Instructional Scanning Tunneling Microscope™

Workbook

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2. OPERATING PRINCIPLES OF THE STM

2.1. How the STM Works

There are five scientific and technical processes or ideas that the STM integrates to make atomic resolution images of a surface possible. Each of these processes was used in other areas of science before the invention of the STM.

- The principle of quantum mechanical tunneling.
- Achievement of controlled motion over small distances using piezoelectrics.
- The principle of negative feedback.
- Vibration isolation.
- Electronic data collection.

This Chapter discusses each of these five concepts. The most detail is provided on the process of quantum mechanical tunneling, since this is the fundamental concept that allows the microscope to work. At the end of the discussion of all these concepts, one can see how they integrate to make an STM.

2.2. Quantum Mechanical Tunneling

Quantum mechanical tunneling is not some obscure process that only occurs under extreme conditions in a crowded basement laboratory of a research university. Quantum mechanical tunneling explains some of the most basic phenomena we observe in nature. One example is the radioactive decay of plutonium. If quantum mechanical tunneling did not occur, plutonium would remain plutonium instead of changing into elements lower on the periodic chart. Plutonium converts to other elements when 2 neutrons and 2 protons are ejected from the nucleus because of tunneling. Even the fundamental force that binds atoms into molecules can be thought of as a manifestation of quantum mechanical tunneling. In this lab, we will look at how tunneling manifests itself in another way. We will attempt to understand how a single electron starts out in one metal and then reappears in another metal, even though they are not touching.

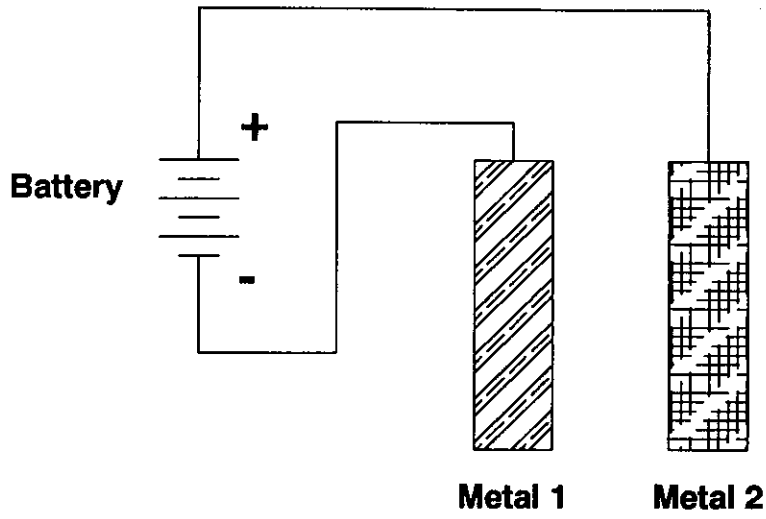


Figure 2.1. Two pieces of metal, each connected to a battery terminal. While the metals are well separated no current flows through the battery.

To begin, let's examine what electron tunneling means in the real world. Consider two pieces of metal. Metals are good conductors of electricity, i.e. electrons can move very easily and quickly from one end of the metal to the other. Imagine connecting one of the pieces of metal to the negative terminal of a battery and the other piece of metal to the positive terminal, as shown in Figure 2.1. If the metals are not touching, no current will flow through the battery. The electrons are free to move around the metal but cannot leave it. The electrons are analogous to water in a reservoir that is blocked by a dam. They can move about the reservoir but have no access to the river below. If the metals are brought together so that they touch, current will flow freely through the contacting area. The electrons have a free path from the negative terminal to the positive terminal of the battery. This current flow is analogous to opening up the gates of the dam and allowing the water to flow down the river into the ocean.

The unusual experimental feature of tunneling is this: when the metals are brought together, but are not quite touching, a small electric current can be measured. The current gets larger the closer the metals are brought together, until it reaches its maximum value when the metals are touching. The concept is analogous to making the dam thinner and thinner by removing cement and noticing that more and more water is leaking through the walls. However, there is a difference between the two analogies. The water physically moves through the pores between the cement, while the *electrons do not move in the space between the metals: they just suddenly appear in the other side.* The metals must be only 10 angstroms apart to produce detectable tunneling current. Figure 2.2 shows current as a function of the separation between metals [a]. Also plotted in this graph is the measured tunneling current if quantum mechanical tunneling did *not* occur [b]. The distances involved are so small that special tools are needed to adjust the distances or the small electric currents will not be detected. We will describe these tools in the section on piezoelectrics (see Section 2.4).

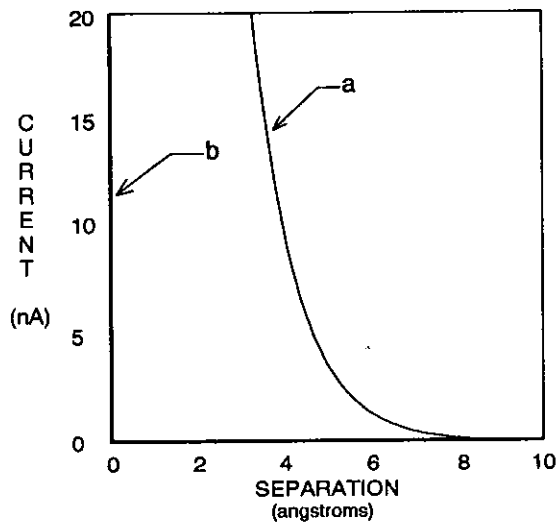


Figure 2.2. This graph shows a plot of current flowing through the battery as a function of the separation of the metals. [a] shows an exponential increase in current as the metals get closer; [b] in the absence of quantum mechanical tunneling, no current flows until the metals touch.

To understand why these small currents occur, the energies involved as the electron moves between the metals must be considered. An electron's energy can be split into two contributions: kinetic energy and potential energy. Kinetic energy (the energy of motion) is large for electrons moving fast and small for electrons moving slowly. Potential energy is the energy available for an electron to convert to kinetic energy if it moves along an electric field. Figure 2.3 plots the potential energy of the electron as it travels from one metal to the other metal. The potential energy shown neglects the complicated aspects of metals, including extra charges from atoms and other electrons on the metals, but does include the general concepts. The potential energy is lower in Metal 2 because this side is connected to the positive terminal of the battery (the terminal to which the electrons are attracted). There is also a large potential energy between the two metals. This is what tends to keep electrons inside their respective metal.

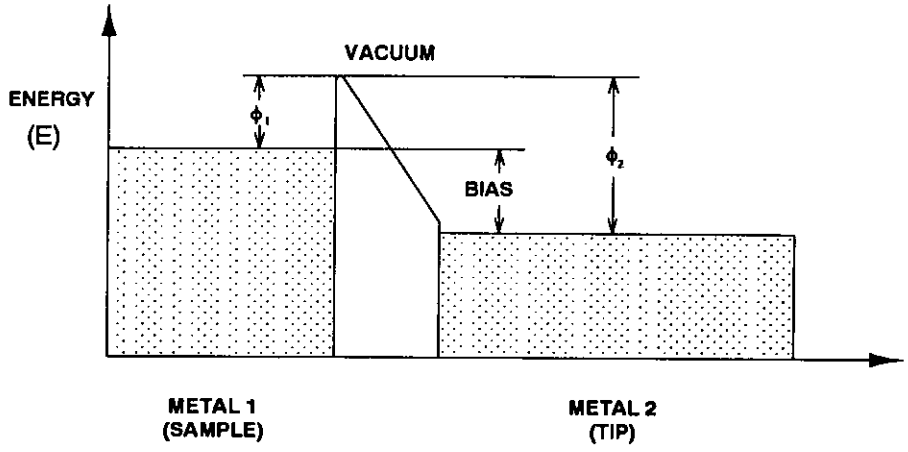


Figure 2.3. The potential energy that the electrons encounter as they travel from one metal to the other. ϕ_1 and ϕ_2 indicate the minimum amount of energy required to remove an electron from the metals (the work function). Note that Metal 2 is attached to the positive terminal, so its electrons have lower potential energy.

This picture shows that electrons are free to move around in their respective metals but cannot leave them. No electron in a metal has sufficient kinetic energy to go over the barrier.

One of the basic tenets of quantum mechanics is that electrons have both a particle and a wave nature. So we should picture the electron not as a hard ball impinging on the barrier, but as a cloud. The size of the cloud is related to the wavelength of the electron (a few angstroms). When the cloud collides with the barrier, part of the cloud may penetrate it. For thick barriers, the cloud will be reflected like a hard particle (see Figure 2.4). For thin barriers, however, part of the cloud may penetrate the barriers and appear on the other side. This process is called tunneling because the electron does not have enough kinetic energy to travel over the barrier, but is able to exist on the other side (see Figure 2.5). It is as if the electron found a way to dig a tunnel *through* the barrier.

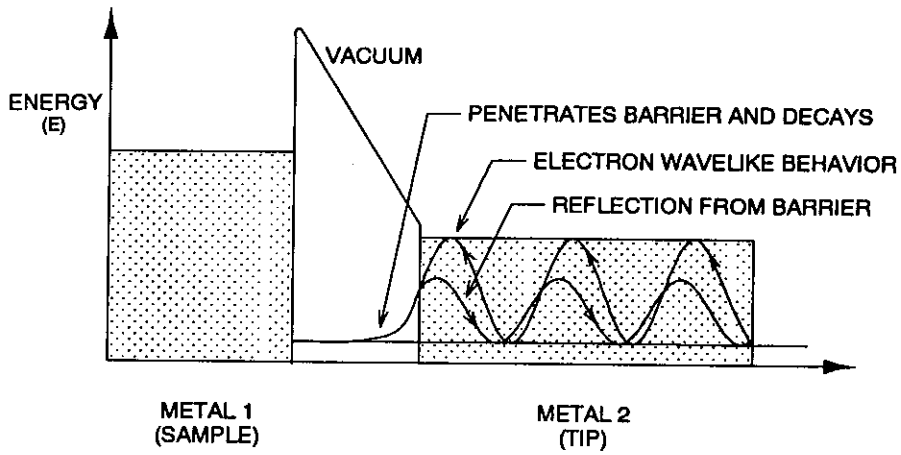


Figure 2.4. Electrons have a wave and particle nature. Upon impinging on the barrier they will be reflected from and penetrate the barrier. If the barrier is too "thick," the electron cloud will decay and no electron will tunnel to the other side of the barrier.

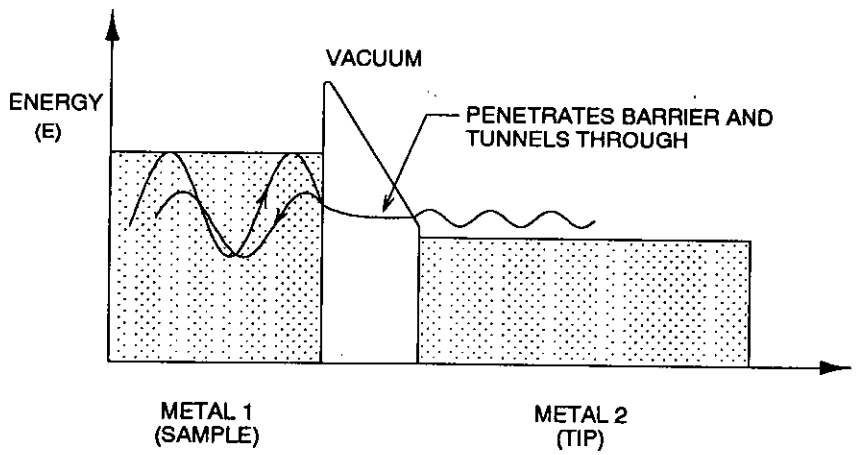


Figure 2.5. If the barrier is "thin," the electrons might tunnel and transmit through the barrier, thus creating an electrical current that can be measured.

In the scanning tunneling microscope, one of the metals is the sample being imaged (sample) and the other metal is the probe (tip). The sample is usually flatter than the probe, as shown in Figure 2.6. If the probe is sharpened into a tip it will most likely have one atom at the end. All of the tunneling electrons will pass through this atom. As we will discuss later, this feature leads to the atomic resolution capabilities of the microscope.

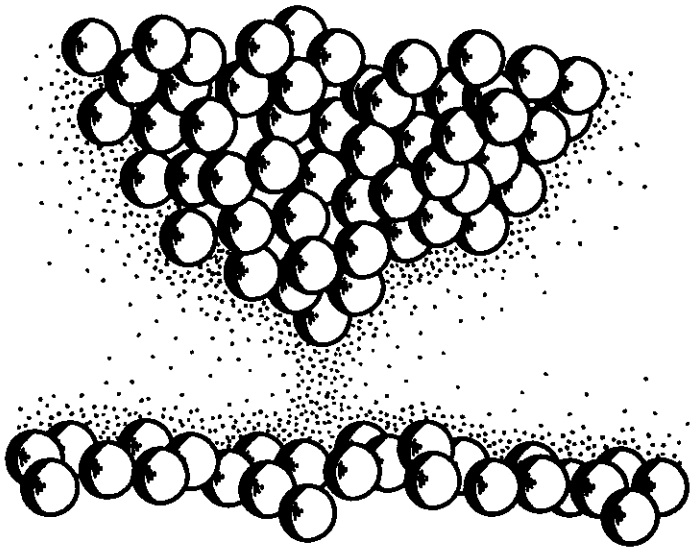


Figure 2.6. The tip consists of clusters of atoms in which one atom usually sticks out more than the others. This atom is primarily responsible for tunneling between the tip and sample.

2.3. Quantifying the Tunneling Process

Using Schrödinger's equation of quantum mechanics, we can actually predict how tunneling current increases as separation between two metals decreases. However, the final results of your tunneling experiments can be understood without knowing quantum mechanics. This more complete description is not necessary for understanding how the STM works; it therefore may be thought of as supplemental.

The Drude model of a metal states that the potential energy of a metal is given by the solid line in Figure 2.3. The energy of all the electrons in the metal is lower than the height of the wall. The difference in energy between the most energetic electron and the vacuum energy is called the workfunction and is denoted by the symbol ϕ.

