. If the jumpers are configured to use the internal voltage source Analog 2, select **di** / **Microscope Select** > **Edit** > **Advanced**.

2. Set Analog 2 to **User defined**. Make sure to click OK to exit both dialog boxes.

    Note: Remember to reset Analog 2 to Atten switch when finished. (For all other configurations Analog 2 should be left on **Atten switch**.)

    Note: *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. (For all other configurations it should be left on Allow.)

Hardware

1. 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. See Figure 231.3b.

    Note: Remember to return this switch to the Gnd/Surface Potential position when finished.

2. Locate the two toggle switches on the backside of the Extender Electronics Module See Figure 231.3b.

3. Verify switches are toggled as shown in Table 231.3a.

Figure 231.3b Toggle Switches on Back of Extender Electronics Module

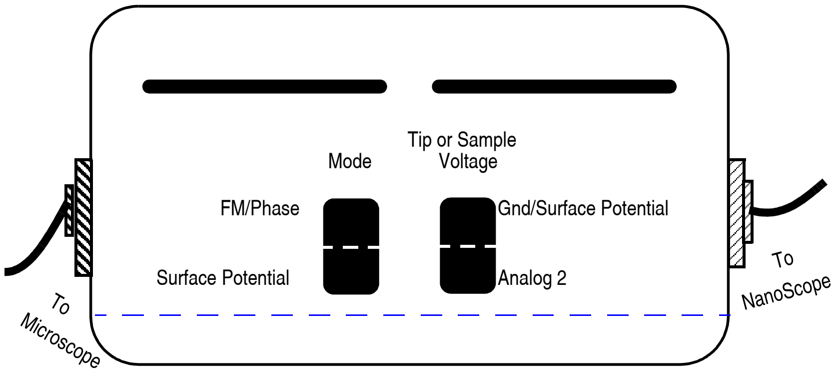


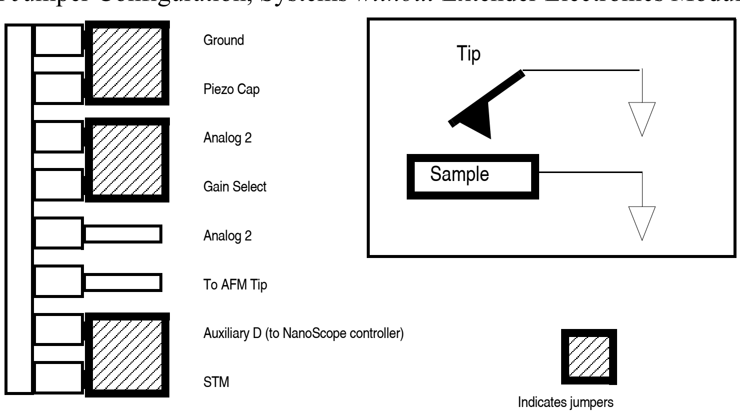
Table 231.3a Extender Electronics Module Toggle Switch Settings

	Mode		Tip or Sample Voltage	
	FM/Phase	**Surface Potential**	**GND/ Surface Potential**	**Analog 2**
EFM with Analog 2 biasing tip or sample	**X**			**X**
EFM in all other configurations	**X**		**X**	
Standard Operation	**X**		**X**	

Jumper Configurations: Systems *without* 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 231.3c.

Figure 231.3c Normal Jumper Configuration, Systems *without* Extender Electronics Module, as Shipped



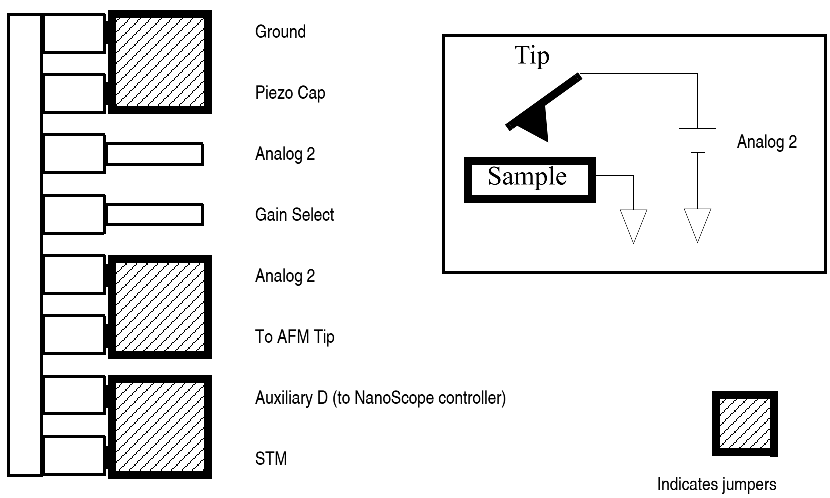
Analog 2 Voltage Applied to the Tip: Systems *without* Extender Electronics Module

The jumper configuration in Figure 231.3d connects the Analog 2 signal from the NanoScope controller (± 12 V range) to the tip.

Note: To apply voltage to the tip, an E-field cantilever holder is required. Contact Bruker Corporation for more information.

ATTENTION: Remember to enable Analog 2 in the software.

Figure 231.3d Jumper Configuration for Application of Voltage to Tip, Systems without Extender Electronics Module

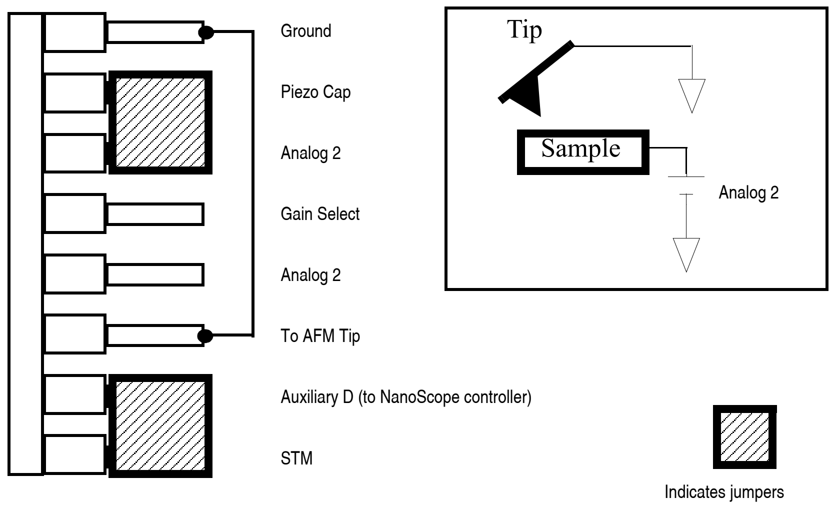


Analog 2 Voltage Applied to Sample: *Systems without* Extender Electronics Module

The jumper configuration in Figure 231.3e connects the Analog 2 signal from the NanoScope controller (± 12V range) to the piezo cap.

ATTENTION: Remember to enable Analog 2 in the software.

