
Standard and Low-Current STM Converters— AFM, LFM and MMAFM Microscopes

232.1 Overview

This support note provides detailed instructions for performing standard and low-current (sub-picoamp) scanning tunneling microscopy (STM) using Digital Instruments scanning probe microscopes (AFM, LFM, or MMAFM) and either the standard STM converter or the low-current STM converter. Sections 232.2-232.6 provide information for standard STM while sections 232.7-232.13 cover low current STM.

232.2 Introduction

STM relies on “tunneling current” between the probe and the sample to sense the topography of the sample. The STM probe, a sharp metal tip (in the best case, atomically sharp), is positioned a few atomic diameters above a conducting sample which is electrically biased with respect to the tip. At a distance under 1 nanometer (.001 μm), a tunneling current will flow from sample to tip. In operation, the bias voltages typically range from 10 to 1000 mV while the tunneling currents vary from 0.2 to 10 nA. The tunneling current changes exponentially with the tip-sample separation, typically decreasing by a factor of two as the separation is increased 0.2 nm. The exponential relationship between the tip separation and the tunneling current makes the tunneling current an excellent parameter for sensing the tip-to-sample separation. In essence, a reproduction of the sample surface is produced by scanning the tip over the sample surface and sensing the tunneling current. The first STM operated in ultrahigh vacuum on cryogenically cooled samples. In the years

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following its invention, many variations on the STM theme appeared. P.K. Hansma and J. Tersoff wrote a good review article on the subject, containing experiments, theory, and over 100 references for *The Journal of Applied Physics*, 61 pp. R1-23, Jan 1987. Digital Instruments, Inc. introduced the first commercial STM, the NanoScope I, in 1986.

232.2.1 Overview of Standard STM

AFM, LFM and MultiMode microscopes require STM adapter heads to perform scanning tunneling microscopy (Figure 232-1).

MultiMode Converter Head



AFM or LFM Converter Head

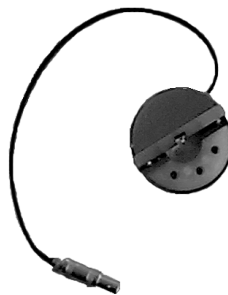


Figure 232-1 Standard STM converter heads.

Note: The plastic semi-circles on the STM converter heads are useful for Electrochemical work, but may be removed for better tip visibility in ordinary STM use.

In addition, the AFM and LFM microscope require the TipView adapter ring as shown in figure 232-2.



Figure 232-2 TipView Adapter Ring for AFM or LFM microscopes

There are two types of STM converter heads available: a standard version which measures current in the nanoampere range and a special, low-current version which measures current in the picoampere range. MultiMode and AFM microscope converter heads are similar except each has different cables.

The STM option is generally employed under the following conditions:

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

STM relies on a precise scanning technique to produce very high-resolution, three-dimensional images of sample surfaces. The STM scans the sample surface beneath the tip in a raster pattern while sensing and outputting the tunneling current to the NanoScope control station. The digital signal processor (DSP) in the workstation controls the Z position of the piezo based on the tunneling current error signal. The STM operates in both “constant height” and “constant current” data modes, depending on gain settings on the **Feedback Controls** panel. The DSP always adjusts the height of the tip based on the tunneling current error signal, but if the feedback gains are low (e.g., **Integral gain** < **1.0**; **Proportional gain** < **0.5**), the piezo remains at a nearly constant height while tunneling current data is collected. With the gains high (e.g., **Integral gain** > **1.0**; **Proportional gain** > **0.5**), the piezo height changes to keep the tunneling current nearly constant, and changes in piezo height are used to construct the image. The exponential relationship between tip-sample separation and tunneling current allows the tip height to be controlled very precisely. For example, if the tunneling current stays within 20 percent of the set-point value (the current to be maintained by the feedback system), the variation in the tip-sample separation is less than 0.02 nm.

232.2.2 Standard STM hardware

Some individual STM components are described below:

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

TipView adapter ring—The AFM or LFM microscope will sit directly on this base. The adapter ring re-routes the wiring within the microscope so that STM scanning is enabled.

Tips— Probes for the NanoScope STM must be less than 0.012" in diameter to fit into the tipholder. The two most commonly used tips are made from either a platinum iridium (PtIr) alloy or tungsten. The PtIr tips are mechanically formed and can be purchased directly from Digital Instruments. In general, PtIr tips provide better atomic resolution than tungsten tips, but tungsten tips are more uniformly shaped. Tungsten tips may perform better on samples with steeply sloped features such as compact or optical disks.

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

Picoamp boost box—Low-current STM operation requires the Picoamp boost box which is installed between the control unit and the microscope base.

232.2.3 Fine points of STM operation

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

232.2.4 STM operating modes

Recall that the two operating modes available on the NanoScope STM are selected with the **Data type** option in the **Channel** panel (refer to figure 232-3). **Height** data reflects the change in tip position required to maintain a constant tunneling current. The DSP interprets the tunneling current, calculates the difference from the desired tunneling current, and determines the voltage that must be applied to the piezo tube to keep the tunneling current constant. Due to the known characteristics of the piezoelectric material, the change in voltage applied to the piezo tube translates directly to a change in distance. This distance data is recorded throughout the scan and displayed on the screen as the “height” of the sample.

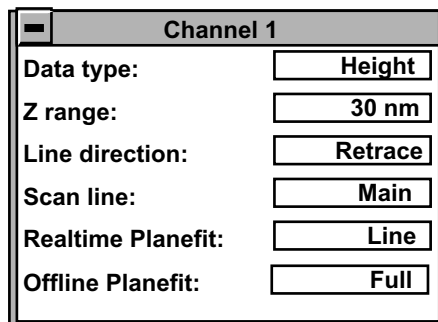


Figure 232-3 Typical STM *Channel 1* control panel parameters.

Current data is a measure of the tunneling current at each point tested on the sample. The DSP measures the voltage drop across a resistor in series with the tip and calculates the tunneling current as the tip scans the surface of the sample. The tunneling current at each data point is recorded and displayed on the screen. It should be noted that if different chemical species are present in a sample, the height data may not be a direct representation of the topography of the surface of the sample. Different atomic species within a sample may produce larger tunneling currents for a given bias voltage.

The two scan modes (height and current) require subtle changes in the menu parameters to operate effectively. The parameter changes also affect the application of the two modes. To operate effectively in collecting height data, the tip must closely track the sample surface. The gains must be maximized to force the piezo to respond quickly to the variations in the sample surface. The height mode is used for most applications. Conversely, the gains must be very low to keep the piezo from responding while collecting current data. After engaging, the tip scans the surface of the sample with very little variation in the piezo height. This constant height provides a reference from which to measure and record the fluctuations in the tunneling current. The current mode is most useful for imaging atoms with relatively small scan sizes.

232.2.5 STM-specific menu parameters

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

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

The **Bias** parameter controls the magnitude and sign of the bias voltage applied between the tip and the sample. A bias voltage encourages the tunneling current to flow. Although settings of **20—100 mV** are typical for conductive samples, the allowable setting ranges from **-10.0—10.0 Volts**. Positive settings of the bias voltage induce negative tunneling currents (i.e., electrons flowing from the tip-to-sample).

232.2.6 Optimization of STM scanning parameters

The process of selecting and optimizing scan parameters can be streamlined. In most cases the scan parameters are dictated by the sample. The **Data type** is usually the first parameter set and the **Proportional** and **Integral gains** are directly related to the **Data type**. The **Scan size** depends on the sample and the features of interest. The maximum **Scan rate** is usually related to the **Scan size**. The **Bias** voltage and the tunneling current **Setpoint** depend on the sample. Usually, they are set at a standard value for engagement and fine-tuned along with the gains and filters to enhance the quality of the image.

As discussed above, a **Data type** of **Current** is the best for atomic-scale images. This mode is not practical for rough surfaces, because the tip will crash into the surface at low feedback gains. Having **Data type** set to **Height** is usually better for all but atomic-scale scans. In general, height data images are best at higher feedback gains and slower scan rates.

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

When **Data type** is set to **Height**, often the best way to set gains is to view the Real Time scan in **Scope Mode** with the **Slow scan axis** set to **Disabled**. This allows the feedback to be tuned while looking at a single scan line of data. First, increase the **Integral gain** until oscillations start to appear, and then, back off a little. Next, adjust the **Proportional** and **Look ahead gains**. High frequency fuzz will appear on the signal when the **Proportional gain** is set too high. Setting the **Look ahead gain** too high will cause oscillations in the scope image and ripples on topview images. Setting the **Look ahead gain** even higher will cause the feedback loop to become unstable. The **Setpoint** current and the **Bias** voltage can also be adjusted using the scope display.

