Scanning Tunneling Microscopy with Au on Ge(111)

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I. INTRODUCTION

The field of surface physics has become increasingly relevant in today's world. It has many applications, including catalysis, corrosion, magnetic storage devices, and integrated circuits. For many decades, the electronics industry has focused on decreasing the size of features on semiconductor devices, in order to increase the number of devices that can be fabricated on a silicon wafer. As the feature size decreases, it begins to approach atomic dimensions, increasing the surface area to volume ratio. In such circumstances, the study of surface physics becomes extremely important, including processes at the atomic level.

Surface physics studies the top few atomic layers of a solid material. We are interested in understanding electrical contacts of metals on semiconductors. To do so, the growth of metals on semiconductors must be closely studied. Metals grow in a variety of ways on the surface of semiconductors. They may grow in flat layers, form islands, or possibly grow in nanowires. There is great interest in low-dimensional surface structures, and one-dimensional structures actually have been seen for platinum on germanium(001) and gold on germanium(001), where (001) is the surface structure of the germanium samples of interest.

There are many analysis methods in surface physics, including low energy electron diffraction (LEED) and x-ray photoemission spectroscopy (XPS), among many others. This research project focused on the growth of gold on germanium(111). In order to examine these surfaces, we use a scanning tunneling microscope (STM) in an ultrahigh vacuum (UHV) chamber. An STM can measure real space images of the sample in three dimensions.

II. TECHNICAL BACKGROUND

A. Motivation

When Au on Ge(111) was observed with a low-energy electron microscope (LEEM), as the coverage increased, the Au grew on the surface in the Stranski-Krastanov growth mode, in which one full monolayer forms, followed by three-dimensional islands. LEEM images have shown structural phases and motion of gold islands. This STM experiment was motivated by the LEEM results, with the goal of studying this growth with the much higher spatial resolution of STM.

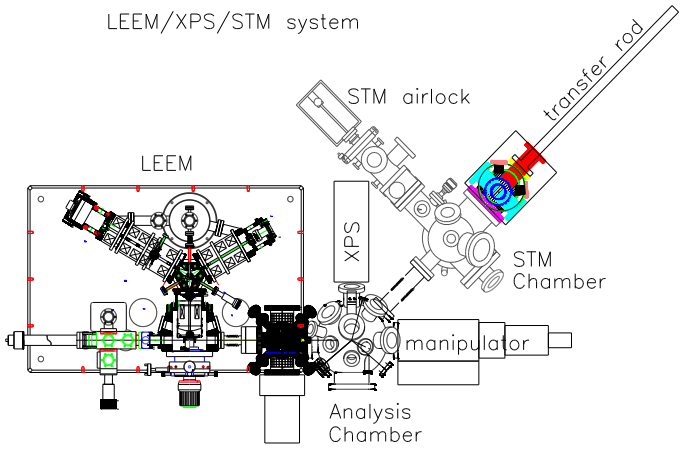

FIG. 1. This is the machinery in the lab. The LEEM was not operational during the duration of this project, and the XPS was only used for sample cleaning. All data for this project was taken in the STM.

B. Vacuum system

The first hurdle in this research project is achieving and maintaining an ultrahigh vacuum (UHV) chamber. UHV is defined as having a pressure on the order of 10^{-10} torr. Since the goal is to measure the top atomic layer of a surface, it is very important that the surface accumulates as little dust as possible. At a pressure of 10^{-10} torr, it takes 10,000 seconds for one monolayer of dust to collect, compared to a time of only 10 seconds at 10^{-6} torr.

Pumping the chamber down to UHV requires several pumping stages. A roughing pump is used for some chambers, which can bring the pressure down to approximately 10 mtorr. A turbo pump is used after the roughing pump. The turbo pump can achieve a pressure on the order of 10^{-9} torr. Finally an ion pump can be turned on, which brings the pressure down to 10^{-11} torr.

The vacuum chambers in the UC Davis lab are arranged as in Figure 1. Throughout the duration of this research project, the LEEM was not operational. There is an airlock chamber that allows for movement of items into and out of the STM without venting the entire chamber to air. The XPS was also not operational during this project, but the chamber was used for cleaning samples and deposition of metal onto samples, which will be discussed in detail later. There is a gate valve between the STM and the XPS which remains closed unless a sample is being moved from one chamber to the other. The transfer rod can be attached to the sample holder and is used to move the sample between chambers. The manip-

ulator has a similar purpose, which is to properly position the sample in the XPS chamber for cleaning and deposition. The manipulator has external controls which allow for adjustment in the x, y, and z directions, as well as for rotating the sample about its axis.

