Growth of Au on Ge (110)

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Abstract

The clean Ge(110) surface is studied using Scanning Tunneling Microscopy (STM) to analyze potential sites for growth. The surface structure and growth mechanism of Au on Ge(110) is characterized with Low-Energy Electron Microscopy (LEEM). Au is dosed at room temperature with approximately 0.5 monolayers (ML) of coverage. The temperature is increased up to 800 °C when the sample is imaged by LEEM. As the temperature increases, the Au islands form into much larger 1-dimensional structures, with all islands growing along the same axis. Island behavior as the temperature decreases is also studied, revealing rapid island contractions which leave traces on the Ge(110) surface.

1 Introduction

Understanding the mechanism by which metals and other materials nucleate and grow on semiconductors has been an area of much interest recently due to the increased usage of such materials for device applications. As such devices get smaller and smaller, so too must the components that make them up. This demand has brought about interest in the ability of self-assembling nanostructures on the surface of semiconductors. Germanium has been of particular interest because of its similar properties to silicon, but has the advantage of having substantially higher electron and hole mobility than silicon. Thus, it has greater potential for use in higher-speed devices. [1] Possible fields of application for such nanostructures include spintronic devices, solar cells, and photonics.

1.1 Overview of Ge(110)

The unreconstructed surface structure of Ge(110) has a rectangular unit cell, with the dimensions of 4.00 x 5.66 Å. [2] Along with this rectangular unit cell, unreconstructed Ge(110) also has zigzag atomic rows which run in the $[1\bar{1}0]$ direction. Each surface atom has two tetrahedral bonds to neighboring surface atoms, as well as a tetrahedral bond to the atomic layer below. The Ge(110) surface will reconstruct to (16 x 2) or c(8 x 10) structures, depending on the annealing temperature. Through analysis of these reconstructions with STM and diffraction, the structural features of these surfaces have been determined. A common feature of both reconstructions is pentagonal clusters of adatoms, shown in Figure 1. The major difference between these 2 reconstructions is in the underlying surface atoms. In the (16 x 2) reconstruction, there is a missing layer of (110) atoms that alternates under the rows of the clustered adatoms. This causes the clusters to differ in height by one layer of the (110) surface in the (16 x 2) reconstruction. In the c(8 x 10), all of the adatom clusters sit on the same (110) plane (Fig 1).

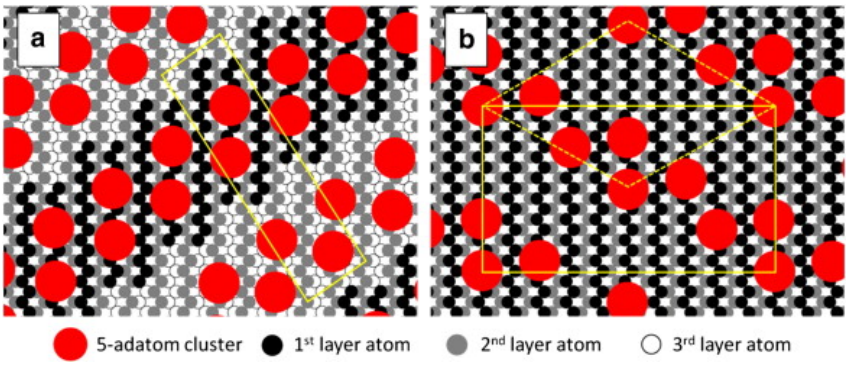

Figure 1: Diagram of both the common reconstructions of Ge (110) with the unit cell outlined in yellow. (a) In the (16 x 2) reconstruction, the underlying surface atoms alternate between missing a layer of Ge(110). (b) In the c(8 x 10) reconstruction, all of the 5-adatom clusters sit on the same plane.[3]

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1.2 Scanning Tunneling Microscope

The STM utilizes the effects of quantum tunneling to map a sample's surface. When the STM tip is brought within a few Angstroms of the surface, electrical equilibrium is reached between the two. A bias voltage is then applied, which allows a tunneling current to occur. This current I is related to the tip height z above the sample by the relationship:

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

where V is the applied voltage, ϕ is the work function of the sample, and A is a constant. The tip is swept across the surface using a piezoelectric scanning tube which moves the tip laterally across the surface in a raster pattern[4]. The STM has two imaging modes, one in which the tunneling current is held constant by a feedback loop. The tip height z above the sample varies over the bumps on the surface as the tip moves laterally. Measuring and plotting the variation in z gives information on the sample. This mode is most commonly used because it prevents the tip from crashing into the surface if the surface isn't atomically flat. In the other mode, the tip height is held at a constant position, and the variation in the tunneling current is measured and plotted. Both these modes create a topographic map of the sample surface with atomic resolution (2 ÅLateral Resolution). With this resolution, the surface reconstructions of Ge(110) can be studied, and the phase(s) present can be identified.

1.3 Low-Energy Electron Microscope

The LEEM uses electrons backscattered off a surface in order to image the sample. The electrons are produced from a tip of lanthanum hexaboride (LaB_6), and then accelerated up to 20 keV.[5] As the beam passes through the illumination column, it is focused by three condenser lenses. The beam then passes through the magnetic separator, which deflects and focuses it onto the back focal plane of the objective lens. The separator also contains an illumination aperture which allows one to select between three aperture sizes: 50 μm, 100 μm, and 400 μm. The correct aperture is chosen by means of a linear translator in order to provide the appropriate brightness and resolution for the image. Once the electrons reach the objective lens, they are then decelerated to about 100 eV, and bombard the sample surface. The backscattered electrons are reaccelerated to 20 keV, and are deflected into the imaging column. The beam passes through a series of lenses which focus the beam onto a microchannel plate, which then amplifies the real-space image onto a phosphor screen. This image is recorded using a digital camera, which is able to record the image at 30 frames per second. The ability to record video of the surface is very useful in studying dynamic processes–such as island growth–on the surface while being able to vary temperature. The LEEM apparatus also has the ability to view Low-Energy Electron Diffraction (LEED) patterns, which allows for further analysis of the surfaces structure.

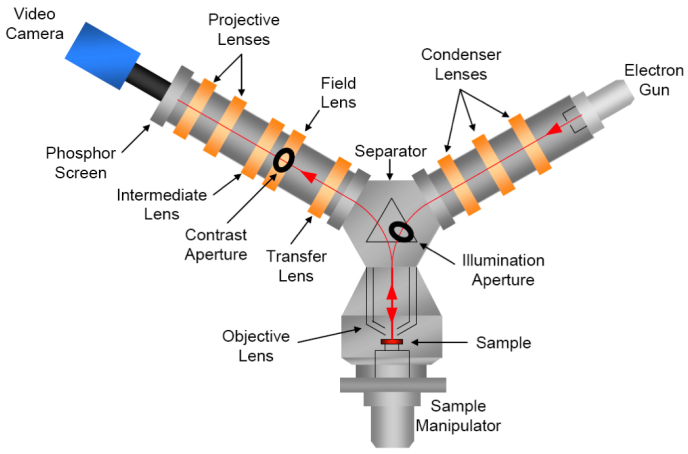

Figure 2: Elmitec LEEM III. Electron path is shown in red. Both the electron source and the sample are at a potential of -20kV, while all the lenses are at ground potential. The sample has an additional voltage which determines the energy of the incoming electrons. [6]

2 Experimental

2.1 Apparatus Set-Up

All measurements were made under ultrahigh vacuum (UHV), in a system containing three chambers: variable temperature STM (Oxford Instruments), LEEM (Elmitec), and x-ray photoemission spectrometer (XPS, Vacuum Generators)(Figure 3). Though the XPS was not actually run, the chamber was used for sputtering and annealing the sample. This system allows for the sample to be moved between measuring devices without having to be exposed to atmosphere, and then cleaned again.