Bias voltages below 20 mV usually provide the best quality images on samples with surface conductivities equal to or better than graphite, but there are exceptions. Resistivity across the surface of a sample can be measured with an ohmmeter. For samples with high resistivity (greater than 1 megohm/cm), bias voltages of 100 mV or even higher may work best. For scans larger than 0.5 μm , it is sometimes better to increase the bias voltage by 50 mV to 100 mV over the value for smaller scans. A higher bias keeps the tip further from the surface, giving the feedback loop greater tolerance in tracking the surface at high speeds.

Increasing the **Setpoint** current can also be helpful for larger scans. This has the effect of raising the gain, but also brings the tip closer to the surface by a small amount. High setpoint currents of 6 nanoamps or more can also be useful in improving the signal-to-noise ratio for atomic images on some materials.

The **Feedback type** can be set to either **Log**, **Boost**, or **Linear** input transformations. Because the tip-to-sample separation is inversely proportional to the log of the tunneling current, the transformation performed on the tunneling current prior to the feedback calculation can have dramatic effects on the performance of the feedback loop. Linear input is more protective of the tip, because the feedback error signal responds exponentially to tip-sample separation. When the tip-to-sample separation decreases, the error signal rises exponentially, quickly driving the tip away. However, the error signal is unsymmetrical. The same sample-to-tip separation change that caused the tip to move away so quickly will generate a small error signal when the tip is higher than it is supposed to be. This unsymmetrical response in linear (**Lin**) mode will distort data. For this reason, the **Boost** and **Log** modes—with $\ln(I)$ used in the feedback calculation—are preferable for most samples, because they respond in a more symmetrical fashion to positive and negative sample slopes. The **Boost** mode is preferable for large scans with high vertical features such as compact disc stampers or integrated circuits. The proportional and integral gain can be reduced greatly when the **Boost** mode is used. The log input has the advantage of having a gain which is insensitive to the value of the **Setpoint** current.

Large scans cannot be taken at the same scan rate as small scans. When using the large scanners with scans above a few microns, the **Scan rate** should be lowered below 10 Hz. Best results can be obtained at scan rates of 1 Hz or less, although image-taking is slow. At these scan rates, the 128 X 128 and 256 X 256 data formats are most useful, quadrupling and doubling the frame rate over the 512 X 512 format for a given scan rate. To ensure there is no image degradation due to a high **Scan rate**, lower the rate and check for changes in the image. Check the **Scope Mode** view to ensure that the scan is not slew-rate limited in Z, as evidenced by an artificial “sawtooth” appearance in the scope trace.

232.2.7 Tunneling tips

Although the microscope will accept any .010" diameter tip, tips made of platinum iridium (PtIr) and tungsten are used most often. PtIr and tungsten tips are supplied by Digital Instruments. Tungsten tips can be electrochemically etched from tungsten wire by following instructions at the end of this chapter. In general, most of the discussion in this manual involving tips and noise reduction applies to both types of tips, but there are some applications which are tip specific.

PtIr tips seem to give better atomic resolution than tungsten in air and liquids, probably due to the lower reactivity of platinum. The PtIr tips are not as uniformly shaped as the tungsten tips, so freshly etched tungsten tips may provide cleaner data

when scanning steeply sloped surfaces such as compact or optical disks. For tunneling on surfaces immersed in conductive liquids, coated tips have been used. Glass coatings are removed from the very end of the tip by briefly applying a high-bias voltage. The quality of the mechanically formed PtIr tips will vary. Some give beautiful images as soon as tunneling starts; others start well but degrade over time; while others start noisy and stay noisy.

232.2.8 Sample surface

Samples to be imaged with a scanning tunneling microscope must conduct electricity to some degree. In many cases nonconductive samples can be coated with a thin layer of a conductive material to facilitate imaging. The sample surface must be conductive enough to allow a few nanoamps of current to flow from the bias voltage source to the area to be scanned. NanoScopes have been used to scan gold, silver, platinum, nickel, copper under oil, chrome plating, doped silicon under oil, conducting polymers, amorphous carbon, blue diamond, diamond-like carbon films, carbon fibers, graphite, iron-oxide compounds, semi-metals, doped semiconductors (molybdenum disulfide), cobalt-chromium compounds, stainless steel, liquid crystals, and other materials. Oxide layers more than a few atoms thick on the sample tend to affect the scanning and wear down the tip as it is dragged through the oxide. The feedback loop will extend the tip until a tunneling current flows, even if it must push the tip through an oxide layer (if it can). If oxide presents a problem, keep the sample covered with oil or operate the microscope in a glove bag filled with nitrogen or argon. The standard NanoScope microscope heads were not designed to operate in UHV.

On samples which are noisy or tend to oxidize, tunneling under oil or scanning in a glove box filled with inert gas can improve the imaging. Silicon oil or paraffin oil (mineral oil) also work well with some samples. The only problem involved with the use of oil is the increased difficulty in the coarse positioning of the tip. The reflection of the tip comes off the liquid instead of the surface of the sample. It is difficult to tell when the tip is close to the sample surface. The best approach is to lower the tip until it just touches the surface of the oil, then select **Engage**.

232.2.9 Vibration isolation

The microscope should be isolated from sources of vibration in the acoustic and subacoustic frequencies. This requirement can be relaxed somewhat for large-scale images, but atomic-scale work is extraordinarily sensitive to ordinary room vibrations.

As a final note, the best way to reduce coupling from vibrations is to eliminate as many sources of vibration as possible. Remember that vibrations can be transmitted to the microscope over the cable. To reduce this phenomenon, prevent tension in the cable and keep it away from fans and other noise sources. Also, keep the micro-

scope away from sources of acoustic noise. Loud conversation can disrupt atomic scale images. Air currents can also disturb atomic images, so it is best to run the microscope with the hood or similar cover on.

232.3 Basic STM Operation

232.3.1 Setting Up the STM Hardware

AFM or LFM Microscopes:

1. If you are using an AFM or LFM microscope, unplug it from the controller. Place the AFM (or LFM) base on the TipView adapter ring while aligning the 25-pin connector as shown in figure 232-4 below.



Figure 232-4 Place AFM base on the TipView adapter ring.

Note: Small magnets in the TipView adapter ring help align it to the AFM or LFM base, however they are not designed to hold the ring when the microscope is lifted. Always support the combined unit by holding both the microscope base and the adaptor ring when lifting it.

2. If you are using an AFM or LFM, plug the scanner into the TipView adapter ring through the 9-pin cable as shown in figure 232-5.



Figure 232-5 Connect the scanner to the TipView adapter ring through the 9-pin cable.

CAUTION! Do not attempt to plug the 9-pin cable from the TipView adapter ring directly into the AFM base. Severe damage will occur to the microscope.

3. After loading the sample and inserting an STM tip (section 232.3.2), place the STM converter head atop the scanner. If you have an AFM or LFM, plug the lemo connector from the head into the TipView adapter ring as shown in figure 232-6.

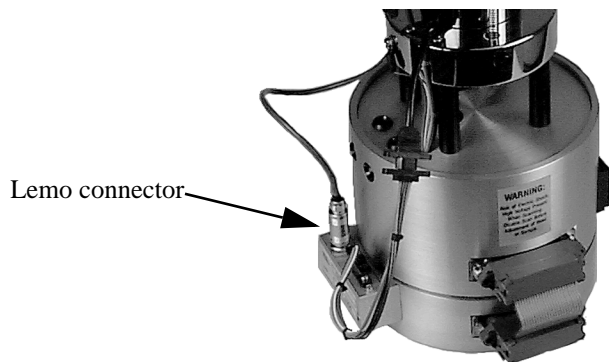


Figure 232-6 Lemo connector from STM head plugs into TipView adapter ring.

MultiMode Microscopes:

If you have a MultiMode microscope, the STM converter head plugs directly into the microscope base. Verify that the mode switch on the MultiMode's base is toggled to STM. The LED on the top of the base should now glow blue.