Figure 231.3e Jumper Configuration for Application of Voltage to Sample, Systems *without* the Extender Electronics Module



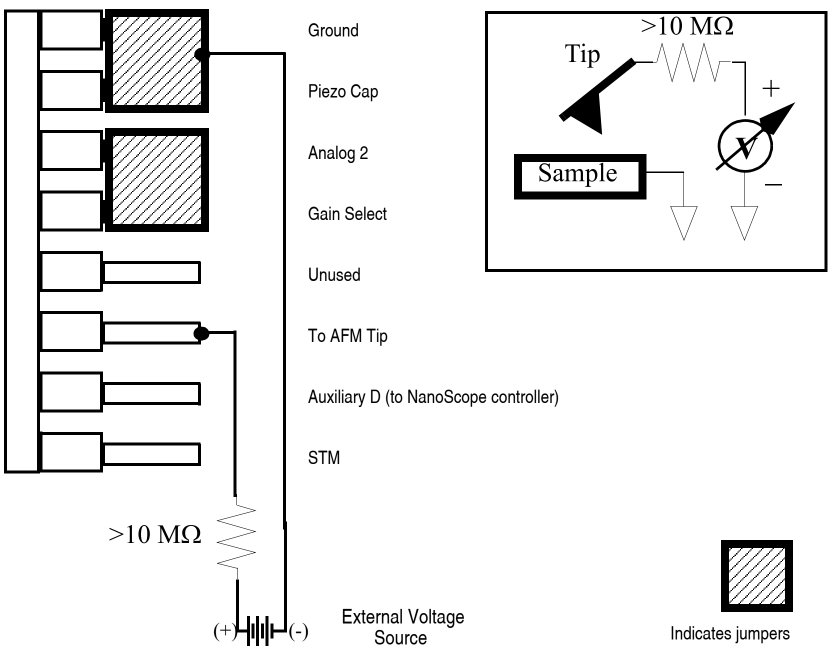
Leads should be connected to the jumper pins using soldered, push-on connectors. *Do not* solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

External Voltage Source Applied to Tip: Systems *without* Extender Electronics Module

In some cases, you may want to use voltages greater than 12V, or to use a pulsed power supply. If an external source of voltage is applied to the tip, configure jumpers as shown in Figure 231.3f.

Note: To apply voltage to the tip, an E-field cantilever holder is required.

Figure 231.3f Jumper Configuration for Applying External Voltage to Tip, Systems *without* Extender Electronics Module



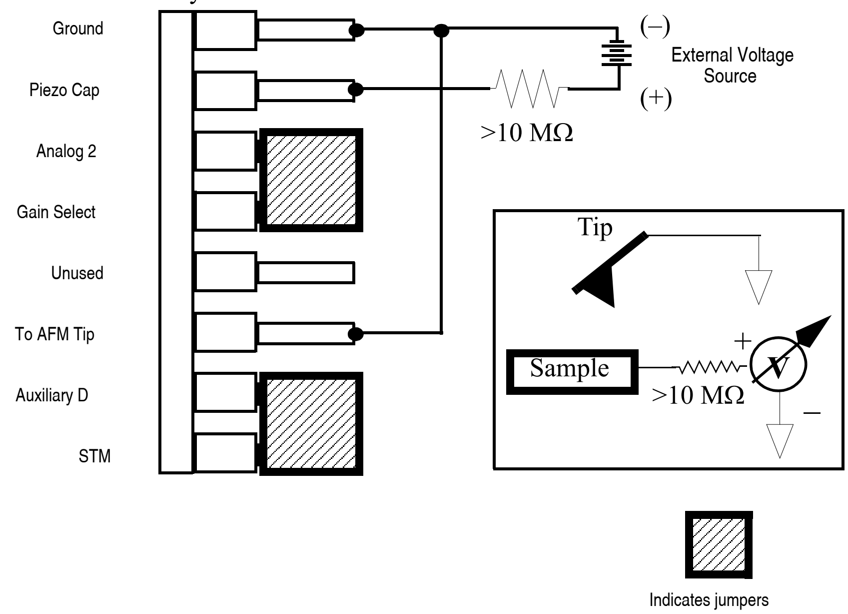
A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to the jumper pins using soldered, push-on connectors.

CAUTION: 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.

External Voltage Source Applied to Sample: Systems *without* Extender Electronics Module

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 231.3g.

Figure 231.3g Jumper Configuration for Applying External Voltage to Piezo Cap, Systems *without* Extender Electronics Module



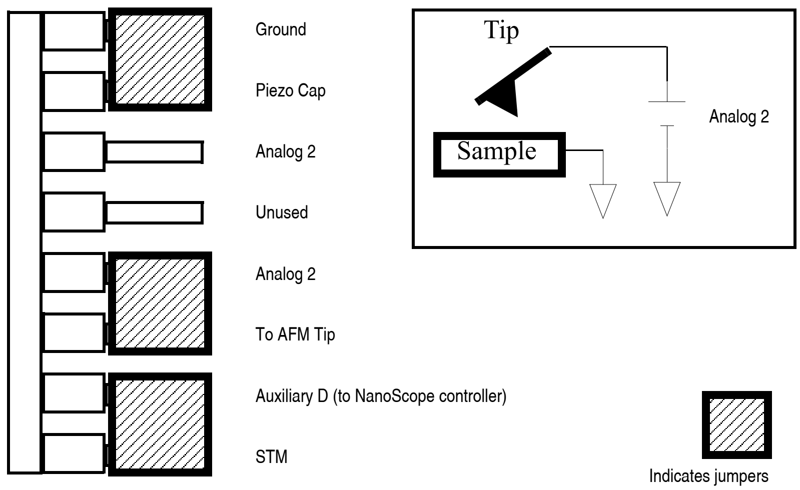
A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to the jumper pins using soldered, push-on connectors. *Do not* solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

Jumper Configurations: Systems *with* Extender Electronics Module

CAUTION: 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 231.3h.

Figure 231.3h Normal Jumper Configuration, Systems *with* Extender Electronics Module, as Shipped



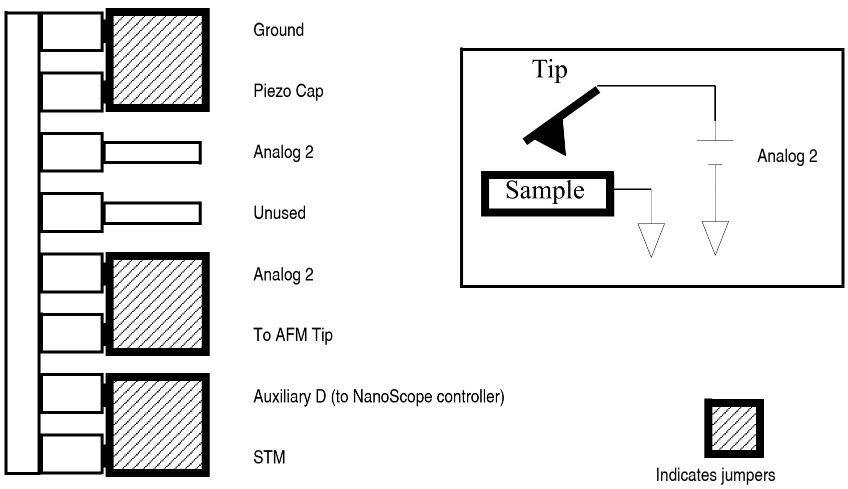
Analog 2 Voltage Applied to Tip: Systems *with* Extender Electronics Module

Notice that the jumper configuration in Figure 231.3i connects the Analog 2 signal from the NanoScope controller (± 12V range) to the tip, and is **exactly the same** as the standard jumper configuration as shipped from the factory.

Note: In all configurations that apply voltage to the tip, an E-field cantilever holder is required.

Note: Remember to enable Analog 2 in the software and to set the Extender module switch to Analog 2.

