Studying Pb Island Growth on Ge(111) Using Scanning Tunneling Microscopy

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Abstract

This paper discusses work done in the Chiang Group as part of the UC Davis Research Experience for Undergraduates. The primary goal of the project was to corroborate earlier results concerning the heights of Pb islands grown in ultrahigh vacuum (UHV) on Ge(111) by measuring scanning tunneling microscope (STM) images of said islands at -24 °C and +3 °C. In light of technical difficulties encountered in operating the STM, this paper will focus on background, instrumentation, and methodology rather than on results. Finally, preliminary results obtained from evaporator calibration experiments done for a different project involving a low energy electron microscope (LEEM) will be discussed.

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1 Introduction

The relevance of surface science has greatly increased in recent years thanks to its importance in the manufacture of electronics. As electronic components continue to shrink in size, their most significant features become their surfaces due to increasing surface-to-volume ratios, as well as due to the fact that chemical reactions occur at surfaces. Of special interest is the study of metal-semiconductor interfaces, as the controlled evaporation of metals onto semiconductor surfaces can result in the formation of potentially useful nanostructures such as nanowires, islands, and thin films, among others.

Both Pb and Ge are of interest to surface physics for their properties. Pb has, in previous studies, displayed collective diffusion: the rapid movement of large numbers of atoms over large distances in relatively short timespans. This phenomenon is poorly understood at a theoretical level, but the resulting diffusion is known to be much more rapid than classical predictions [2].

In addition, Pb has the property of displaying quantum size effects (QSE). In this context, QSE refers to the confinement of electrons within crystal structures due to some dimensions of the crystal becoming comparable to the electron wavelength [4]. Related to this, Pb has a rather unusual property which may be mathematically expressed as follows:

$$2d \approx \frac{3}{2}\lambda_F \tag{1}$$

where d is the thickness of a single layer of Pb and λ_F is the Fermi wavelength [4]. Every two layers of Pb add approximately three nodes to the electron wavefunction spanning the thickness of a Pb island or thin film. This wavefunction is, therefore, a close approximation of a perfect standing wave inside the structure it spans, making said structure particularly stable [4]. This property is likely the explanation for the observed tendency of Pb islands to grow to be a particular number of layers in height at a given temperature. It is this temperature-dependent height selection which is the main interest of this project.

Ge was chosen as the substrate for its properties as a semiconductor. Germanium, like Silicon, is a Group IV semiconductor. While Si is currently the dominant semiconductor in modern electronics, Ge has the additional property of very high electron and hole mobility, which makes it the semiconductor of choice for certain specialized applications such as high speed integrated circuits.

2 Background

The primary motivation for this project was to continue work done in the doctoral thesis of Dr. Andrew Kim. Island height selection in the Pb/Ge(111) system was studied at various temperatures. However, the instrument of choice for measuring island heights - the scanning tunneling microscope (STM) - was not operational when the study was conducted. Instead, an alternative method for measuring island heights was used. The method involved computing intensity vs. voltage (IV) curves from a modified Kronig-Penney (KP) model representing the layers of Pb on the Ge substrate. This was done for a number of different potentials, where the parameter being changed was the number of Pb layers assumed to comprise an island. Then, an experimental IV curve was measured from a particular Pb island as seen under a low energy electron microscope (LEEM). An example of a modified KP model and IV curve are shown in figures 1 and 2, respectively.

The experimental curve was then compared to the various simulated curves. The simulated curve which was the closest fit to the experimental data was thus concluded to indicate the number of layers making up the island. Using this method, it was found that Pb islands tended to be 7 layers in height at a temperature of -24 °C and 10+11 layers at +3 °C. The notation 10+11 indicates that the island examined had a hybrid height, likely due to growing over a step edge [4].

