Atomic Resolution with Scanning Tunneling Microscopy

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I. Introduction

The scanning tunneling microscope (STM) has the capability to directly image atoms on a surface. It was developed in the early 1980's by two scientists at IBM. Since its invention, the STM has become a widely-used tool to study surfaces at the atomic scale. For example, it has been used to investigate deposition and etching of materials, to map surface electronic structure, and to investigate electron transport in thin films. The STM can even be used in some cases to manipulate individual atoms and molecules and build designer atomic structures.

In this lab, our goal will be to obtain an atomic-resolution image of a graphite surface and then use this image to determine the distance between nearest-neighbor carbon atoms. In order to achieve atomic resolution, the STM utilizes a quantum mechanical effect known as tunneling. The atomically sharp tip of a STM is brought to within 1 nm of a surface. If a small positive voltage ~100 mV applied to the tip, electrons will tunnel from the surface to the tip, even though the region between the tip and the surface is insulating. The tunneling current of electrons, typically ~1 nm, is extremely sensitive to the distance between the tip and the sample. See Appendix J for further information on tunneling.

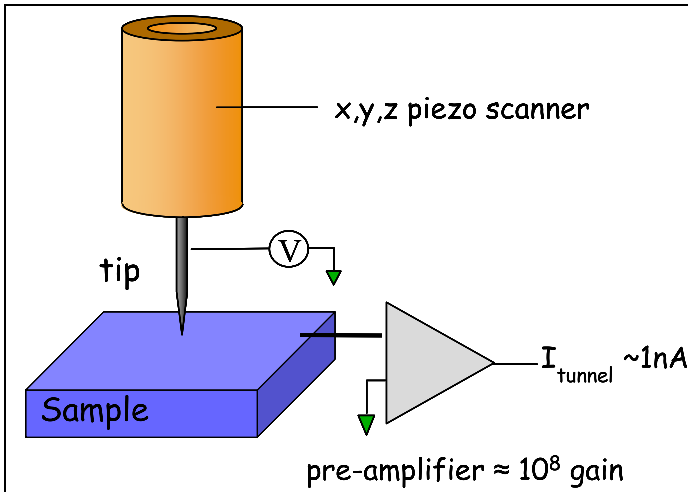


Figure 1. Simple STM schematic. Electrons tunneling between the tip and the sample flow through the feedback circuit. The computer adjusts the Z piezo to keep the tunnel current constant.

In normal operation, the STM maintains a constant tunnel current, and thus a constant distance between the tip and the sample, as the tip scans back and forth. If the tip scans across the surface and encounters a high point, the computer will send a signal to the scan head to retract the tip in order to keep the tunnel current constant; a low point will cause the scan head to extend.

The STM scan head is based on piezoelectric ceramics, which can be precisely compressed or stretched on the nanoscale by applying modest voltages to the material. Separate voltage signals control the X, Y, and Z motion of the tip. The topographic image displayed by the STM software is a recording of the Z motion of the scan head as it changes to keep the tunnel current constant.

The achievement of atomic resolution with the STM also depends on the stability of the tip and the sample. Vibration isolation systems designed to reduce relative motion between tip and sample is common to all STMs. For example, for the STM you will use in lab, the scan head rests on a heavy granite slab. The slab is mounted on four rubber-like legs that somewhat decouple the motion of the slab from the table it rests on. The inertia of the granite block helps the scan head remain stationary even if the table beneath is moving. Temperature also affects the stability. Many research grade STMs are cooled to low temperature to reduce thermal motion of the atoms on the tip and the sample. In addition, most STMs operate in ultrahigh vacuum to reduce contamination of surfaces and unwanted chemical reactions with the atmosphere.

II. Procedure

A. Acquire STM Images

Obtaining atomic resolution images is not necessarily easy, but it is possible with some persistence and luck. If you do not obtain a good image in the time allotted, go on to parts B and C. Use the images located in the desktop folder labeled *STM Images.*

1. Follow the instructions on pp. 17-30 in the *Nanosurf STM system* manual for obtaining atomic resolution images of your graphite sample.
   a. If you crash your tip (LED turns red), call the instructor to replace your tip.
   b. If your images look like those on pp. 32-33, call the instructor to replace your tip.