Figure 231.3i Jumper Configuration for Application of Voltage to Tip—Standard Configuration, Systems *with* Extender Electronics Module

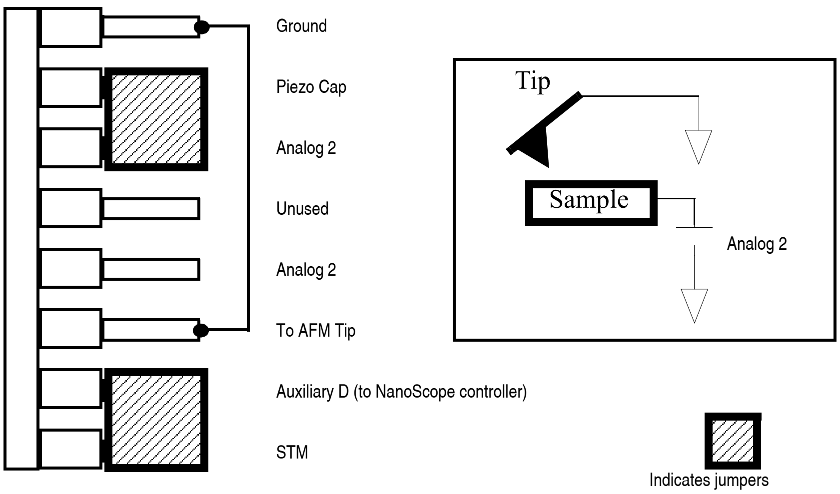


Analog 2 Voltage Applied to Sample: Systems *with* Extender Electronics Module

The jumper configuration in Figure 231.3j connects the Analog 2 signal from the NanoScope controller (± 12 V range) to the piezo cap.

> **Note:** Remember to enable Analog 2 in the software and to set the Extender box switch to Analog 2.

Figure 231.3j Jumper Configuration for Application of Voltage to Sample, Systems *with* Extender Electronics Module



Leads should be connected to the jumper pins using soldered, push-on connectors.

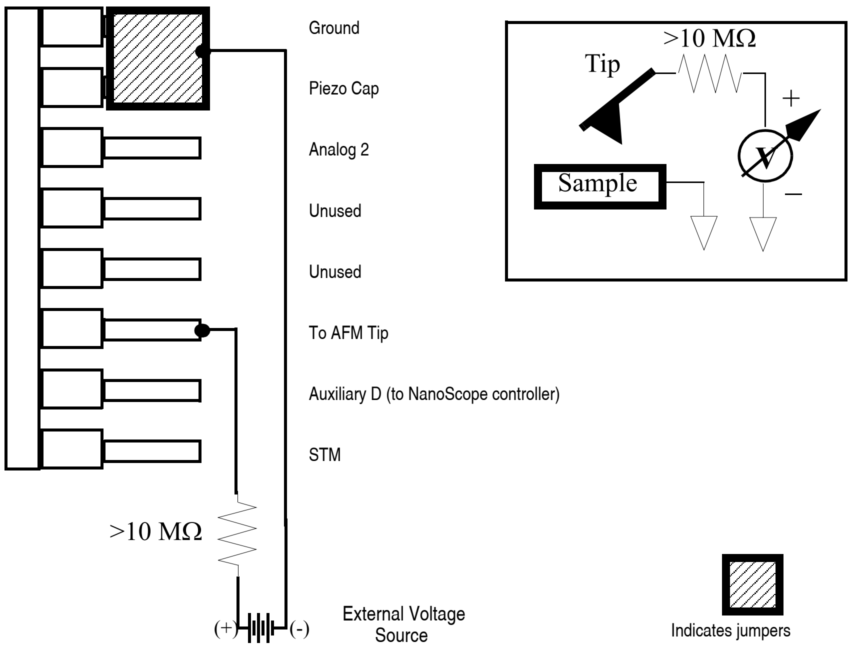
CAUTION: Do not solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

External Voltage Source Applied to Tip: Systems *with* Extender Electronics Module

In some cases, you may want 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 231.3k.

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

Figure 231.3k Jumper Configuration for Applying External Voltage to Tip, Systems *with* Extender Electronics Module



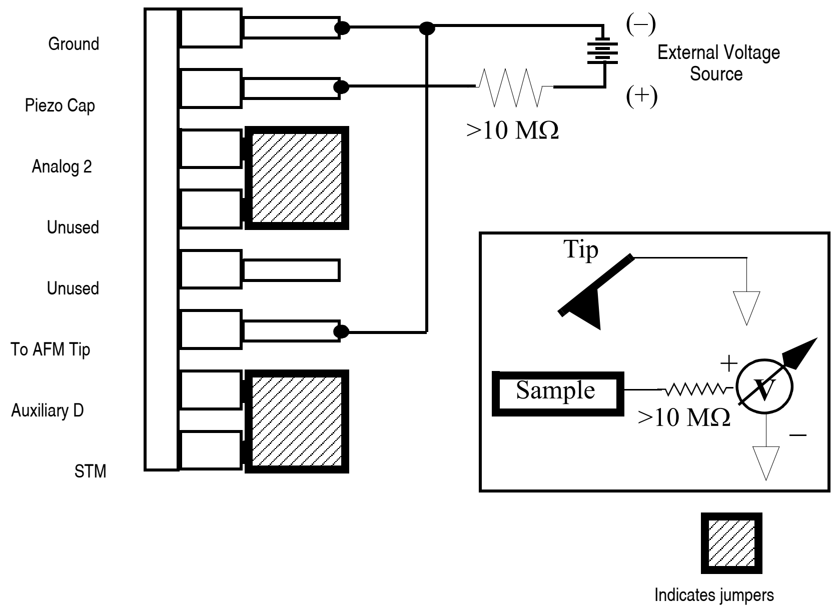
A current-limiting resistor (e.g., 10–100 MΩ) should be placed in series with the external voltage supply as shown to protect the tip and sample from damage. Current-limited power supplies may also be used. Voltage leads should be connected to the jumper pins using soldered, push-on connectors.

CAUTION: Do not solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

External Voltage Source Applied to Sample: Systems *with* Extender Electronics Module

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 231.3l.

Figure 231.3l Jumper Configuration for Applying External Voltage to Piezo Cap, Systems *with* 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.

CAUTION: Do not solder leads directly to the pins, as the heat may cause damage and/or make jumpering the pins difficult.

231.4 EFM Procedures

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

1. Verify the following electric force microscopy preparation is complete:
   a. Jumper Configurations
   b. Extender Electronics Module Settings (see Figure 231.3b and Table 231.3a)
   c. Analog 2 settings in software (see Page 7)
2. Electrically connect the sample by mounting it to a standard sample disk or stage using conducting epoxy or silver paint.
3. Verify the connection is good (a poor connection introduces noise).

   Note: If an external power supply connects directly to leads on the sample itself, it is important to electrically isolate the sample from the piezo cap. A piece of Kapton tape covering the bottom of a sample puck works well.
4. Mount a metal-coated NanoProbe cantilever into the **electric field cantilever holder**.

   Note: MFM-style cantilevers (225 µm long, with resonant frequencies around 70 kHz, models MESP and SCM-PIT) usually work well. It is also possible to deposit custom coatings on model FESP silicon TappingMode cantilevers. Make sure that any deposited metal you use adheres strongly to the silicon cantilever.
5. Set up the AFM as usual for TappingMode operation.
6. Select **View > Sweep > Cantilever Tune.**
7. Follow the procedure below for the type of electric force imaging desired, **Phase Detection**, **Frequency Modulation** or **Amplitude Detection** (see sections 231.4.1 through 231.4.3).

231.4.1 Phase Detection

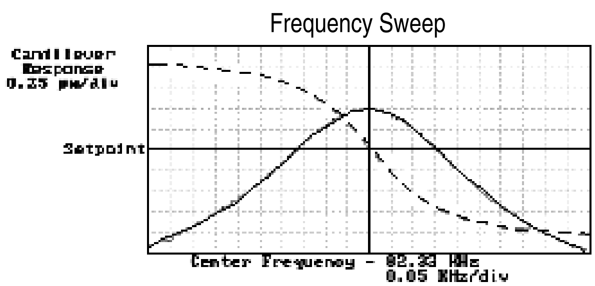
Phase Detection is only available when the Extender Electronics Module has been correctly configured into the system.

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).

