The Scanning Tunneling Microscope at UC Davis, California

Cicely Potter

Utah Valley University

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Abstract

As computer chips and components become smaller, their surface are to volume ratio becomes larger. This means that most of the chip is surface, and surface physics becomes very relevant. Scanning Tunneling Microscopes have a resolution on the order of angstroms and are very useful for observing the structure of atoms on surfaces. In particular, observing how metals form on semiconductors can yield insight to building better devices. The STM is a very intricate and sensitive piece of equipment, requiring lots of maintenance and repair; the experiment consisted primarily of repairing the components. The machine was functional by the end of the experiment, and an image of a gold film on a mica sheet was recorded. Further experiments are planned to take more images of gold on germanium.

Introduction

In the interior of any material or object, the atoms that compose it interact with each other. There are models and theories that describe these interactions, and the structures that the atoms form in solid objects. Atoms on the surfaces of these objects however, are not as easily described. These atoms not only interact with the atoms in the interior, but atoms of the surrounding material. Surface physics is a subfield of condensed matter physics, and is the study of the behavior of atoms on the surfaces of objects. This applies to chemical reactions, semiconductor manufacturing, magnetic disk drives, and computer chips. As computer chips become smaller, surface physics becomes more relevant. The growth of metals on semiconductors is related to the formation of metal contacts for devices, and understanding the physical mechanisms relating to these phenomena can help to improve materials and make better devices. This is the motivation behind the experiment; to observe a gold film on a germanium surface using a Scanning Tunneling Microscope.

Scanning Tunneling Microscopes, or ‘STM’s, record the topography of surfaces by bringing a very fine metal tip extremely close to a surface. Because of the wave nature of particles, electrons have a certain probability of traveling through an area of higher potential energy where, classically, they’d be forbidden to be; this is called quantum tunneling. In this way, electrons will tunnel through the space between the sample and the tip, creating a current. With the quantum current recorded versus the 2D (X and Y) position of the tip, we can construct a 3D image of the surface.

Equipment and Techniques

The apparatus that was used was a set-up of 3 connected chambers containing the STM, the XPS (X-ray Photospectroscopy), and the LEEM (Low Energy Electron Microscope) respectively. (Figure 1) These chambers were made to be pumped out, leaving vacuum inside. The connections in the chambers allow the sample to be transferred from one chamber to another; this means that the sample can be studied with all 3 instruments without being taken out of vacuum.