232.3.2 Imaging samples

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

1. Attach a sample to a metal puck using an electrically conductive (e.g., silver-based) epoxy such as Dynaloy 325 or equivalent. Ensure that the sample itself is conductive and in electrical contact with the top of the scanner tube (the scanner cap).
2. Insert a new tip in the STM converter head tipholder. The tips come in a small, plastic snap-box filled with foam. Grip the tip with tweezers near the sharp end, then insert the blunt end of the tip into the tipholder. Do not bend the tip, tipholders feature a curved tube to grab the tip. The tip must be inserted so that it protrudes beyond the bottom profile of the tipholder; otherwise, the tipholder will bottom out on the sample surface before the tip engages. Ensure that the tip is tightly held.
3. Place the converter head atop the scanner, checking for clearance between the tip and the sample as the head is lowered. If the tip appears to be too low and threatens to crash into the sample, adjust the scanner screw(s) to obtain more clearance. The head will sit securely atop the scanner under its own weight, assisted by the pull of magnets mounted in the scanner. Newer converters have pins for connecting retaining springs to increase rigidity.
4. Select the STM option. Click the left mouse button with the cursor on the **Real Time** icon in the menu bar to select the Real Time operating mode. Pull down the **Microscope** menu by clicking on the **Microscope** item in the menu bar, click on the **Select** command, then click on **STM** and **Ok** to place the microscope into STM mode.
5. For "J" scanners, set the **Scan Controls** panel parameters to the values shown below. It is sometimes helpful to start in Scope Mode (**Real Time/View/Scope**

Mode) at a reduced **Scan size**. Once gains are adjusted for the small scan, the **Scan size** may be increased.

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

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

NOTE: The **Bias** and **Setpoint** parameters must be set according to the conductivity of the sample. Samples which are excellent conductors (e.g., gold) exhibit high electron flow; therefore, the error signal is relatively large. Good conductors can operate with a **Bias** of **1–50 mV**. Conversely, if the sample is a semiconductor, the **Bias** may have to be set higher (e.g., **1000 mV**) to obtain an image. Beware of high bias values! If the **Bias** is set too high for a given conductor, electrical arcing may occur, damaging the tip and/or sample.

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

- Verify that the tip is withdrawn from the sample surface.
- Invoke the **Microscope/Offset** command to observe the offset current of the preamplifier in nanoamps. The offset should read between $\pm 0.1 \text{ nA}$. If the value is too large, check that all cables are plugged in and that the tip is not contacting the sample.

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



Depending on the condition of the tip, the image on the screen will be either clear or noisy.

The **Z Center Position** bar graph on the display monitor reflects the Z position of the piezo during the scan. Under most circumstances, the white bar should be in the center of the graph, indicating that the Z voltage for the piezo is fluctuating around zero volts. If the sample is not level, or if there is drift in the mechanical system after the tip has engaged, the Z center position will fluctuate during the scan or drift off-scale.

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

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

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

A poor tip—either because it has touched the sample, has contamination on the end, or because of its manufacturing—will render a poor image. In this case, there is little likelihood that the tip will recover and the best procedure is to replace the tip. Pull down the **Motor** menu by clicking on **Motor** in the menu bar, then click on **Withdraw** (or click on the **Withdraw** icon) to stop scanning, and retract the probe from the surface. After the tip is withdrawn, the “Secured” message will appear in the status bar of the control monitor. Remove the bad tip and install a new one. Verify there is sufficient clearance between tip and sample after installing the new tip—a new tip will extend a different distance out of the tipholder than the old tip.

To Improve Image Quality...

- Vary the **Scan size** and the **X offset** and **Y offset**. Move to a new location on the sample by changing the X offset and/or Y offset of the scan. The X and Y offset parameters define the center of the scan. Often, a better image can be obtained on a different portion of the sample. The X and Y offsets can be changed by altering the values in the control panel, or by using the **Offset** command in the menu bar on the display monitor.



- Alternate the commands **Withdraw** and **Engage** a couple of times. This raises and lowers the tip and may get rid of contamination on the end of the tip.
- Use the **Step Motor** command to single-step the tip up a step or two. Be sure to minimize the **SPM step size** before stepping the motor down. Watch the **Z Center Position** scale on the display monitor to verify that the piezo has sufficient room to retract so the tip will not harm the surface of the sample. Stepping the tip down until the **Z Center Position** goes to the retracted end of the scale where the indicator turns red will usually destroy the tip.
- Vary the **Setpoint** current over the range 2 to 10 nA. If needed, current may be increased up to 48 nA for a very brief period of time.
- As a last resort, oscillate the tip by setting the **Integral gain** high (about **100**) for a second. The oscillation will show up on the display as a grainy pattern of light and dark. Set the **Integral gain** back down and check if the signal is quieter than before. Setting the Integral gain high for a brief period is also useful for cleaning debris from the tip.
- If none of the above procedures improves the quality of the signal, replace the tip and try again. If that doesn't work, call Digital Instruments for technical assistance.

232.4 Spectroscopy with the STM

The NanoScope STM performs limited spectroscopic operations under the two scanning tunneling spectroscopy (STS) modes of operation. The variation of the tunneling current due to variations of the bias voltage or tip-to-sample separation can be tested and recorded at a single point with the **View / STS Plot** modes. These are discussed in this section. A related form of imaging, current imaging tunneling spectroscopy (CITS) is described in an appendix of the *Command Reference Manual*.

232.4.1 STS plot modes

In the **STS Plot** modes, the tip is positioned at a point on the surface, and a spectroscopic plot is acquired and displayed in a scope format. Between plots, the feedback is run to establish the tunneling current to the setpoint value. The different types of STS Plots that can be acquired are:

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

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

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

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

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

232.4.2 Operation of STS

In the following sections, the operation of the spectroscopic functions of the NanoScope III STM will be discussed. Additional information can be obtained from the *Command Reference Manual*.

STS Plot

There are several items that you should be aware of when using the NanoScope to acquire spectroscopic plots with the **STS Plot** commands. The spectroscopic capabilities can provide information that can help to distinguish different species although exact species identification is difficult, especially in air. The spectroscopic plots should aid in comparative studies between samples or between different regions on a sample, but they will not reveal the precise make-up of that sample.

A comparison to STM imaging reveals two somewhat conflicting requirements. As a good starting point, the sample and tip should produce consistent STM images. The images should repeat well from frame to frame and be fairly free of noise or areas on the surface that appear unstable. For current-versus-voltage type plots, the spectroscopic plots may be nice and smooth and repeat well, but switching back to the STM imaging mode reveals images which are quite noisy. This can be attributed to the fact that the quality and uniformity of the tip is probably more critical for imaging than for making the rather simple spectroscopic plots. This is generally not true for the I vs. S plots. Some general recommendations for acquiring spectroscopic plots are:

1. Low settings for the **Integral gain** are preferred. Since the tip is not tracking any topography, the lower gains are acceptable and tend to make the plots more stable.

2. The maximum input range of ± 100 nA on the NanoScope with the standard preamps will restrict the current-versus-voltage plots.
3. The spectroscopic functions do not adjust for any loss of bias voltage due to IR losses caused by the input impedance of the preamplifier. This has different effects for the STS $i(v)$ and STS $i(s)$ functions which are discussed individually below:

STS $i(v)$

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

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

232.4.3 Continuous Imaging Tunneling Spectroscopy (CITS)

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

232.5 Obtaining Images with STM

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

1. Highly ordered pyrolytic graphite (HOPG).
2. A gold calibration ruling.

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

232.5.1 Imaging highly ordered pyrolytic graphite (HOPG)

This section describes how to use the NanoScope to image graphite atoms in highly ordered pyrolytic graphite (HOPG) with an AFM or LFM. MultiMode users should ignore references to the TipView and watch for notes specific to MultiModes.

1. Set the AFM or LFM microscope onto the TipView adapter ring and connect it to the base using the 25-pin cable. Connect the scanner to the STM base using the 9-pin wire cable. The microscope should then be placed on a vibration-isolation platform and connected to the controller.

2. As it comes from Union Carbide, the HOPG sample is ready to be cleaved. Carefully cleave a thin layer of the sample with a sharp razor blade, then cut it into fourths. Mount it on a puck using conductive adhesive. Apply sticky tape to expose a fresh layer. You will notice that graphite is slippery and easily dropped. Be careful! Verify that the sample is electrically bonded to the puck.

3. Insert a new tip in the tip-holding tube mounted on the white ceramic cross-member of the converter head. The tips come in the small plastic snap-box filled with foam. The tips are inserted into the foam blunt end first so the sharp end of the tip protrudes from the foam. Grip the tip with a tweezers near the sharp end and insert the blunt end of the tip into the tipholder. The tip should be inserted so that it protrudes about 2 mm and should be fairly tight in the tipholder. (**NOTE:** Handle the converter head as little as possible during these operations to prevent thermal drift.)