The validity of the above-described method is not well-established, as it is not widely used in the literature. Therefore, the primary motivation behind the REU project was to use the

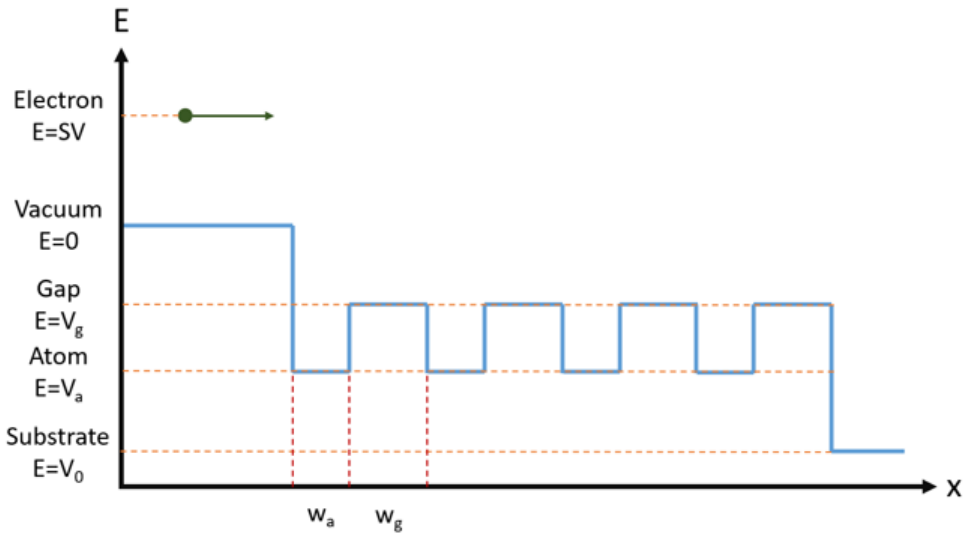


Figure 1: Modified KP potential for the case of a 4-layer island. Modification lies in the insertion of non-periodic regions at the vacuum and substrate levels [4].

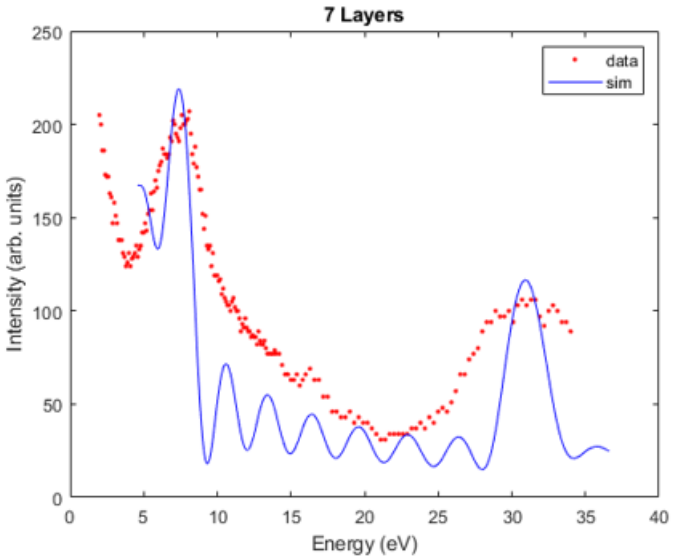


Figure 2: Simulated IV curve in blue and experimental data in red. This particular IV curve was the closest fit to the data, which were collected at a temperature of -24 °C [4].

now-operational STM to accurately measure island heights and corroborate results obtained using the LEEM/KP method.

3 Instrumentation

The Chiang Lab has three primary instruments: the scanning tunneling microscope (STM), low energy electron microscope (LEEM), and x-ray photoelectron spectroscope (XPS). Their respective functions will now be described.

3.1 STM

Scanning tunneling microscopy uses the phenomenon of quantum tunneling to form three dimensional real space images with atomic resolution. To operate an STM, one brings an atomically-sharp tip to within a few ångströms of the sample surface and applies a bias voltage between the tip and the surface. Electrons will then tunnel between the surface and the tip according to the direction of the bias. This results in a current described by the following relationship:

$$I \propto Ve^{-A\sqrt{\Phi}z} \tag{2}$$

where I is the tunneling current, V is the bias voltage, Φ is the work function of the sample and the tip, z is the height of the tip above the sample, and A is a constant [2]. The tunneling current is very sensitive to changes in z. Thus, by keeping current constant using a feedback loop while using piezoelectric materials to move the tip laterally across the surface, one can record how z changes to keep I constant, yielding an outline of the surface. By performing many such scans in parallel, one can then reconstruct a 3D image of the surface topography. An example of such an image is shown in Figure 3.