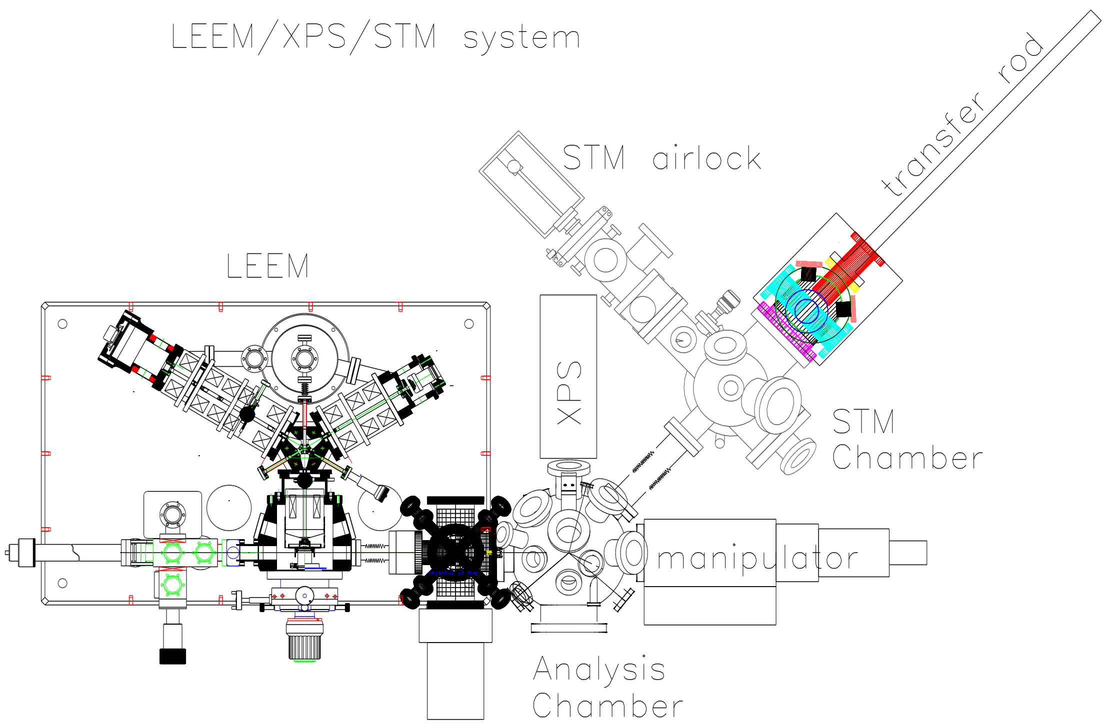

Figure 1. Diagram of the apparatus used as seen from above.

The STM was the primary instrument for the experiment; the XPS was not used, and the LEEM had only been used for previous experiments. The LEEM allows for deposition of one material onto another during observation, and can record video. While the STM can do neither of these things, it has its own advantages. The STM has a spatial resolution on the order of angstroms, thus it is capable of detecting and recording the positions of individual atoms. (Table 1)

	STM	LEEM
Spatial resolution	2 Å lateral, 0.01 Å vertical	10 nm lateral, interference of atomic steps
Image Size	100 Å-1 µm	10 µm field of view
Time resolution	20 min per image	Video rate (8 fps)
Real time deposition	Cannot deposit	Can measure island growth during deposition
Features measured	Atoms, steps, defects	Islands, steps, diffraction patterns

Table 1. Properties of the STM compared with properties of the LEEM

The STM is removable from its chamber and is secured in place with 24 bolts. The STM itself is composed of a copper block suspended with springs, a sample holder, and a scanner. The scanner is the component that moves, allowing the STM to map out the surface of the sample. It is placed on top of the copper block, where its 8 electrical pins are inserted into their ports; these are wired through the bottom of the STM to allow access from the outside. This is where the cables from the electronics box connect to the STM. The sample holder is secured in the copper block below the scanner. (Figures 2 and 3)

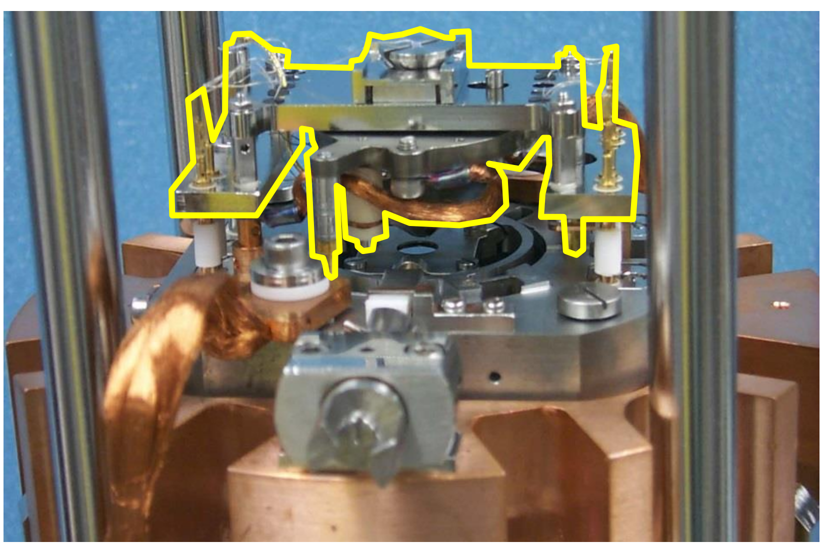

Figure 2. The scanner (outlined in yellow) in its place on the copper block. The copper block is roughly 10 inches in diameter.