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

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

5. Looking at the tip with a 25X magnifier, lower the tip down with the scanner screw until the tip is about a tenth of a millimeter or less (about .004 inch or less) above the sample. For non-vertical engage scanners, try and adjust the screws such that the head is not tilted too severely. It is usually easier to use only one coarse-adjustment screw to do this if you do not need to move the tip too far. If necessary,

adjust the rear screw so that there is a minimal amount of tilt in the head from front to back. A reflection of the tip may be visible on the sample. Using the coarse-adjustment screws, lower the tip until there is only a slight gap between the end of the tip and its reflected image — do not bring the ends together. If you do, you will almost certainly have to replace the tip. Don't worry at this point if the head is tilted.

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

7. Go to **Real Time** mode. Click on the **Microscope / Select** command, then click on **Small Sample** ("Base" box), **STM** ("Head" box) and the appropriate "Scanner" selection (e.g., **TVEI** if the "E" scanner is being used).

Note MultiMode users: If you are using an extender, select **Small Sample Extended MultiMode STM,E**. If not, use small sample, **STM**, **MMSTME** (if an "E" scanner is being used).

- To look at graphite atoms, it is necessary to set the **Scan size** to about **5–20 nm**. Use the mouse to change the parameter.
- Set the remaining parameters so that the **Feedback Controls** panel is as shown in figure 232-7 below.

8. As a final check, click on the **Offset** command in the **Real Time / Microscope** menu. The offset should be between ± 0.1 nA. Proceed to the next step if the system reports a reasonable value.

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

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

Figure 232-7 *Feedback Controls* panel for graphite.

9. Now, let's start tunneling. Click on the **Real Time / Motor** item and then click on the **Engage** command, or use the **Engage** icon. The status bar on the control monitor shows how many microns the tip has traveled. If the travel distance gets up to the default limit, engagement will halt and the software will prompt you to check the tip alignment. If the tip is not at the surface yet, repeat **engage**. When the tip engages with the surface and tunneling occurs, the computer will beep and an "Engaged" message will appear in the status bar.

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

NOTE: If version 4.20 or higher software is used, the STM must have the **In polarity** parameter set to **Forward** on the **Microscope / Calibrate / Detector** panel. In previous versions of software, the setting didn't matter and may have been set to **Reverse**. This setting will cause false engagement with newer Z.exe programs. Each **Microscope/Select** file that does STM should have its **Microscope/Calibrate/Detector** set to forward polarity.

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

- A good signal is one which clearly shows the repeating pattern of the carbon atoms. Compare this image with the graphite image that may be stored in the C:\DATA directory. Check to see how the image repeats on scans from bottom-to-top and top-to-bottom. Usually, when the tip is first engaged, there will be a drift in the piezo which will cause the image to repeat poorly. The most obvious sign of drift is features which are compressed vertically in one frame and vertically elongated in the next frame. This drift will decrease with time.

Click on a scan parameter and drag the mouse to change the parameter. Lower the **Integral** and **Proportional** gains near zero and vary the **Bias** voltage to see how the image changes. Vary the **Scan size** to get a feel for how the image can be magnified. Instead of displaying an image of the tunneling current, select **Height** at the **Data type** line to display the Z (vertical) signal. You will have to increase the gains and decrease the scan rate to get a good Z-height image.

To use an "E" or "J" scanner on an atomic scale with the **Data type** set to **Height**, the **Z limit** parameter (**Scan Controls** panel) should be decreased to **55 Volts**.



Click on the **Scope Mode** command in the **Real Time / View** menu to see what the signal looks like for each trace across the sample. Return to the **Image Mode** by clicking on the **View / Image Mode** command, or icon.



- A poor tip, either because it has touched the sample, has contamination on the end, or because of its manufacturing, will give a noisy signal. In the worst case, the image will have no pattern at all, and Z on the display will run off scale in both directions. In this case, there is little likelihood that the tip will recover, and the best procedure is to replace the tip. Pull down the **Real Time / Motor** menu, then click on **Withdraw** (or click on the **Withdraw** icon) to stop scanning, and retract the probe from the surface. After the tip is withdrawn, the "Secured" message will appear in the status bar of the control monitor. Before removing the head, it's a good idea to execute the **Withdraw** command several times to protect the sample from being scratched when the head is picked up. Extend the scanner screws a few turns, unplug the preamp, remove the head, and replace the tip. Try to leave the new tip protruding the same amount as the old one. Be sure there is sufficient clearance after installing the new tip, because the new tip will extend a different distance out of the tipholder than the old tip.
- The nominal Z position of the piezo provides useful diagnostic information. The bar graph to the right of the image on the display monitor reflects the Z position of the piezo during the scan. Under most circumstances, the "Z center position" bar should be in the center of the graph to indicate that the Z voltage for the piezo is fluctuating around zero volts.



If the sample is not level or if there is drift in the mechanical system after the tip has engaged, the "Z center position" will fluctuate during the scan or drift off scale. If the "Z center position" reaches the "Extended" end of the scale, the maximum voltage is being applied to the piezo, but the tip still cannot reach the surface. Similarly, the "Retracted" end of the scale means that the maximum voltage of opposite polarity has been applied to the piezo, and the tip cannot be lifted from the surface. The "Retracted" end of the scale is more dangerous, because damage to the tip or sample can result.

If the "Z center position" gets close to either limit, use the **Withdraw** command followed by the **Engage** command to reposition the "Z center position" near the middle of its range. If there is drift in the Z center voltage, use the **Real Time / Motor / Step Motor** command with a small **Step size** to keep the tip within the Z piezo range. This drift will eventually settle out.

EXCEPTION: The **Z limit** represents only a portion of the maximum **Z range** (440 V). When using a **Z limit** of less than **440 V**, version 4.xx software will attempt to recenter the Z-axis if you double-click on the **Z limit** value or change it.

Sometimes the tip will show atomic structure on graphite with noise which is large enough to reduce the definition of the structure. In these cases, it may be possible to

reduce the noise without replacing the tip. Any or all of the following procedures may be tried:

- Vary the **Scan size** and the **X** and **Y offsets** (**Scan Controls** panel). Move to a new location on the sample by changing the **X** and/or **Y offset** of the scan. The **X** and **Y offset** parameters define the center of the scan. Often, a better image can be obtained on a different portion of the sample. The **X** and **Y offsets** can be changed by altering the values in the control panel or by using the **Offset** command in the menu bar on the display monitor.
- Alternate the commands **Withdraw** and **Engage** a couple of times. This raises and lowers the tip and may get rid of contamination on the end of the tip.
- Use the **Real Time / Motor / Step Motor** command to index the tip down a step or two. Be sure to minimize the **Step size** before stepping the motor down. Watch the "Z center position" scale on the display monitor to be ensure the piezo has enough room to retract so the tip will not harm the surface of the graphite. Stepping the tip down until the "Z center position" goes to the retracted end of the scale where the indicator turns red will usually a tip. Sometimes, though, bad tips can be reconditioned by being driven several steps into the surface and then withdrawn and re-engaged.
- Vary the **Setpoint** current over the range **2—10 nA**. If needed, you can increase it up to **48 nA** for a very brief period of time.
- As a last resort, oscillate the tip by setting the **Integral gain** high for about one second. The oscillation will show up on the display as a grainy pattern of light and dark. Set the **Integral gain** back down and check if the signal is quieter than before.
- If none of the above procedures improves the quality of the signal, you should cleave the sample, replace the tip, and try again. If that doesn't work, review all parameter settings or call Digital Instruments for technical assistance.

232.5.2 Imaging a gold calibration ruling.

A gold calibration ruling is supplied with systems purchased with the 12- μm range, "E" scanners. This section describes how to image the ruling and assumes you have performed the previous section for imaging graphite. An alternative is the platinum coated 10 μm x 180nm standard supplied with all J scanners and with MultiModes when purchased with NanoScopes. It too, must be made as described in step number two of this section.

1. Although it is not as important for large-scale scans, the microscope should be put on a vibration isolation platform. Also, connect the microscope to the controller according to the instructions in the earlier part of this chapter.

2. Attach the calibration ruling to a sample puck with the ruled side facing up. Use carbon glue or silver epoxy to provide a conduction path between the sample puck and the top surface of the ruling. This step is very important because without a conduction path the **Bias** voltage will not be applied to the sample surface and the tip will crash into the sample surface in a effort to detect the tunneling current. If the sample is already attached to a puck with sticky tab adhesive, use Naptha to clean the excess off. Then apply conductive material from puck to gold.

3. Verify that a tip is in the tip-holding tube attached to the cross-member on the converter head.

4. Place the head on the three balls of the scanner. Make sure the screw(s) are extended enough that the tip does not contact the sample.