2. Select **Auto Tune**.

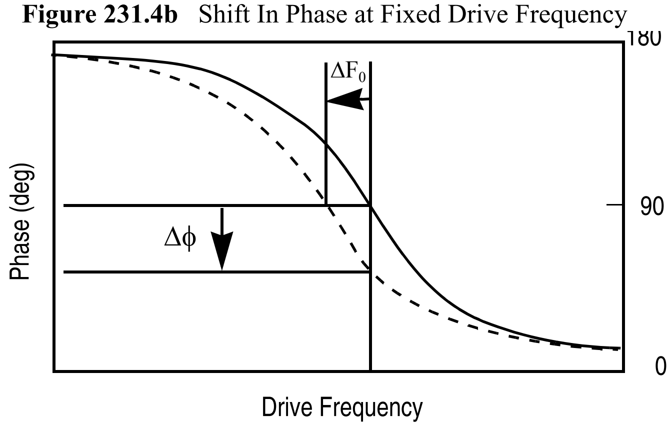
Note: Two curves appear on the Cantilever Tune graph (see Figure 231.4a): the *amplitude* curve in white (the solid line in Figure 231.4a), and the *phase* curve in yellow (the dashed line).

Figure 231.4a Phase Detection Cantilever Tune
(Extender Electronics Module Installed)



Note: The phase should decrease with increasing frequency and cross the center line (0° point) at the peak frequency. The phase curve then correctly reflects the phase lag between the drive and the cantilever response. Gradients in the electric force cause a shift ΔF_0 in the resonance frequency. Resonance shifts also give rise to phase shifts $\Delta\phi$ used to generate an image of the electric force gradients; see Figure 231.4b.

Figure 231.4b Shift In Phase at Fixed Drive Frequency



3. Quit **Auto Tune**.

4. Select **Back to Image Mode**.

5. Engage the AFM and make the necessary adjustments to obtain a good topography (**Height**) image on Channel 1.

6. 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.)

7. Set the remaining Interleave parameters (**Setpoint**, **Drive amplitude**, **Drive frequency**, **Gains**) to the main Feedback Controls values.

   Note: This can be done by setting the flags to the left of each parameter to “off” (grayed bullets).

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

9. In the Interleave Controls panel set **Interleave mode** to **Lift**.

   Note: For software versions 4.23 and lower set Interleave scan to **Lift** and switch **Interleave mode** to **Enable** in the Interleave Controls panel.

10. 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.

    Note: 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.

    Note: For software 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. For more details, refer to EFM Troubleshooting/ Pointers, Section 231.5.

11. Optimize the **Lift scan height**. For high-resolution, make the **Lift scan height** as small as possible without crashing the tip into the surface.

231.4.2 Frequency Modulation

For more quantitative results, use frequency modulation (FM). This technique provides a direct measure of change in resonant frequency sensed by the cantilever.

1. Follow all the steps required to perform phase detection EFM.
2. in the **Interleave Controls** panel set the **Input feedback** to **Frequency**.
3. Switch the **Data Type** for Channel 2 to **Frequency**.

Note: 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).

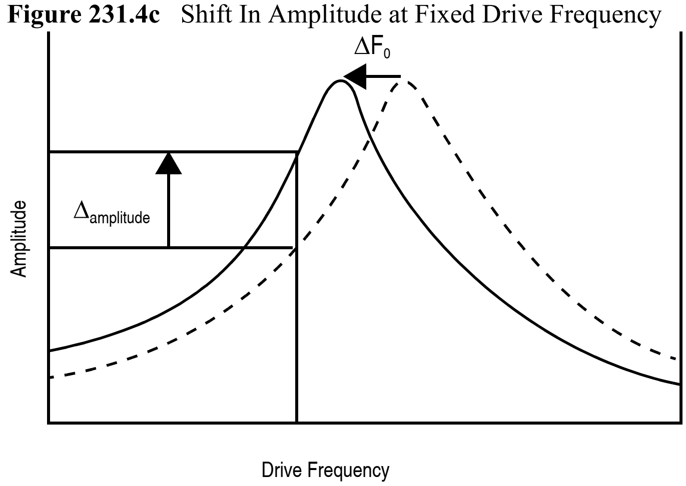
Note: For software Versions 4.23 and lower, there is no **Input feedback** setting; just switch the **Data Type** for **Channel 2** to **Frequency**.

231.4.3 Amplitude Detection

Amplitude detection is inferior to the phase and frequency detection methods described previously and is not the recommended technique for systems with an Extender Electronics Module.

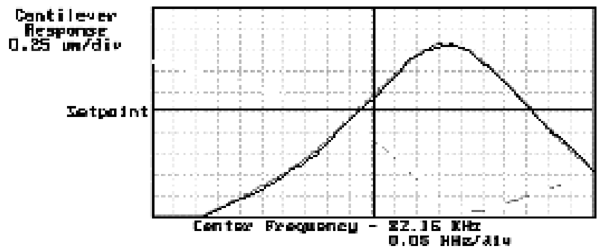
Amplitude detection is the only technique available for systems without the Extender Electronics Module. This section describes the differences in set up for amplitude detection. Changes in the cantilever amplitude provide an **indirect** measure of shifts in the cantilever resonance frequency as shown in Figure 231.4c.

Figure 231.4c Shift In Amplitude at Fixed Drive Frequency



1. Set the **Drive frequency** to the left side of the cantilever resonance curve, as shown in Figure 231.4d.

Figure 231.4d Amplitude Detection Cantilever Tune



Frequency Sweep

2. For maximum sensitivity, set the **Drive frequency** to the steepest part of the resonance curve.

Note: As the tip oscillates above the sample, a gradient in the electric force shifts the resonance frequency F_0 (see Figure 231.4c). Tracking the variations in oscillation amplitude while in LiftMode yields an image of the electric force gradients. You may use either side of the resonance, though we have obtained slightly better results on the low side, as shown in Figure 231.4c.

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 EFM Troubleshooting/Pointers

231.5.1 Use Low Setpoint When Tapping in Electric Field

If a voltage is applied during the main TappingMode line (as well as the interleave line) either through Analog 2 or an external source, be careful to ensure proper operation. It is possible to enter a mode in which the tip is never actually touching the surface, even though the amplitude setpoint is being met. As the feedback moves the tip closer to the sample surface, the resonant frequency of the tip is shifting (see Figure 231.4c). The closer the tip is to the surface, the larger the force gradient and the more the resonant frequency shifts. It is possible for the resonant frequency to shift enough so that the amplitude setpoint is met without the tip touching the surface. The tip can be a significant distance away from the sample. The height image has comparatively poor resolution as does the corresponding electric field gradient image. A significantly lower amplitude setpoint is needed to really tap the surface in the presence of a strong electric field.

231.5.2 Verify Electric Field at Surface

If applying Analog 2 voltage to tip or sample to generate an E-field, set the phase channel **Realtime planefit** to **None**. While in **Scope Mode**, vary the interleave **Analog 2** value and verify that phase signal shifts accordingly.

231.5.3 Fine Tune Lift Scan Height

Set the lift height to be as small as possible without hitting the surface, because lateral resolution of EFM improves with decreased tip/sample separation. The minimum lift height depends on the roughness of the sample, the difference between the amplitude setpoint and free air amplitude, and the quality of the height image. Hitting the surface usually produces phase data with extremely high contrast (either black or white pixels).

231.5.4 Fine Tune Interleave Drive Amplitude

Decrease the drive amplitude in the interleave line to further minimize the lift height. Be aware that the S/N ratio also decreases with decreasing drive, so there are diminishing returns after a point.

231.5.5 Optimize Tune in Vicinity of Surface

Select **View > Sweep > Cantilever Tune** to open tune panel while engaged. A tip offset of 30nm usually works well for smooth surfaces (if the tune has a noisy straight area in the middle instead of a smooth peak, the tip is hitting the surface and a larger tip offset is neededtypically for rough surfaces). Adjust drive frequency to center of peak or just to the left. If phase data is to be collected during the main line, zero the phase at the drive frequency.