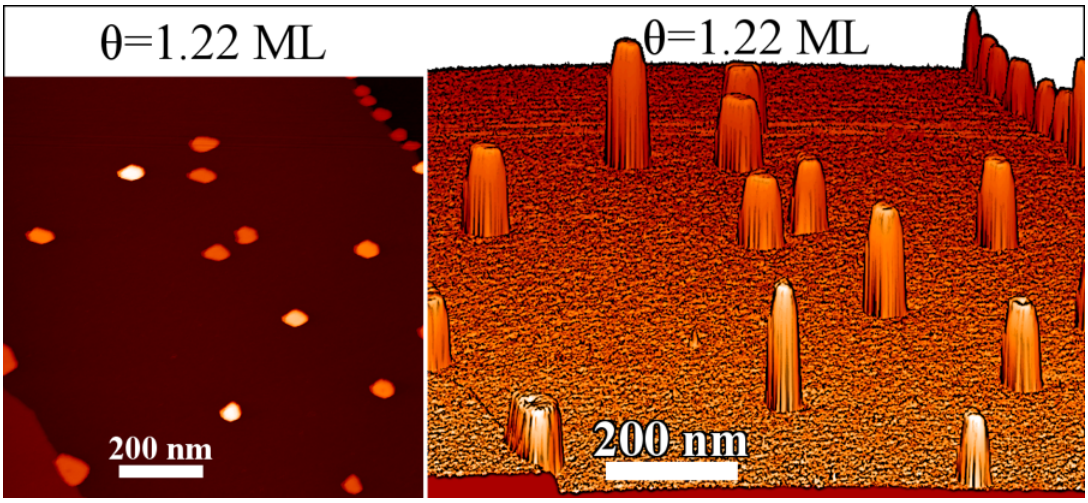
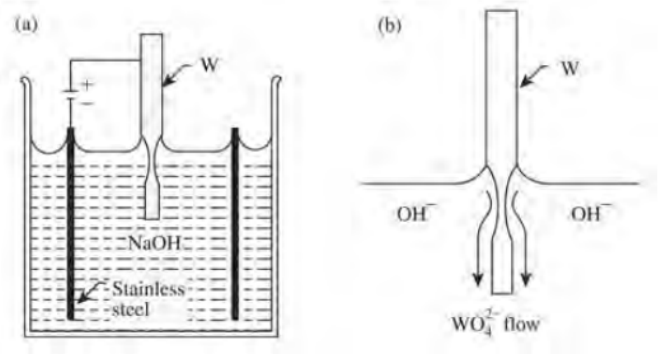


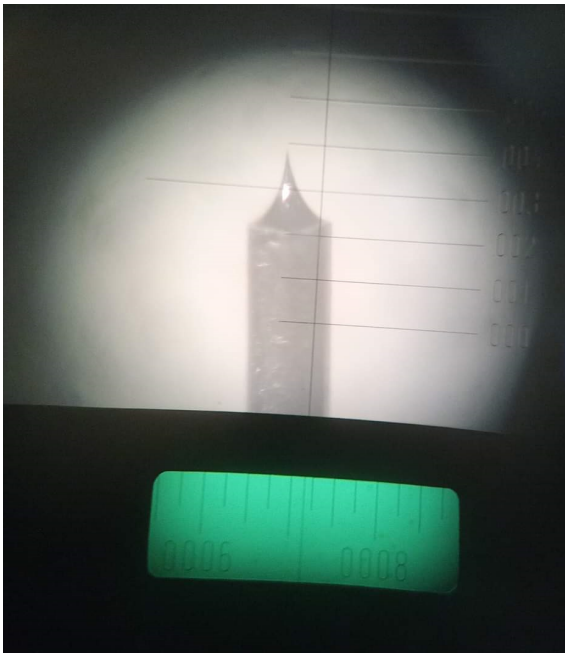
Figure 3: Example of a 3D image obtained using STM. From a study of height-selected Pb islands grown on Si(111) [3].