5. Looking at the tip with a 25X magnifier, lower the tip down with the coarse-approach screws until the tip is about a tenth of a millimeter or less above the sample. This can be done by bringing the end of the tip as close as possible to the reflected image without allowing them to meet. If the tip does touch the sample, replace it, as it will almost certainly be unusable.

6. Be sure the power switch in the right rear of the controller is in the on (up) position. The power lamp on the front panel of the controller will be illuminated.

7. If you have not already done so, start the main program by entering "Z".

- Go to the **Real Time** mode.
- Click on the **Microscope / Select** command, then click on **Small Sample** ("Base" box), **STM** ("Head" box) and the appropriate "Scanner" selection (e.g., **TVEI** if the "E" scanner is being used).
- Note MultiMode users: If you are using an extender, select **Small Sample Extended MultiMode STM,E**. If not, use small sample, **STM, MMSTME** (if an "E" scanner is being used).

Set parameters in the **Feedback Controls** panel as shown in figure 232-8. The **Scan Controls** panel can have its parameters set as follows: **Scan size 3 μ m; Z limit 440**

V; Number of samples 256; Scan rate 2.44 Hz. Set the **Data type** parameter on the **Channel 1** panel to **Height**.

Feedback Controls	
Integral gain:	10.00
Proportional gain:	5.00
Look ahead gain:	0.00
Bias:	500.0 mV
Setpoint:	5.00 nA
Analog 1:	0.00 mV
Analog 2:	0.00 mV

Figure 232-8 STM control panel for calibration ruling

9. Click on the **Real Time / Motor / Engage** command to begin scanning. At this point, an image should begin to form on the screen; the supplied gold sample has cross rulings of 1 μ m pitch. Go to the **Scan Controls** panel and increase the **Scan size** item to 10 μ m. If the picture appears grainy or noisy, the tip is probably poor and should be replaced. In any case, you can try the following procedures to improve the image:

- Change the **Integral** or **Proportional gain** parameters. Gain values which are too high will cause graininess in the image, whereas low gains will cause image smearing, particularly where the surface has sharp, abrupt features.
- Vary the **Scan rate**. A **Scan rate** which is too high will cause smearing (like a low gain setting) and lower scan rates may test your patience.
- Vary the **Setpoint** current and **Bias** voltage items.

232.6 Troubleshooting for STM

This section addresses errors or malfunctions encountered during the operation of the AFM while performing STM.

232.6.1 Head and microscope-related problems

This section deals with problems related to the scanners or the microscope. If a problem exists with a scanner, try a second one under the same conditions, if possible. Otherwise, the following list of symptoms and cures may be helpful:

232.6.2 Head engages immediately

If the STM engages immediately after initiating the **Motor / Engage** command, then one of the following probably occurred:

tip on surface— Make sure the tip is not touching the surface of the sample. Adjust the scanner screw(s) upward until the tip is far from the surface.

controller off— Verify that power to the controller is on and that the controller is connected to the computer workstation via the 25-pin cable.

TipView adapter ring is not connected— Verify that the 25-pin cable on the TipView adapter ring is connected to the AFM base.

head has offset— Follow directions in section 232.3.2 to determine whether the scanner requires adjustment to its offset.

In polarity parameter set to Reverse— Check to see that the **In polarity** parameter on the **Detector Calibration** panel (**Microscope / Calibrate / Detector**) is set to **Forward**.

disconnected— Verify that the microconnector from the converter is plugged into the TipView ring and the TipView ring is plugged into the controller.

232.6.3 Tip never engages

If the tip never engages, test for the following:

disconnected—Verify that the lemo connector for the head is plugged into the TipView adapter ring and the 25-pin connector on the TipView adapter ring is plugged into the AFM base. Make sure the microscope is connected to the controller.

binding on drive screw— Feel the rear, motor-driven adjustment screw during engagement to verify that it is rotating. If the motion is erratic, then the screw is fouled with debris and binding. Remove the screw, clean threads and regrease per instructions provided in the maintenance chapter of your product instruction manual.

bias shorted—Measure the bias by using a voltmeter between the tip and sample. If this is not in agreement with the settings in the *Bias* voltage item in the STM Parameter control panel, then check to see if anything is providing a conduction path between the sample and the microscope base or any other ground.

bias not applied to sample surface— In some cases (e.g., the calibration reference), the sample consists of a layer of electrically conductive material on top of an insulator. In these cases, a conduction path must be provided between the sample

puck and the sample surface. Carbon glue or silver epoxy can be used to connect the bias voltage to the sample surface.

232.6.4 Tip crashes

If the tip always crashes into the surface with the **Z Center Position** either changing erratically or stuck in the fully retracted position, try the following:

check sample conductivity—There are two problems associated with sample conductivity. First, the bulk conductivity of the sample may make it difficult to image. If the resistance of the sample is greater than 1 Kohm/cm, higher bias voltages should be tried. If the resistance is greater than 1 Megohm, bias voltages of 100 mV or more should be used. Samples with resistances 1 Megohm or greater will be difficult to image even with high bias voltages.

Measuring the bulk conductivity of the sample with probes may not tell the whole story. Probes may easily penetrate oxide or contamination layers on the sample surface yielding reasonable resistance measurements. However, oxide and contamination layers on the sample surface can make imaging very difficult. Higher bias voltages are required for these types of samples.

clean contacts— The magnetic balls of the scanner tend to attract small metallic particles which may make the surface gritty. Clean them with adhesive tape and then wipe with alcohol. Also, clean the kinematic mating surfaces on the underside of the head.

check for binding in the motorized screw— Make certain the motorized adjustment screw rotation is smooth and not erratic, which can cause the screw to advance in jumps. If this is the problem, clean the screws as described in the maintenance chapter of your product instruction manual. On newer scanners, the set-screw used to adjust tension on the motor driven screw's plastic threaded bushing may need to be loosened slightly.

check the polarity of the Z-axis piezo— Use the **Calibrate** command in the **Real Time / Microscope** menu to review the calibration parameters for the head in use. The **Z polarity** should be set to **Forward**. (**EXCEPTION:** A few rare scanners have serial numbers ending in "RP," reverse polarity; these should be set to **Reverse** instead.)

232.6.5 Problems during *Real Time* operation

This section deals with problems that are associated with the Real Time aspects of the NanoScope software although they often point to the scanning head or microscope.

232.6.6 Z Center Position

If the Z center position tends to go out of range at the edges, top and bottom or right and left of the scan, then try the following:

Level sample to scan plane—The Z center position is affected by sample tilt, and larger (e.g., "J") scanners may be less tolerant of sample tilt due to their having small vertical range compared to larger lateral scan range. Set the **Realtime Plane-fit** for **Offset** or **None**, then go to **Scope Mode**. Note the trend in **Z Center Position** across the sample scan profile using **View / Scope Mode**. Verify that the sample surface is parallel to the sample puck before mounting it atop the scanner cap.

Increase Z limit parameter— If set too low, try increasing the **Z limit** parameter value (**Scan Controls** panel).

232.6.7 Z drift

If the Z center voltage tends to drift out of range rather quickly after the head is engaged, it may be a result of the following:

thermal drift— Allow some time for the temperature to stabilize if the microscope and/or scanner has been stored in a cold place overnight. Drift can be minimized by keeping the STM in a thermally stable environment. Avoid operating the microscope near windows and air ducts.

tip not tight— Make sure the tip is held tightly in the tipholder. Push and pull on the tip with tweezers to see that it is not loose. If it is loose, push the tip in farther.

sample hold-down— Verify that the sample is flat against the puck and cannot move vertically. This may become problematic if the sample has been affixed using uncured, conductive epoxy, and/or if foam adhesives are used.

scanner screws/converter head contact— Clean mating surfaces between the underside of the converter head and the scanner balls. If dirt or oil are evident, carefully remove with a clean wipe and alcohol. Some scanners utilize plastic threaded bushings in the scanner body through which the adjustment screws are threaded. If the bushings become fouled with grit, or if they are loose, drift may result. If loose, gently tighten setscrews in the scanner body to increase tension on the adjustment screw bushings. (Do not overtighten!) For more information regarding removal and replacement of adjustment screws, refer to the maintenance section of your instruction manual.

stiff rear screw— If the rear screw is difficult to turn (due to an accumulation of dust in the threads, etc.), the drive shaft can wind up while driving the screw and unwind after the stepper motor has stopped. This will cause the tip to continue towards the surface. Clean the threads and screw per the maintenance section of your product instruction manual.