2. Check for drift.
   a. Read p. 31 in the *Nanosurf STM system* manual for information about drift. Can you see evidence of drift in your images?
   b. How can you minimize the effect of drift on the accuracy of your atomic measurements? Think of all possible strategies and implement those that you can.

3. Be sure to save images as you go along. Your tip can go bad at any time.
   a. Click the *Photo* button before the image is complete.
   b. When the scan is complete, a window with the image will appear behind the scan window.
   c. To save an image to the hard disk, click on the save icon.

4. Maximize image quality.
   a. Often drift will diminish over time and the image quality will improve. Continue to scan as long as you can to take advantage of increasing stability over time.
   b. Optimize the *I-Gain* and *P-Gain* values. These values control the feedback loop that adjusts the tip to maintain a constant tunneling current. Increasing these values will give a sharper image up to a point; increasing them too much leads to instability and a noisy image.
   c. As time allows, try adjusting other parameters to optimize your image.
   d. Any time you get a better image than previous images, be sure to click *Photo*.

B. Remove Noise from Images

The typical STM image has artifacts such as random noise and scan lines. These artifacts can be removed by a Fourier transform filter so the real data is more easily seen and analyzed. See Appendix K for an introduction to Fourier Transforms (FT).

1. Open one of your graphite images in WsXM.
   a. Start the program WsXM.
   b. Change the *File Type:* to *All Files (*.*)* in order to see the **.ezd* file which contains your data.
   c. Two images will open. Close the one with a grainy appearance and less contrast.

2. Open the *2d FFT Filter* dialog box (see Fig. 2 below).
   a. Click on your image to make its window active. Press the Fast Fourier Transform (FFT) button 

 to activate the *2d FFT Filter* dialog box.

   b. Adjust the slider bar on the *Zoom* control so that all the bright peaks are clearly visible in the FT of your image (to the right of your original image).

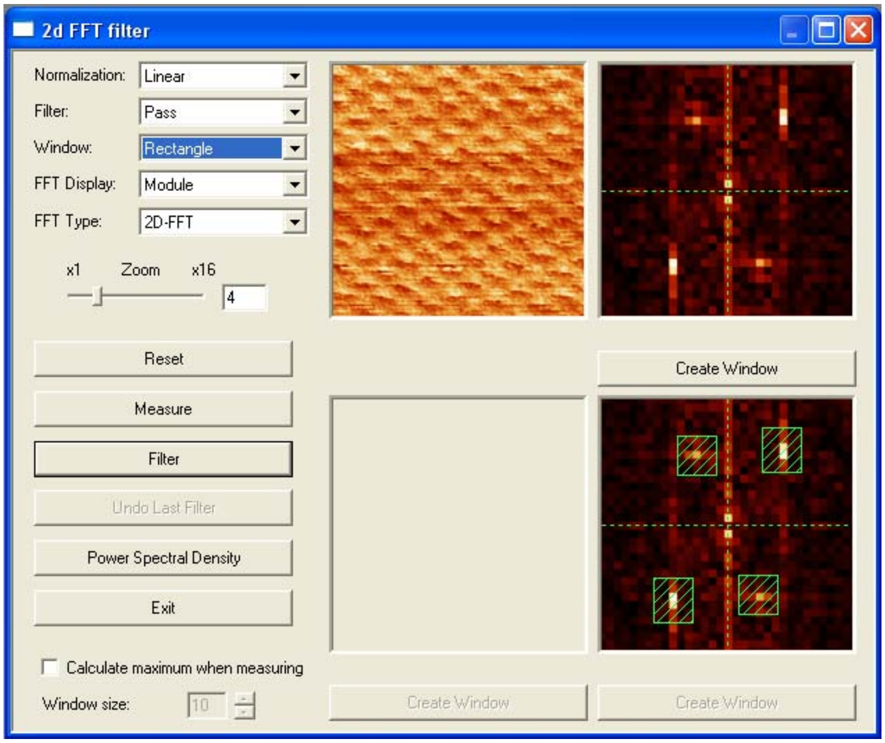


Fig. 2 *2d FFT Filter* dialog box.