Tips for the STM were made using electrochemical etching. In this technique, a thin tungsten wire is immersed in KOH. A counterelectrode is submerged elsewhere in the solution. When a voltage is placed on the tungsten wire, it acts as an anode and a chemical reaction occurs

on the surface of the wire at the level of the meniscus. This reaction generates tungsten anions which gradually flow down the wire until gravity finally causes the bottom of the wire to fall off due to it becoming depleted at the meniscus. The final result is, ideally, an atomically-sharp tip. A diagram illustrating the process is shown below, alongside an image of a completed tip under a microscope.



(a) Diagram outlining electrochemical tip etching [1].



(b) Example of a completed STM tip as seen under a microscope.

Figure 4: Tip etching procedure and final product.

3.2 LEEM

Low energy electron microscopy is in some ways analogous to optical microscopy: electrons take the place of photons and magnetic lenses replace glass ones. The electron source is a

LaB_6 crystal. Being thermionic, the crystal emits a large number of electrons upon heating, thereby acting as an electron gun. The resulting electron beam is accelerated to an energy on the order of 20 keV, before passing through several condenser lenses. The beam is then redirected using a beam separator onto the target. Crucially, the target is kept at a potential near that of the gun, causing the electrons to decelerate to an energy on the order of 10 eV. At this stage, these low energy electrons are said to be surface sensitive in that they will only penetrate the surface of the sample and will not reach the bulk. After penetrating the surface, the electrons will elastically backscatter, before being reaccelerated to 20 keV. The beam once again passes through the separator, which now directs it through a number of projector lenses. The beam then strikes a microchannel plate, which acts as a signal amplifier, before finally striking a phosphorescent screen being recorded by a video camera. In this manner, LEEM is capable of making short movies with a resolution of around 10 nm.

3.3 XPS

X-ray photoelectron spectroscopy makes use of the photoelectric effect to determine what elements are present in a sample and in what relative quantities. X-rays are produced by bombarding a magnesium or aluminium anode with high energy electrons. These X-rays will, in turn, strike the sample and cause a core electron to be ejected with kinetic energy given by

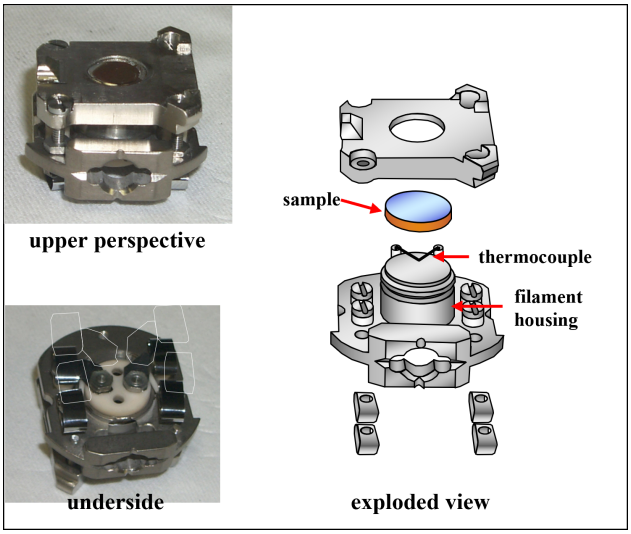
$$KE = h\nu - E_b - \phi \tag{3}$$

where $h\nu$ is the incident photon's energy, E_b is the electron binding energy, and ϕ is the work function of the entire sample [4]. The kinetic energy can be measured using an electron energy analyzer, from which the binding energy E_b can be calculated by rearranging equation (3). Because the binding energy is unique for every core level of every atom, knowing the value of E_b provides a direct way of determining the composition of the sample. The XPS performs a scan over a range of binding energies, producing a spectrum of peaks whose relative heights indicate the relative amounts of different substances in the sample. In the context of the project, such a spectrum would also be used to calibrate the evaporator inside the STM chamber by computing the ratio of the heights of the peaks corresponding to Ge and Pb and comparing this experimental ratio to one computed by the software Simulation of Electron Spectra for Surface Analysis (SESSA).