232.6.8 **Real Time image hides features**

If the real time image appears flat, but captured images reveal detail, try rotating the **Scan angle** to 90°. The real time image leveling software tends to hide features that are parallel to the fast scan direction.

232.6.9 **Image is streaky or wavy**

If high-resolution images appear streaky or wavy, it may be a result of the following:

Insufficient vibration isolation— Atomic-scale scans are the most susceptible to vibration in the acoustic and subacoustic frequencies. Verify that the AFM is acoustically isolated with a functioning isolation pad and, if required, an acoustic hood. If vibrational noise persists, try moving the microscope to a less noisy location or using an isolation tripod.

Bad tip— Many strange effects have been pondered and hunted down for hours only to find that changing the tip fixes the problem. Try a different tip to see if the problem persists.

Low scan rates— A simplistic method for reducing the effects of drift and even low frequency vibration is to increase the **Scan rate** and reduce the **Number of samples**.

232.6.10 **Image is triangular over step-like features**

The performance of the STM over large scans with high vertical features is very dependent on the ability of the feedback loop to force the tip to track the sample surface. The digital feedback used in the NanoScope has been designed to maximize performance on a variety of samples. Try the following to improve the image quality over step-like features:

Log or Boost feedback—The most dramatic increase in performance of the feedback over large scans is achieved using the **Log** or **Boost** feedback modes. These make the error signal symmetrical for conditions with the tip too far or too close to the surface. **Boost** mode further optimizes the feedback performance for large scans.

Gain—The settings of the **Integral** and **Proportional gain** parameters also tend to be critical for large scans. They should be high enough to produce a sharp image, but not so high as to cause oscillations.

Scan rate—The **Scan rate** should be lowered for large scans, especially, if the sample surfaces are rough or contain large steps. Moving the tip quickly along the sample surface at high scan rates with large scan sizes will usually lead to a tip crash.

Setpoint current—Raising the **Setpoint** current will effectively raise the gain of the feedback loop which can be quite helpful for large scans. It will also bring the tip closer to the surface but only by a small amount ($i \approx e^{-5}$).

232.7 Overview of Low-Current STM

The following sections provide detailed instructions for performing low-current (sub-picoamp) STM using two Digital Instruments devices: the Low Current STM Converter for the Contact AFM and the LFM (Model #CSTMLC); and, the Low Current STM Converter for MultiMode AFM (Model #MMSTMLC), or with the Extender™ Electronics Module (Model #MMSTMLCE). The reader will find a general description in Section 232.8, followed by detailed instructions for use of Digital Instruments' low-current converters.

232.8 Description

Operation of conventional STMs with current (I_{tun}) in the nanoamp range is characterized by strong tip-sample force interactions. The forces applied to the sample during STM imaging in air actually exceed the forces in atomic force microscopy (AFM). The effects of high STM forces were found when imaging materials which included graphite [1a], inorganic layered compounds [1b], organic conducting crystals [1c] and organic adsorbates on conducting substrates [1d].

For metallic surfaces, the tip-sample gap resistance, $R_{\text{gap}} = V_{\text{bias}}/I_{\text{tun}}$, can be used as a qualitative measure of tip-sample separation. Where

V_{bias} = bias voltage

I_{tun} = setpoint current

In general, decreasing I_{tun} will increase the tip-sample separation and decrease the tip-sample force.

By operating at R_{gap} in the $\text{G}\Omega$ range, for example, $V_{\text{bias}} = 1.0 \text{ V}$ and $I_{\text{tun}} = 1 \text{ pA}$ the tip-sample distance increases enough to allow less-destructive imaging of a variety of surfaces.

The ability to conduct STM measurements in a wide range of R_{gap} can also be useful for experiments aimed at observing the image variations as a function of R_{gap} .

Picoampere-level STM provides the opportunity to image insulating samples deposited on conducting substrates. Indeed, it was shown that using STM at ultra high R_{gap} allows imaging of the topmost layers of alkanethiol adsorbates on gold surfaces and to reveal earlier unknown surface features.

Picoampere-level STM also opens the possibility of imaging biological macromolecules such as DNA deposited on a non-conducting mica substrate. Though such imaging was explained by lateral conductivity across the water overlayer covering surfaces in air, a true mechanism of electron transfer is not yet established.

232.9 Description of Hardware

The Low Current STM head, which allows STM measurements with I_{tun} in the pA range, is a new product of Digital Instruments, Inc. This head is constructed for operation with our MultiMode and AFM bases.

Low-current STM operation also requires the Picoamp Boost Box, which is installed between the control unit or extender box and the MultiMode or AFM base.



Figure 232-9 Low-Current Converter components: MultiMode head and Picoamp Boost Box.

The gain is switchable between 10^{10} V/A or 10^{11} V/A and the bandwidth is switchable among 400 Hz, 1.5 kHz and 4 kHz. Lower bandwidths allow for lower noise levels which are essential for operation at sub-picoampere I_{tun} .

IMPORTANT: The default calibration value of the detector in standard STM mode is 10 nA/V. This value should be changed to either 0.1 or 0.01 nA/V for operation of the low-current head with gain factors of either 10^{10} and 10^{11} respectively. To change values, click on **Real-time / Microscope / Calibrate / Detector**. The **In sensitivity** parameter is listed on the **Detector Calibration** panel and can be changed by simply entering the new value.

232.10 Precautions

Due to the low-current head's highly sensitive electronic components, special precautions must be taken.

1. The input stage operational amplifier is extremely vulnerable to electrostatic discharge (ESD) and is easily destroyed. It is recommended that the user be grounded with a wristband at all times that the low current head is handled, especially when tips are installed.
2. The feedback resistor and capacitor in the input stage are extremely vulnerable to contamination. At no time should these delicate components ever be touched with the hands. If these components are accidentally touched, they must be rinsed clean in a highly purified degreasing fluid immediately.
3. Good grounding is essential for low-noise performance. A good contact between the SPM base and the metallic cover (can) included with the low current head is needed to reduce the electrical noise level. A simple grounding kit is included with the converter which includes a length of wire and connecting lugs. Make certain that the can cover and SPM base are electrically connected by the wire and that the can cover is in place. The can cover acts as a Faraday shield.
4. Leakage and offset current:
 - Offset current values vary slightly from head to head but are typically in the range of 0.5-0.1 pA. Therefore, minimum operating currents are just slightly below the 1 pA value.
5. Please note that cables connecting the SPM base, the Picoamp Boost Box and the NanoScope controller should not be lengthened if possible.

IMPORTANT: Always disconnect the Picoamp Boost Box and Low-current STM Converter head before performing other types of imaging.

232.11 Installation

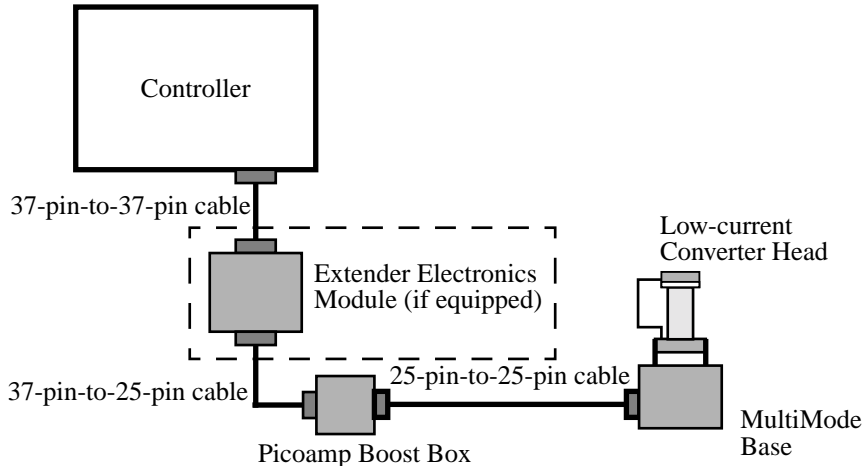
The low-current STM converter is designed to be installed onto one of four Digital Instruments devices: 1) the MultiMode SPM (standard); 2) the MultiMode SPM (with Extender™ base); 3) the dedicated AFM; and 4) the dedicated LFM. Generally, installation consists of cable connections and nothing more; however, cabling differs somewhat between the devices.

232.11.1 MultiMode SPM

IMPORTANT! If you are installing on a MultiMode SPM equipped with Extender™ Electronics Module for performing phase modulation MFM and EFM work, you must install the Picoamp Boost box with the jumpers inside set for extended. A label on the outside of the Boost box indicates whether it was set for standard (Model MMSTMLC) or Extender (Model MMSTMLCE) at the factory. If you are not certain which base your MultiMode has, contact Digital Instruments for guidance.