3. Filter your image.
   a. Press the *Filter* button. The lower right window will become active.
   b. Select the brightest peaks in the FT image. Place the mouse on the center of the bright peak, click and hold the left mouse button, and drag outward to make a small box around it. Release the left mouse button. Repeat for other bright peaks.
   c. Activate the filter by right clicking on the FT image. A new image will appear in the lower left window with only the frequencies you selected in the FT image.
   d. Click on *Create Window* to open a larger filtered image. Close the *2d FFT Filter* dialog box.

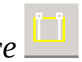
4. Print out your best filtered image
   a. Experiment with filtering images. When you agree on your best image, print one for each person in your group.
   b. Be sure to label your image with sample type, magnification (or a scale bar), date, and your name.

<u>C. Analyze Images</u>

1. Identify the hexagonal rings of carbon atoms on your filtered image.
   a. Find the center of a hexagonal ring of carbon atoms (it should be lower than anything else around it). Count the number of atoms around the center—can you find six? See p. 33 in the nanoSurf STM manual for help with interpretation of your graphite image.

b. Draw a hexagon on your image that shows the location of a hexagonal ring of carbon atoms. There should not be any atoms in the center of the hexagon!

2. Determine the distance between a carbon atom and its nearest neighbor.

   a. Use the *Profile* tool  and the *Measure Distance*  tool to measure the distance between atoms in your image. You may not be able to directly measure the distance between nearest neighbor atoms.
   b. In general, it is more accurate to measure the distance across several atoms and divide by the number of atoms.
   c. Your determination of the nearest neighbor distance should include an uncertainty value. This means that you will need to make more than one measurement!

APPENDIX J

Brief Introduction to Quantum Mechanical Tunneling

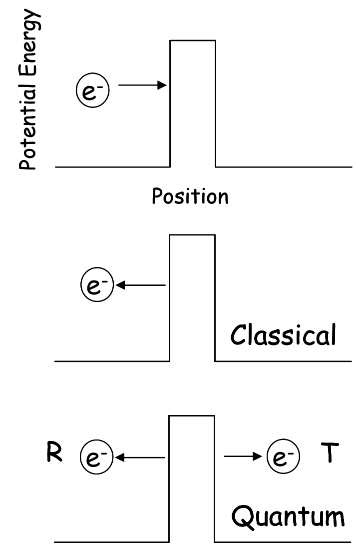
The scanning tunneling microscope (STM) is based on a quantum mechanical phenomenon known as tunneling. In tunneling, a particle, in this case an electron, can jump from one location to another without spending any time in between. A diagram about tunneling can be seen in the figure to the right. If a particle is incident upon a barrier in our everyday macroscopic world, it will always reflect from the barrier. In other words, its probability of reflecting from the barrier R is always R = 1. In the microscopic world of quantum mechanics, a particle will not necessarily reflect; rather, it has some probability T of tunneling through the barrier. The probability of reflecting from the barrier R is less than one. R and T are related by:

$$R+T = 1$$

The probability of transmission, or tunneling, is

$$T \propto e^{-\beta w}$$

Where β is a constant that depends on the energy of the particle and the barrier, and *w* is the width of the barrier.



A particle incident on a barrier. In the macroscopic world it always reflects. In quantum mechanics, it can be transmitted with probability T, or reflected with probability R.

