LEEM and LEED Analysis of Ge(100)

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Germanium is a semiconductor that has possible applications in modern electronics. As electronics get increasingly small, understanding the behavior of materials at the top few layers of atoms is vital. This paper describes an ultra-high vacuum system that can be used to study Ge(100). Within the system is a chamber where samples can be cleaned using the sputtering and annealing technique. Another chamber is outfitted with equipment for low-energy electron microscopy and diffraction, making real time analysis of samples possible. Although no final results were obtained within the time span of this project, initial steps were taken to create a phase diagram of Pb on Ge(100).

I. INTRODUCTION

With the growth of the electronic and technological fields, surface science has seen increasing relevance. Surface science is concerned with the deviations from standard properties of materials that occur on interfaces. Much of the experimental work done in this field involves numerous forms of spectroscopy, including low energy electron diffraction (LEED), low energy electron microscopy (LEEM), scanning tunneling microscopy (STM), and X-ray photoelectron spectroscopy (XPS). Dr. Chiang's lab at the University of California, Davis uses all these techniques, most recently for the study of various surface properties of germanium. This paper details an experimental technique for LEEM and LEED analysis of Ge(100).

II. BACKGROUND

Germanium is a material of interest in part due to its similarities to silicon, a popular semiconductor in modern electronics. One similarity is the diamond cubic structure that both materials possess. With this structure, it is natural to consider Ge(100), Ge(110), and Ge(111). Much of Dr. Chiang's previous work has involved Ge(110) and Ge(111), and a cleaning technique for these materials has been well established. The technique, called sputtering and annealing, is commonly used for ultra-high vacuum (UHV) experiments.

Firing argon ions at the sample's surface acts as a means of dislodging any contaminants. Because argon is a noble gas, it will not combine with the sample itself. The force required for the argon to remove particles is also enough force for the argon to cause dents in the sample face. These divots are addressed by completing an annealing cycle. Heating the sample to slightly less than its melting point causes the top layers of the crystal to melt and reform smoothly. Repeating these cycles 30-40 times gives a surface that is free of contaminants and suitable for study on an atomic level.

Using this method Dr. Chiang's lab was able to sufficiently clean Ge(110) and Ge(111) for experimentation, but Ge(100) samples were left with large bumps on the surface. The effectiveness of the cleaning can be viewed in real time through the use of LEEM. The LEEM and LEED design that is incorporated into the lab's UHV system is shown in Fig. 1. The LEEM operates by sending electrons down one channel where a series of lenses slow the electrons which then pass through a separator. The electrons must be sufficiently slowed, such that they only penetrate the first few layers of the sample before bouncing back through the separator. They are redirected down a second channel to a video cam-

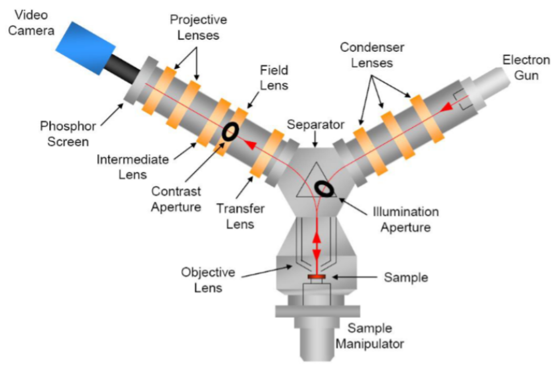

FIG. 1: LEEM and LEED. This is a diagram of the equipment used for LEEM and LEED analysis. It can be used for both real world imaging and taking diffraction images of samples place into the UHV chambers.

era which can display an image of the sample in real time. In this way, samples may be inspected after cleaning, but one can also view the sample during deposition experiments.

One aspect of surface science is ascertaining the structure of crystals that form when a material is deposited on a substrate. LEED analysis makes this possible by creating a diffraction pattern that is a reconstruction of the sample's surface. The LEEM apparatus can be used for this purpose, by placing an additional lens within the second channel. From the diffraction pattern, one can develop a phase diagram that shows the structure of formed crystals dependent on temperature. There is disagreement in previous literature on the phase diagram of Pb on Ge(100), meaning further exploration on this topic is necessary [1] [2]. The goals of Dr. Chiang's lab therefore include finalizing a sputtering and annealing regime that adequately cleans Ge(100) samples and using these cleaned samples to create a phase diagram of Pb deposits via LEED analysis.