C. Tunneling

The tunneling current is the current between the surface of the sample and the tungsten tip. The tip is given a bias voltage, which allows for a quantum mechanical tunneling current. For this reason, the STM can only be used to observe samples that have conducting properties. The tunneling current I is proportional to bias voltage V such that:

$$I \propto Ve^{-A\sqrt{\phi}z}, \tag{1}$$

where A is a constant, ϕ is the average work function of the tip and sample, and z is the separation between the tip and sample in Ångstroms. The tunneling current therefore has the functional form:

$$I \propto 10^{-z}. \tag{2}$$

It is then evident that the tunneling current decreases rapidly with a slight increase in separation between the tip and sample. The atom right on the end of the tip therefore has the largest contribution to the current.

D. Samples

The goal of these experiments is to scan the surface of samples after deposition of a metal. The first task is therefore to construct sample holders for the germanium samples.

A sample holder is displayed in Figure 2. Underneath the sample is a filament through which a current can be run. The filament then heats the sample via electron beam heating, which is used for cleaning purposes. In order to measure the temperature of the sample, a chromel-alumel thermocouple is used. The sample holder is supported by four feet, and the back two are used for thermocouple wiring. One back foot is made of alumel and the other of chromel. Alumel and chromel wires are attached to their respective metal feet, and fed through the sample holder until they meet under the surface of the sample to form a thermocouple. With this arrangement, the temperature of the sample can be determined by the feet of the sample holder. The top plate is screwed on top of the sample to secure it.

Many difficulties in this project are due to the sample holder. There are fragile parts that often need to be repaired after moving a sample holder around inside

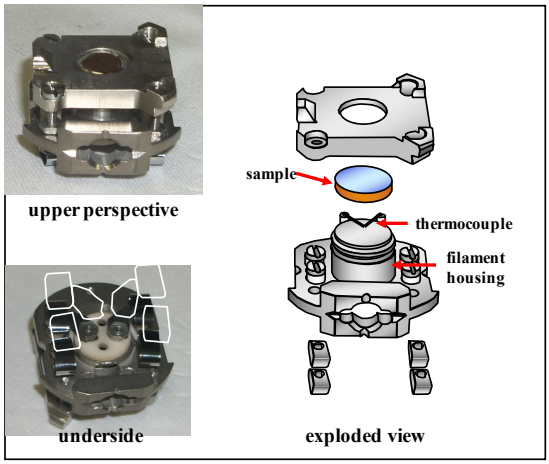

FIG. 2. This is a sample holder, which is used for securing the sample during deposition and scanning.

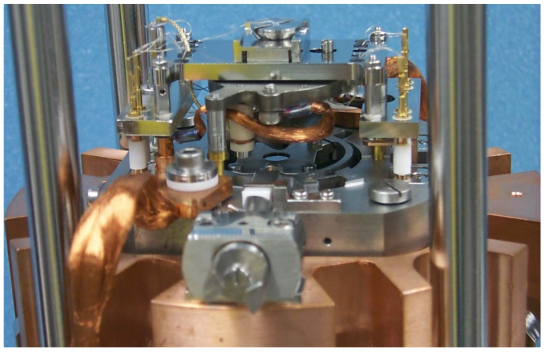

FIG. 3. STM scanner placed on top of a sample.

the chambers. There is also the possibility for short circuits of the thermocouple wires, which can potentially prevent a measurement of the sample temperature. Another potential issue is a break in the filament circuit, which would prevent heating of the sample.

E. Scanner

The scanner of the STM is placed on top of the sample, as displayed in Figure 3. A tungsten tip is inserted to the bottom of the scanner so that it is suspended just above the surface of the sample. This tip detects a tunneling current from the surface of the sample. For precision purposes, this tip must be extremely sharp, ideally one atom wide at the end. Making these tips is another major component of this project.

Manufacture of the tips is done by creating the circuit in Figure 4. As the current travels through the KOH solution, the tungsten tip is etched at the level of the solution's meniscus.