231.5.6 Optimize Tune in Interleave

Find optimal lift height. Use view/sweep/cantilever tune to open the tune window while engaged. Choose a tip offset equal to the lift height. Make any changes to the drive frequency and drive phase to adjust the main tune (tip #3). In the Sweep controls panel, select the interleave controls button. Activate (green button) the drive frequency and phase, and Analog 2. With Analog 2 set to operating voltage, set the frequency to the center of the peak and zero the phase.

231.5.7 If Voltage is Needed, Use Analog 2 When Possible

It is often very difficult to get a high quality tapping mode image with an electric field present. Analog 2 has an important advantage over an external power supply – it can be used to create an E field only in interleave and be set to 0v in the main line (> v4.31). Its range is limited to +/- 12v.

231.5.8 Try Uncoated Si Tip

EFM has been successful with a variety of tips, including standard si tips with no metal coating (fesp, tesp, ltesp). The si is highly n-doped and is often conductive enough for EFM measurements. Metal coatings make the tip less sharp, decreasing lateral resolution. The standard tips also seem to give cleaner images over longer periods of time. There is likely a reduction in sensitivity to small
E fields with the decreased conductivity.

PART II: Surface Potential Detection

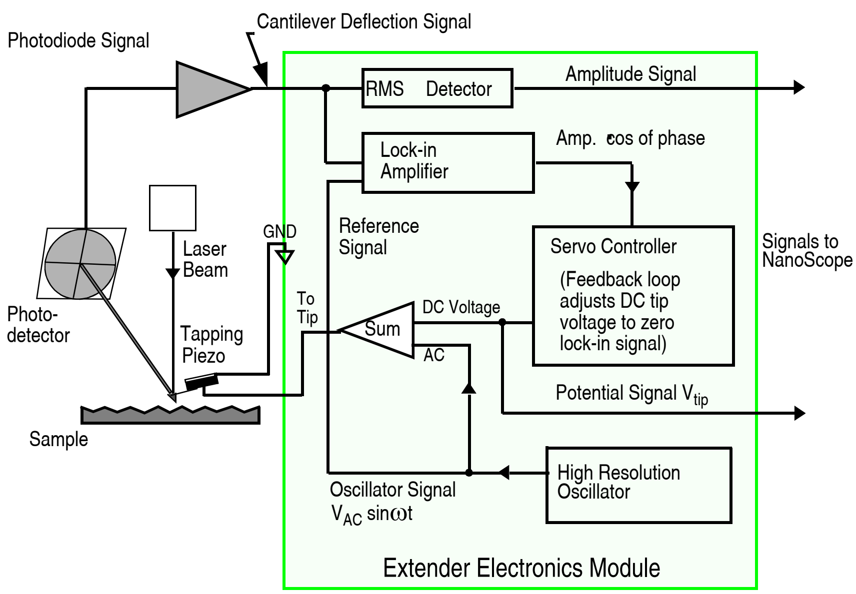
231.6 Surface Potential Detection—Theory

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 not equipped with the Extender Electronics Module.

231.6.1 Theory Overview

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 (See Figure 231.1a). 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 231.6a. On the first pass, the sample topography is measured by standard TappingMode. In TappingMode the cantilever is mechanically vibrated near its resonant frequency by a small piezoelectric element. On the second pass, the drive 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. If there is a DC voltage difference between the tip and sample, then there will be an oscillating electric force on the cantilever at the frequency ω. This will cause the cantilever to vibrate, and an amplitude can be detected.

Figure 231.6a Simplified Block Diagram of Extender Electronics Module in Surface Potential Mode



If the tip and sample are at the same DC voltage, there is no force on the cantilever at ω and the cantilever amplitude will go to zero. 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 surface potential. The voltage applied to the cantilever tip is recorded by the NanoScope Controller to construct a voltage map of the surface.

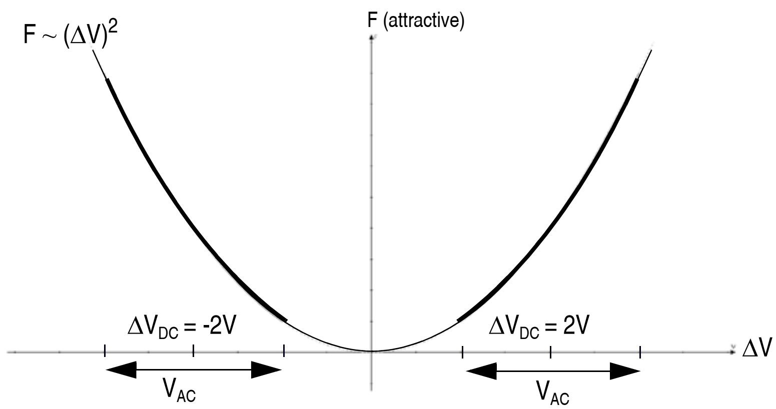
231.6.2 Theory Details

A good way to understand the response of the cantilever during Surface Potential operation is to start with the energy in a parallel plate capacitor, *U*. $U = \frac{1}{2}C(\Delta V)^2$

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:

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

Figure 231.6b Force as a Function of Voltage



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, $V_{AC}\sin\omega t$; ω is the resonant frequency of the cantilever. $\Delta V = \Delta V_{DC} + V_{AC}\sin\omega t$

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

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

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 oscillation of the cantilever. In regular TappingMode, the cantilever response (RMS amplitude) is directly proportional to the drive amplitude of the tapping piezo. Here the response is directly proportional to the amplitude of the F_ω drive term: $\text{amplitude of } F_\omega = \frac{dC}{dZ}\Delta V_{DC} V_{AC}$

The goal of the Surface Potential feedback loop is to adjust the voltage on the tip until it equals the voltage of the sample (ΔV_{DC}=0), at which point the cantilever amplitude should be zero (F_ω=0).

The larger the DC voltage difference between the tip and sample, the larger the driving force and resulting amplitude will be. But the amplitude alone is not enough to adjust the voltage on the tip. The driving force generated from a 2V difference between the tip and sample is the same from a negative 2V difference (see Figure 231.6b). What differentiates these states is the phase. The phase relationship between the AC voltage and the force it generates is different for positive and negative DC voltages (see Figure 231.6c through Figure 231.6f).