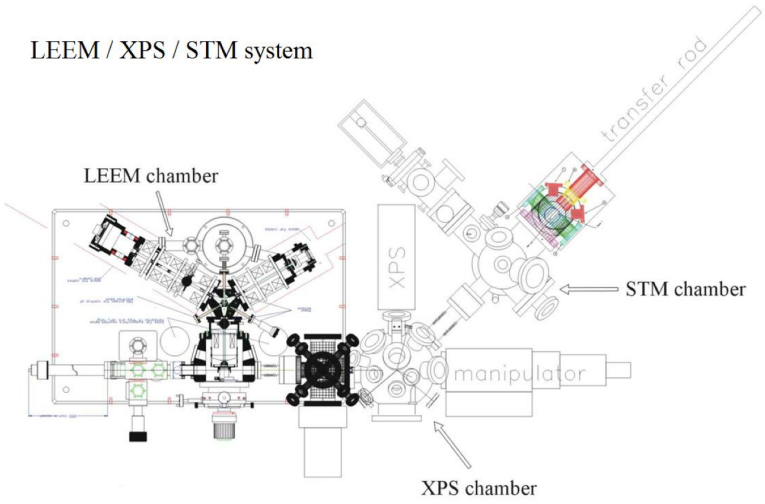

Figure 3: Configuration of LEEM/XPS/STM system. All chambers are under ultrahigh vacuum. Sample transportation was accomplished using two magnetic transfer rods. Samples are put into the system using a small entry chamber attached to the STM. [7]

2.2 Sample Holder Design

The sample holder used is unique to this system. It is a hybrid design between an Elmitec LEEM III holder and an Oxford Instruments VT-STM Sample Holder. The base is a normal Elmitec cartridge while the top plate was designed by Oxford Instruments in order to allow the holder to be compatible with both the STM and LEEM. This allows for the sample to be easily moved between both imaging devices. Another important aspect of this sample holder is its internal components. Directly underneath the sample, there is a tungsten filament, which is used to heat the sample in any of the three chambers. The sample holder also contains a thermocouple, which allows the temperature of the sample to be measured by measuring the voltage across the thermocouple.

2.3 Cleaning Procedure

The XPS chamber (Figure 3) contains an argon ion sputtering gun which was used to clean the sample. The sample was sputtered with Ar ions with energies of approximately 400 eV [1] for 15 minutes. The sample was then heated up to $\approx 800^{\circ}$C and annealed for 10 minutes [2]. The high temperature was achieved with a tungsten filament that is located directly under the Ge sample in the holder. A current of 2.7 amps was run through the filament, and an additional voltage was applied between the filament and the sample. This voltage allows for electrons to flow from the filament to the sample, thus increasing the temperature. The sample was put through cycles of sputtering and annealing, with the last anneal usually being 30 minutes.

[1] Sputtering gun used was a SPEC Ion Source IQE 11/35
[2] Temperature was measured using a infrared pyrometer

2.4 Scanning Tunneling Microscope Procedure

Once the sample has been cleaned a sufficient amount, it is placed into the sample stage in the STM chamber, and the entire three chamber system is supported by 3 laminar flow isolators to vibrationally isolate it. The tip holder is then put in place directly above the sample. Before taking any measurements, the tip and sample are allowed $\approx$ 1-2 hours to reach thermal equilibirum to minimize thermal drift. The stage is then lowered so that it rests on a four-spring system, which uses eddy currents to dampen any oscillations. Using a video camera focused on the sample surface, the tip is brought towards the sample in a rough approach procedure. The fine approach is controlled by the software, which slowly moves the tip until the correct tunneling current is reached. Once the tip is close enough, the tip is swept across the surface, and the software creates an image. [3]

The tips used in the STM are electrochemically etched from tungsten wire. This was accomplished by bringing the tip of the wire into contact with a solution of 3M potassium hydroxide (KOH) and applying a voltage between the filament and another electrode in the solution. The KOH conducts a current and reacts at the meniscus with the wire. A cutoff current is set to 8 mA. Once the current drops, and the reaction is complete, the resulting tip is examined with a microscope to check for quality in the sharpness of the tip. Once the sharpness is ensured, the tips are manually inserted into a syringe needle in the holders and are held in place by friction.

2.5 Low-Energy Electron Microscope Procedure

The dosers used to deposit Au onto the Ge sample are located in the LEEM chamber. Before dosing, a LEED pattern for the clean sample of Ge(110) was found. Then, the sample is dosed at room temperature for 15 minutes, which achieves approximately half of a monolayer of coverage. Initially, a diffraction pattern is measured from the newly dosed surface. The imaging method is then changed over from LEED to LEEM. Once the image is in focus, a current is run through the tungsten filament inside the sample holder in order to heat the surface. The temperature reaches a maximum value of 825 $^{\circ}$C [4], at which time the current is turned off, and the sample is imaged as it cools down. Once the surface cools, further analysis is conducted by changing the focus of the image to probe the underlying Ge(110) surface.