The wave nature of an electron, illustrated in Figures 2.4 and 2.5, is critical to explaining tunneling. The movement and shape of the electron wave is governed by Schrödinger's equation, which might be thought of as the quantum mechanical analog of Newton's equation of motion, $F = ma$.[1]

In the STM, tunneling takes place between the tip and the sample. A complete description of the tunneling process requires a solution of the three-dimensional form of Schrödinger's equation, which has the general form:

$$[H]\psi = E\psi \tag{2.1}$$

where $[H]$ is the Hamiltonian operator and E is the total energy eigenvalue. The time dependent Schrödinger's equation can be expressed as:

$$\frac{-\hbar^2}{2m}\nabla^2\psi(r,t) + U\psi(r,t) = i\hbar\frac{\partial\psi(r,t)}{\partial t} \tag{2.2}$$

In this equation:

$\hbar$ is Plank's constant, a fundamental quantity in quantum mechanics, divided by 2π

m is the mass of the electron

$\psi(r,t)$ is the wave representation of an electron

U is the potential barrier function form

∇^2 is the Laplacian operator

$\frac{\partial}{\partial t}$ is the partial time derivative

i is $\sqrt{-1}$

For our purpose it is sufficient to use a one-dimensional analysis, which the Schrödinger equation above is given by:

$$\frac{-\hbar^2}{2m}\frac{\partial^2\psi(x,t)}{\partial x^2}+U(x)\psi(x,t)=i\hbar\frac{\partial\psi(x,t)}{\partial t} \tag{2.3}$$

where the equation:

$$\psi(x,t) = A\,e^{i(-kx-\omega t)}+ B\,e^{i(kx-\omega t)} \tag{2.4}$$

is the plane wave representation for an electron wavefunction of wavenumber $k = 2\pi/\lambda$ and angular frequency ω.

In addition, we assume a steady-state (time-independent) situation in which electrons of energy $E(x, t) = E$ encountering a uniform potential barrier of height $U(x, t) = U(x)$ are continuously flowing from one metal to the other. It is then necessary to solve only the one-dimensional steady-state Schrödinger equation, given by:

$$\frac{-\hbar^2}{2m}\frac{\partial^2\psi(x)}{\partial x^2}+U(x)\psi(x)=E\psi(x) \tag{2.5}$$

where E is the kinetic energy of the electron. Note that $U(x)$ is the potential energy of the electron as a function of position, as shown in Figure 2.3. $U(x)$ is smaller than the electron energy in the metals and larger than the electron energy in the barrier. For simplicity we can assume $U(x) = U_0$ a constant in the barrier.

In the metal, the general solution to the above equation is given by:

$$\psi(x) = Ae^{-ikx} + Be^{+ikx}, \quad k = \sqrt{\frac{2m(E - U_0)}{\hbar^2}} \qquad \text{(Metal 1)} \qquad (2.6)$$

$$\psi(x) = Ee^{-ikx} + Fe^{+ikx} \qquad \text{(Metal 2)} \qquad (2.7)$$

and in the barrier (the classically forbidden region) the solution is:

$$\psi(x) = Ce^{-\mu x} + De^{+\mu x}, \quad \mu = \sqrt{\frac{2m(U_0 - E)}{\hbar^2}} \qquad \text{(barrier)} \qquad (2.8)$$

Equations 2.6 and 2.7 show that the phase of the electron wavefunction varies uniformly in the metals. The wavelength is $\lambda = 2\pi/k$. Higher energy electrons have a smaller wavelength. When a high energy electron wave encounters the boundary of the metal, it "leaks out" a small amount, as discussed in the previous section. The "intensity" of the electron wave decays as a function of distance from the boundary. Mathematically, the argument of the exponential function becomes real and the electron wavefunction decays. (For imaginary arguments, the wave function would have oscillatory behavior.)

To gain a quantitative insight into the electron tunneling phenomena, it is necessary to derive an expression for the transmission coefficient, i.e. the transmitted flux from the sample to the tip through the barrier of width *L*. The barrier is considered wide but finite, such that the electron wavefunction exponential decay in the barrier is significant. Furthermore, the electron wavefunction and its first derivative must be continuous (join smoothly) at the sample-barrier and tip-barrier boundaries to conserve energy and mass. If we set up a coordinate system in which the surface of the sample (Metal 1) is at $x = 0$ and the tip (Metal 2) is at $x = L$, and apply the boundary conditions for continuity:

$$A + B \approx C$$

$$ik(\mathrm{A} - \mathrm{B}) \approx -\mu \mathrm{C} \qquad (2.9)$$

(at the sample surface, $x = 0$) where D, the amplitude of the reflected wavefunction at the sample-barrier boundary, is neglected, since $D << A, B, C$. However, D is *not* insignificant at the tip-barrier boundary. At the tip-barrier boundary, $x = \mathrm{L}$, continuity would require:

$$Ce^{-\mu L} + De^{\mu L} = Fe^{ikL}$$

$$-\mu Ce^{-\mu L} + \mu De^{\mu L} = ikFe^{ikL} \qquad (2.10)$$

Solving for B/A at $x = 0$, by solving for C and substituting for it, we get:

$$\frac{B}{A} \approx \frac{-(1 + ik\delta)}{1 - ik\delta} \qquad (2.11)$$

where δ is $1/\mu$, A is the amplitude of the electron wavefunction in the sample surface incident on the barrier, and B represents the amplitude of the reflected wavefunction. The reflection coefficient (R) for the wavefunction is then defined as:

$$R \approx \left|\frac{B}{A}\right|^2 \qquad (2.12)$$

where $|\ \ |^2$ represents the product of a complex number and its conjugate. In this case, it represents the relative intensities of the incident and reflected wavefunctions.

An electron incident at the barrier will either be reflected or transmitted through the barrier. In terms of probability or frequency of occurrence, $R+T = 1$, where R and T are the reflection and transmission coefficients. Thus:

$$R \approx \left|\frac{B}{A}\right|^2 = \frac{-(1+ik\delta)}{1-ik\delta} \cdot \frac{-(1-ik\delta)}{1+ik\delta} \approx 1 \tag{2.13}$$

and, therefore:

$$T = 1 - R \approx 0 \tag{2.14}$$

which indicates that, for an infinitely wide barrier, no electrons would be found in the barrier region. Nevertheless, dividing the first of the sample vacuum-barrier boundary conditions by A results in:

$$1 + \frac{B}{A} \approx \frac{C}{A} \tag{2.15}$$

The probability of finding an electron in the barrier region at $x = 0$, due to quantum tunneling, is given by:

$$\left|\frac{C}{A}\right|^2 \approx \frac{4(k\delta)^2}{1+(k\delta)^2} \tag{2.16}$$

To find the effective tunneling transmission coefficient, $\left|\frac{F}{A}\right|^2$ i.e. the relative probability or frequency of occurrence of an electron tunneling out of the sample surface, across the sample-tip-barrier region, and into the tip, combine the tip-barrier boundary equations (at $x = L$) and Equation 2.16 to get:

$$\frac{F}{A} \approx \frac{-4ik\delta}{(1-ik\delta)^2} e^{-L(\mu+ik)} \tag{2.17}$$

which produces the desired quantitative result:

$$T(E) \approx \left|\frac{F}{A}\right|^2 \approx \left(\frac{4k\delta}{\left(1+(k\delta)^2\right)}\right)^2 e^{2\frac{L}{\delta}} \propto e^{-L\sqrt{2m\phi/\hbar^2}} \tag{2.18}$$

where:

$$k^2 = \frac{2mE}{\hbar^2}$$

$$(k\delta)^2 = \frac{E}{(U_0 - E)} = \frac{E}{\phi} \tag{2.19}$$

Substituting typical numbers of $\phi = 5\text{x}10^{-19}$ joules, $m = 9\text{x}10^{-31}$ kilograms, and $\hbar = 1.05\text{x}10^{-34}$ joules-seconds, results in:

$$T(E) = e^{-2L}, \quad L \text{ in angstroms} \tag{2.20}$$

This formula shows that for each angstrom change in separation, the probability that an electron tunnels decreases by an order of magnitude. This demonstrates mathematically that tunneling current is indeed a sensitive measure of the distance between the tip and sample.

In the STM, one of the metals is the sample being looked at and the other metal is the probe. The sample is usually flatter than the probe, as shown in Figure 2.6. Because the probe is formed of atoms, if it is sharpened into a tip, it will most likely have one atom at the end of the tip. The spacing between atoms is about 3 angstroms. Therefore, any tunneling through atoms that are one atom back from the closest atom is a fraction [$e^{-(2)(3)} = 0.002$] of tunneling through the atom at the tip, as shown in Figure 2.6. Virtually all of the tunneling electrons will pass through the single atom closest to the surface. This feature produces the atomic resolution capabilities of the microscope. The transmission probability of electrons integrated over time manifests as nanoamp currents for the sample and tip separation of few angstroms, as indicated in Figure 2.2.

2.4. Angstrom Motion Using Piezoelectrics

2.4.1. What are Piezoelectrics?

In the last section, we claimed that electron tunneling occurs only when the sample and tip are separated by 10 angstroms. How can two pieces of material be moved together with this precision? To image a surface with atomic resolution, the surface must be moved with the precision of less than that of an atom's length. This motion is accomplished using piezoelectric ceramics (in general called PZT).[2]

If an electric field E is applied across a piezoelectric ceramic, it will expand in one direction and contract in the other. This process is illustrated in Figure 2.7. Piezoelectricity is quite common in nature. Quartz, for example, exhibits this effect. Materials which exhibit the piezoelectric effect are all electrets. An electret is analogous to a magnet, but instead of having a permanent magnetic field that causes it to attract iron bits and other magnets, an electret has a permanent electric field. The reason an electret has a permanent electric field is that it is composed of at least two different kinds of atoms, one that is positively charged and another that is negatively charged. Certain arrangements of these charged atoms can lead to a permanent electric field in the crystal, as illustrated in Figure 2.8. When another electric field is applied across the crystal, the negatively charged atoms will move up and the positively charged atoms will move down, leading to a net expansion of the crystal. In general, PZT ceramics consist of such crystalline grains which align in response to external electric fields and cause the dimensions of the PZT to change.

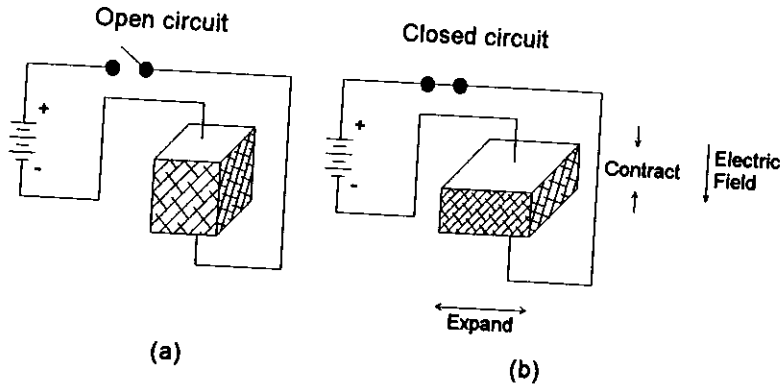


Figure 2.7. PZT in response to applied electric fields changes dimensions.

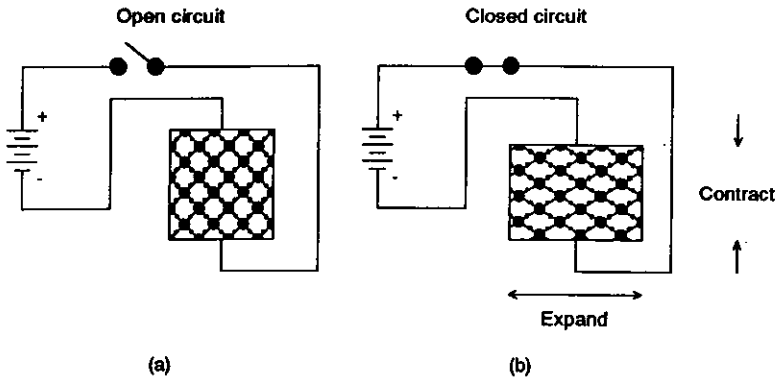


Figure 2.8. Large and small circles represent two different kinds of atoms. These atoms are arranged to have a permanent electric field (an electret). When an electric field is applied, the atoms realign in response to the field. This can cause a physical change in the crytstal's dimensions.

The sensitivity of piezoelectric ceramic depends on the particular arrangement of the atoms in the crystal and its thickness. The sensitivity of the piezoelectric materials used in the STM is about 150 angstroms/volt. Because the computer running the microscope can control voltages with millivolt accuracy, it can control the motion of a PZT material to less than an angstrom sensitivity.

2.4.2. Scanning with Piezoelectrics

Three-dimensional motion is achieved by shaping a piezoelectric ceramic into a hollow cylinder. This piezoelectric device is illustrated in Figure 2.9. The tip is attached to the bottom of the cylinder, while the top of the piezoelectric and the sample are fixed. Four electrodes are formed on the outside of the cylinder, as shown in the figure, and one electrode is formed on the inside of the cylinder. By independently controlling the voltages applied across these electrodes, the piezoelectric cylinder can bend in any direction and be extended and retracted. We show two examples of this in Figure 2.10.

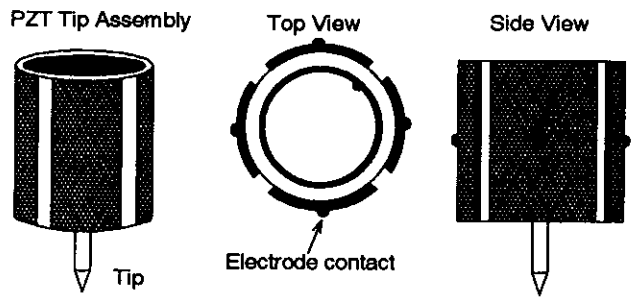


Figure 2.9. A PZT ceramic made into a hollow cylinder with quadrants is for "fine" motion control of the tip.

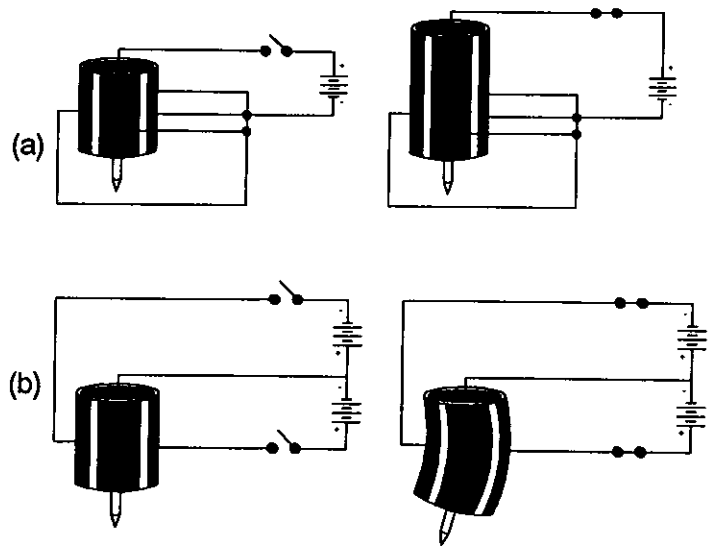


Figure 2.10. Applying a potential between the inner electrode of the PZT cylinder and its outer electrodes [a] will cause the PZT to become thinner and to elongate. Applying a potential between opposite outer electrodes of the PZT cylinder with respect to the inner electrode [b] will bend the PZT.

