PicoLE STM
(Scanning Tunneling Microscopy)
User's Manual

v1.0

Molecular Imaging

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Low Current STM Operation

Speed and sensitivity are, in theory, mutually exclusive requirements in the current-to-voltage amplifiers used in STM. In practice, however, the dominant limitation is pickup of stray signals (such as high-voltage scanning ramps). The careful layout of the PicoSTM has eliminated this problem. At the standard sensitivity of 10 nA/V, the PicoSTM has a bandwidth of 3 kHz, permitting scanning at up to 100 Hz (line frequency with 256 pixels) for high signal levels on relatively flat surfaces. Therefore, slower scanning under feedback control permits operation at substantially lower currents. Using the PicoScan program controller and reasonably clean substrates, the PicoSTM has been operated down to sub-pA. This performance can be obtained in electrolytes if the highest grade of STM tip is used.

Low current operation is essential for application such as STM imaging of weakly adsorbed organic molecules, imaging via surface conduction in thin water films, or measurements of conductance over a wide range of currents. On the other hand, the head amplifier should have an adequate bandwidth to permit scanning at speeds that approach the resonance frequency of the piezoelectric element so that rapid imaging is possible at high currents. Thus, it would seem that different head amplifiers must be used for high and low current applications; however, the performance of the amplifier is usually limited by DC and AC coupling of the high voltages used for scanning. If this problem is solved, low noise levels and reasonable bandwidths are attainable. Furthermore, when used in a feedback system with a controller, the STM will operate at currents substantially below the RMS noise level of the front-end amplifier. This is because of the signal averaging effect of the integrator. Scanning rates have to be reduced substantially as the noise-floor of the complete system is approached, but this is facilitated by the low drift of the PicoSTM. Thus, the one scanning head can be used for imaging at up to 3 kHz bandwidth and for imaging at pA current levels.

For ultra low-current imaging, the following steps should be taken:

1. Prior to loading the sample stage, adjust the scanner to have the offset nulled. One way to do this is to use the oscilloscope window in the PicoScan software to monitor the tunneling current. If the reading on the oscilloscope reads null offset, and does not in the software, then this indicates the controller has on offset at its ADC. Contact Molecular Imaging if this is significant.
2. If a large leakage offset cannot be nulled by adjustment, clean up the tip holder area with methanol and let it dry completely.
3. After loading the sample stage, check the offset again. It should not have increased. In many cases the attachment of the sample stage helps to stabilize the tip current reading and make it easier to null the offset because of the shielding by the stage.
4. If imaging in solution make sure that the leakage is lower than the setpoint current. If using extremely low leakage STM tips, make sure they are relatively fresh, for they can degrade over time.
5. Engage the microscope at a setpoint current of 50 to 100 pA, scan size approximately 100 nm, and a bias voltage of 50 mV.
6. After engaging, gradually lower the current to the desired value, and then lower the gain and the scan rate accordingly to optimize image quality. For imaging at sub-pA, the scan speed used can be around 1 line/s.

STM Specs

Sensitivity	10 nA/V	1 nA/V
Bandwidth	3 kHz	300 Hz
Lowest Imaging Current	3 pA	.4 pA
Noise RMS	1.5 pA	.3 pA
Current Range	± 100 nA	± 10 nA

Table 6.1

The standard sensitivity is 10 nA/V. The 1 nA/V module is available from Molecular Imaging. Be sure that the sensitivity of the scanner matches the Pre-Amplifier Conversion Coefficient setting in PicoScan.

STM Nose Assembly

The STM nose assembly plugs into a socket within the scanner. Be sure to tighten the setscrew once the nose is in place.

Inserting STM Tip

The microscope is designed for operation with a 0.25 mm diameter wire. STM tips are available from Molecular Imaging and are recommended for all STM applications. To insert an STM tip into the nose assembly:

1. Mount the scanner module or nose assembly on the mounting jig as in AFM operation.
2. With a tweezers, grasp the STM tip firmly. If using insulated tips, be careful not to hold the insulated section of the tip too tightly or you could damage the insulated portion, resulting in current leakage.
3. Trim the butt end section of the tip so at least 6.5 mm will protrude out of the scanner when using the ambient cell, and 8 mm will protrude when using the liquid cell.
4. Place the tip into the holder.
5. Be certain that the tip is snug in the tip holder by gently trying to move the tip up and down with the tweezers. If the tip is not snug, the tip may be pulled out to an extent in order to bend the wire (slightly) against the holder wall. Then, reinsert the tip into the holder.
6. The tip should stand straight up.

Notes

- If the tip is bent prior to insertion, it usually causes noticeable drift for some time, and you should allow the drift to settle if it affects your imaging.

Inserting the Scanner Module

The procedure for inserting the scanner module into the microscope is the same as in AFM operation (see the **PicoLE System module**).

Placing the Sample

The procedure for placing the sample is the same as in AFM operation, except that an electrode must be placed between the working electrode terminal on the sample plate and the sample. Additionally, the sample must be electrically isolated from the sample plate. The sample may be secured to the sample plate by several methods. An ambient plate may be used to secure the sample, a liquid cell may be used for operations in solutions, or the sample may be simply attached to a glass plate. Choose the method that best fits the needs of the particular scanning application. All the sample stages except heating and cooling stages have a glass plate glued on the sample plate to provide insulation from the metal plate, which is grounded. Therefore, when using heating and cooling stages with a conductive sample for electrochemical experiments, the sample should not be in electrical contact with the sample plate or it will be grounded out. In this case, put an insulating material between the sample and the sample plate. To properly affix a sample to the sample plate:

1. Place the sample on the sample plate. The sample can be attached using a liquid or ambient cell, a magnetic backing, or a piece of two-sided tape (be aware that using 2-sided tape can cause the sample to drift). If using a cell, push the two retaining pins on either side of the sample through the holes in the cell assembly and secure them with the retaining clips. This will hold the cell in place.
2. If using a cell, push the short end of an L-shaped pogo electrode into the hole in the wall of the Teflon cell nearest to the electrode clamping assembly on the sample stage. Push up the rectangular nut on the electrode clamping assembly from underneath and place the end of the pogo under the nut.
3. Connect the EC cable between the underside of the microscope and the sample plate.
4. Mount the sample plate on the microscope as in AFM operation.

Note: For imaging in STM Mode is will be necessary to switch the AFM/STM switch on the front of the PicoLE **Head Electronics Box** to STM. This will allow the microscope to function correctly in STM Mode.

Freestanding Operation

Freestanding operation mode (FSOM) can be used to image larger samples, or samples that require special setup which do not fit into the standard sample stage. In this mode, the microscope is placed onto a larger, flat, hard surface, which can be a sample surface itself, or a platform with the sample placed on it.

1. Make sure the sample is electrically isolated from the microscope body and connect the sample to the working electrode. If a large sample is being used and the microscope is sitting on the sample, the microscope must be electrically isolated from the sample by an insulating material.
2. The two translation pegs must be removed. To do this, remove the screws holding the pin assemblies in place from the bottom side of the microscope. There are 4 pieces of hardware per leg assembly (screw, leg assembly, curved washer and bronze top washer). Be careful not to lose any of the hardware. It is now possible for the microscope to rest on the three plunger screws.
3. Using the sample plate, adjust the height of the microscope with respect to the sample, by using a dummy sample of similar height. This is to ensure the real sample is not higher than the surface on which the microscope will be resting.
4. Position the STM tip so the gap is less than 0.5 mm, remove the plate, and set the microscope onto the surface.
5. Follow all procedures, as previously discussed, to mount the cantilevers and operate the STM scanner and microscope.