Figure 231.6c V_{AC} at ω, $\Delta V_{DC} = 2V$

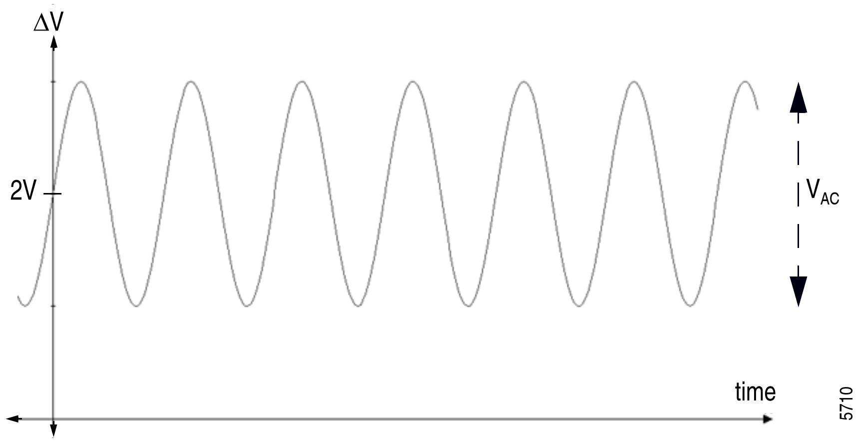


Figure 231.6d Force has major component at frequency ω, $\Delta V_{DC} = 2V$

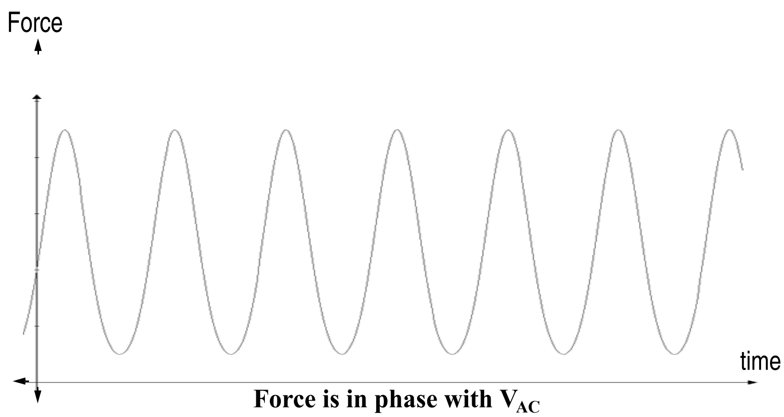


Figure 231.6e V_{AC} at ω, $\Delta V_{DC} = -2V$

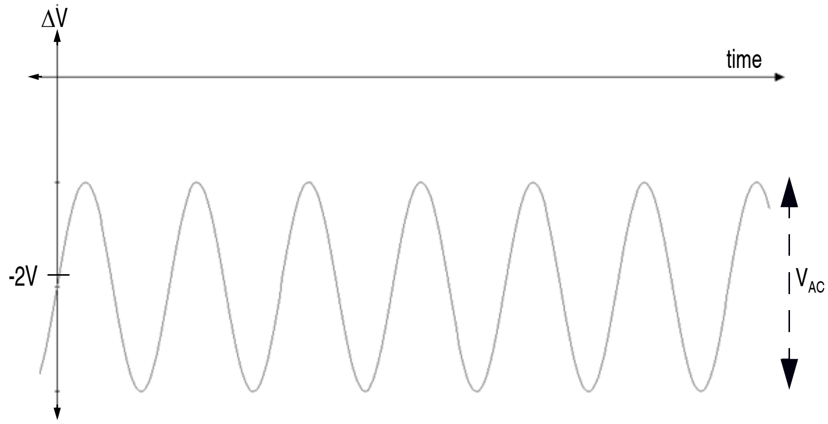
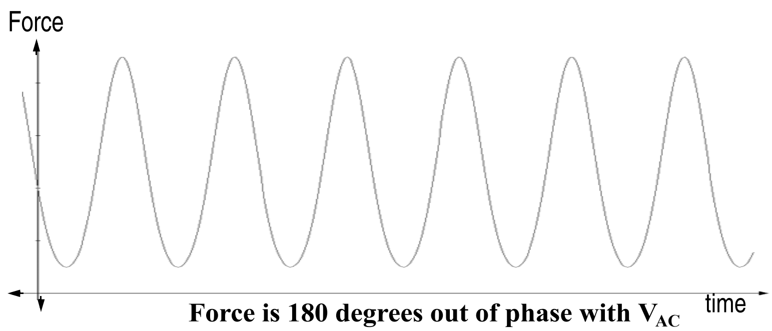


Figure 231.6f Force has major component at frequency ω, $\Delta V_{DC} = -2V$



Force is 180 degrees out of phase with V_{AC}

In the case where $\Delta V_{DC} = 2V$, the force is in phase with V_{AC}. When $\Delta V_{DC} = -2V$, the force is out of phase with V_{AC}. Thus, the cantilever oscillation will have a different phase, relative to the reference signal V_{AC}, depending on whether the tip voltage is larger or smaller than the sample voltage. Both the cantilever amplitude and phase are needed for the feedback loop to correctly adjust the tip voltage. The input signal to the Surface Potential feedback loop is the cantilever amplitude multiplied by the cosine of its phase. This signal can be accessed in the software by selecting Phase (interleave scan line) in one of the channel panels (note this is NOT the standard phase signal).

If $\Delta V_{DC} = 0$, the electric drive force is at 2ω. The component of the force at ω is zero, and the cantilever does not oscillate (see Figure 231.6g and Figure 231.6h). The Surface Potential feedback loop adjusts the applied DC potential on the tip, V_{tip}, until the cantilever's response is zero. V_{tip} is the Potential data that is used to generate a voltage map of the surface.

Figure 231.6g V_{AC} at ω, ΔV_{DC}= 0V

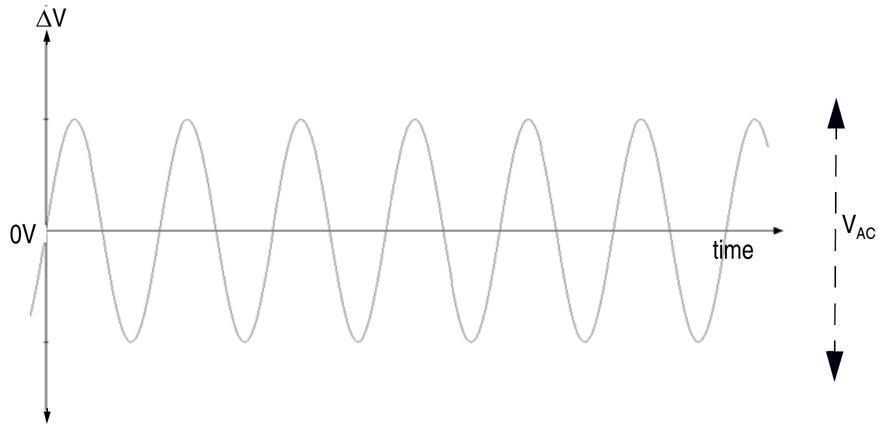
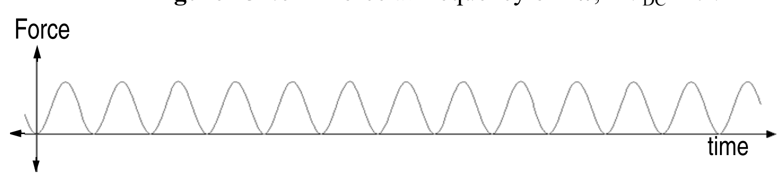


Figure 231.6h Force at frequency of 2ω, ΔV_{DC} = 0V



231.7 Surface Potential Detection, Preparation/Applying Voltage

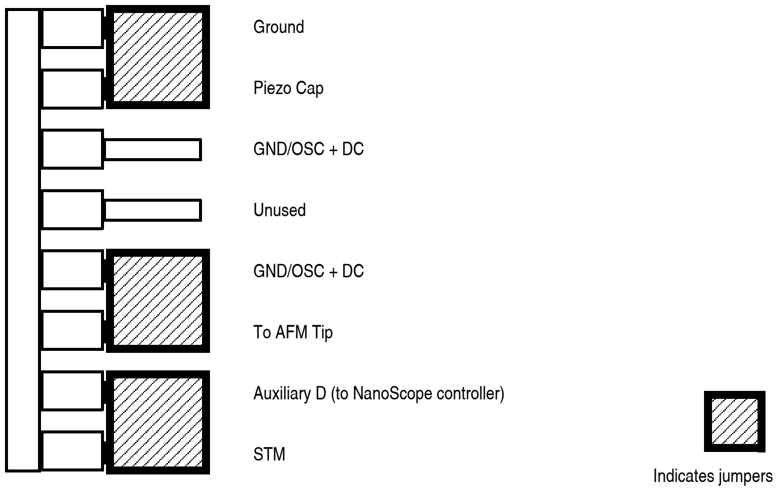
If the sample does not require voltage to be applied (e.g., measuring work function difference on a sample made up of several metals), skip to Section 231.8. It is often desirable to apply a voltage to one or more areas of a sample. This may be done in two ways—by connecting a voltage to the sample through 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. Review sections 231.1 & .2 and decide which set-up is most appropriate for your experiment.
2. Power down the NanoScope controller. Unplug the cable from the base of the MultiMode.
3. Disconnect and remove microscope head and scanner.
4. Locate the jumpers on the board visible underneath the base of the MultiMode through a cut-out in the steel as shown in Figure 231.3a. As shipped from the factory, the baseplate jumpers should appear as shown in Figure 231.7a.
5. Reconfigure jumpers as needed.