In Figure 2.10[a] a negative voltage is applied to the outside electrodes and a positive voltage is applied to the inside electrode. The electric field, therefore, points from the inside to the outside of the cylinder. This is opposite the poled direction of the tube. The cylinder will shrink between the electrodes (i.e. the wall will become slightly thinner) and, to keep the volume constant, the tube will become longer. If an opposite polarity is applied to the electrodes, the reverse will happen and the tube will shrink. Applying voltages in this way gives control of the motion parallel to the tube's centerline.

In Figure 2.10[b] a positive voltage is applied to the left electrode, a negative voltage to the right electrode, and the other electrodes are held at ground. In this example, the left side of the tube will shrink and the right side of the tube will lengthen, with the front and back of the tube staying the same length. The tube can accomplish this shrinking and growing by bending to the left. If an opposite polarity is put on the left and right electrodes, the tube will bend to the right. For small amounts of bending, the fact that the tip also moves down a little, as well as to the right and the left, can be neglected. If voltages are applied in the same way across the front and back electrodes, the tip bends to the front and to the back. The voltages to the left and right electrodes and to the front and back electrodes can be applied simultaneously to move the tip at any angle; a voltage applied to the inside electrode will cause up and down motion along with this bending. By applying voltages in this way, the end of the piezoelectric cylinder can be positioned anywhere within a three-dimensional region with subangstrom precision.

2.5. Negative Feedback

Measuring tunneling current provides a way to sense the location of the sample relative to the tip. If there is no tunneling current, the tip is too far from the sample. If there is a small tunneling current, the tip is near the sample. Negative feedback turns this position sensor into a microscope.

The device that measures the tunneling current is connected to electronic feedback circuitry that obeys the following rule: *if it senses a decrease in the current, it moves the tip closer to the sample, and if it senses an increase in the current, it moves the tip away from the sample.*

Now imagine a surface with some bumps on it. This surface is illustrated in Figure 2.11. The microscope is operated by positioning the tip somewhere over the left-hand side of the sample and lowering it toward the sample until a tunneling current is detected. The tip is then moved to the right. When the tip approaches a bump, the tunneling current increases. The feedback loop senses this increase and raises the tip to maintain a constant tunneling current. The tip will continue to rise until it is over the top of the bump. As the tip moves farther to the right, the current decreases and the tip must be lowered to maintain a constant tunneling current. By the time the tip has reached the far right, it has traced out a cross section of the topography of the sample, as illustrated in Figure 2.11.

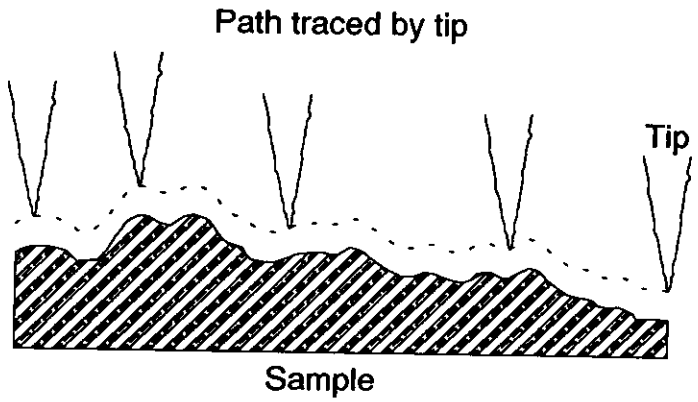


Figure 2.11. The tip traces the sample topography from left to right. When the tip gets close to a bump, tunneling current increases. The feedback loop, in response to an increase in the current above a preset reference tunneling current, moves the tip up. Conversely, if the feedback loop senses a decrease in current, it moves the tip in to compensate. Thus the tip traces the surface topography as it scans the sample, as shown by the dashed line.

2.6. Vibration Isolation

One of the problems that delayed the development of the STM was the intuitive feeling that it would be impossible to hold two objects only 10 Å apart without having them crash into each other. Even though it cannot be seen by the eye, objects are always in motion relative to each other. Walking around a room causes desks and chairs to vibrate with an amplitude of about one micron, or 10,000 angstroms! To have the feedback loop operate as described above, these vibrations must be eliminated.

At first it may seem to be an impossible task to stop materials from vibrating on the angstrom-length scale of the STM sensitivity. However, it turns out to be quite easy to isolate the STM from these vibrations. Three methods of vibration isolation are illustrated in Figure 2.12.

Historically, the first method of vibration isolation used was superconducting levitation. Superconducting levitation is the phenomenon that if a magnet is placed above a superconductor, the superconductor will repel the magnet and not allow it to sit on its surface. If the STM is placed inside this magnet, it will essentially float and thus remain isolated from noise in the room.

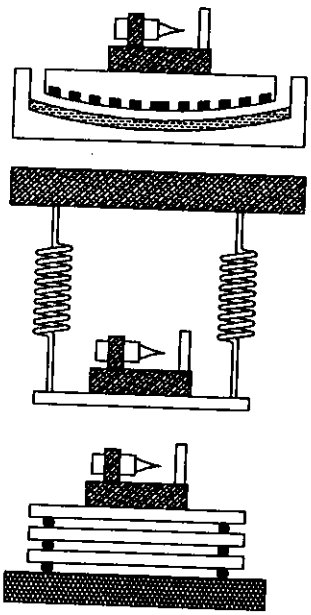


Figure 2.12. Three methods of vibration isolation. In [a] the microscope floats on top of a container filled with liquid nitrogen. In [b] springs, in conjunction with magnets for Eddie Current damping, provide an effective means of vibration isolation. Stacked plates with elastomer spacers [c] has become the standard configuration to isolate vibrations in most compact microscopes.

Another way to isolate the microscope from vibrations is to hang it by springs from a fixed surface. If the fixed surface vibrates, the springs will stretch to accommodate this vibration and the plate on which the tunneling microscope is placed will remain fixed.

A third way to isolate the microscope from vibrations is to place it on top of a series of metal plates. The plates are separated by elastomer spacers ("O" rings). The spacers act as springs and dampers. If a lower plate vibrates, the plate above it will vibrate less because the elastomer will compress and expand. The more plates that are stacked up, the smaller the vibrations will be by the time they get to the top plate. Assume that each plate will vibrate with 1/5 less amplitude than the plate below it because of the spacers. If four plates are stacked, as illustrated in Figure 2.12, the top plate will vibrate with 1/125 the amplitude of the bottom plate. Actual microscopes using these three different techniques are shown in Figure 1.8.

2.7. Electronic Data Collection

The final result of running the STM must be a picture. So how do we get from the concept of negative feedback and quantum mechanical tunneling to a picture? With an optical microscope the magnified image, due to reflected light, is something you can look at directly. With a scanning tunneling microscope, the image is a collection of voltages. The tunneling microscope must be interfaced to a computer with graphic capabilities to look at the image. This interface is shown in Figure 2.13.

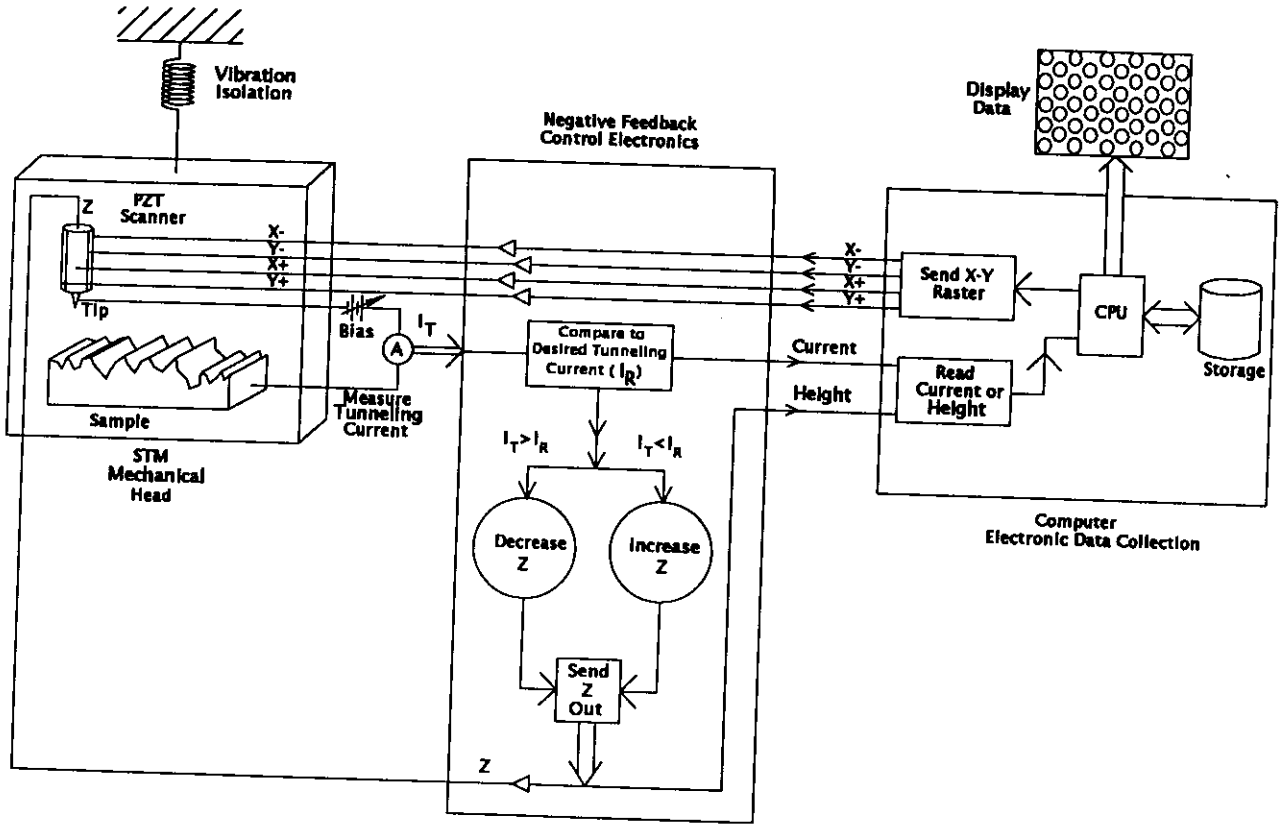


Figure 2.13. A block diagram of the STM system: the STM Mechanical Head, consisting of vibration-isolation mechanism, PZT, sample, and tip; Negative Feedback/Control Electronics, consisting of the feedback loop and X, Y, and Z power supplies; and Computer Electronic Data Collection, consisting of a computer, data acquisition card, and graphics display.

An STM image is built up one point at a time. A bias voltage is applied to produce a tunneling current between the sample and tip. (The direction of current flow depends on the sign of the bias voltage.) The tip is made to move across a surface from left to right (in the X direction), moving up a little bit each time (in the Y direction) as shown in Figure 2.14. The computer generates the voltages that move the tip in the X-Y pattern ("X-Y rastering") and sends this information to the control electronics, which subsequently amplifies the voltage to a large enough value to move the PZT scanner. The control electronics uses negative feedback to apply a voltage to the PZT scanner to move it away from and toward the surface (in the Z direction), so that the tip traces a contour of the surface. The control electronics simultaneously sends the Z axis voltage required to maintain a constant tunneling current back to the computer.

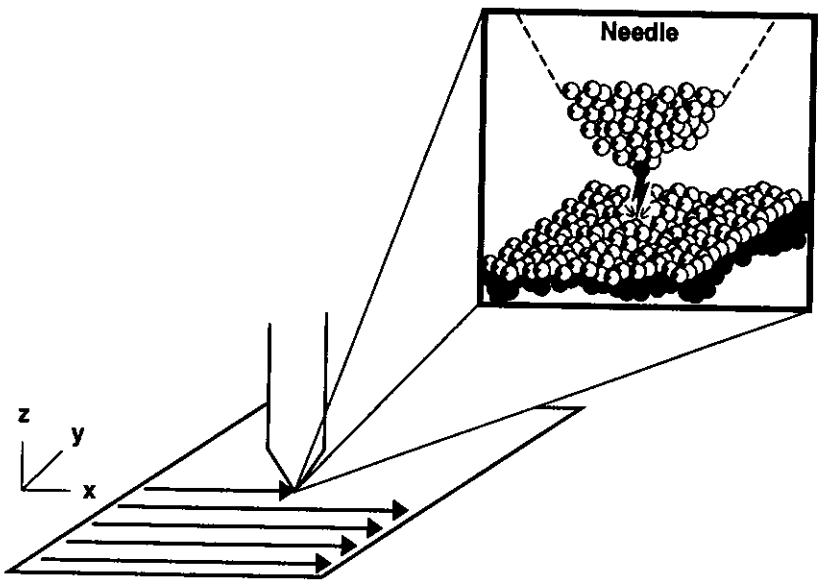


Figure 2.14. "X-Y raster": the tip moves across the surface from left to right (X) and up a little (Y) at the end of each X-raster.

From the computer's point of view, it sends out a voltage in the X and Y directions and retrieves a voltage in the Z direction. The X and Y voltages correspond to the lateral position along the sample and the Z voltage corresponds to the height of the sample. Thus, after the computer scans, it knows the height of the sample as a function of lateral position. This information can be plotted on the computer's display. The X and the Y position are the Cartesian coordinates of a point on the display, and the gray level is proportional to the Z value that the computer received. Plotted on the computer using this gray scale, high areas of the sample will appear white and low areas of the sample will appear dark.

3. OPERATING INSTRUCTIONS

We assume that the Instructional Scanning Tunneling Microscope (ISTM) is already unpacked and installed. If not, please refer to the *Instructional Scanning Tunneling Microscope Operating Manual and Quick-Start Procedures* for detailed instructions on how to unpack and install the system.

This section will give you an overview of the operation of the ISTM and basic setup procedures to start your exploration of the exciting world of scanning tunneling microscopy. It will also give you the necessary knowledge and skills for setting up the experiments which follow in Chapter 4.

3.1. A General Overview of the Burleigh ISTM

The ISTM is comprised of three subsystems: the electromechanical tunneling assembly, the control system and power supplies, and the display device. These parts will be referred to throughout the manual as: the head, the control electronics, and the computer. These parts are shown in Figure 3.1. In addition to the microscope itself, the system includes a set of samples and tips, described in Section 3.2.