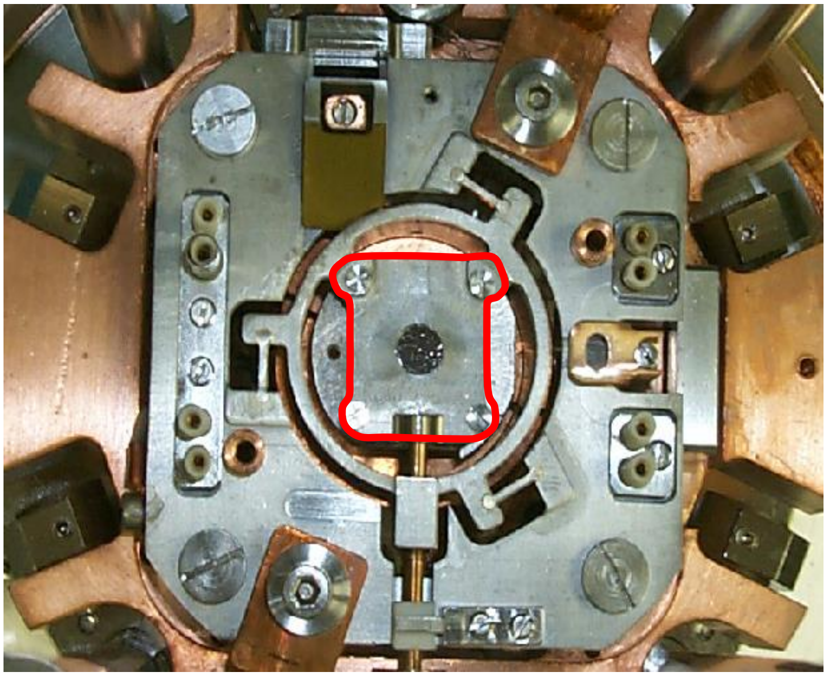

Figure 3. The sample holder (outlined in red) secured in place in the copper block. The copper block is roughly 10 inches in diameter.

Both the sample holder and scanner are placed in the STM using a claw. The claw is on the end of a rod that extends through the ceiling of the chamber and out. On the other end of the rod is a lever that contracts the claw arms when pressed. Directly connected to the STM chamber is the load lock, which is used to load items into the chamber. To load items into the STM, they are placed on a tray, which is in turn placed on a copper rail, which can be transferred down the length of the chamber via a knob on the outside of the load lock. Once the rail reaches the STM chamber, the items are picked up from the tray using the claw.

This specific device has a couple of systems meant to dampen mechanical noise. The first of these is that the copper block is suspended with springs. There are magnets mounted below the copper block that induce eddy currents in it when it moves. When currents are induced by a magnetic field, the current creates a magnetic field in opposition to the original one; this is called Lenz's Law. The opposing magnetic field serves to dampen the movement of the copper block. The second of the mechanisms is that the chamber itself is mounted on laser table legs, or laminar flow isolators. Thus, when the floor vibrates, the legs isolate the chamber from the movement, keeping it as still as possible.

Vacuum is important for the STM because in air, the sample would be covered with deposited matter in less than a second. Higher vacuum means less deposition, i.e. a longer time to conduct an experiment before the sample becomes covered. An ion gauge is used to measure the vacuum in the chamber. Ion gauges work by ionizing the gas in the chamber, attracting them via a negative voltage, and then measuring the ion concentration, thus indirectly measuring the gas concentration. In order to bring the chambers down to vacuum, there are 4 types of vacuum pumps used. The mechanical pumps are the most basic, and are used first; the mechanical pumps must get the chamber down to 60 millitorr before any other pumps can be used. Next, the Turbo pumps are activated; these are the pumps that, when working properly, can achieve Ultra High Vacuum (around 10^{-9} torr). If the vacuum doesn't reach the required level with the turbo pump, the 3rd pump can be used: the ion pump. The ion pump works by ionizing the gas and attracting it with a high voltage. When the ion pump is turned on, the ion gauge must be turned off, otherwise it will be overloaded with ions; it is very sensitive. The Titanium Sublimation pump, or TSP, is the final type of pump used to create a vacuum. The pump releases titanium atoms into the chamber, coating the walls with a thin film. Since titanium is very reactive, the gasses still in the chamber will form a solid product with it when they collide. This reduces the pressure by reducing the amount of gas in the chamber. Baking the chamber is another method that helps to create a vacuum. The chamber is wrapped in heating tape, and covered with foil to hold the heat in. The heat tape is plugged in, and the chamber is allowed to bake (while being pumped) for several hours; often over 24 hours. Heating the air makes it easier to pump out, and turns liquid water into vapor, allowing it to be pumped out also.