3 Results

3.1 Examining Clean Ge(110) with STM

After a Ge(110) sample had been cleaned using the process described above, the sample was allowed to return to room temperature, and then imaged with the STM. Images (Figure 4) show a zigzag configuration that is consistent with the c(8 x 10) reconstruction in the 3D model, and rows on the left which are consistent with the (16 x 2) reconstruction.

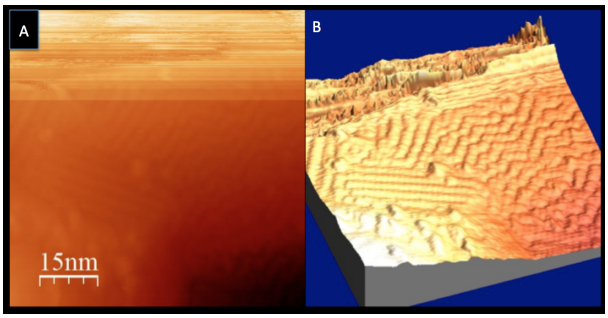

Figure 4: STM images of cleaned Ge(110) at room temperature. (A) Raw image. (B) 3D model created by STM analysis software. In (B) the zigzag configurations of the anatoms show a c(8 x 10) reconstruction, and the rows on the left appear to have the (16 x 2) reconstruction. (Diagram in Figure 1)

[3] Acquisition software used was Oxford Instruments TOPS3 program. Analyis was done with Nanotec WSxM sofware.
[4] Temperature is determined using voltage readings from a thermocouple in the sample holder.

Other features found on the surface of cleaned Ge(110) sample were two-sided, mound-like structures. It appears that these structures are completely composed of Ge, and do not contain any foreign material. As seen in the derivative image (C) in Fig. 5, one side of the mound is steeper than the other. This is most likely due to the angle at which the Ar ions hit the surface during sputtering, which causes one side of the mound to be more eroded than the other by the bombardment. Images (B) and (C) also show two smaller mounds. These could be the beginnings of larger mound structures, and upon further sputtering, they increase in size. If this were the case, then irregularities in the surface structure could be the origins of small mounds that become compounded upon further cleaning cycles and eventually lead to the larger mound structures seen in Figure 5.

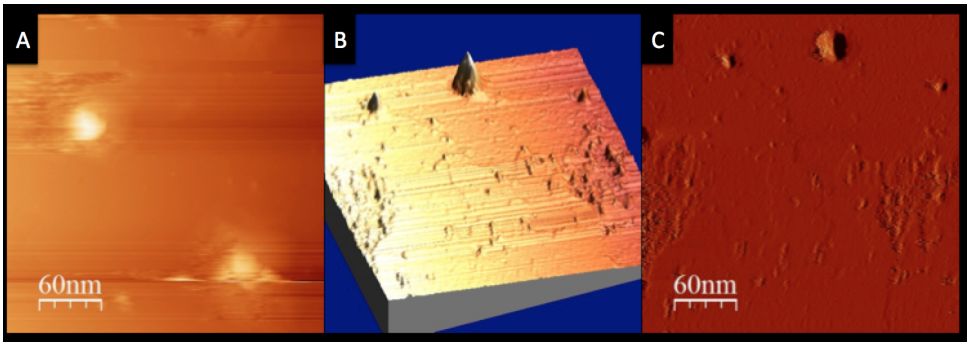

Figure 5: STM images of mound-like structures found on a cleaned Ge(110) surface at room temperature. Image (B) is the 3D model of (C) generated by the analysis software. Image (C) is a derivative image generated from the raw image. In (C), darker areas correspond to a steeper gradient. (B) contain two smaller mounds which could get larger with more sputtering.