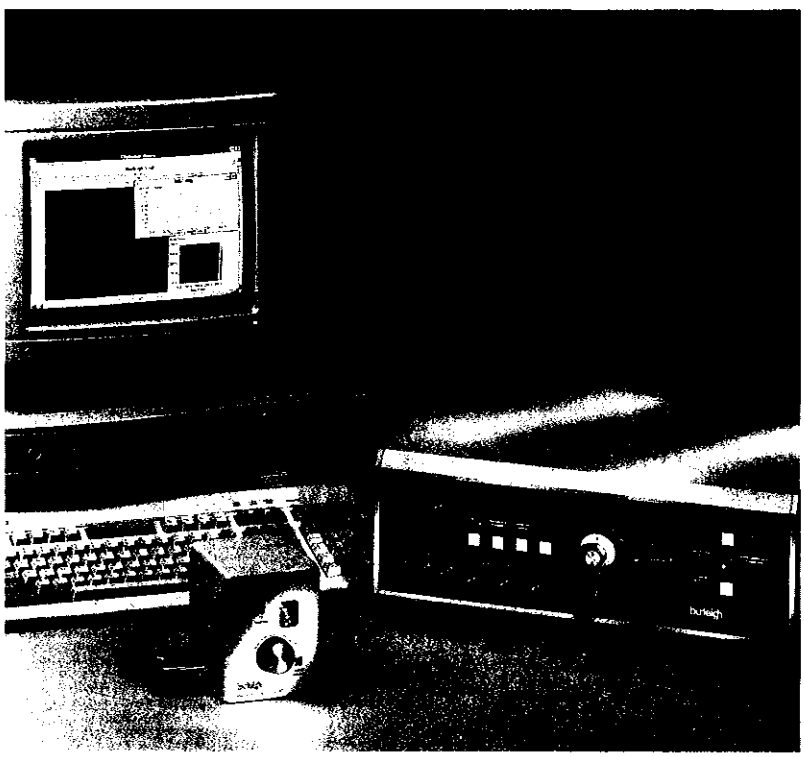


Figure 3.1. The Burleigh Instructional STM System.

The head houses the sample and tip, both of which are easy to install and remove. The tip is attached to a tubular PZT for X and Y rastering over the sample surface and "fine" Z movement perpendicular to the surface. These components are mounted on a vibration-isolation mechanism, also housed inside the ISTM Head. The head also contains an amplifier circuit that converts the small tunneling current to a voltage and then amplifies it to a usable level before sending it to the control electronics.

The control electronics uses the concept of "negative feedback" to keep the sample and tip within tunneling distance as the tip is rastered over the sample. The control electronics accomplishes this by increasing the voltage to the Z element of the PZT if the tunneling current falls below the desired value or decreasing the voltage if the tunneling current rises above the desired value.

As the control electronics applies corrections to keep tunneling current constant, an error signal is generated. This error signal and the amplified tunneling current are sent to the computer. In real time the computer reads this data, sends X and Y raster signals to the control electronics, and, synchronous with the raster, displays the data on its monitor, forming the image.

3.2. The ISTM Head

3.2.1. Head Characteristics

Figure 3.2 shows the ISTM Head. Though rugged and compact, the ISTM Head is a sensitive precision instrument and should be handled with care. The head has a unique design, allowing you to exchange samples and tips in seconds. Both the tip and sample are held in place magnetically and can be accessed through the small window on the front of the head. Figure 3.3 shows the sample holder and its carriage.

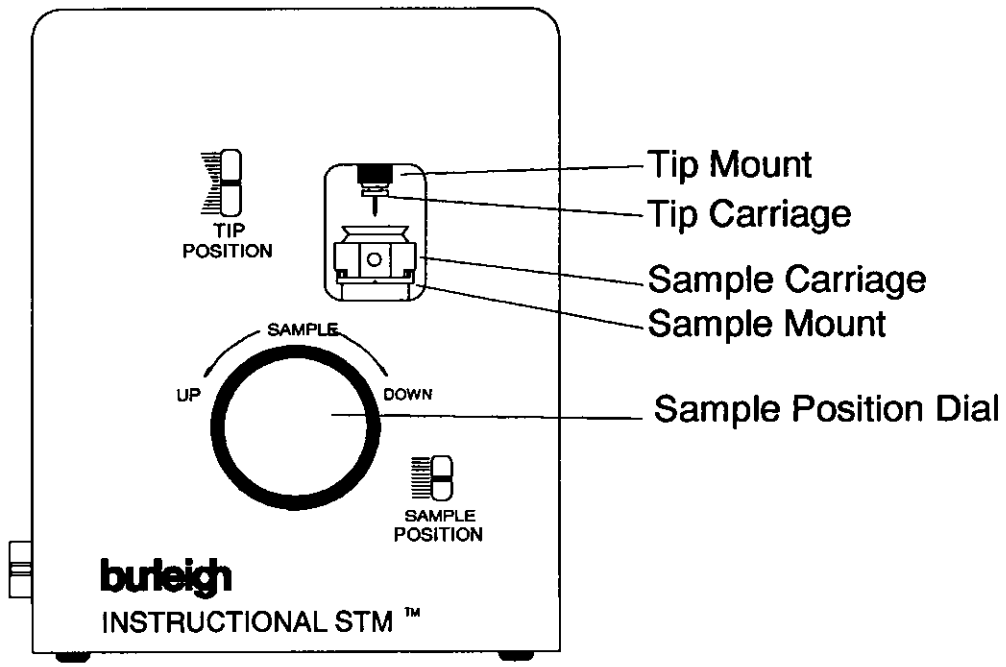


Figure 3.2. The ISTM Head and its components: the tip, fixed in a tip carriage, which is magnetically held in place by the tip mount; the SEM sample holder, which is plugged into the sample carriage and held magnetically in place on the sample mount; and the sample dial, which moves the sample up or down with respect to the tip.

The sample holder is a standard 1 cm aluminum SEM plug that has been incorporated into the design of the sample carriage. The sample holder inserts into the carriage (Figure 3.3) and is fixed by a small set screw (for detailed instructions see Section 3.2). Samples are changed by simply removing the SEM plug and replacing it with another. Figure 3.2 shows the ISTM Head loaded with a sample and a tip. The tip is also mounted in a removable holder as shown in Figure 3.4, and is held magnetically.

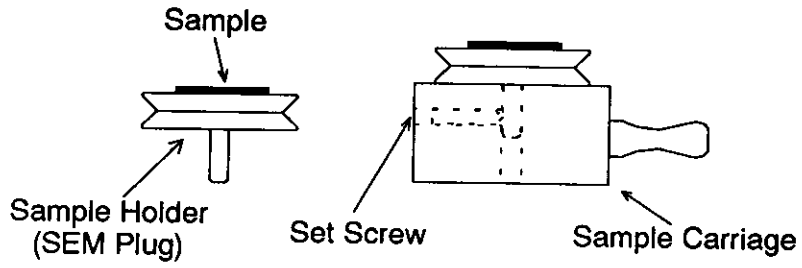


Figure 3.3. The sample is glued onto the Sample Holder (SEM Plug) and is inserted into the Sample Carriage. A #2 Set Screw holds the Sample Holder in place.

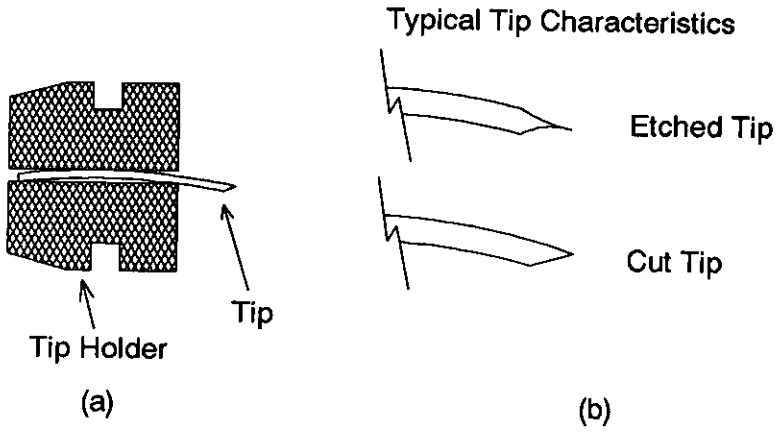


Figure 3.4. The tip is inserted into the Tip Holder [a]. Note that the tip is slightly bent to keep it more secure. Typically etched tips [b] look visually sharper than mechanically cut tips.

The tip can automatically be brought to within atomic distances of the sample in a matter of seconds. One of the issues not discussed in previous sections was the fact that PZT motion alone is not large enough to move the tip away from the surface for easy access to the sample or tip. The PZT used here has a range of about 1.2 microns in Z and about 3 microns in X and Y. (The actual range of your scanner is saved in the software parameter page.) Given the range of motion of the PZT, then, how can the tip be brought close to the sample from macroscopic distances? This is accomplished using a stepper motor in combination with a motion-reduction lever mechanism similar to the one shown in Figure 3.5. The motion-reduction mechanism allows a finer motion than the step size of the motor allows. The tip is

stepped toward the sample automatically. The control electronics drives the motor and terminates this motion when a preset tunneling current is sensed. A mechanical indicator on the upper left-hand side of the head shows the tip position and its sense of direction (see Figure 3.2).

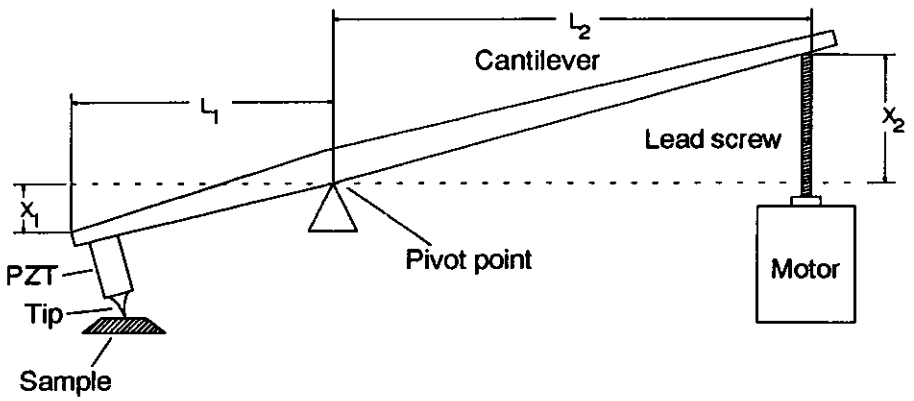


Figure 3.5. The tip coarse-reduction approach is controlled by a motor through a reduction of motion mechanism. The motion X_2 of the motor is reduced by a factor of L_2/L_1 in moving the tip.

The sample can also be moved toward or away from the tip by turning the Sample Position dial on the front of the head. Turning the dial counterclockwise moves the tip and sample closer together; turning it clockwise moves them apart. *Be careful when using this dial: you can easily run the sample into the tip and damage both sample and tip.* Observe the mechanical indicator on the lower right-hand side of the head showing the relative position of the sample to the tip. Do not attempt to go beyond the available range.

The vibration-isolation mechanism used in the ISTM Head consists of a stack of four metal plates with special rubber damping material between them, as discussed in Section 2.6. To achieve the best results, place the head on an optical table or vibration-isolation table. You can use a variety of techniques to decrease environmental effects. One of the easiest is to place a heavy metal plate on top of a small, partially inflated inner tube and then place the head on top of the plate. You should also avoid noisy environments for the operation of the ISTM. Acoustical noise can couple into the sample-tip junction, causing unwanted interference in the image, or possibly no image at all! Again, remember that the sample and tip are only angstroms away when tunneling. Even though the internal vibration-isolation mechanism of the ISTM is very effective, attention to reducing outside sources of vibration will result in much better images.[1]

The head is connected to the control electronics via a connector on its side. The connecting cable carries the sample-tip bias voltage and the X, Y and Z PZT voltages to the head and returns the amplified tunneling current to the control electronics. Since the cable can conduct unwanted mechanical vibration into the head, try to position it with a slight loop or bend near the head to absorb vibrations.

3.2.2. Setting up the Head

To set up the head, first make sure the control electronics is turned off. At this point the sample carriage and/or tip may or may not be loaded into the head. Figure 3.2 shows the head with a sample and tip holder already loaded. If no sample is loaded, refer to Sections 3.2.2.1-4. If a sample and tip are already loaded, make sure you find out from your instructor what the sample and tip material are before proceeding.

A note about tools: you will need a pair of tweezers or needle nose pliers for the following operation. Since the head contains powerful magnets to retain the sample and tip, it is best to use non-magnetic tools. Unfortunately, non-magnetic tools may not always be available. If you must use magnetic (steel) tools, be careful that they are not pulled away from you, damaging the sample or tip. For best results, rest a thumb on the outside of the head and use it to support the tool.

To remove any preloaded samples and tips:

1. Watching the sample position indicator, located on the lower right-hand corner of the ISTM Head, move the sample down by turning the Sample Position dial clockwise. *Do not exceed the range of the indicator.*

NOTE: Do not turn the dial if you feel any resistance.

2. Stop when there is 1 - 2 mm space between the sample and tip.

3. Gently slide the sample carriage (Figure 3.3) out of the sample mount using your fingers (Figure 3.6).

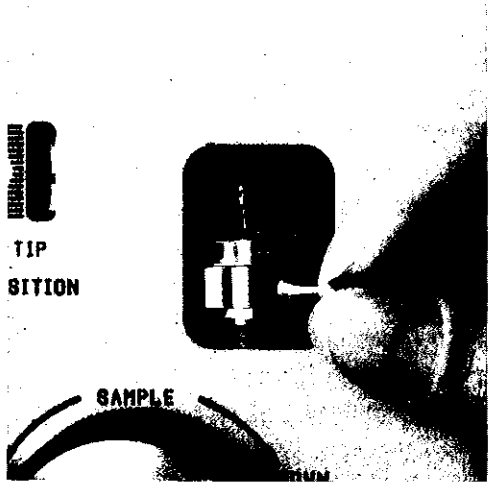


Figure 3.6. Use your fingers to take hold of the sample carriage handle and carefully slide out the carriage.

4. Avoid contacting the sample with the tip when removing the sample: this will scratch the sample and destroy the tip.

5. Set the sample aside in an upright position. Do not touch the surface of the sample: doing so will destroy the surface.

6. Hold the pliers or tweezers firmly and be sure that they are not pulled to the sample magnet while removing the tip holder (Figure 3.7). An optional pair of non-magnetic tweezers is provided with the sample-tip set.