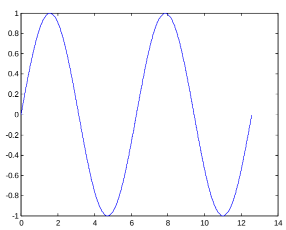
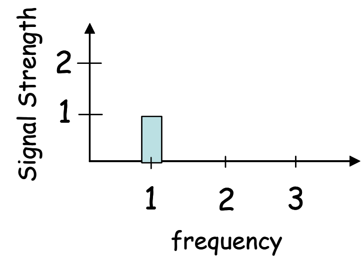
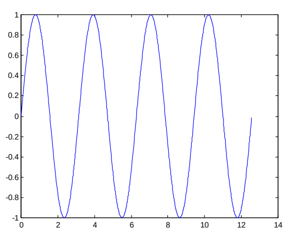
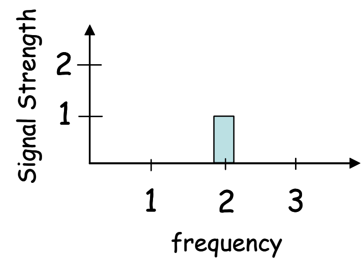
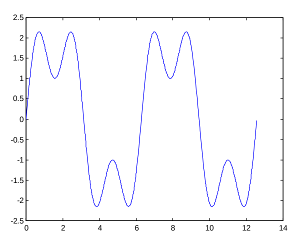
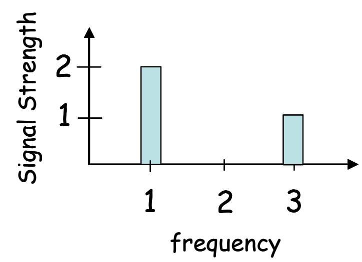
The exponential dependence on distance is what makes the STM such a sensitive instrument for exploring surfaces. If the tip contains one atom that is slightly closer to the surface than the other atoms, most of the tunneling current will flow through that one atom. The sensitive tunneling current allows the STM to have ~0.01 nm resolution vertically and atomic (~0.2 nm) resolution horizontally.

Electrons are allowed to be in either the STM tip or the sample. The space between the two is a barrier. However, if the distance between the two is small enough, electrons will tunnel from one to the other. If no voltage is applied between the tip and the sample, electrons will tunnel back and forth with equal probability and no net current will flow. However if a voltage is applied, electrons will prefer to tunnel from the lower voltage to the higher one.

APPENDIX K

Brief Introduction to Fourier Transforms

This is a basic conceptual introduction to the fourier transform (FT), which can be useful in understanding the FFT data processing step for removing noise from images. The idea behind the FT is that any signal can be written as the sum of sine waves, with each frequency sine wave having its own amplitude. The FT signal is the amplitude corresponding to each frequency. For example:

Signal	Plot of signal	Fourier Transform
y = sin(x)		
y = sin(2x)		
y = sin(3x) + 2sin(x)		

In WsXM, the software goes through the image and breaks it into sine waves (in the case of a spatial image the 'frequency' has units of 1/length) and maps the amplitude of each sine component. Since the image is two dimensional, the FT is also two dimensional. Features with regular spacing will appear much brighter than those without regular spacing. You can choose to keep only those signals with a specific frequency by selecting them on the FT. Since graphite has a regular structure that repeats spatially, this is a good strategy. Such frequency filtering tends to remove noise, which appears at all frequencies.

<u>Additional information on Fourier transforms</u>
http://www.med.harvard.edu/JPNM/physics/didactics/improc/intro/fourier1.html
http://www.cs.unm.edu/~brayer/vision/fourier.html
http://online.redwoods.cc.ca.us/instruct/darnold/laproj/Fall98/KrisCrg/Fourier.pdf

III. Analysis

1. Typically, samples must be imaged in vacuum with the STM. Why? What properties of graphite allow us to image it in air?
2. What are the requirements to establish a quantum mechanical tunneling current between a tip and a surface? What are the advantages of using a tunneling current to image a surface?
3. Explain the concept of constant current imaging. What does it mean if two regions of your image are the same brightness?
4. The nanosurf STM is designed to reduce the impact of external mechanical vibrations and prevent unwanted motion of the tip relative to the sample. Describe the features of the STM that reduce mechanical noise.
5. Define "drift." How does drift affect an image? How can you orient your measurements of the distance between atoms to minimize the effect of drift?
6. Attach your best filtered image to your report (make sure it is properly labeled). Describe how the fourier transform (FFT) filter allow you to remove random fluctuations from the image.
7. Draw a hexagon on your image that shows the location of an individual ring of six carbon atoms. Why do some of the atoms appear lower, even though all the ion cores lie in the same plane?
8. Determine the distance between a carbon atom and its nearest neighbor (show all work). Your number should include an uncertainty value. How does it compare with the accepted value of 0.14 nm?