The sample holder is a small component that holds the sample, and is transferable between chambers. It has 4 feet, 2 of which are used as a thermocouple to determine the temperature of the sample. The sample holder also has a tungsten filament inside that, when heated, emits electrons. (Figure 4) An electrical bias is put on the sample, attracting the electrons to it from the filament. This process is what makes the quantum current flow possible, and will be revisited.

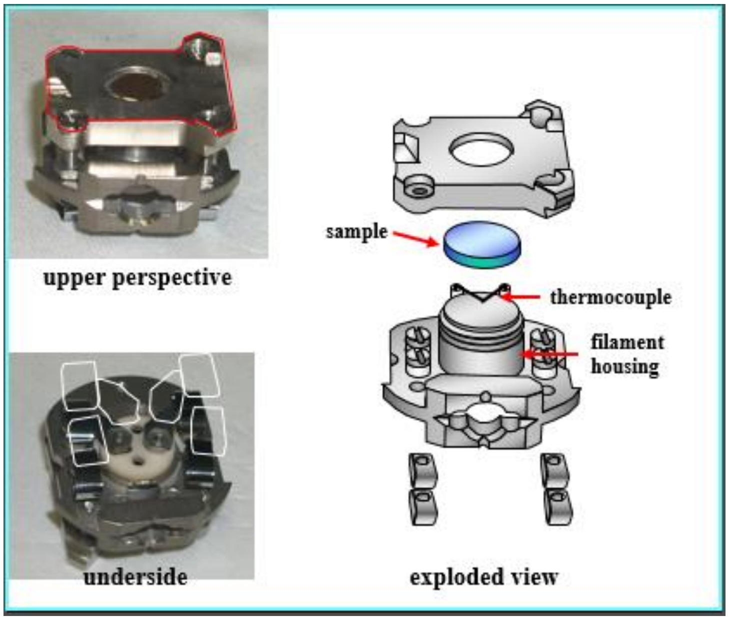

Figure 4. Images of a sample holder from the top (top left) and bottom (bottom left). Included is an expanded diagram of the sample holder (right)

At the start of the experiment, there were two working sample holders. Another one was constructed; with more sample holders, more samples can be examined without going through the process of replacing the sample in a single sample holder, which can break the sample if not done carefully. Building a sample holder was a challenge, as the pieces had to be handled not only with gloves on, but also with tweezers, because of their size. First, the tungsten filament was placed on a small rod that was spot welded to a base. This base was secured in the base of the sample holder itself, and a protective cover called the filament housing placed over it and the tungsten filament; this is the step that had to be repeated a few times, as the pieces were very small and delicate, often falling apart while being assembled. Next, the feet are cut and screwed on. The smaller 2 of the 4 feet were made of tungsten and the larger were made of 2 different materials to function as a thermocouple. Depicted in the diagram on the right in figure 4 is another thermocouple above the filament housing. This component was not included in the assembly of this sample holder. This extra thermocouple can be helpful for more precisely determining the temperature of the sample, but is not necessary to the design. Once all of this is in place, the top of the sample holder can be screwed on; the top is removed frequently, as it must be removed to place a sample in the sample holder. (Figure 5)