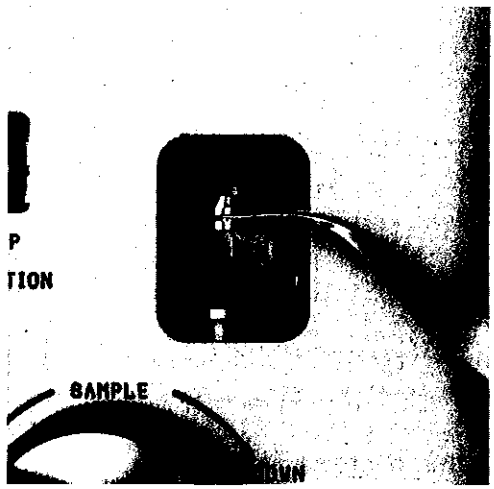


Figure 3.7. Use a pair of tweezers or pliers to remove the tip holder carefully.

7. Place the tip holder down on the table with the tip pointing up, to avoid damage.

Once the tip and sample are removed, you can load a new sample and tip.

3.4. True-Image™ Software Overview

The physical hardware necessary for scanning tunneling microscopy existed in the 1940s or even earlier, although the concept had yet to be imagined. Even the early STM experimenters used X-Y chart recorders or storage oscilloscopes to display images they acquired. However, it is modern computer technology that makes STM practical and allows those who are not specialists to acquire and manipulate meaningful images.

Burleigh True-Image software was written as a Microsoft® Windows™-based product, taking advantage of the ease of use provided by one of the most popular graphic-user interfaces (GUIs) and the inexpensive processing power of the latest generation of 386 and 486 PCs.

The True-Image software manages the scanning signals sent to the ISTM Control Electronics and displays the resulting images in close to real time. It also incorporates sophisticated image-processing functions specifically chosen for their applicability to STM images.

If the True-Image software is not installed, refer to the *Instructional Scanning Tunneling Microscope Operating Manual and Quick-Start Procedures.*

To start the True-Image software:

1. Type WIN at the Microsoft DOS® prompt.
2. Double-click on the Burleigh icon
3. Double-click on the STM icon.

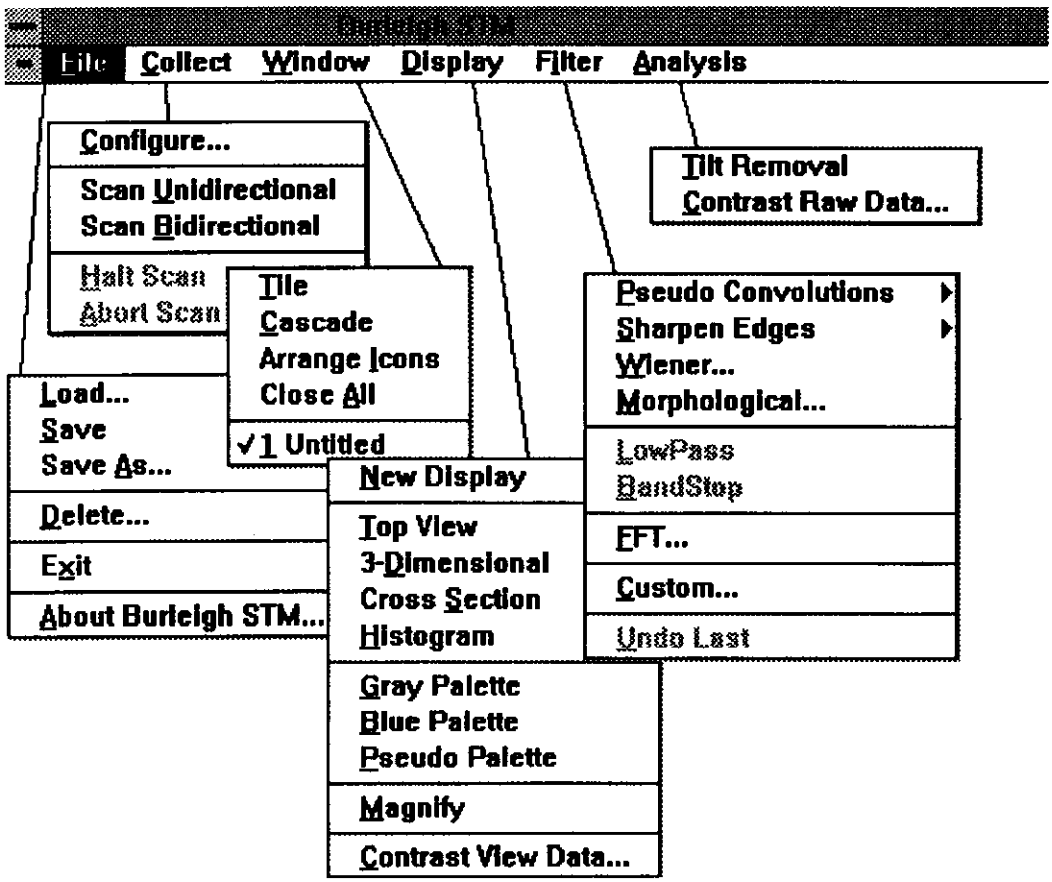
To shutdown the True-Image software:

1. Close the True-Image software by selecting Close from the File menu. You can also double click on the upper left-hand corner minus sign.
2. A menu box may appear prompting you to save your image data. If you wish to keep the image, choose YES and follow the prompts.
3. You will now be back in the Windows Program Manager. Double click on the upper left-hand corner minus sign to exit Windows.
4. One last message box will appear and Windows 3.0 will give you the option to keep any Windows changes. Windows 3.1 does this automatically. Choose the desired option. Note: do not "shell" out of Windows by choosing a DOS icon. You must fully exit Windows.
5. You should now be back at the standard DOS prompt. Almost all systems can be shut off at this point; however, very few may require manual head parking or cache flushing. See your system documentation.

NOTE: You need to make an orderly exit from both the True-Image software and Windows before turning off the computer. Failure to do this may result in loss of work, or even hard disk corruption, if the computer is shut off before data is properly updated.

3.4.1. Menu Structure

The True-Image software is a Windows program and operates according to the same general rules as all Windows software. For reference, the first two levels of the menu structure are shown below:



3.4.1.1. The File Menu

The File menu contains commands that are used to manipulate image files. The commands in this menu allow you to load existing files, delete existing files, and save new or existing files.

Load is used to automatically open an image window and load an existing image file into that window. If an image window is already opened and active, the Load command will allow an existing image file to be loaded into the opened window for analysis. To load an image from the image directory:

1. Click on the File menu.
2. Select and click on the Load submenu.
3. Another menu containing the directory and image files will appear.
4. Use the mouse to select one of the image files and double click on the desired name.
5. The image should appear on the screen.

Save is used to save the image file associated with the currently active image window. If the image file is already named, the file is saved under its current name. If the file is not already named, you will be prompted to enter a valid image filename under which the image file will be saved. The file extension used should be *.img*.

Save As can also be used to save the image file associated with the currently active image window. However, Save As always prompts you to enter a valid image filename under which the image file will be saved. This allows you to save a previously named file under a new name and to save a modified file without losing the original. The file extension used should be *.img*.

Delete allows existing image files to be deleted. You will be prompted to enter a valid image filename to delete from the disk. *Delete should be used cautiously: any files that are deleted by this command are unrecoverable by the system.*

Exit causes the Burleigh STM software to be terminated, sending control back to the operating system. Before performing this action, all valuable images in the system should be saved under appropriate filenames so they can be used in future sessions.

About Burleigh STM displays software version information about the current Burleigh STM software.

3.4.1.2. The Collect Menu

The Collect menu contains commands used for configuring data collection scans. To set up for a new page and scan:

1. Click on the Display menu.
2. Select the New Display submenu.
3. Select the Collect menu.
4. Select the Configure submenu. The parameter entry page will display.

Configure is used to set all of the available parameters pertaining to the scanning of an image. When this command is selected, a parameter entry dialog box is displayed. For a new display, the parameter values in the dialog box are set to default settings. For an image that has been loaded from disk, the parameters are set to the values that were entered when that particular image was scanned.

A description of each of the scanning parameters follows:

Data Points in X Direction is the number of actual samples (the number of data points) that will be taken as the microscope scans in the X direction. The number of samples can be powers of 2 up to 256.

Data Points in Y Direction is the number of actual scan lines (the number of data points) that will be taken as the microscope scans in the Y direction. The number of samples can be powers of 2 up to 256. The number of X and Y points should be equal.

Max Scanner Range in X is the maximum number of angstroms that the microscope can cover as it scans in the X direction. You may recalibrate and change this number. Your microscope is calibrated prior to shipment and the exact maximum scanner range in X is reflected in this menu.

Max Scanner Range in Y is the maximum number of angstroms that the microscope can cover as it scans in the Y direction. You may recalibrate and change this number. Your microscope is calibrated prior to shipment and the exact maximum scanner range in Y is reflected in this menu.

Max Scanner Range in Z is the maximum range of the motion of the PZT along the Z direction (perpendicular to the sample). The range of this scanner in Z is about 40 percent of the range in X and Y. Your microscope is calibrated prior to shipment and the exact maximum scanner range in Y is reflected in this menu.

Scan Range in X is the actual number of angstroms that the microscope will cover as it scans in the X direction is this number divided by the zoom factor. This value must not exceed the value for the Max Scanner Range in X. For a scan range of 5000 Å and zoom of 50 you scan a 100 Å area of the surface.

Scan Range in Y is the actual number of angstroms that the microscope will cover as it scans in the Y direction is this number divided by the zoom factor. This value must not exceed the value for the Max Scanner Range in Y.

Bias Voltage is the number of millivolts that is used to bias the microscope tip. This value should be set to match the bias voltage displayed on the front panel of the instrument.

Tunneling Current is the number of nanoamps that is flowing though the microscope tip. This value should be set to match the tunneling current displayed on the front panel of the instrument.

Scan Delay is the number of milliseconds that will elapse between each sample as the microscope scans in the X direction. This value can be set between 0 and 10 milliseconds. You need to adjust the scan speed depending on constant current or height mode of operation. This parameter can be changed with the slide ruler.

Zoom Multiplier is the value that is used to determine the actual number of angstroms encompassed when scanning in the X and Y directions. This value should be set to match the value of the zoom switch on the front panel of the instrument.

Scan Mode: selecting the **Single** option will cause the image to be scanned only once before stopping. Selecting the **Continuous** option will cause the image to be scanned continuously until you halt or abort the scan.

Data Type: the **Current** scan data option should be selected when the data that is being taken represents values in current. The **Topographic** scan data option should be selected when the data that is being taken represents values in angstroms.

Plane Removal: selecting this option will cause the raw data that has been scanned to be modified such that a plane is fit through the data and subtracted out to rectify any physical tilt of the sample material. The results of plane removal will only be seen after the current scan has been halted.

Line Removal: selecting this option will cause the temporary view data that is being displayed during a scan to be modified such that a line is fit through the data for each scan line and subtracted out to rectify any physical tilt of the sample material. This operation is done on a scan line by scan line basis, so the results will be seen as the image is being scanned. This operation does not affect the raw data of the image in any way.

OK will accept the current scan parameters for any subsequent scanning operations.

Cancel will abort any changes to the current scan parameters.

Default will save the current scan parameters to the application initialization file so that the current scan parameters will be used as the default scan parameters in the future.

After setting the parameters on the parameter page, you may initiate a unidirectional or a bidirectional scan. Type *A* or *Alt A* to abort the scan. Type *H* or *Alt H* to save the image in the memory.

Scan Unidirectional will cause an image to be scanned according to the current scan parameters. Each scan line will be sampled as the microscope moves in the X direction, from left to right, before retracing.

The **Scan Bidirectional** command will cause an image to be scanned according to the current scan parameters. The first scan line will be sampled as the microscope moves in the X direction from left to right and the next scan line will be sampled as the microscope moves in the X direction from right to left. The bidirectional mode operates twice as fast as the unidirectional mode.

Halt Scan will halt the scanning process after the current scan is complete. The results of the scan and any data manipulation will then be displayed. (This command may be accelerated by typing *H* during a scan.)

Abort Scan will stop the scanning process immediately. Any image data sampled will be lost. (This command may be accelerated by typing *A* during a scan.)

3.4.1.3. The Window Menu

You may have multiple images on the screen. Use the Window menu to select either the Tile or Cascade option to display multiple images on the screen. This is a standard Microsoft Windows option.

Tile displays all the opened displays on the screen in a mosaic fashion.

Cascade stacks all the opened displays on top of each other.

Arrange Icons rearranges the "minimized" displayed images on the bottom of the screen. To "minimize" an image, click one on the minus sign in the left-hand corner of the image, then select the "Minimize" option. Refer to your Microsoft Windows operations manual for further information.

Close All closes all the currently open displays.

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3.4.1.4. The Display Menu

The Display menu contains commands that are used to modify only the view data for an image. The commands in this menu do not modify the raw data for the image in any way.

NOTE: When the software is first started, an opening screen will be displayed. You must first click Display on the menu bar, then select New Display.

New Display is used to open a new image window into which an image may be scanned or an existing image file may be loaded. All other commands in the Display menu are available only when a new image window has been opened or an existing image has been loaded from a disk.

Top View will cause the current image to be displayed in its default view which is as if looking at the sample from the microscope's point of view. The image is displayed from top with topographic or current data as a gray scale.

3-Dimensional View allows the current image to be viewed in perspective. A dialog box with parameters pertaining to the perspective view will be displayed. You can choose between a **Skeleton** view (low-resolution perspective) or a **Full** view (high-resolution perspective). Also the Z-axis multiplication factor may be entered as an integer from 1 to 20. Note that to use 3-Dimensional View, you should first normalize the data between its minimum and maximum (see Data View below).

Cross Section allows you to take a cross section of a top view image. With a top view display, a line may be stretched to indicate the boundary for the cross-sectional data.

Histogram the data for any image may be displayed as a histogram with this command. The resulting graph displays the number of actual samples at a particular data level.

The three **Palette** commands allow the system palette to be reset.

> **Gray Palette** will display the current images in a monochrome range of grays with differing intensities.
>
> **Blue Palette** will display the current images in a range of blue-green with differing intensities.
>
> **Pseudo Palette** will display the current images in a pseudo coloring scheme that ranges from green to blue to red.

Magnify a portion of a top view may also be displayed magnified with the Magnify command. In top view a rectangle may be stretched to enclose a portion of the total image. The selected portion of the image will be displayed in a magnified form to further increase the detail that can be viewed within the selected region. (Note: only one level of magnification is supported.)

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Contrast View Data provides the ability to change the contrast of the view data for an image in three different ways. A dialog box allows you to select the type of contrasting desired.

In **Minimum/Maximum** contrasting the view data for an image is scaled between the minimum and maximum data values in the image and then normalized to the range of colors in the selected palette. The minimum data value in the image is mapped to the darkest color in the palette and the maximum data value in the image is mapped to the brightest color in the palette.

In **Standard Deviation** contrasting the view data for the image is scaled between plus and minus two of the value obtained as the image data standard deviation and then normalized to the range of colors in the selected palette.