After a couple of minutes, the end of the tip falls off, as seen in Figure 5. This leaves the piece of tungsten with an extremely sharp tip. While this is a fairly simple

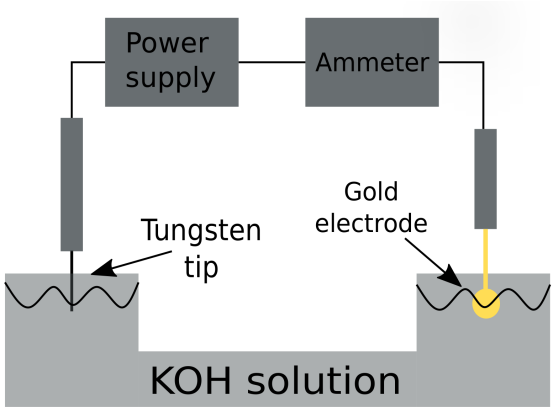

FIG. 4. This circuit is used to create very sharp tips on pieces of tungsten wire.

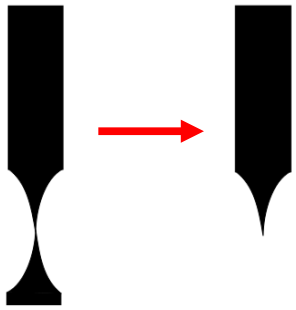

FIG. 5. The bottom of the tungsten wire eventually falls off, leaving a sharp tip for use in the STM.

procedure, the tungsten wire needs to be inserted to the solution just the right amount. If it is too low, the part that falls off will be too heavy and create a spring effect, causing the tip to bounce up into the wire and is therefore ruined. On the other hand, if the wire is too high, the bottom part will not be heavy enough to drop off and we will be left with something resembling the first part of Figure 5. It is difficult to measure exactly how deeply the wire is suspended in the solution, but it should be only a couple of millimeters.

The sharp tip then needs to be inserted into the bottom of the scanner. This is another matter requiring precise manipulation, since the tip needs to be positioned so that it sits just above the sample surface when the scanner is placed above the sample holder. If the tip is too far down, it will crash onto the sample surface as soon as the scanner is set down. There is also the possibility of the tip being too high too get a tunneling current. There is some room for displacement in the z direction, but only a few millimeters.

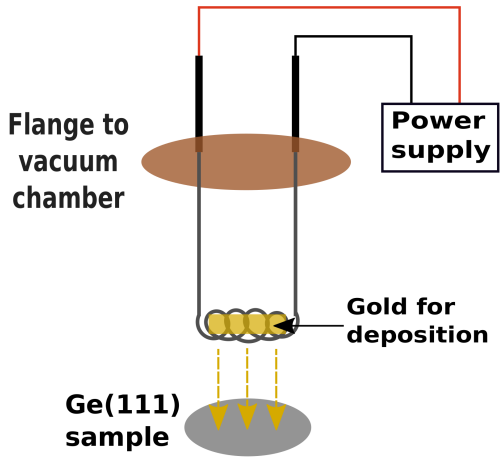

FIG. 6. An evaporator is formed by a tungsten coil with leads that can be externally connected to a power supply. A small amount of gold is secured in the coil, which evaporates onto the surface of the germanium sample inside the vacuum chamber when a current is sent through.

III. EXPERIMENT

A. Deposition

Once there is a new germanium sample inside the STM, it must be thoroughly cleaned before deposition of another metal. To do this, we use alternating cycles of sputtering and annealing. Sputtering is the bombardment of the surface with argon ions. The goal of this process is to clean the surface of any contamination. Since this bombardment damages the surface, it is followed with annealing, in which we heat the sample via electron beam heating from the filament. We heat the sample until it is close to the melting point of germanium, at approximately 800°C, so that the surface recrystalizes, leaving a smooth surface. The sputtering cycle lasts fifteen minutes, and the annealing lasts ten. Since it takes about forty cycles to sufficiently clean the sample, this is done automatically overnight.

After cleaning, the sample is finally ready for deposition of gold. An evaporator is shown in Figure 6. An external power supply is connected to the leads, which sends a current through the tungsten coil inside the XPS vacuum chamber. Once the gold has heated up enough, it evaporates onto the germanium sample.

Calibration of the gold coverage is very important, since we want to deposit a specific amount of gold. This is done using a quartz crystal deposition monitor in a separate vacuum chamber. The evaporator is positioned in this vacuum chamber above a quartz crystal. The crystal vibrates with a frequency proportional to the amount of material on its surface. Therefore, as we heat the evaporator, we can tell how much gold has collected on the

quartz crystal after a given amount of time based on the frequency. This calibration allows us to determine the current necessary for the desired deposition rate. For example, one experiment had a desired deposition rate of one monolayer per hour, and based on the quartz crystal calibration, this rate was achieved with a current of 9.5A. However, the calibration is a rough estimate, since the evaporator is then moved into a different chamber for deposition, in which parameters are likely to differ. This method therefore does not ensure an accurate deposition amount, but it is still useful as a guideline.