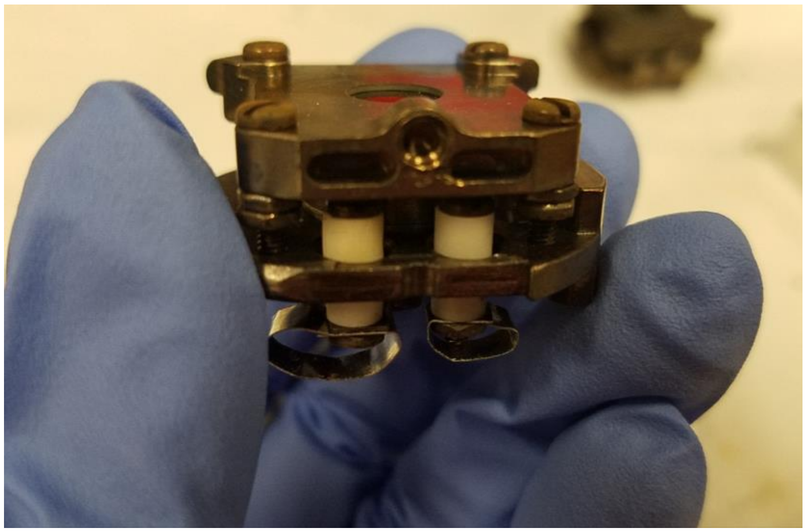

Figure 5. The 3rd sample holder after being assembled

The scanner is made of 2 main parts; a table-like piece with 4 legs and 8 pins that plug into ports on the copper block, and a tripod piece that is attached to the table piece, but is allowed a range of movement. The tripod is the component that holds the tip, and the part that scans across the surface of the sample to create a map. It does this via a mechanism for coarse movement, and another for fine movement. The coarse movement is achieved via the tripod legs and a wedge that can be moved forward and backward to lift or lower one of the legs; it has a range of 1.63 mm of vertical movement. (Figure 6) A piezoelectric tube split into four quadrants is used for fine movement, and has a range of 700 nm. Piezoelectric materials are those that stretch or contract when electricity is run through them. (Figure 7)

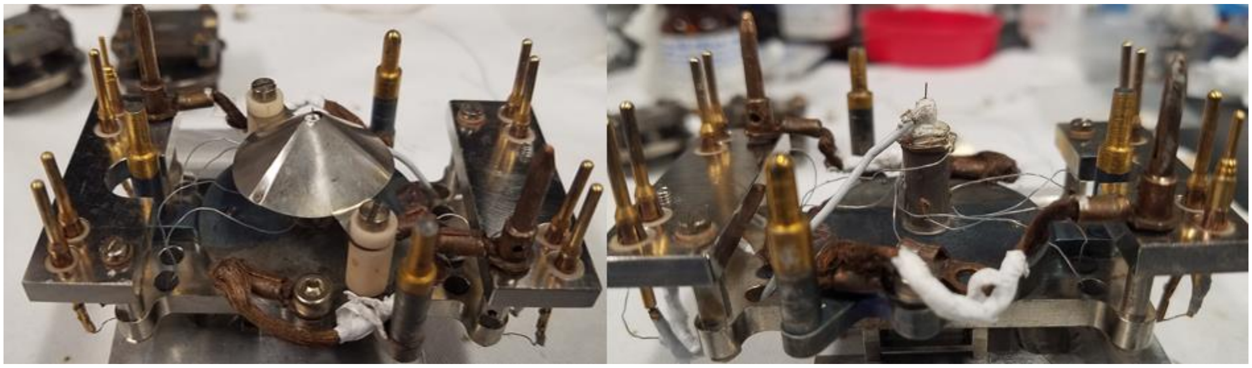

Figure 6. Bottom view of the scanner both with (left) and without (right) a protective cover

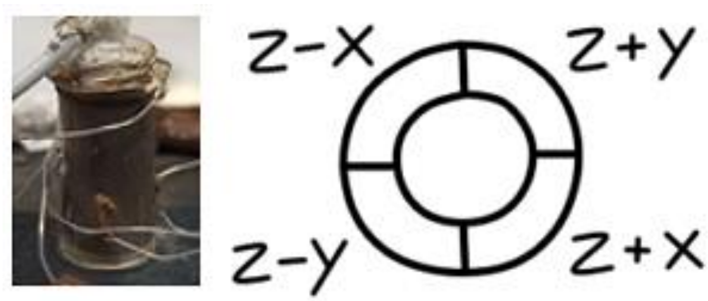

Figure 7. On the left, is a close-up of the piezoelectric tube. On the right, a diagram of the quadrants of the piezo, as seen from above. Each quadrant is electrically isolated from the others.