3.2 LEEM Analysis of Au Growth

Au island growth on a Ge(110) sample was examined using the LEEM in the temperature range from 650 °C to 825 °C. As the temperature increases, these islands begin to coalesce into larger, 1-dimensional structures. At the highest temperature of about 825 °C, the islands are at their maximum size and are lowest in number. This behavior is similar to that previously studied for Ag on Ge(110) (Figure 6(D)). A notable difference is in the dimensions of the islands. The islands of Ag on Ge(110) appear to be much longer, and have stronger 1D character. This could be due to the higher Ag coverage of about 7.6 ML, compared with the 0.5 ML coverage of the Au.

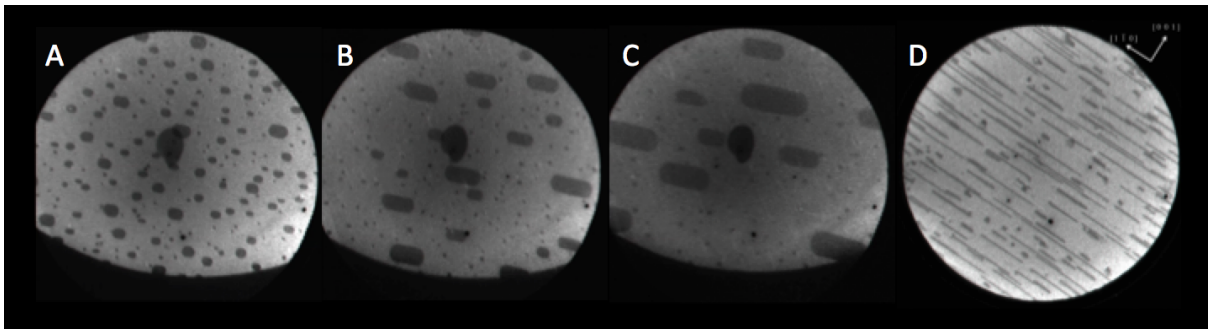

Figure 6: LEEM images of Au island growth as temperature increases. The dark spot in the center of each image is a burnt out component of the microchannel plate in the LEEM. This feature is present in all of the LEEM results presented in this paper. These images are in order of increasing temperature, with image (A) at the starting temperature of $\approx$ 650 °C, and image (C) measured at the maximum temperature of 825 °C. Image (D) is an earlier LEEM image of Ag islands on Ge(110) from Ref [5]. Both Ag and Au have 1D islands that increase in length and reduce in number with the temperature.

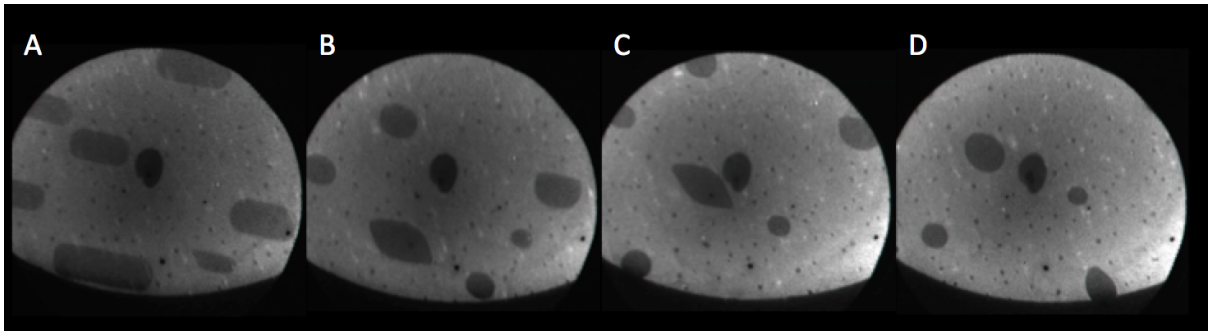

Figure 7: Island contraction seen with LEEM. As the surface temperature decreases, islands rapidly contract into more circular structures. Image (A) shows islands at maximum temperature (825 °C). As the sample cools, the islands reform into those seen in (B). Images (C) and (D) highlight the rapid reforming of the ovular island in image (C) into the circular island seen in (D).

