

EXPERIMENTS WITH THE SCANNING TUNNELING MICROSCOPE: CALIBRATION AND IMAGING

Introduction

The goals of this experiment are to learn the operating principles of a scanning tunneling microscope (STM), accurately calibrate the STM by imaging an aluminum reflection grating, and to study the surface structure of graphite. With proper experimental technique and suitable use of image processing filters, you should be able to resolve individual carbon atoms and determine bond lengths and angles.

Theory

The theory behind the STM is grounded in the quantum mechanical process of barrier tunneling. Classically, we suppose that a particle with total energy E cannot enter a region of space where the potential, V , is larger than E (a so-called repulsive barrier). Quantum mechanics, however, permits a finite probability of the particle entering the classically forbidden zone. If a bias potential is applied across two conductors separated by a small insulating gap, such as air, then there is a finite probability that an electron will tunnel through the gap from one conductor to the other. We can calculate the transmission probability, T , using the WKB approximation:

$$T \simeq \exp \left[-2 \frac{\sqrt{2m(\phi - V_b/2)}}{h} a \right]$$

where m is the mass of the electron, ϕ is the work function, V_b is the bias voltage, and a is the barrier width. This function tends to be quite sensitive to the value of a .

The main idea is to employ an extremely sharp tip as one of the conductors and to use the sample for the other conductor. Positioning of the tip is critical; this is accomplished in our STM with piezoelectric ceramics. These piezoelectric elements not only permit accurate scanning through short distances parallel to the sample surface, but they also allow the sample-tip separation to be controlled by negative feedback from the tunneling current. In this way, the tip position relative to the sample can be controlled to within a few Angstroms!

There are two basic modes of operation for the STM. First, we can scan horizontally at a constant height above the average surface level and monitor the variation in the tunneling current which is proportional to the sample-tip separation (constant height mode). Second, we can specify that a constant tunneling current be maintained so that the tip is forced to

follow the topography of the sample (constant current mode). You should become familiar with the advantages and disadvantages of both operating modes during the course of the experiment.

Procedure

Precautions:

There are no safety hazards associated with this experiment, although the equipment and samples can be damaged by incorrect operation. Obviously, avoid touching the surfaces of the samples or the pointed end of the scanning tip. Only gentle assembly is necessary for proper preparation of the STM tip and sample. Slow and deliberate movements will reduce the chance of accidentally dropping or scratching the samples. Carefully monitor the tip position indicator and *do not exceed the indicated range*. Do not mechanically force any parts of the STM head. If jamming occurs see the instructor for help.

The most common problem is “crashing” the wire tip into the sample. This is not serious as long as the sample is not extensively scarred, but the tip will have to be re-cut or replaced. Try to be frugal with your PtIr wire for making tips; this wire is very expensive (\approx \$100 per ft!) and there will be no seconds. *The special wire cutters are for preparing PtIr wire tips only*. Use for any other purpose can permanently damage the cutters.

You may save your images in the “C:\ISTM\2009” directory on the hard drive. You can also transfer any images you acquire to a 3.5 floppy (a: drive) or 5.25 floppy (b: drive).

Calibration using an aluminum grating

The horizontal scanning range of the STM can be controlled from the Collect/Configure settings in the STM software. The actual scanning range is determined as a fraction of the maximum range allowed by the STM tip controllers. This maximum range is the quantity you will need to determine using the aluminum grating sample. Follow the procedures listed in this handout under “Quick-Start”. Use data type=Topographic (constant current mode) for this exercise. Notes: (1) set the bias voltage to 1.5 volts for the grating, instead of 1.0 volts as originally printed in the manual (which is based on a gold-coated grating). (2) you should start out in type=Current (constant height mode) until you are sure that you have a good tip, then switch to Topo mode for analysis.

Once you have been successful in obtaining images of the grating, you can move on to adjusting *Max scanner range in X* and *Max scanner range in Y* so that the imaged distance

between grating rulings corresponds to your grating sample. Assume that the grating specification is 1800 ± 0.5 lines/mm. You may find the cross section option under the analysis menu useful in this regard (particularly the LEN option under the statistics package).

Imaging graphite

In order to obtain useful images of HOPG (highly oriented pyrolytic graphite) you will need to prepare an appropriate tip. The best way to test your tip is by using the Al grating sample from the calibration. Once you are certain that you have a single atomic site on the tip that is responsible for the tunneling (i.e. there are no ghosts or dual images) and that the tilt of your image is minimal, then proceed to the section under “HOPG” in this handout.

In addition to tip considerations, you must also make every attempt to limit stray vibrations and acoustical noise. It may be necessary to enclose the STM head in a soundproof container. This is best done after setting up the head but before doing the approach to tunneling. You will have to experiment with the Gain, Time Constant, and Filter settings on the electronics controller, and also with the Scan Delay on the software in order to find the optimal combination for your sample and tip.

The filter software will be very useful to clean up any excess noise in your images. You are strongly encouraged to take advantage of these in order to obtain the best HOPG images possible. Some form of filtering will most likely be required in order to determine the bond lengths and angles of the surface atomic sites.