The tip itself is made of tungsten wire, and chemically etched to be the most efficient shape. The ideal tip will have a cusp shape with a single atom for a point. In reality, this won't happen, but a good tip will still have a well-defined cusp shape to give a single tunneling point; there is no way to definitively know if the tip is ideal. Tip etching methods are designed to create good tips as consistently as possible. (Figure 8)

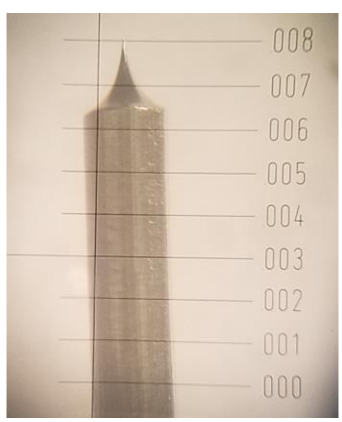

Figure 8. An image taken through a microscope of a chemically etched tungsten tip. The markers on the right indicate millimeters

To chemically etch a tip, a short length of tungsten wire is immersed in a 3 molar KOH solution, and a current is run through. The wire is etched at the surface of the solution until that point of the wire is thin enough that the immersed part stretches the remaining wire into more of a cusp shape, and then falls off. The factors that affect the quality of the tip are the concentration of the solution, the length of wire immersed, proper cleaning before and after, and annealing the wire before etching. (Figure 9)

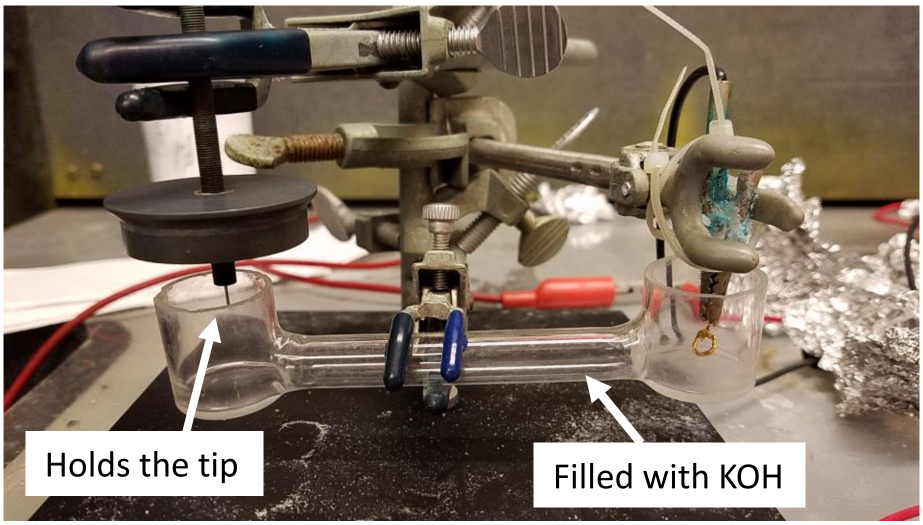

Figure 9. The equipment used to chemically etch the STM tips

When the tip is brought close to the surface of the sample, a current is created via quantum tunneling. This current is made possible by the tungsten filament located in the sample holder. The filament emits electrons when heated, and these electrons are attracted to the sample, replenishing the electrons that leave the sample via quantum tunneling. By converting the current to a voltage and recording it, the distance between the tip and the specific point on the surface can be found using the following equation:

$$I = \Delta V e^{-A\sqrt{\emptyset}z} \approx 10^{-z} \qquad \text{Eq. 1}$$

Where ΔV is the voltage between the tip and sample, z is the height above the sample, A is a constant, and ϕ is the average work function between the tip and the sample. These variables are shown in Figure 10 below.

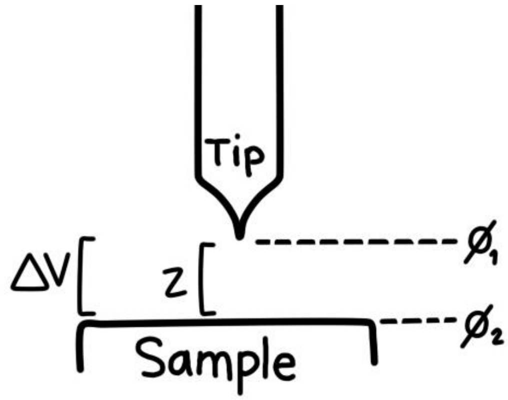

Figure 10. A diagram of the tip and the sample, showing the variables used in the equation to find the distance between the tip and the sample.