The sample was allowed to cool once it reached the maximum temperature of 825 °C. Upon doing so, the 1D islands began to collapse back into the more spherical structures that had previously been observed. As seen in Figure 7, the islands observed at 825 °C immediately contract into the more circular forms that are common at lower temperatures. These contractions presumably occur in order for the Au to find lower energy configurations. Overall, the islands that remain after the sample had returned to a low temperature are still considerably larger than they had been upon initially being dosed. The number of islands is also closer to that which was observed at higher temperatures rather than those present after initially dosing. This shows it is more energetically favorable for the Au islands to coalesce into fewer islands that are larger in size. This observation is consistent with the commonly observed phenomenon of Ostwald ripening.

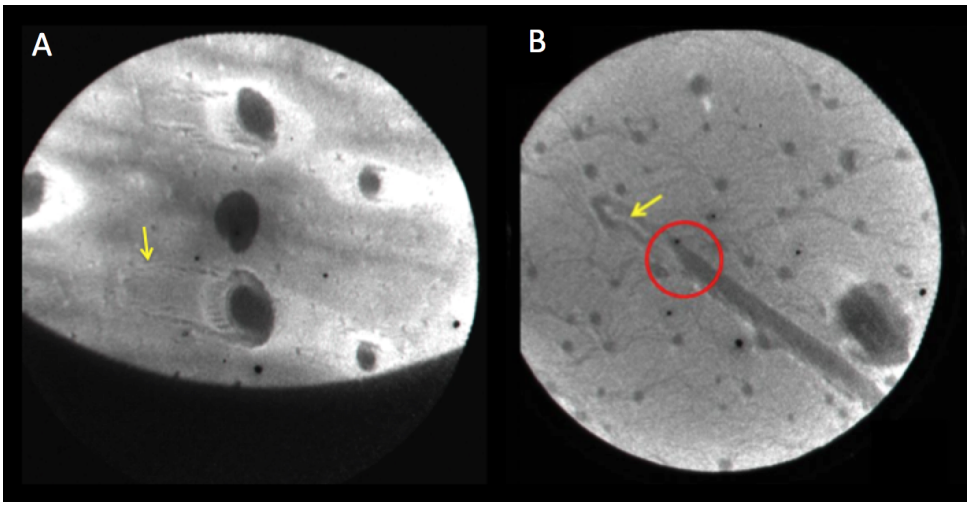

Figure 8: Island traces observed with LEEM once the Ge(110) sample had cooled down. Yellow arrows point to the the traces left on the Ge(110) surface. The red circle marks the end of the Ag island. (A) Shows the trace left behind by Au. Outlines can be seen around the remaining circular islands that resemble the 1D islands formed at high temperatures. (B) Previously observed "trails" left behind by Ag after contraction.[5]

Once the sample reached a low temperature, the surface was examined further with LEEM. After a change of focus, it was noticed that there were traces in the underlying surface that appeared to be outlines of the once-present 1D islands (Figure 8). These traces could have been caused by the rapid contractions of the islands as the temperature decreased. The speed of the contractions could have exposed the Ge(110) phase that is formed under the islands, and the outline seen could be a border between these two different phases.

4 Conclusion

The growth behavior of Au on Ge(110) was studied with LEEM. As the temperature increased, the Au islands culminated in larger, 1D structures elongated along the same axis. The high temperature behavior of the Au was notably similar to that of Ag on the same surface[5] (Au and Ag growth are compared in Figure 6). Once the temperature began to decrease, the islands rapidly collapsed back into more circular structures (Figure 7). Upon further examination of the cooled sample, outlines of 1D islands which had been present at high temperatures were observed (Figure 8). This feature had also been found for Ag growth on the Ge(110) surface. Overall, there were several similarities between the growth of Au and Ag on the Ge(110) surface, including island shape and behavior. There were also differences between the two, most notably the relative dimensions of the islands.

Future research could be conducted to analyze the mechanism by which metals nucleate on the surface of a semiconductor using not only LEEM, but STM as well. The high resolution STM could offer more details and information about the structure of the Au islands, as well as the structure of the underlying Ge(110). Also, the effects of varying some of the aspects of this process could be studied. The effect of the substrate temperature during Au deposition on the island formation could be studied, as well as the effect coverage on the relative sizes of the resulting islands. Further research could also investigate the variation of the cleaning parameters, such as studying the effect of sputtering energy on island formation. Understanding how these factors contribute to island size and shape would be beneficial in future work to self-assemble nanostructures on the surface of semiconductors.

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