6. Replace and reconnect the scanner and head. Reconnect cable to base of MultiMode. Switch the NanoScope Controller power back on.

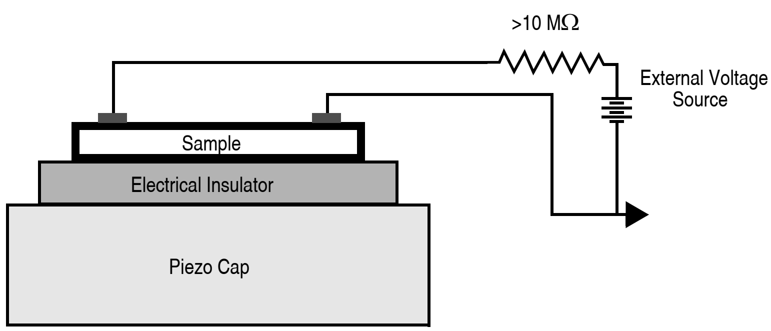
Figure 231.7a Normal Jumper Configuration as Shipped from Factory for MultiMode *with* Extender Electronics Module Installed. Piezo Cap is at Ground.



231.7.1 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 231.7a), and the sample should be electrically insulated from the piezo cap (see Figure 231.7b). 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). A piece of Kapton tape covering the bottom of a steel sample puck works well.

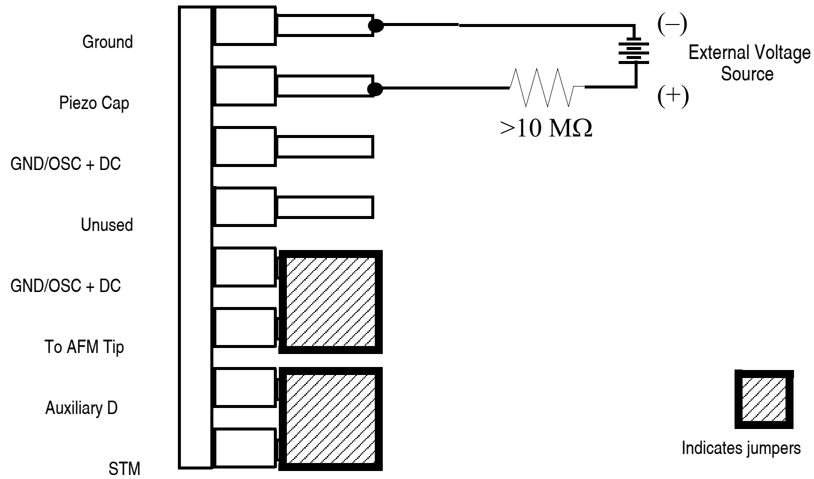
Figure 231.7b Insulating Sample from Piezo Cap



231.7.2 Applying Voltage to the Sample Through Piezo Cap

When voltage is to be applied to the sample via the piezo cap, configure the jumpers as shown in Figure 231.7c.

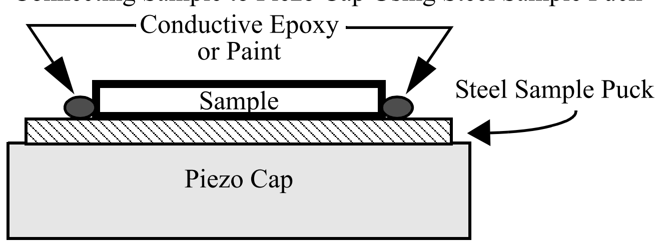
Figure 231.7c Jumper Configuration for Application of Voltage to Sample via Piezo Cap



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 to a standard sample puck using conductive epoxy or silver paint, as shown in Figure 231.7d.

Figure 231.7d Connecting Sample to Piezo Cap Using Steel Sample Puck



231.8 Surface Potential Detection—Procedure

Locate the two toggle switches on the backside of the Extender Electronics box (Figure 231.8a), and verify that they are toggled as shown in Table 231.8a.

Figure 231.8a Toggle Switches on Back of Extender Electronics Module

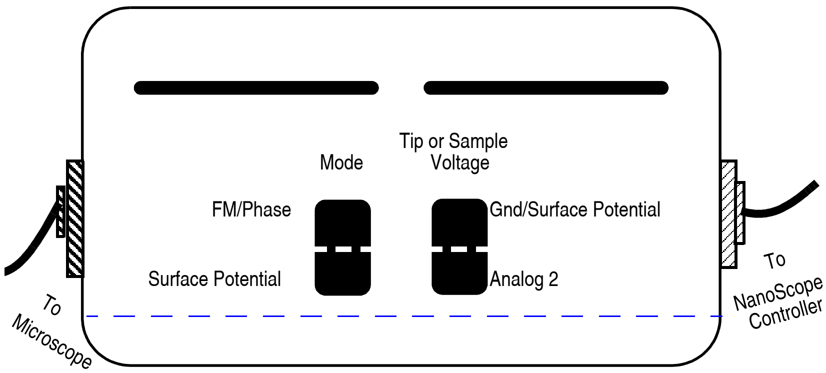


Table 231.8a Extender Electronics Module Toggle Switch Settings for Surface Potential Detection

	Mode		**Tip or Sample Voltage**	
	FM/Phase	**Surface Potential**	**GND/Surface Potential**	**Analog 2**
Surface Potential		X	X	
Standard Operation	X		X	

1. Mount a sample onto the piezo cap. Make any external electrical connections that are necessary for the sample.

2. Mount a metal-coated NanoProbe cantilever into the **electric field cantilever holder**. MESP or SCM-PIT model cantilevers (metal coated, 225 µm long, with resonant frequencies around 70 kHz, model MESP) usually work well.

   Note: 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.

3. Set up the AFM as usual for TappingMode operation.

4. Use Cantilever Tune and **AutoTune** (as described in Section 231.4.1) to locate the cantilever resonant peak.

5. Engage the AFM and make the necessary adjustments for a good TappingMode image while displaying height data in Channel 1.

6. Under the Panels menu select Interleave Controls. 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).

7. The Interleave Controls **Drive amplitude** is the AC voltage that is applied to the AFM tip V_{AC}. To start, choose a Drive Amplitude of 6V and set the corresponding button green.

8. Set the **Drive phase** in the Interleave Control panel. For MESP, SCM-PIT or FESP cantilevers (resonant frequency ~60-80khz) enter a **Drive phase** of –90 degrees. For TESP cantilevers (with resonant frequency ~300khz), try a **Drive phase** of –160 degrees. Make sure the corresponding button is green.

    Note: *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.

9. Choose a **Lift start height** of 0 nm and a **Lift scan height** of 100 nm. The **Lift scan height** can be readjusted later.

10. In the Interleave Controls set the **Input feedback** to **Potential** (set green button). 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.

    Note: *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. Adjust the FM gains. In the **Other Controls** panel set **FM igain** to 10 and **FM pgain** to 100 as a 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 231.9.

12. Optimize the **Lift scan height**. 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. See Section 231.9.2 for more details.

231.9 Surface Potential Detection Troubleshooting/Pointers

231.9.1 Unstable/Railed Potential Signal

1. Potential Signal Oscillates or is Unstable:
   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 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.

2. Potential Signal is Railed:
   If the Potential signal is perfectly flat and shows no noise even with a small data scale, the feedback loop is probably railed 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, 20V. Go into Scope Mode (View/Scope Mode) and check whether the data is railed at one of the limits.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 MESP or SCM-PIT cantilevers, 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.* For more deails on optimizing the drive phase, see Section 231.9.5.
   - 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.