In **Manual** contrasting you can enter the minimum and maximum values to be used in the algorithm. The image view data may be scaled between any two values of image data before being normalized to the range of colors in the selected palette. The actual values that appear in the Minimum/Maximum edit fields are the values that correspond to the current minimum and maximum values of the view data. Therefore, after any of the three contrasting operations, the resulting values may be obtained by selecting Contrast View Data and viewing the values in the Minimum/Maximum edit fields.

3.4.1.5. The Filter Menu

The True-Image software package includes very extensive image-processing tools. The structure of the filtering allows user programs to interact directly with the program. Under the Filter menu you will find a set of image-smoothing routines.

The Filter menu contains commands that are used to modify the raw data of an image. Each of the commands in the Filter menu is available only when a new image has been scanned or an existing image has been loaded from a disk.

Pseudo Convolutions: Mean performs a convolution on the raw data of the image using the mean value of the data and an integer value for Strength between 1 and 500. A value of 1 has the minimal effect; a value of 500 has the maximal effect. Pseudo Convolutions Mean effectively performs a low pass filtration on the image.

Pseudo Convolutions: Median performs a convolution on the raw data of the image using the median value of the data within a user-specified window of samples and an integer value for Strength between 1 and 500. The median window can specify any number of samples by scantiness (X by Y) between two (1 by 2) and 81 (9 by 9). A value of 1 has the minimal effect; a value of 500 has the maximal effect. Basically this filter consists of a sliding window encompassing a matrix with an odd number of data points. The center data point of the matrix is replaced by the weighted median of the data points in the window. Pseudo Convolutions Median effectively performs a low pass filtration on the image.

Sharpen Edges: Gradient performs edge sharpening on the raw data of the image using a one-step Gradient mask on the data. Sharpen Edges Gradient effectively performs a high pass filtration on the image.

Sharpen Edges: Laplacian performs edge sharpening on the raw data of the image using a two-step Laplacian mask on the data. Sharpen Edges Laplacian effectively performs a high pass filtration on the image.

Unlike Pseudo Convolutions Mean and Median filtration, the Sharpen Edges Gradient and Laplacian filters have negative matrix elements. A typical mean filter would be:

1	2	1
1	4	1
1	2	1

while the Gradient and Laplacian Matrices used are:

-1	-1	1
-1	-2	1
1	1	1

0	-1	0
-1	4	-1
0	-1	0

Wiener Filter performs an optimal Wiener smoothing filter on the raw data of the image. The strength of the filtering algorithm is entered as the Wiener filter Strength, which can be any integer between 1 and 200. The Wiener filter used here removes the 1/frequency noise along the Y direction dominantly. This is particularly important in STM applications because the majority of uncorrelated noise is along the Y direction of the scan. The filter uses the Fourier transform of the image to find power spectra of image lines along the X direction for all the scan lines. These spectra are then averaged. The background on the average spectra is used as the optimal Wiener filter. The Wiener filter multiplies each pixel in the Fourier image by this filter. The effects of this filter can be very dramatic, so be careful in choosing the strength.

Morphological Filter performs a morphological filtering algorithm on the raw data of the image. You can choose between a filter that **Dilates** or **Erodes** the data. Each of the two filtering choices also has the option to use either a three-point or a five-point mask while filtering. The filtering is based on the degree that the masks fit the data. The points in the data that do not fit the mask are removed and replaced by it. The scheme of operation is graphically demonstrated in Figure 3.10. There are numerous advantages of this filter over Fourier transformation techniques. This filter does not distort the image during processing.[5]

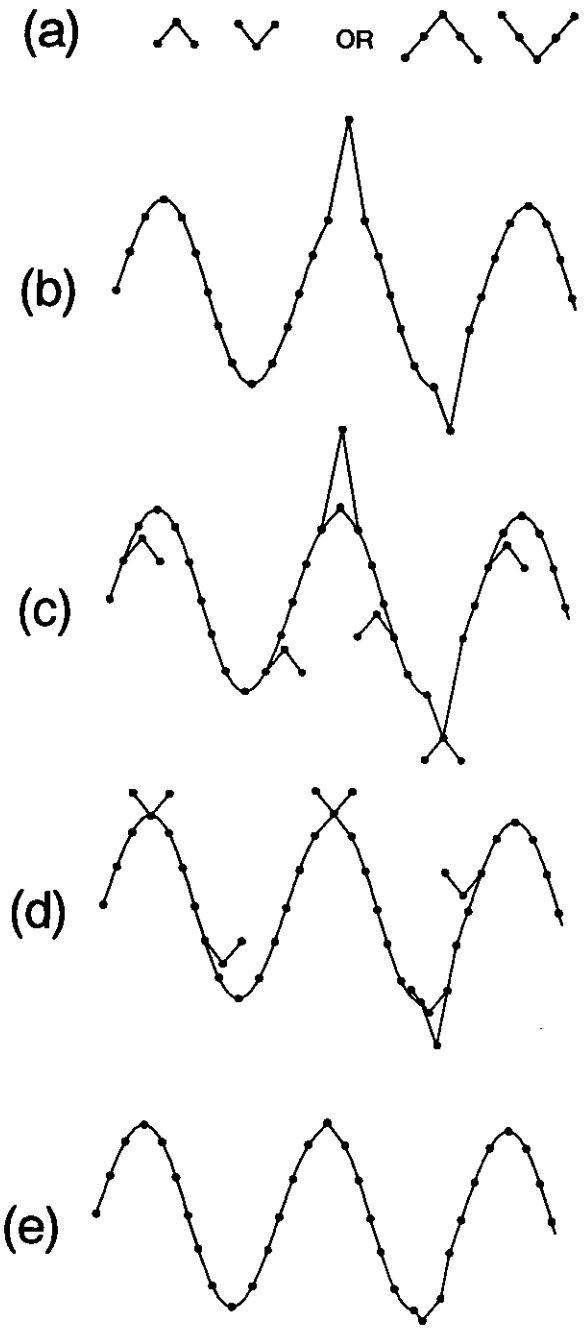

Figure 3.10. The masks are shown in [a] and the signal to be processed in [b]. The shape (slope) of the masks is determined by the strength factor. In [c] the erosion mask slides over the data points and removes all "positive" spikes. In [d] the dilation mask removes all the "negative" spikes. The resultant is shown in [e].

FFT (Fast Fourier Transform) transforms the raw data of the image into a representation of the spatial frequencies present in the image data. The resultant is the power image of the data. The lowest frequencies present in the data are at the center of the FFT image; the frequencies represented are higher as the image is traversed in both the X and Y directions out from the center. This image can be displayed in a linear or logarithmic gray scale. To see the details better, you should use logarithmic scale. Once an FFT has been executed, two other filtering operations become operational. These are the Low Pass and Band Stop Filter commands.

> **Low Pass Filter** allows you to stretch a rectangle symmetrically about the center of an FFT image. The bounds of this rectangle represent the cutoff points for the spatial frequency filtering in both the X and Y directions. Since the lowest frequencies are at the center of an FFT image, this filtering operation will retain (pass) all spatial frequencies within the bounding rectangle and will exclude (attenuate) all spatial frequencies outside it. The results of the Low Pass Filter can be seen by performing an Inverse FFT on the filtered spatial image.
>
> **Band Stop Filter** allows you to stretch a rectangle symmetrically about any point within an FFT image. The bounds of this rectangle represent the cutoff points for the spatial frequency filtering in both the X and Y directions. This filtering operation will exclude (attenuate) all spatial frequencies within the bounding rectangle and will retain (pass) all spatial frequencies outside it. The results of the Band Stop Filter can be seen by performing an Inverse FFT on the filtered spatial image.
>
> **Inverse FFT:** after a transform, the FFT command changes to Inverse FFT. Selecting Inverse FFT after a filtering operation will transform the image back to a representation of discrete data values which will then show the results of the applied filter.

Custom Filter allows you to program a filter algorithm — specifically, a DOS executable file that conforms to the custom filtering specifications — to be performed on the raw data of an image. This executable file must reside in the Filters directory on the system's hard drive. When selected, the Custom Filter command will list all available filter algorithms that may be performed. After a custom filter is selected, the raw data will be modified by the selected algorithm and subsequently displayed as a filtered image. During the execution of a custom filter no other commands are operational. The program must wait for the algorithm to be completed before commencing any other functions. An example program written in "C" is given in this directory.

Undo Last Filter becomes operational after any filtering operation has been performed. Selecting this command will cause the previous raw data of the image to be restored. *The current filtered data will be lost.*

NOTE: Only one filtering operation may be executed before the original raw data of the image is lost. After this point (i.e. two consecutive filtering operations), the previous filtered data will become the new raw data for subsequent functions.

3.4.1.6. The Analysis Menu

The Analysis menu contains commands that are used to modify the raw data of an image. Each of the commands in the Analysis menu is available only when a new image has been scanned or an existing image has been loaded from disk.

Tilt Removal provides a means of fitting a plane to the raw data of an image and subtracting the value of each point on the plane from each corresponding data point on the image. This operation has the effect of modifying the raw data of the image such that the effects of any physical tilt of the original sample material are removed from the scanned data. The resulting image data is thus normalized to be relative to a flat plane (similar to Plane Removal in the Collect/Configure submenu). The best-fit plane constant is represented and can be changed by the user.

Contrast Raw Data has the same capabilities as the corresponding Contrast View Data command in the Display menu, except that Contrast Raw Data directly modifies the raw (rather than the view) data of the image.

4. LABORATORY EXPERIMENTS

This chapter describes a number of experiments based on the STM with an emphasis on obtaining meaningful results within the time constraints of a normal undergraduate teaching lab. These experiments seek to convey various concepts of molecular and electronic structure in a very direct manner. There are seven laboratory experiments, involving five different classes of material, described in this section. The experiments include:

- imaging of gold surfaces
- graphite
- two-dimensional semiconductors (MoS_2)
- molecular ordering of liquid crystals (8CB, to be defined below) and alkanes ($C_{32}H_{66}$) on surfaces
- the phenomena of charge density waves (TaS_2; not provided with the prepared sample set)

The prepared sample set is used in applications of electron tunneling to metals, semimetals, and semiconductors. All of the materials and the experiments suggested below were chosen because they are relatively straightforward, require little sample preparation, and had high success rates. Our experience at the University of Rochester and Burleigh Instruments has been that approximately 80 percent of students obtain atomic resolution images of graphite, all students get good, though not atomic, images of gold, 80 percent of students get images of MoS_2 with reduced clarity relative to graphite, and 30 to 50 percent of students get reasonable images of liquid crystals or alkanes on graphite. These percentages give some guideline as to which experiments might be more suitable for a particular course.

In this regard, the distinctive properties of graphite, gold, and semiconductor surfaces can be studied and each material can be related to the electronic band structure of the crystal. In particular, the ordering of liquid crystals and alkanes gives a good example of the cooperative effects between molecules that can lead to self assembly and overcome entropic barriers; and the charge density wave exhibited by TaS_2 provides a very dramatic example of the effect of small bond distortions on electron density and band structure. The set of experiments using adsorbed liquid crystals and alkanes also demonstrate how electron tunneling can be modified by insulators.

Each sample is discussed in detail below as a self-contained experiment with example data and general instrument settings to get started. Figures from prior experiments are also provided for your reference. Together these experiments can be incorporated into a full laboratory course on materials based on the STM alone.

4.1. Gold Surfaces — An Example of a Metal

Based on their relative resistance to conducting electricity, solid state materials are broadly classified as metals, semiconductors, and insulators. Metals have very low resistance due to the availability of a continuous distribution of electronic levels to transport electrons, i.e. a conduction band. The resistance of a metal is determined primarily by lattice vibrations that impede the electrons' motion. Insulators have very large resistance and basically do not conduct electricity; this high resistance results from the large energy gap between valence molecular orbitals and the conduction band of unoccupied states. The valence orbitals of insulators are completely filled with electrons. Since no two electrons can occupy the same orbital (i.e. electrons are fermions) there can be no *net* motion of electrons in an electric field. The resistivity of semiconductors falls between that of metals and insulators. For semiconductors, conduction occurs through the thermal promotion of electrons, which creates unoccupied electronic levels that enable net electron motion. These three classifications are shown schematically in Figure 4.1.

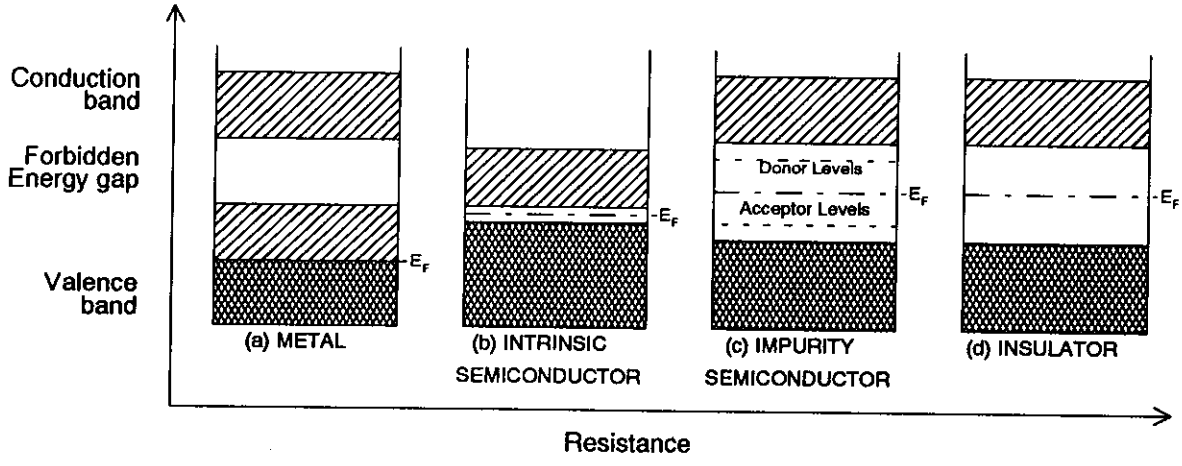

Figure 4.1. Schematic band models of solids classified according to electronic properties.

The element gold (Au) exists at room temperature as a metal that is extremely inert. Most metals, such as iron, aluminum, and alkali metals, react rapidly with oxygen in the air to form an insulating metal oxide on their surfaces. This oxide layer makes these metals unsuitable for STM studies under ambient conditions. Gold, on the other hand, does not tarnish (oxidize) and retains its metallic luster for years, which is one of the main reasons for its prominent role in history in coinage, jewelry, and art. Gold, silver, and platinum are referred to as the noble metals because of their relatively low reactivity with oxygen. Of the noble metals, gold is the most inert.