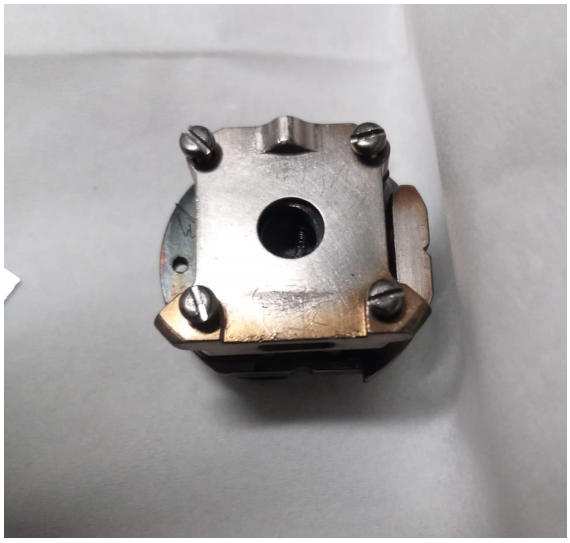
3.4 Sample Holder Design

Finally, the sample holder should be briefly mentioned, as this component is common to all three instruments. The sample holders used in the lab are made of either molybdenum, stainless steel, or titanium. Two important features are visible in the figure below. The first is the pair of thermocouple wires attached to a ring supporting the sample. The thermocouples

are of type K or C, and are one of the methods of measuring sample temperatures (the other being by means of an infrared pyrometer when at very high temperatures). The second feature is a tungsten filament beneath the sample, just visible in Figure 5b on the right side of the central hole. The filament heats the sample by thermal radiation, as well as by bombardment with electrons emitted by the filament and accelerated towards a biased sample.



(a) Diagram of the sample holder. Thermocouple wires and feet visible [2].



(b) Example of sample holder used. As this holder lacks a sample, the tungsten filament (shiny metallic coil) is visible in the central hole.

Figure 5: Sample holder design.

4 Experimental Procedure

While no substantial experiments were conducted for reasons which will be explained in the following section, the general procedure which would have been carried out was known and will now be described.

Initially, all relevant chambers - in this case, those of the STM and XPS - would be brought to an ultrahigh vacuum (UHV) of 10^{-10} Torr. This would be a multistep process involving several pumps, such as a mechanical pump, a turbo pump, an ion pump, and a titanium sublimation pump. In addition, the chambers would be baked above the boiling temperature of water overnight in order to remove any residual water molecules adsorbed onto the interior of the chambers.

Once the chambers are ready, a sample of Ge(111) would be cut out of a germanium wafer to fit inside a sample holder. This sample would then undergo *ex situ* cleaning, which consists of sonicating the sample in a methanol bath for one minute and soaking it in H_2O_2 to create a uniform oxide layer. The Ge sample would then be mounted on its holder and brought into

the XPS chamber, where *in situ* cleaning would be performed using sputter-annealing cycles. These cycles consist of bombarding the sample surface with Ar^+ ions and then heating the sample to 800 °C in order to reconstruct the surface.

The STM chamber would then be brought to the target temperature for the experiment by first cooling the chamber using liquid nitrogen before heating it back up to the target temperature. The temperature would be maintained using a feedback loop.

The sample would then be brought into the STM chamber. This would be followed by a tip approach procedure, wherein the STM tip would gradually be brought to within a few Å of the surface in order to be ready to perform scans. The Pb evaporator would then be turned on, beginning Pb deposition; the evaporator is presumed to have already been calibrated as described in §3.3. Finally, STM scans over a particular region would be performed at regular time intervals.

5 Results

Due to unforeseen technical difficulties, no results were obtained in the primary Pb/Ge(111) project. An earlier project involving the LEEM also encountered such problems, but some calibration data was obtained. These problems will now be described, as will the calibration work done using the LEEM.