1. Turn off all power to the SPM controller. Disconnect and remove the MultiMode head.
2. Connect the picoamp booster box (male receptacle) to the controller using the 37-pin-to-25-pin connector cable. If an Extender™ Electronics Module is being used with the system, insert it between the controller and picoamp booster as diagrammed below in this section.
3. Connect the picoamp booster to the SPM base using the 25-pin-to-25-pin cable connector.
4. Connect the low-current converter head to the MultiMode's support ring using the micro-D, 15-pin connector cable. Set switches on the picoamp booster to the desired current level and bandwidth.
6. Before repowering the system, double-check all cable connections. If atomic-level scans are intended, verify the cable between the SPM and picoamp booster box is unstrained (a taut cable will transmit vibrations). The cable should rest

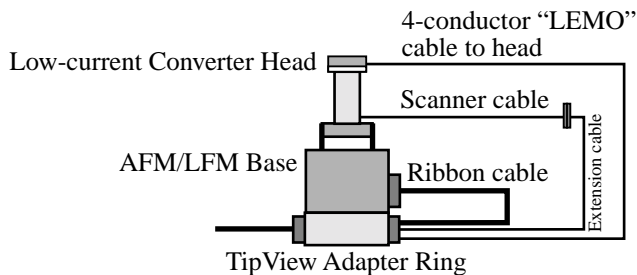
loosely. Before loading samples and running the SPM, attach the ESD wristband to prevent sudden electrical discharges.



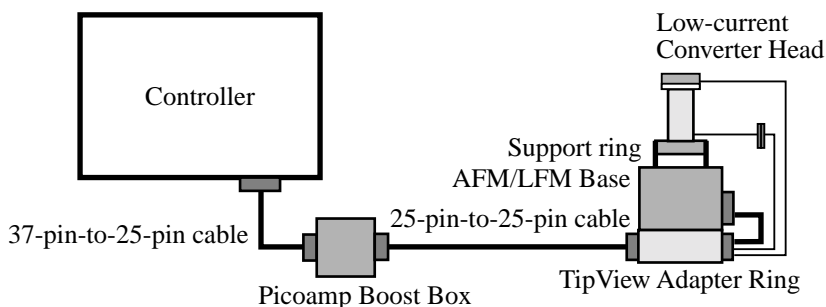
232.11.2 AFM & LFM

NOTE: The Low-current STM Converter Head is installed on AFMs and LFM's having the TipView™ option. If your AFM or LFM does not currently have a Tip-View adapter ring, use the one supplied with your installation kit.

1. Turn off power to the AFM controller. Disconnect and remove the AFM or LFM head.
2. Verify that your AFM has a TipView adapter ring attached to the bottom. If the adapter ring is not present, you will have to add one.



3. Connect the picoamp booster box (male receptacle) to the controller using the 37-pin-to-25-pin connector cable. Connect the picoamp booster to the AFM base using the 25-pin-to-25-pin cable connector. (See diagram below.)



4. Connect the low-current converter head to the AFM’s TipView adapter ring using the “LEMO” connector cable. The scanner cable should be plugged into the adapter ring’s D-receptacle. Set switches on the picoamp booster to the desired current level and bandwidth.

CAUTION: Do not plug anything into the support ring receptacles. Both support ring receptacles should be left unused.

5. Before repowering the system, double-check all cable connections. If atomic-level scans are intended, verify the cable between the AFM and picoamp booster box is unstrained (a taut cable will transmit vibrations). The cable should rest loosely. Before loading samples and running the AFM, attach the ESD wristband to prevent sudden electrical discharges.

232.12 Operation

Once you check that leakage is absent and the offset current is small, the operation of the low-current STM head is typically checked in the atomic-scale imaging of graphite and the large-scale imaging of a gold-coated grating. The atomic-lattice of graphite is well resolved in the images obtained with I_{tunn} in the 2-20 pA and V_{bias} in the 20-100 mV range. Mechanically-sharpened Pt/Ir tips are generally used for such measurements, and a tripod is good enough for the vibrational isolation of the SPM base.

232.12.1 Peculiarities

The transition to low current, however, brings some limitations, which are absent in STM measurements with standard heads. The bandwidth of the amplifier and, consequently, of feedback is substantially narrower than that used for standard measurements in the nA range (12 kHz). Therefore, the feedback mechanism can be less effective at high scan speeds. This must be taken into account, especially during imaging of corrugated surfaces.

However, the low current STM head can be used for imaging of atomically flat surfaces such as graphite and inorganic layered crystals at line frequencies up to 50 Hz and still achieve atomic-scale resolution.

NanoScope II Users: The low-current STM converter head may be used with the AFM base and the NanoScope II version 5.5 software. The software should be run in TipView mode. Readings will be seen in nanoamps; however, actual values are in picoamps.

232.12.2 Illustrative Examples

Different prototypes of the low-current STM head were used for measurements in several laboratories. In addition to the control measurements routinely conducted on graphite, the operation of the low-current STM head has been checked on several layered crystals and alkanethiol layers deposited on gold.

Several images obtained using low-current STM are presented below.

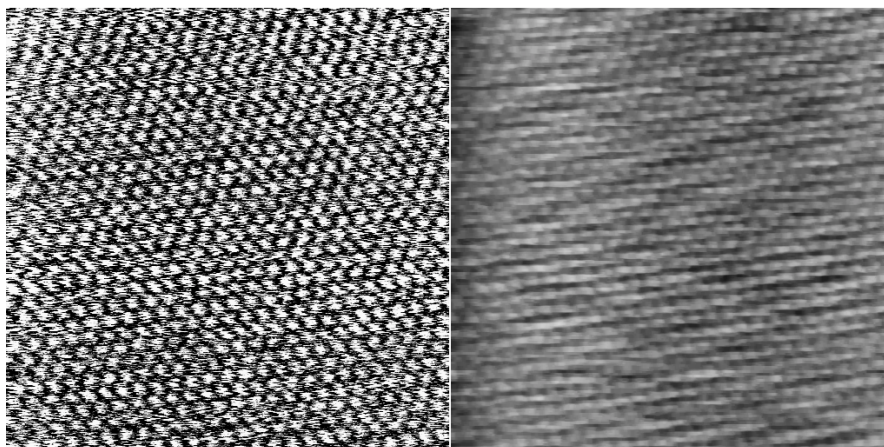


Figure 232-10 STM current and height images of HOPG surface.
Scan size = 6.0 nm, $I_{\text{tun}} = 1.6$ pA, $V_{\text{bias}} = 29$ mV.

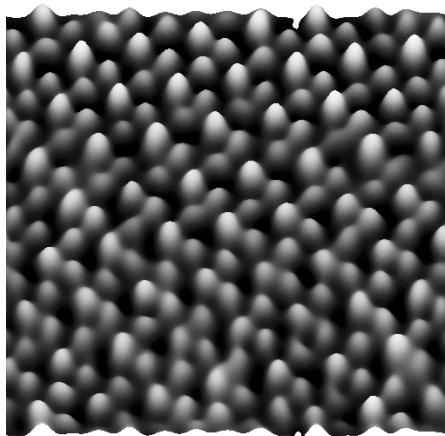


Figure 232-11 STM current image of layered crystal α - RuCl_3 .
Scan size = 4.48 nm, $I_{\text{tun}} = 1.5$ pA, $V_{\text{bias}} = 42$ mV.