III. EXPERIMENTAL APPARATUS

All experimentation is done within a ultra high vacuum chamber. The apparatus consists of four connected chambers, as shown in Fig. 2. Samples on

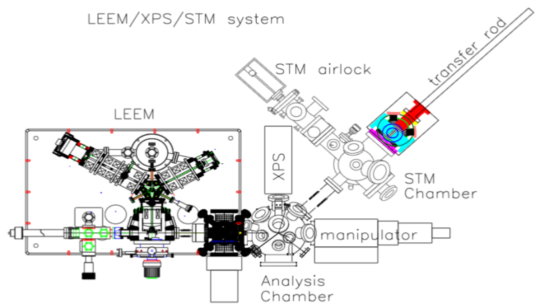

FIG. 2: UHV System. The system is composed of four interconnected chambers. Samples are placed in the system via the airlock and can be transferred to other chambers for cleaning and for LEEM, LEED, XPS, or STM analysis.

a tray are placed in the first chamber, labeled STM airlock, which can then be sealed and pumped down to a pressure on the order of 10^{-9} torr. The tray can then be transferred to the second chamber, referred to as the STM chamber. The STM located in this chamber was not used for this experiment. Samples can then be moved from the tray and onto the first transfer rod which can be pushed into the third chamber. This chamber can be used for XPS analysis, as well as sputtering and annealing. The second transfer rod can then be used to pull the sample into the fourth chamber, where samples can be analyzed via LEEM and LEED. Note that the large square in Fig. 2 labeled LEEM is equipment on the exterior of the fourth chamber.

IV. PROCEDURE

A. Sample Preparation

To prepare samples for analysis, we began with wafers of highly polished Ge(100). The wafers are cut to size using a diamond scribe. To remove contaminants, the cut samples were placed in a beaker of hydrogen peroxide which was placed into a water bath to undergo five minutes of ultrasonic cleaning. The hydrogen peroxide was then replaced with methanol and another five minute cleaning cycle was completed. Samples were dried with lens paper and mounted onto the sample holders, as shown in Fig. 3. A laser was used to ensure that the sample's surface was aligned such that once within the vacuum chamber it would be properly angled for LEEM and LEED analysis.

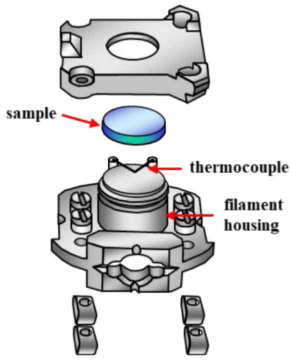

FIG. 3: Sample Holder Diagram. The sample holders are specially machined to be compatible with all measurement systems within the UHV system. The sample is secured above the thermocouple wires, which are attached to the feet of the holder. Underneath the sample is a housing for the filament which is used to heat the sample for cleaning.

Additionally, since the sample holders were delicate, the connections between the feet and the base were checked every time a new sample was mounted. Connections were measured with a handheld ohmmeter, and a measured resistance indicated that the filament housing had loosened during the sample placement. Repairs for this issue were made with a spot welder. Wafers, samples, and sample holders were all handled only while wearing gloves to prevent contaminants, including skin oils, from coming into contact with anything that would be placed into the UHV system.

B. UHV Handling

All analysis is performed in a UHV system due to the scale that measurements are made at. Dust particles and contaminants present at room pressure are large enough to interfere with analysis and will appear in the LEEM and LEED images. In UHV, a cleaned sample can be considered free of contaminants for several hours. Once samples have been placed into the STM airlock, the pressure of the first chamber must be reduced. For all transfers between chambers, the relevant pressures must be near the same order of magnitude to prevent harm to the equipment or damage to the sample. The pressure of the airlock is generally about one order of magnitude larger than the STM chamber meaning that the pressure of the overall system rises slightly over time, but several transfers can be completed before the STM chamber's pressure must be corrected.

In order to reduce the pressure of the airlock, a turbo pump is used in addition to heating the chamber over the boiling point of water in order to remove water molecules. This heating is done by heating tapes wrapped around the chambers and a covering of aluminum foil. This process is completed overnight and brings the pressure of the airlock to 10^{-9} torr. If any other chamber is opened to atmosphere, the procedure to sufficiently reduce pressure is more lengthy and is therefore only done when internal repairs to the chamber are necessary. As stated above, if there are frequent transfers between the airlock and the first chamber, this process must also occasionally be completed to maintain UHV throughout the system.