5.1 Technical Difficulties Encountered in STM Project

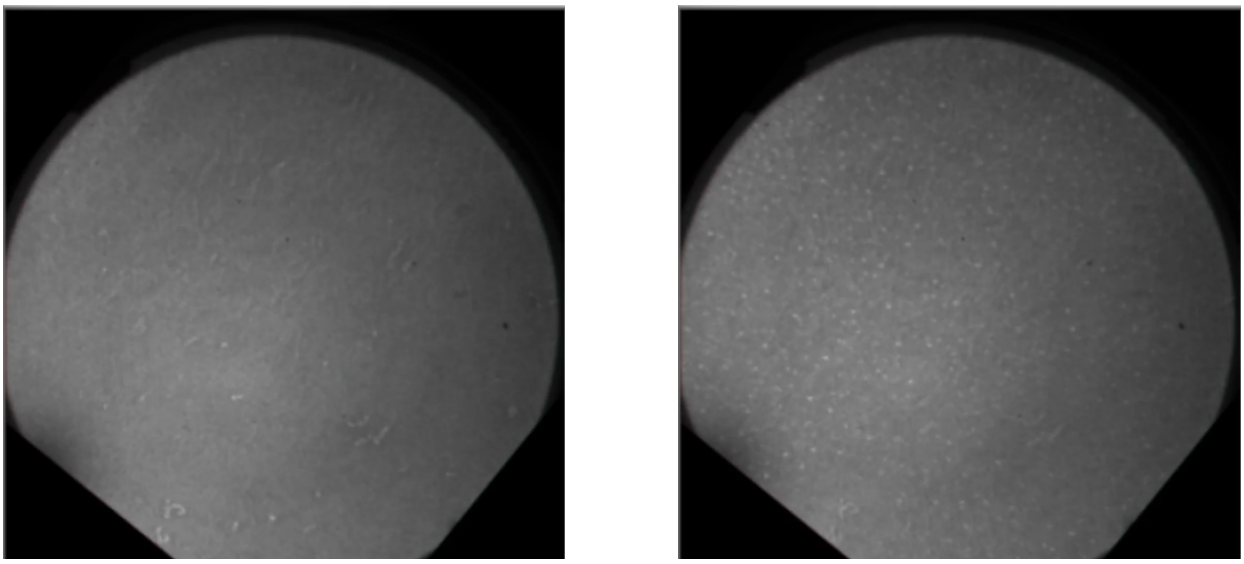
Before beginning the experimental procedure described in §4 in earnest, it was first necessary to confirm that the STM was functional and could attain atomic resolution. For this purpose, an attempt was made to image the surface of a graphene sample. Despite efforts, the tip could not be made to approach the sample closely enough. Upon closer inspection of the STM, it was found that all piezoelectric tubes were broken. The cause for this was most likely the inexperienced placing of the STM scanner onto the stage, whereby excessive force when pushing the scanner down onto the stage caused the tubes to snap. As a result of having to replace all the piezoelectric tubes, no experiments could be performed in time.

5.2 LEEM Project: Evaporator Calibration

Initially, the REU project was to be concerned with Pb on either the (110) or (100) face of Ge, where the primary instrument of investigation would have been the LEEM. A preliminary step before studying either face was to calibrate the Pb evaporator to have a deposition rate close to 0.3 ML/min. To achieve this, the Pb evaporator was turned on at a specific current and the sample surface was imaged using LEEM. The sample being imaged was Ge(111),

on which Pb exhibits a high-contrast phase transition from the α-$(\sqrt{3} \times \sqrt{3})$R30° phase to the (1x1) phase at a coverage of $\frac{1}{3}$ ML at temperatures between 200 °C and 300 °C. As soon as this transition was observed, the time elapsed from the start of deposition was recorded and used to compute the average deposition rate. The evaporator current was adjusted to approach the desired rate. An example of this phase transition is shown in Figure 6.

For the purpose of calibration, a Ge(111) sample was used in place of Ge(110) and Ge(100), with the implicit assumption that the deposition rate will remain unchanged when using other faces. The reason for the substitution is that Pb does not undergo a suitable high-contrast phase transition on either of these faces [4].



(a) Dark background indicates presence of α-phase.

(b) Bright spots indicate formation of (1×1)-phase.

Figure 6: LEEM images of sample surface before and after phase transition. FoV = 15 μm.

In the case of the LEEM, the issue which prohibited further progress with the project was the wearing out of the threads on the sample manipulator. The sample manipulator adjusts the distance between the sample and the objective lens. Due to the age of the manipulator, the threads had become worn out to the point that the sample could not be moved at all, rendering the LEEM unusable until a replacement manipulator could be installed.

Acknowledgements

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References

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