A computer program records the data and creates an image. The cable that comes from the tip and is fed into the computer contains a pre-amplifier, which converts current to voltage for the computer to read. Before entering the computer, the cables enter a circuit board that controls power to the operational-amplifier, keeping the current as steady and clean as possible. This circuit board also controls the gain. There are two ways to map the surface. For one method, the tip is kept at the same height above all points. To accomplish this, the tip must move up or down as it comes across peaks and valleys respectively; this movement is recorded versus the X and Y position of the tip. Another way to map the surface is to hold the tip at a constant height regardless of the peaks and valleys on the sample; this way the quantum current, and thus the voltage, changes with the topography. Again, the changes in voltage are recorded versus the X and Y position.

Problems and Resolutions

The STM is a sensitive device, and numerous repairs had to be made before any results could be found. First, the STM had to be removed from its chamber, and 2 of its wires replaced. This was done simply by cutting a new length of wire, and altering it to match the faulty wire. The pieces that connected the wire to the STM were soldered onto the wires with unfluxed solder, as flux is bad for the vacuum.

Additionally, one of the two scanners had an electrical connection between the tip, and the ground, which rendered it useless until the cause of this was found. Eventually it was discovered that the insulation on the wire to the tip had been coated in titanium from TSP runs. The scanner was placed in a tub of hydrogen peroxide which, while harmlessly dying the scanner blue, removed the titanium.

The other scanner was also found to be malfunctioning later on, and this was determined to be the result of a disconnection between a quadrant of the piezoelectric crystal and the wire meant to provide a voltage to it. This was resolved with silver epoxy, after the original silver paste was tried a few times, and proved ineffective. The silver epoxy dried more quickly, and was stronger. However, it was also less conductive, and likely less flexible, reducing the flexibility of the piezo itself.

The vacuum of the STM was initially unable to get below 10^{-6} torr, and the conclusion was that there was a leak. The leak was thought to be because of the O-ring in the load lock. The O-ring is a rubber ring that fits in a groove between the load lock door and the load lock itself, and is meant to keep air from entering the load lock when the door is closed. The O-ring may not have been the proper dimensions. Trying an O-ring with different dimensions improved the situation but didn’t entirely solve the problem. The vacuum could then be brought down to 10^{-7} torr; even reaching 10^{-8} torr occasionally.

Perhaps the most devastating issue that arose, was when the turbo pump on the STM was irreparably damaged. The filter meant to keep objects from entering the pump and damaging the blades was sucked into the pump and, ironically, destroyed the blades itself. This was due to faulty manufacturing; it took a few days to receive the replacement pump from the company.

Once other repairs were complete, it was found there was a problem with the signal itself; it contained a lot of noise. The mechanical noise isolators were effective, as stomping, opening and closing the door to the lab had no significant effect on the signal. The noise was also around 60 Hz, implying it was electrical in origin. The attempted solutions for this were to examine the circuit board and the pre-amplifier. Some components of these circuits were replaced, but the problem remained; though it was somewhat reduced. Another electrical problem, occurring in conjunction with the noisy signal, was the scanner and STM refusing to communicate with the computer; i.e. the computer could not read the signal, or move the tip across the surface. The computer continuously displayed the tip as crashed into the sample, despite there being a visible distance between the tip and the sample. It was eventually found that the communication problem was due to a bent pin in one of the cables; bending it back into place resolved the issue. The noise, however, was never completely removed.

Additional, more easily resolved problems were caused by the copper rail slipping into the STM chamber, the scanner slipping out of the claw and falling into the STM chamber, tightening a bolt so hard that the head broke off, unscrewing a valve too soon and ruining the vacuum inside the XPS chamber, samples breaking due to the sample holder screws being tightened too much, and dropping tips while trying to insert them in the scanner.

Results and Discussion

Towards the end of the allotted time, an acceptable image was recorded. This was after most issues had been resolved, despite the slight electrical noise remaining. The image was taken of a gold film on a thin mica sheet. (Figure 11) The original intention was to record gold on germanium, as this had been observed in the LEEM, and not yet with the STM, but a sample with gold deposited on germanium had not yet been prepared.