231.9.2 Lift Scan Height

The smaller the tip/sample separation, the better the lateral resolution and signal to noise ratio of the Potential data. To minimize the separation, decrease the lift scan height to just above the point where the tip begins to touch the surface. When the tip crashes into the surface during the Potential measurement, dark or light streaks or dots 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** is affected by the sample roughness, scan speed, target amplitude used during tuning, etc.

A disadvantage of a lower lift scan height is that height artifacts tend to be more pronounced.

231.9.3 Tip Choices

It is 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 MESP and SCM-PIT tips make them well suited for sensitive electrical measurements. However, the coating increases the 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, H.O., Knapp, H.F., Stemmer, A., “Practical Aspects of Kelvin Probe Force Microscopy,” Rev. Sci. Instrum. 70 (1999) 1756.) that these changes in tip shape account for many of the DC shifts observed in Surface Potential images. Uncoated silicon probes have been successfully used for making some Surface Potential measurements. The standard TappingMode probes (Models TESP) and the Force Modulation probes (Model FESP) are highly n-doped silicon, often conductive enough for Surface Potential Detection. 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 Detection. 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.

If tips with higher resonant frequency are used, such as TESP (~300kHz) are used, a different drive phase must be used, see Section 231.9.5.

231.9.4 Tuning

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 usually require readjustment after the cantilever is moved.

231.9.5 Drive Amplitude

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 6V.

231.9.6 Drive Phase

The Drive phase adjusts the phase of the reference signal to the lock-in amplifier. The correct phase relationship must exist between the reference and the input signal to the lock-in for the Potential feedback loop to perform correctly. For cantilevers with resonant frequencies from 60-80kHz (such as MESP, SCM-PIT, and FESP), use an interleave Drive phase of –90 degrees. For cantilevers with higher resonant frequencies there is an increased lag in the electronics that must be compensated for. For cantilevers around 300khz (such as TESP) an interleave Drive phase near –160 degrees often works well.

To find the appropriate Drive phase for a specific cantilever, one can follow the method prescribed by Jacobs (Jacobs, H.O., Knapp, H.F., Stemmer, A., "Practical Aspects of Kelvin Probe Force Microscopy," Rev. Sci. Instrum. 70 (1999) 1756). Set up Surface Potential in Open Loop mode (see Section 231.9.7) by setting the FM gains to zero. Use a data channel to look at Phase during the interleave scan line. Set the Realtime Planefit equal to None for that channel. Adjust the interleave Drive phase until the Phase is centered at zero degrees. Subtract 90 degrees from this value and enter that for the interleave Drive phase to be used during the experiment. For example if a drive phase of –10 degrees centers the phase signal in open loop, then use a value of –100 degrees.

231.9.7 Open Loop Operation

Sometimes it is useful to run Surface 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.

Note: 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 9, select **Data type** to **Phase**.

231.10 Surface Potential Electronics

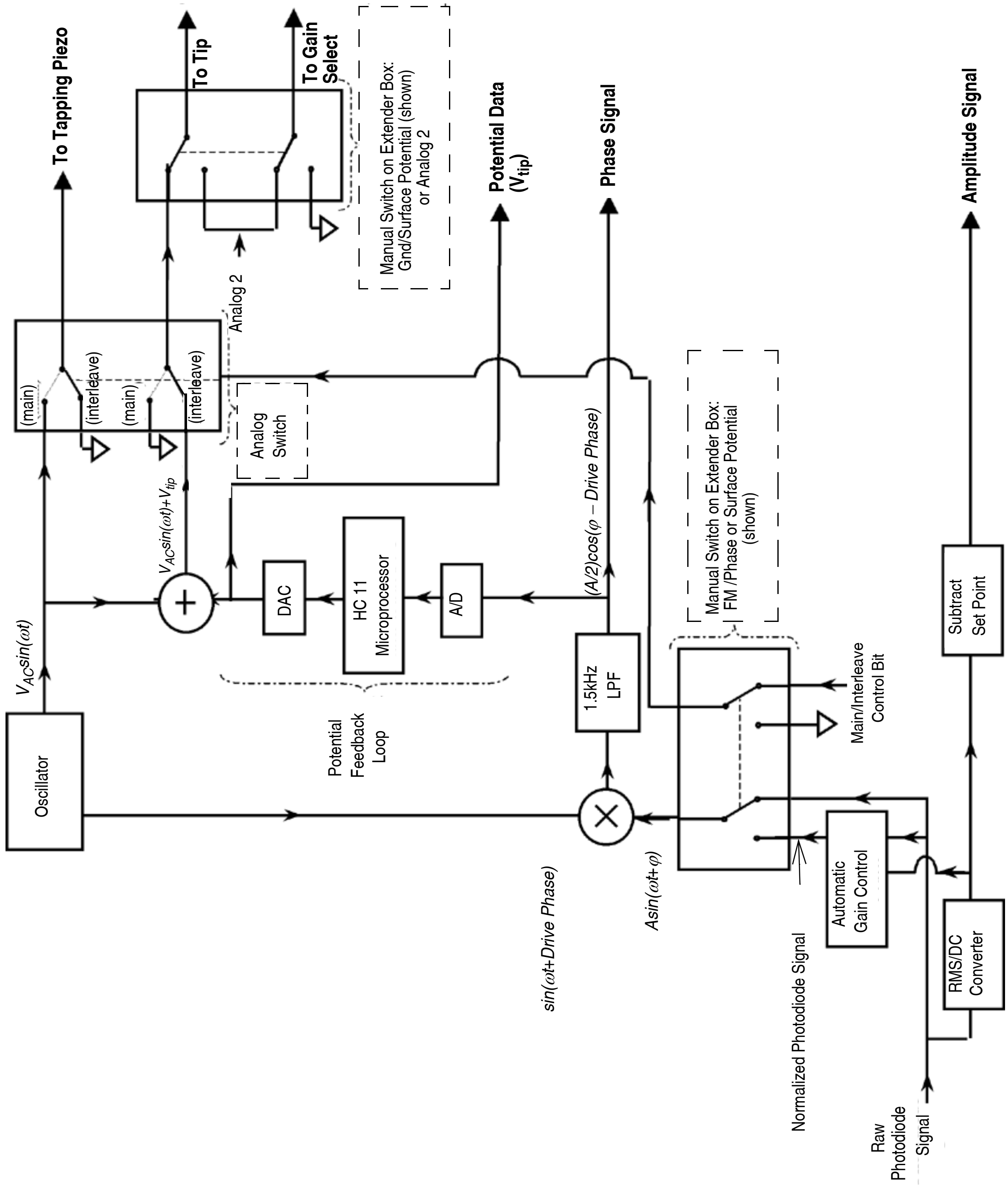


Figure 231.10a Surface Potential Extender Box Electronics

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 (see Figure 231.8a).

Selecting 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, $V_{ac}\sin(\omega t)$, 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, $V_{ac}\sin(\omega t)$, has an adjustable amplitude set in the software by the **Drive Amplitude**. The other output is a reference signal with fixed amplitude and adjustable phase, $\sin(\omega t + \mathrm{DrivePhase})$. This reference signal is multiplied with the photodiode signal, $A\sin(\omega t + \varphi)$, giving an output of:

$$\sin(\omega t + \mathrm{DrivePhase}) \times A\sin(\omega t + \varphi) = (A/2)\left[\cos(\varphi - \mathrm{DrivePhase}) - \cos(2\omega t + \varphi + \mathrm{DrivePhase})\right]$$

The resonant frequencies of tapping cantilevers are well above 1.5 kHz, so the low pass filter reduces the signal to $(A/2)\cos(\varphi - \mathrm{DrivePhase})$. 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 $\cos(\varphi - \mathrm{DrivePhase})$.