To bring one of the main chambers to UHV, the first steps are the same as those above used for the airlock. After the chamber has cooled, titanium sublimation pumps are used. Any gas particles remaining in the chamber bind to Ti atoms and are therefore easier to remove via the pumps. At this time, any filaments in the relevant chamber are degassed. Additional use of the titanium sublimation pumps may be necessary after the degassing. Finally, an ion pump is utilized to bring the chamber to 10^{-10} torr.

Although a base cleaning was performed before mounting samples, a second cleaning procedure is performed in the third chamber. The sputtering and annealing technique was performed using a tank of argon affixed to the outside of the chamber. Annealing was completed by heating the filament housed within the sample holder and applying a high voltage bias on the sample. As each holder and sample are slightly different, a calibration must be done for each sample put through the sputtering and annealing process. This was done by using the manipulator to angle the face of the sample towards the viewing window of the analysis chamber, setting the current to 2.7 A, and slowly increasing the voltage until the sample reached a temperature between $800°$ C and $850°$ C. The sample temperature was determined using an infrared pyrometer. When the appropriate voltage setting was determined, the manipulator can then be used to rotate the sample to face the sputtering gun.

While argon is in the chamber, the ion pump must be turned off, meaning that the pressure must be maintained by a turbo pump for the duration of the sputtering and annealing. Argon was released into the chamber until the pressure reached between 5×10^{-6} torr and 1×10^{-5} torr. The sputtering gun was set to 45 eV and one cycle of sputtering was completed over 15 minutes. If being done manually, the argon canister was closed off and the sample was heated for 10 minutes. Because of the time needed to complete 30-40 cycles, the lab also contains an Arduino microcontroller that can control the sputtering gun and the annealing settings. This automation allowed samples to continuing cleaning overnight or throughout a weekend. In this case, the argon canister is not closed during the annealing cycle. After the completion of the sputtering and annealing cycles, the sample can be moved into the final chamber for LEEM and LEED analysis.

V. RESULTS

Due to the time frame of this project, we were not able to obtain quantitative results. There were several issues with equipment caused by age and frequent use that had to be addressed before experimentation could occur. Firstly, a new technique for securing the filament housing was developed. The fragility of the original sample holders meant that any jolting motion during transfers inside the UHV system could cause the filament housing to loosen, causing a connection between the body of the holder and the filament. This renders annealing impossible, but frequently occurred due to the motion of placing the holder in the manipulator in the analysis chamber. To overcome this, strips of tantalum foil attached via spot welder were used to strengthen the structure and greatly increased the amount of movement, and therefore the number of transfers and sample mountings, a sample holder could endure before repairs were needed.

The sample holders also contain several ceramic pieces around the bolts attaching the feet and around the thermocouple wires. Due to age, several of these ceramics had become dirty and begun acting as conductors. Faulty ceramics were located with a hand-held ohmmeter and removed for cleaning. The top layer of each ceramic was removed with a diamond file and the pieces were then put through the ultrasonic cleaning process used for samples before being refitted on the holders. In some cases, thermocouple wires were also replaced and reattached with a spot welder.

Finally, the analysis chamber had to be opened to atmosphere in order to fix a wire that had loosened. This repair, as well as closing the chamber and recreating the UHV, took a significant amount of time during which no samples could be cleaned. In the final stages of the project two samples were successfully put through 40 sputtering and annealing cycles and transferred to the LEEM chamber. Once in the chamber, some time was given to aligning the lenses within the LEEM apparatus. In the final days of this project, it was determined that the high voltage display was damaged and would need repairs.

VI. FUTURE WORK

After repairs are completed on the LEEM apparatus, it will be possible to make further progress on the goals outlined above. Firstly, a satisfactory cleaning process can be established for Ge(100). As mentioned in the Background section, sputtering and annealing has produced good results for Ge(110) and Ge(111) in this lab, but left the surface of previous Ge(100) samples rough. It is possible that lengthening the time of the annealing cycle could resolve this issue. Another possible solution is increasing the number of cycles performed.

With sufficiently cleaned Ge(100) samples, the LEED apparatus can be used to determine the phase diagram of Pb deposits. Gathering LEED data over a range of temperatures and finding the crystal structures that form on the Ge(100) surface will provide clarity on the disagreement in previous literature on this topic.

Acknowledgements

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[1] Zhang Y. et al., "Surface superstructures of the Pb/Ge (001) system," *Surface Science Letters* **293** (1993).

[2] Falkenberg G. et al., "Lead-induced reconstructions of the Ge(001) surface," *Surface Science* **372**, (1997).