The general definition of a metal is that it is shiny, ductile, and highly conductive. All of these material properties can be understood from the atomic structure and the nature of the atomic orbitals defining the bonding between atoms. The metal shine comes from the extremely high index of refraction at virtually all wavelengths, from the visible to the infrared. The conduction band electrons can absorb light and be promoted to higher-lying unoccupied levels at virtually

any wavelength. These optical transitions, and the enormous concentration of electrons that can interact with light, impart the high index of refraction that makes metals highly reflective. The metal's gold color originates from variations in absorption in the blue and red regions of the visible spectrum (it is not perfectly reflective). The ductile property of gold and other metals arises from the strong overlap of the valence atomic orbitals, which leads to the formation of electronic bands. The curvature of their interatomic potential, which defines the interatomic forces that hold the lattice together, is smaller than that of an insulator. Basically, as a force is applied to increase the interatomic spacing, there is still enough orbital overlap to maintain a degree of bonding between the lattice plains. However, the ductile nature of metals actually comes from dislocations in the lattice. No crystal is perfect; there will always be locations where the lattice has a break in its symmetry that essentially increases the structural void space of the lattice. Any stress applied to a metal deforms the dislocations of the crystal as the atoms maintaining a net bonding interaction move past one another. The ductile nature of metals is much like rolling out air pockets in dough to stretch the dough. It is this property that enables metals to be pulled into wires. In the absence of defects, most metals would have a tensile strength 10^2 to 10^4 higher than naturally occurring crystals.

High conductivity is the primary characteristic of metals. An atomic level understanding of this phenomena requires a discussion of how atoms bond together to form solids. Whether or not a material is conductive depends on the relative energy positions of the interacting valence atomic orbitals that bond the lattice of atoms together. To understand this point, consider what happens when two atoms are brought together spatially, as shown in Figure 4.2. If the energy of the two orbitals is similar, the two levels will combine to form a lower energy (bonding) molecular orbital and a higher energy (antibonding) molecular orbital. This coupling between two levels is a classic problem in quantum mechanics. In the bonding molecular orbital, a build-up of electron density occurs between the two atoms (the atomic orbitals add constructively). The two-atom system enters a condition of quantum resonance in which the electrons are shared by both atoms. In this manner, the electrons experience the coulombic attractive forces of both nuclei and the total energy decreases to form a lower energy bound state referred to as a molecular bond.

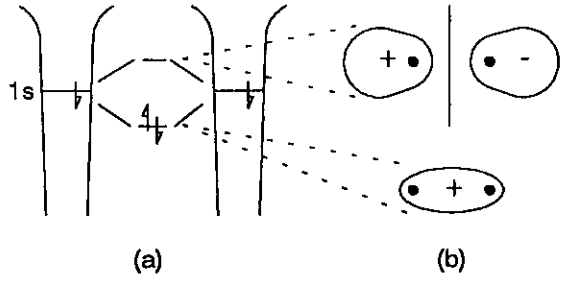

Figure 4.2. The creation of two new molecular orbitals from the spatial overlap of two 1s hydrogenic orbitals.

Now, consider the case in which, instead of two atoms interacting, n atoms are interacting, where n is a very large number. The atoms can again be defined to have a minimum energy point at some characteristic interatomic separation or bond length. Each atomic orbital contributes one electronic level to a highly congested overlap of electronic levels that merge to form a *band,* as shown in Figure 4.3.

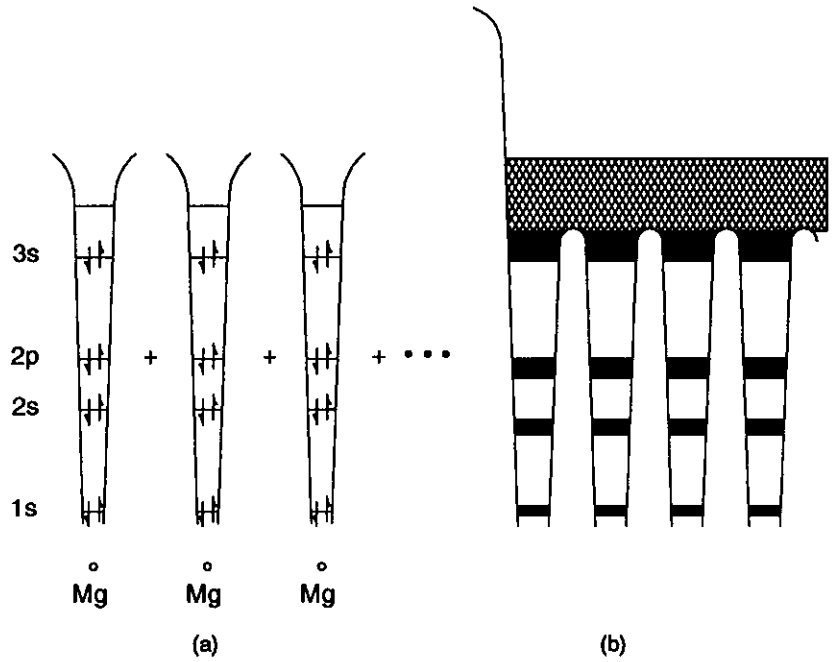

Figure 4.3. Energy levels in magnesium: [a] isolated atoms; [b] section of crystal. The sharp energy levels in the atoms have merged to form bands in the crystal. Double arrows represent filled levels.

One other important feature of the solid state that merits discussion before proceeding is the nature of the electronic wavefunctions. As opposed to atoms in which the electron experiences a coulombic attractive potential from a single nucleus, electrons in the solid state experience a periodic potential of period **a**, the dimension of the crystal unit cell, $U(r + a) = U(r)$, where the vector $r = x + y + z$. The wavefunctions must reflect this symmetry. Refer to Schrödinger's equation in Chapter 2.5. The wavefunctions would have the form:

$$\psi(r + a) = e^{ik \cdot a}\psi(r), \quad k = 2\pi/\lambda \tag{4.1}$$

where k is referred to as the momentum vector and λ is the effective wavelength of the electron. The electrons would have a periodic wavefunction, governed by the periodic potential of the crystal. Wavefunctions expressed in this form are known as Bloch wavefunctions and can be understood by considering a simple particle in a box problem of length L where L corresponds to the length of the crystal. This is a fairly good analogy. There are no barriers between the atoms as the atoms are covalently bonded and the electron behaves as if it were a free electron within the confines of the crystal dimensions. The quantum mechanical solution

to this problem gives $k = n\pi/L$ where n is an integer. Larger n integers have shorter effective wavelength and thus higher energy, i.e.,

$$E = \frac{p^2}{2m} \tag{4.2}$$

where p is the momentum and m is the electron mass. From the de Broglie relation equating the correspondence between particle and wave properties: $p=h/\lambda$ or alternately $p = \hbar k$ the energy relation can be rewritten in terms of the momentum vector as:

$$E = \frac{\hbar^2 k^2}{2m} \tag{4.3}$$

This is the usual manner for depicting the different energy levels in a solid. An isotropic solid would give a parabolic dependence of energy on momentum, as shown in Figure 4.4[a]. There are discrete steps in the energy, just as in a molecule's energy diagram. However, the steps are so small (10^{-20} times smaller than kT at room temperature for a 1 cm^3 crystal) that the conduction or valence band appears as a continuous function or a continuum (of states).

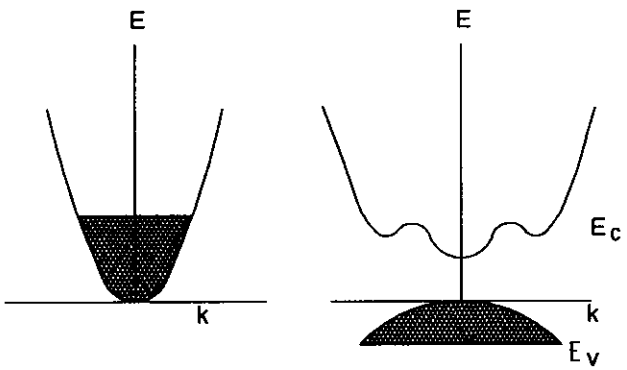

Figure 4.4. In [a] the free-electron-like behavior of electrons in metals compared to [b] the more complex behavior of electrons in semiconductors.

The nature of electrons is such that they would occupy lowest possible energy states. Finite increases of temperature excites these electrons to higher energy states. Since the electrons defined by k states are fermions, the probability that a particular level is occupied is given by Fermi-Dirac statistics:

$$P(E) = \frac{1}{1 + e^{(E-E_F)/kT}} \tag{4.4}$$

where E_F is called the Fermi energy, the energy at which there is a 50 percent probability of finding a state occupied by an electron. This distribution is shown in Figure 4.5. For a metal, E_F and the position of the highest occupied molecular orbital coincide.

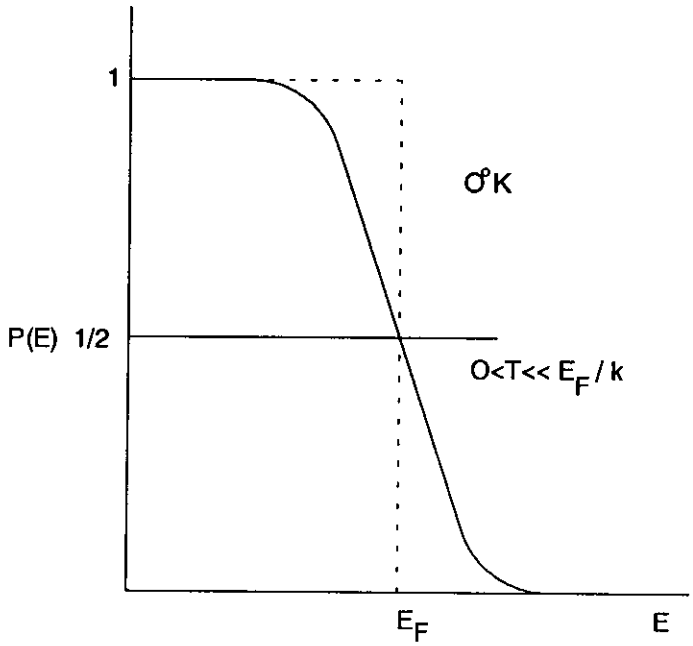

Figure 4.5. The Fermi-Dirac distribution function for energies of electrons in metal.

The property of electrical conductivity requires that the electron be free to move spatially throughout the crystal in response to an applied field. This is only possible when there are close-lying, unoccupied orbitals that enable the electron to move spatially and pick up translational energy from the applied field. In this regard, the bands from the core orbitals are comprised of closed-shell atomic orbitals and are completely filled states. The electrons from these states do not contribute to the conduction process. Only the bands formed from the valence orbitals can meet this condition. It is the energy level of these bands that defines the Fermi energy, as explained above.

The high electrical conductivity for a metal such as sodium (Na), whose electronic configuration is $1s^2 2s^2 2p^6 3s^1$, is easy to understand. The valence band is only half occupied, leaving accessible higher-lying levels for electron motion. The case of magnesium (Mg) is more difficult to understand. Because its atomic electronic configuration is $1s^2 2s^2 2p^6 3s^2$ one would expect the valence band to be filled. However, the higher-lying conduction band, formed primarily by the 3p orbitals, overlaps energetically with the valence band to form a continuous band of states capable of supporting conduction. This is shown schematically in Figure 4.3.

It should be noted that the above picture is somewhat oversimplified. In the case of diamond, for example, the valence band is formed by a combination of both carbon 2s and 2p orbitals and there is a large gap between the valence and conduction bands for the interatomic separation characteristic of the diamond structure (which we discuss further, below). This large band gap makes diamond a very good insulator, even though it has open shell 2p levels for

the constituent atoms. This latter point is made to illustrate that the exact nature of the solid state electronic levels depends on the details of the interatomic spacing (lattice structure) and extent of wavefunction overlap. Nevertheless, this discussion points out that a metal can be formed through either partially filled valence band states or the overlap of the valence band with higher-lying molecular orbitals referred to as conduction bands.

In STM experiments you are measuring the spatial variations in the electron density of the highest occupied levels in the band. These are the states near the Fermi level of the metal. The electrons near the Fermi level have the lowest barrier and can tunnel to unoccupied states in the tip. Gold has a valence electronic configuration $6s^1 5d^{10}$. The conduction band of this metal is formed primarily from these 6s and 5d atomic orbitals, as shown schematically in Figure 4.6. The STM images the spatial modulation in electron density associated with these band states.

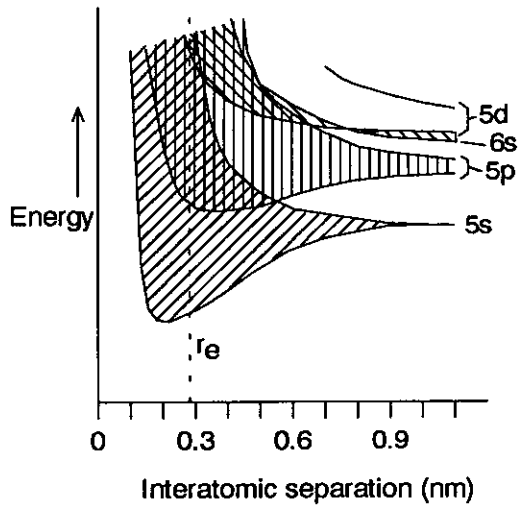

Figure 4.6. Approximate quantum mechanical calculation of the formation of energy bands as atoms are brought together into a crystal.[1]

The minimum energy crystal structure for gold is a simple face-centered cubic structure with an average interatomic separation of 2.9 Å. There are several different surfaces that can be studied with the STM. The different surface orientations relative to the lattice structure are defined by the orientation of a vector perpendicular to the plane using Miller indices for the nomenclature. The different crystal structures and surface orientations are shown in Figures 4.7 and 4.8. Au(100) was the first surface studied by the STM[2] and Au(111) was the first metal surface to be observed with atomic resolution.[3] Since then, other orientations have been studied and a variety of phenomena have been observed. Atomic resolution has been obtained in air[3] and even in electrochemical environments.[4]

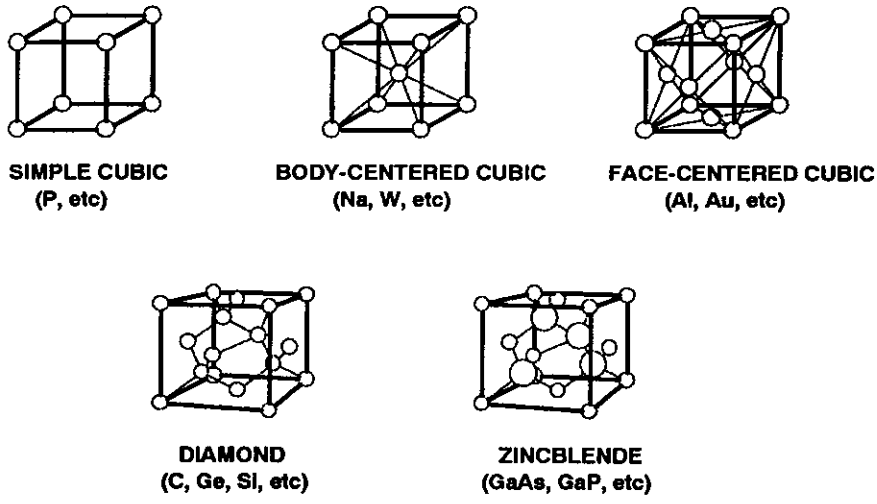

Figure 4.7. Some important unit cells (direct lattices) and their representative elements or compounds.