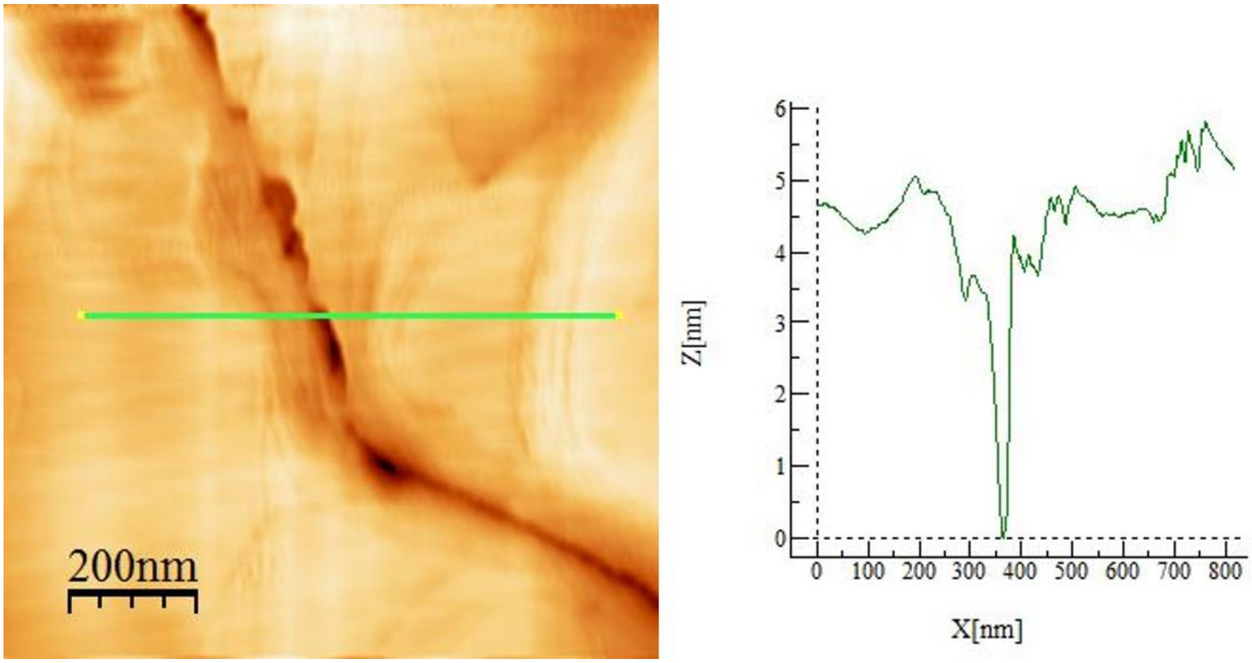

Figure 11. On the left is the image recorded by the STM after some noise was removed. This is a feature formed in a gold film deposited on a mica surface. The size scale is indicated in the bottom left corner. The green line indicates where the data on the right comes from. This graph shows the height (Z) versus the horizontal position (X)

The image is focused on a feature on the surface resembling a crevice. This "crevice" was formed when the gold was deposited on the surface of a small mica sheet. This was accomplished by vaporizing the gold and allowing it to collect on the surface.

This is a pretty good image, considering the noise and technical issues that had plagued the project from the start. While it is disappointing to only have recorded one image, it was an exciting moment, and did serve to prove that the apparatus was once again in functioning condition.

Conclusion and Future Work Planned

The STM is a sensitive machine, with many components; it requires a lot of maintenance to keep it functioning properly. Cleaning, repairing, and setting up the equipment is an important part of any experiment, and can sometimes take more time and work than carrying out the experiment itself. Thus, it's not uncommon that the entirety of the REU was spent on bringing the STM back into working order.

Future work will include imaging gold on a germanium surface as originally planned. Any kind of images taken of samples already studied in the LEEM can provide insight, as the two machines have different resolutions, and their own advantages and disadvantages.

References

C. L. H. Devlin, D. N. Futaba, A. Loui, J. D. Shine, and S. Chiang, Mat. Sci. Engin. B96, 215 (2002).