In data taken with a LEEM, gold has been shown to grow on Ge(111) in a Stranski-Krastinov growth mode, in which gold forms one full monolayer and then goes on to form three-dimensional islands atop the monolayer. This experiment utilized an STM to record data of the same interaction. An STM has a higher spatial resolution, so the growth behavior is can be monitored more precisely.

B. Data acquisition

Once gold has been deposited onto the germanium sample, it is secured in the STM with the scanner placed above it. We use an optical microscope connected to a camera to view the tip above the surface. Using a handheld controller, we can first manually approach the tip to the surface until it seems it is very close, but this is not very precise. There is danger of crashing the tip if it is moved too far down, which is difficult to gauge by eye based on the optical microscope. After manually approaching as close as possible, we use a computer program. The program takes a bias voltage and set point for the tunneling current as inputs. It then moves the tip in small steps toward the surface until a tunneling current is detected. The tip then scans laterally over surface while the program's feedback loop maintains constant tunneling current by changing vertical distance from the sample. While scanning, the computer program measures the topography of the surface and displays an image.

IV. RESULTS

The scan shown in Figure 7 displays on a relatively large scale the growth of gold on germanium(111). The large bright feature in the center appears to be a gold island. At the bottom of the image, along the line drawn, is a slight dark spot. The z-position of the sample surface is plotted along this line in Figure 8. The dip around X = 115 nm indicates a crater in the germanium surface, likely due to a tip crash from a previous attempt at scanning.

Data taken showed that gold actually began to form islands on germanium before completing one full monolayer, which contrasts with the Stranski-Krastinov growth mode. Based on the limited amount of data that was taken during this summer project, the Stranski-Krastinov growth mode is not necessarily the method of growth of gold on Ge(111).

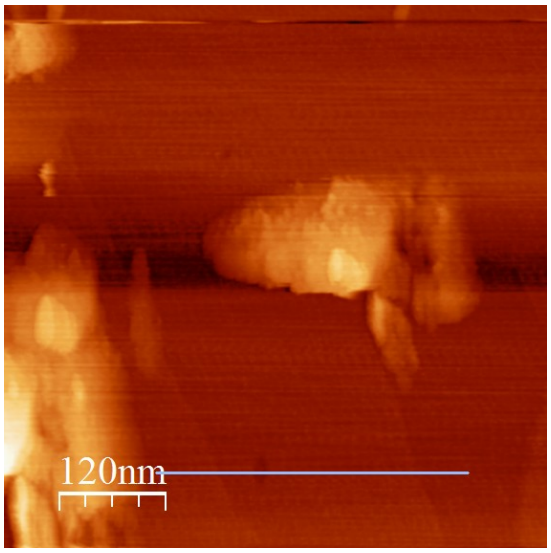

FIG. 7. This STM scan is Au on Ge(111). The gold was deposited at room temperature and had a coverage under one monolayer.

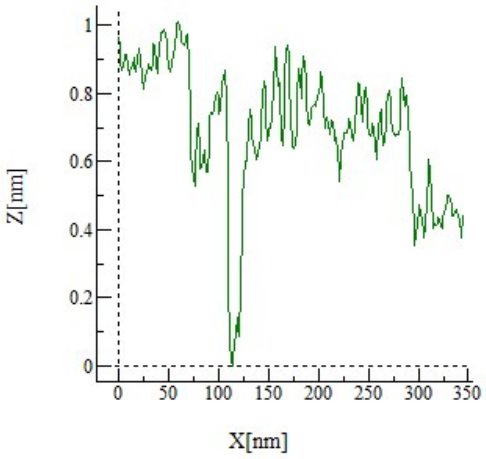

FIG. 8. The z-position of the sample surface is plotted along the line drawn in Figure 7.

V. CONCLUSIONS

This project was not able to verify the Stranski-Krastinov growth mode, but if this had not been limited to a ten-week project, much more data would have to be taken to further investigate. Another improvement would be to increase the precision of the quartz crystal calibration method. Due to the lack of precision with the current method, it was difficult to deposit an exact amount of gold onto the germanium samples. Doing this

would allow for more precise deposition of gold onto the germanium surface.

ACKNOWLEDGMENTS

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