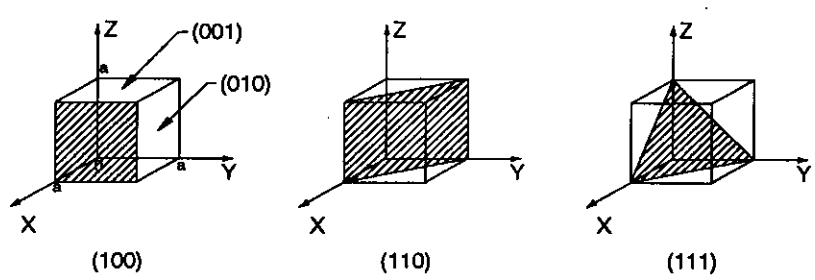

Figure 4.8. Miller indices of some important planes in a cubic crystal.

4.1.1. Experiment

To attain atomic resolution for gold, the STM signal has to be particularly low in noise. The very nature of the metal means that the electrons will be strongly delocalized between the atoms and there will only be small variations in the electron density with atomic position. The periodic modulations are typically on the order of 0.1 Å, so one should not expect to image gold atoms in normal room conditions. The purpose of this lab is to introduce the concepts of tunneling and the extremely delocalized nature of electrons defined by the metallic state.

4.1.1.1. Holographic Gold Grating: STM Magnification

The first sample to examine is the gold-coated holographic grating. This sample is a single-period hologram with a sinusoidal spacing. A hologram of the type seen on credit cards is composed of many such sinusoidal patterns of varying periods and orientations, which recreate the original image when viewing the diffracted or scattered light off the surface. This sample illustrates the piezoelectric tube scanner range and reinforces the level of magnification possible with the STM. The reference images are: grating1.img, grating2.img grating3.img, grating4.img, and grating5.img. These are images of a grating with spacing of 0.2 μm (2000 Å). The grating you are provided with has a spacing of 2400 lines/mm or about 0.4 μm (4000 Å) line spacing. It is easier to obtain high quality images with this grating.

Procedure:

Head Preparation

1. Prepare either a PtIr or W tip and mount the tip.
2. Select the gold grating from the sample set. To determine the direction of the grating lines. Look at the surface of the sample along one of its edges. Light would diffract by the grating along a direction perpendicular to the grating lines. Mount the sample so that the grating lines are parallel to the sample carriage handle.
3. Turn the Sample Position dial until the sample range indicator is close to the middle of the range or the sample-tip spacing is less than 0.5 mm. Be careful not to damage the tip and the sample. You may have to use the Coarse Retract button to reposition the tip. By moving the tip with the controller make sure there is enough range for the tip to reach the sample during approach.

Software Preparation

4. Load grating3.img (File/Load). This image is shown in Figure 4.9.

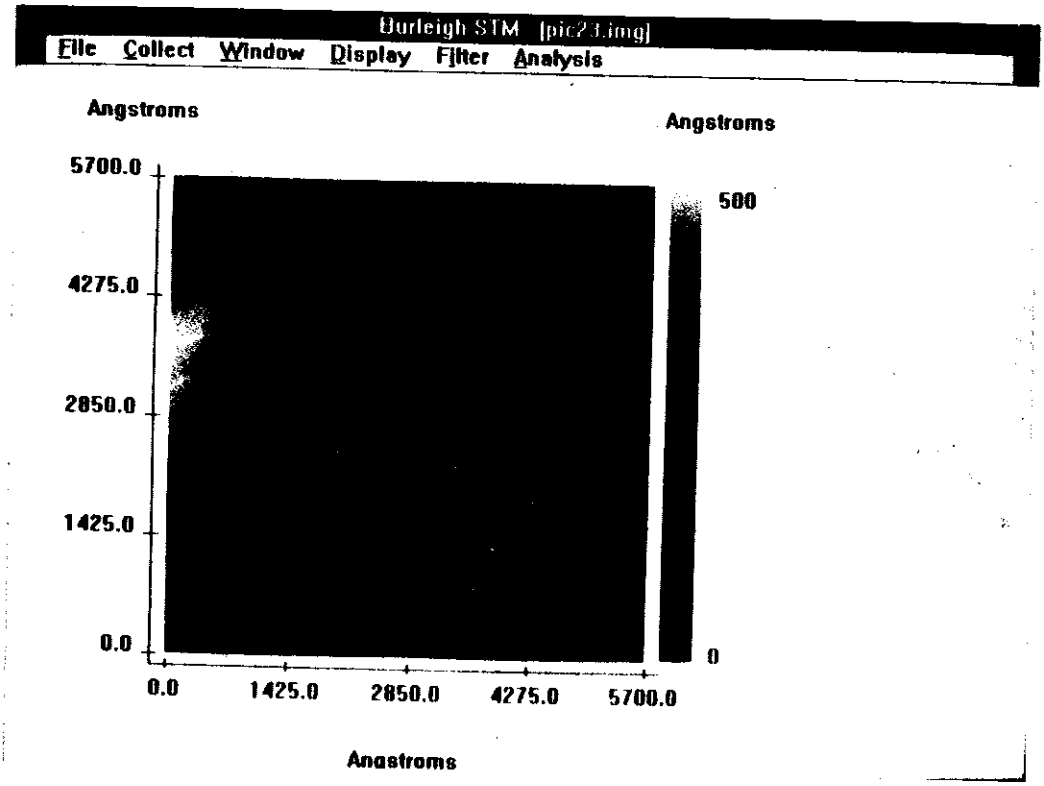

Figure 4.9. An ISTM image of a 5700 Å x 5700 Å area of gold grating obtained in constant current mode.

5. Set the Scan Delay (Collect/Configure) to 0.2 mS/Sample.

6. In this scan you are monitoring height variations, so set the Data Type to Topographic (Collect/Configure).

Electronics Preparation

7. Set the Bias Voltage to about 1 volt.

8. Set the Reference Current to 8 nA.

9. Set the Servo Loop Response for constant current mode of operation. Set the Gain close to maximum. Set the Filter close to maximum. Set the Time Constant to minimum.

10. Set the magnification to X1.

11. Set the X and Y offset slides at their middle range.

12. Press the Tunneling Current button to monitor tunneling current (it should read about zero).

Tunneling

13. Press the Coarse Retract button momentarily to reset the motor controls.

14. Press the Auto Approach (Tunneling) button for approach and wait.

15. Monitor the tunneling current until it reaches about 8 nA (equal to the reference current). If the tunneling current oscillates, reduce the Gain and Filter or increase the Time Constant to stop the oscillation. Oscillation manifests itself as periodic variations of the PZT voltage (J2 BNC on the front panel of the controller) which you may monitor on an oscilloscope.

16. Once tunneling is achieved, start a unidirectional scan (Collect/Scan Unidirectional).

17. Collect images and save one at this range.

NOTE: Typing (*ALT C* then *A*) or just *A* will abort the scan without saving the image. Typing (*ALT C* then *H*) or just *H* will stop at the end of the image and save it in the computer memory.

18. Change the scanner range by turning the magnification dial. Set the software size correctly in the menu by setting the zoom factor (Collect/Configure). Collect an image at each setting.

The large scan range should reveal a sinusoidal pattern on the surface with a period of 4000 Å and height variations of 400 - 500 Å. Figure 4.10 shows large scans of a grating with a periodic spacing of 2000 Å. As you zoom in, the details of the gold crystallization process on the hologram should become apparent. The evaporated gold tends to rapidly diffuse to form random crystallites with grain sizes of approximately 60 - 100 Å. You can prepare a montage of all the collected scans (from the Windows display mode) to visualize an overall factor of magnification of ~ 10^8 at the highest magnification (such as in the images grating1.img through grating5.img).

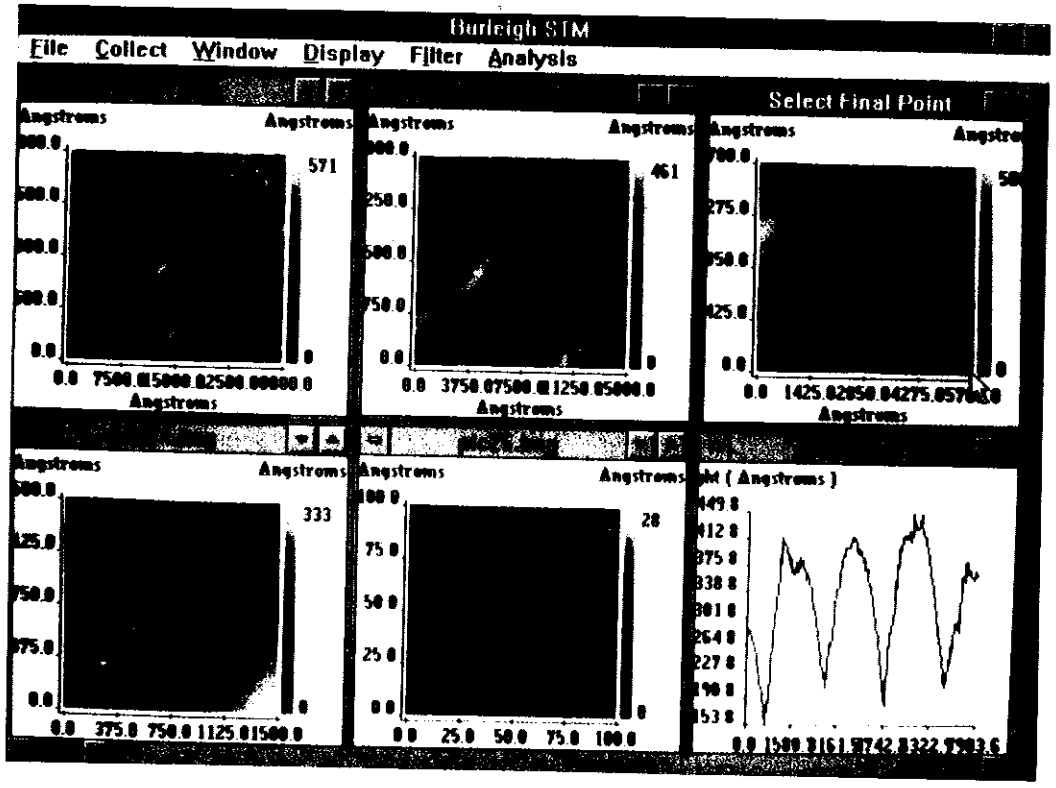

Figure 4.10. STM images of gold grating at different magnification settings. It should be possible to take images of atomic steps in extremely quiet tunneling conditions.

You may use the grating spacing to calibrate the range of the PZT motion. The grating has 2400 lines/mm (Your sample kit may indicate a different number of lines/mm, use that number for calibration). This corresponds to a line spacing of about 4167 Å, the prependicular distance between center of the lines. To calibrate the PZT acquire images of the grating with the following software settings (Collect/Configure): Set Scan Range in X equal to Max Scanner Range in X, and set Scan Range in Y equal to Max Scanner Range in Y. Set the Zoom Multiplier to X2. Take an image with the magnification set to X2 on the control electronics.

From the image calculate the scale in X and Y directions. The sample may be oriented to give only vertical grating lines on the display. You may assume the scan to be square, and just count the number of grating lines you see on the screen. Then, multiply this number by 4167. Set the Max Scanner in X and Y equal to this number multiplied by 2. The Max Z Range is typical 40% of the Max Range in X.

Only for extremely quiet tunneling conditions will it be possible to discern atomic features. The best chance of obtaining atomic features is from the constant height mode at high scan speeds. Set the tunneling current to 4 - 5 nA and use a bias voltage of ~100 mV. You should compare this image to the constant current scans. The constant current mode cannot operate at as high a scan speed because it is limited by the response-time of the feedback loop; it is also more prone to acoustic noise than the constant height mode. The tip is extremely important in this regard, so you may want to try different tips. However, you should not expect to attain atomic resolution in this experiment. The main point here is to get an approximate determination of the maximum periodic variation in the electron density across the surface.

Load a representative image (File/Load). Use the cursor and draw a line through the data to visualize the fluctuations in tunneling current (Display/Cross Section). The current should be fairly constant across the surface, with variations of less than 0.1 Å in the tip position, to maintain constant current. The noise on your data may exceed this value, so you may want to filter the data to reduce the noise level in the image. The main point is that the low degree of current variation across the surface illustrates the highly delocalized nature of electrons in metals. This study should be contrasted to that of graphite in the next section.

NOTE: Be sure to store the images you want to keep on a diskette as backup. You may want to analyze or filter the images later and put them into perspective view or other formats. You should also make a hard copy for your reports. These can be made by either photographing the computer monitor or using a video printer, if available.

4.1.1.2. Questions

1. Sometime during the use of the STM, the tip may have "crashed." This is observable as a sudden large change in the current. This occurs when there is a change in the surface topology to which the feedback loop does not respond quickly enough and the tip touches the surface. Calculate the effective resistance of the tunneling gap for the conditions used in your experiment and compare that to the expected resistance if the tip was in direct ohmic contact with the surface. The resistivity of gold is 10^{-8} Ω/cm. This comparison should graphically illustrate the tunneling effect you are observing.

2. As the temperature of metals is raised, the resistance to current flow increases. Discuss the mechanism of resistance in metals and compare this mechanism to electron tunneling. How would the temperature-dependence of the two mechanisms differ?

3. Because these experiments were conducted in air, adsorbed water, solvents, and gases are undoubtedly on the surface. How do these molecules affect the tunneling process and how might the tip perturb their distribution? Contaminants on the tip are also likely problems. Explain how this would affect the noise on your STM experiment.

4. Consider the problem of the electron source in these experiments. If the tip is not scanned but left stationary over the surface, at a fixed distance that corresponds to a tunneling current of 1 nA, calculate the number of electrons/second that flow through the atoms that participate in the tunneling process between the gold and tip surfaces.

5. Calculate the expected variation in tunneling current for workfunction of 5 eV and sample height variations of 0.1 Å.