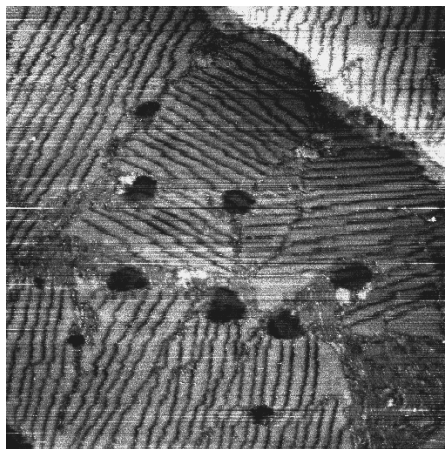


Figure 232-12 STM height image of alkanethiol layer on Au (111) substrate.
Scan size = 178.5 nm, $I_{\text{tun}} = 2$ pA, $V_{\text{bias}} = 1$ V. (Courtesy of Dr. I. Tuzov, NCSU)

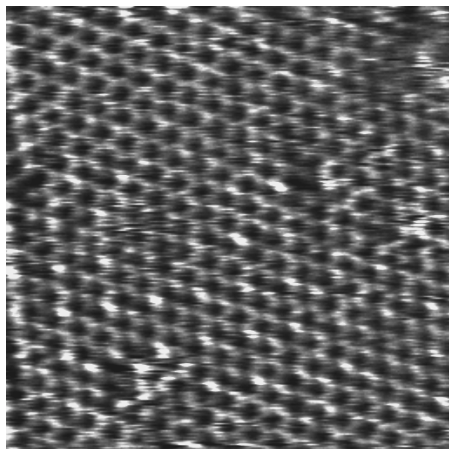


Figure 232-13 Molecular-scale STM current image of alkanethiol layer on Au (111) substrate. Scan size = 10.0 nm, $I_{\text{tun}} = 13$ pA, $V_{\text{bias}} = 1$ V. (Courtesy of Dr. I. Tuzov, NCSU)

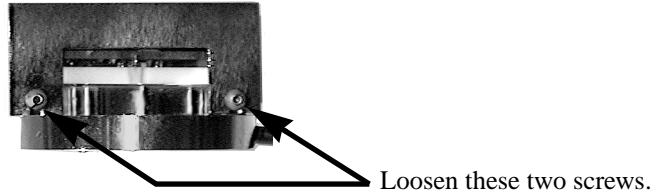
232.13 Servicing the Converter

The Low-current STM Converter features sensitive components which may be damaged if exposed to sudden voltage spikes. Spikes may be due to electrostatic discharge (ESD), or from external voltage sources. Operators should always wear an anti-static, grounding wristband (included in Low Current STM converter kit) while handling the head to protect against ESD.

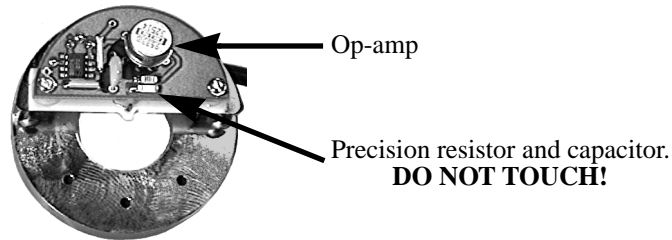
The op-amp is the most vulnerable component; several spares are shipped with the unit. If it is damaged, it may be replaced by doing the following:

1. Turn off the microscope. Disconnect the Low-current STM Converter head by unplugging its cable from the support ring. Set the head on a workbench area grounded against ESD. Ensure that an ESD wristband is being worn.

2. Use the 0.050" allen wrench, included in the converter kit, to loosen the two retaining screws which secure the head's cover. It is not necessary to remove the screws.



3. Pull the cover straight up and off to expose the PC board and electronics. It will appear as shown below.

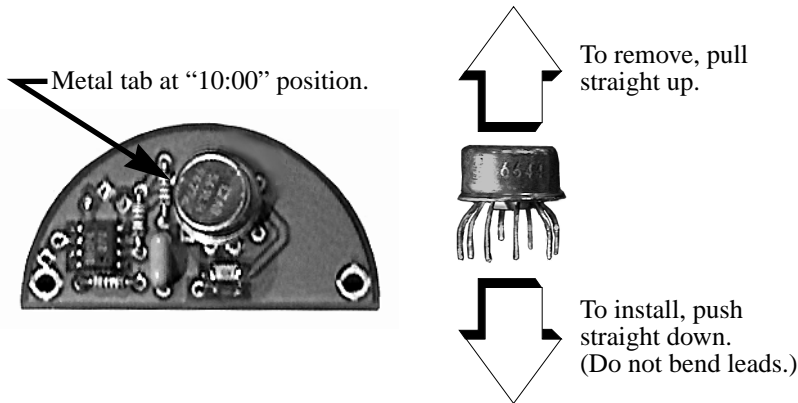


NOTE: Throughout steps #4-6 below, avoid touching the precision resistor and capacitor. Any contaminant from the hands, including body oils, may cause excessive current flow, disabling the head. If these components are touched, they will have to be recleaned immediately using highly purified degreasing compounds.

4. Remove the op-amp by grasping it and pulling straight up. It should slide out of its socket.

5. Remove the new op-amp from its protective package. The op-amp is stored in conductive foam and its leads have been preformed to fit the socket. Be careful not

to bend any of the leads. Orient the op-amp so that the metal tab on the edge of the can is in the “10:00” position as shown below.



6. Verify that each of the op-amp’s wire leads is properly aligned with the appropriate hole, then press the op-amp gently into its socket. Do not bend wire leads.

7. Replace the metal cover on the head. Retighten retaining screws to secure.

232.14 Etching Tungsten Tips

You can purchase tungsten tips from Digital Instruments or make them yourself. This section describes the process of etching tungsten tips for use with the Nano-Scope.

Materials Equipment Required

- Variac auto transformer
- Optical microscope (20-100X)
- Sodium nitrite (NaNO_2)
- Distilled water
- Ethyl alcohol
- WD-40 (anti-oxidant)
- Two 50-ml beakers
- Tipholder (see below)
- Platinum wire
- Tungsten Wire, 0.010" Diameter
- Miscellaneous Wire/Clips

232.14.1 Procedure

1. Mix a 5 percent (by weight) solution of Sodium Nitrite in water.
 2. Pour ≈ 40 ml of the Sodium Nitrite solution into a beaker.
 3. Pour ≈ 40 ml of WD 40 into a beaker.
 4. Construct an electrode out of the platinum wire and insert it into the beaker.
 5. Adjust the variac for 30V, and with it Off, connect one output to the platinum electrode.
 6. Cut 10 to 12 pieces of tungsten wire ≈ 1.25 cm long. Before etching, check to make sure that at least one end of the wire has not split by inserting the ends into a tip holder. If an end has split, you will not be able to insert it into the tipholder. You can etch the end that has split, however, preserve the unspoiled end.
 7. Place the tungsten tips into a holder. We like to use an IC socket (the low cost, edge-grip square contact type, not the machine-grip round contact type), with all the pins soldered together. The tips will then be held in place while inverting them over the solution. We also find it helpful to solder the IC socket to the back of a proto-board (perf-board). You can then invert the tips over a beaker with the proto-board sitting on the rim of the beaker.
 8. Invert the tips over the Sodium Nitrite solution with ≈ 2 mm of the tips' surface submerged. More than 2 mm will cause excessive foaming of the solution during etching, and less than 2 mm will result in tips that are too blunt.
 9. Connect the other output of the variac to the common of all the tips.
 10. Turn on the variac and etch the tips. While the tips are etching, the solution will foam, and the tips will start to glow. As the tips etch towards the surface, the foaming will be reduced. Continue to etch the tip until it stops.
 11. Re-submerge the tips ≈ 1 mm into the solution for ≈ 15 seconds at 30V. Turn on the variac and re-etch the tips. There should be only slight bubbling from the tip and it should not glow.
- Note:** It possible to vary the tip shape at this point by lowering the voltage on the variac and increasing or decreasing the amount of time the tips are submerged. Longer time gives blunter tips.
11. Dip the tips into ethyl alcohol to clean them. If you plan to keep the tips around for more than a day, then dip them into the WD 40 after cleaning.

12. Examine the tips under the optical microscope. Ones that are too long, too blunt, or split at the end will hardly ever be good tips and can be thrown out at this time. Of course, this is a subjective process. As your experience in etching grows, you will get better at throwing out the bad ones.

13. Repeat the etching procedure. Replace the etching solution when a fairly large amount of residue is present. Typically, you can etch 60 to 80 tips in a 40 ml solution.

232.15 References

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(b) B. A. Parkinson, "Layer-by-Layer Nanometer Scale Etching of Two-Dimensional Substrates Using the Scanning Tunneling Microscope" *J. Am. Chem. Soc.* **1990**, 112, 7498

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(d) A. Wawkuschewski, H.-J. Cantow, S. N. Magonov "Multilayered Structure of Alkanes Adsorbates at Liquid/Graphite Interface" *Langmuir* **1993**, 9, 2778-2781.

2. (a) C. Schönenberger, J. A. M. Sondag-Huethorst, J. Jorritsma, L. G. J. Fokkink, *Langmuir* **1994**, 10, 611-614.

(b) J. A. M. Sondag-Huethorst, C. Schönenberg, L. G. J. Fokkink, "Formation of Holes in Alkanethiol Monolayers on Gold" *J. Phys. Chem.* **1994**, 98, 6826-6834.

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(b) F-R. F Fan, A. J. Bard, "High Resolution SECM in Humid Air. Imaging of Nafion and DNA on Mica Surface. Publication pending in *Science*.