Surface Imaging of Iridium on Ge(110)

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Epitaxial growth of metals on semiconductors is difficult to predict theoretically, making it best investigated on a case-by-case basis via experiment. This study looked at how the surface structure of Ge(110) changed when dosed with increasing amounts of Iridium at a variety of annealing temperatures. Previous work has examined the Ge(110) surface, but this is the first to use Iridium as the dosing metal. We used a scanning tunneling microscope to obtain detailed images of the surface, and found that Iridium dosed upon the sample formed small islands in one direction on the surface, except at high temperatures and coverages where the Iridium formed 1-dimensional islands in two directions.

Introduction

In the realm of surface physics, the study of epitaxial growth is important especially in an experimental context. One simple theoretical model assumes knowledge of the surface tensions of the deposit and substrate as well as the interface tension between them (Ibach, 180). From these quantities, the growth mode (Frank-van-der-Merwe, Vollmer-Weber, or Stranski-Krastanov) can be predicted. However, the tensions are not easily determined experimentally, and the number of possible patterns of epitaxial growth on a substrate far exceeds these three basic models. Therefore the experimental approach to investigating epitaxial growth provides the most insight for a given surface and deposit from which a physical explanation can then be hypothesized.

The experiment we ran used a Ge(110) surface deposited with Iridium. It was inspired by a previous study which found that Platinum and Gold deposits on Ge(001) formed atomic lines on the surface (Kockmann). Earlier studies in our laboratory examined Silver dosed on Ge(001), Ge(111), and Ge(110) (Mullet). As a transition metal near these elements in the Periodic Table, Iridium is a logical choice for further investigation. The choice to use a Ge(110) surface was made to provide more insight into this semiconductor substrate.

Sample Preparation

A single Ge(110) sample was used for all the measurements in this study. The sample was cleaned by a process of sputtering and annealing. The sample was sputtered by Ar at 0.3 keV for 15 minutes. The sample was then annealed by running a current through the sample holder at a high voltage. We calibrated this each time a new sample was put into the vacuum so that any location on the sample's surface would reach a temperature of at least 800 C during the annealing process. For our Ge(110) sample, this correlated to an applied current of 2.7 A and a high voltage of 190 V.

When the sample was first placed into the vacuum it was cleaned for eleven cycles of sputtering and annealing. Between experiments, the sample was cleaned six times, with a final half-hour anneal, to remove all deposited Iridium (and other contaminants) from the surface. The sample was then immediately dosed with the desired amount of Iridium. Previous calibrations using LEEM showed that 8 minutes of exposure would result in 1 monolayer (ML) of Iridium on the surface, and from this we could control the approximate coverage.

Once dosed, the sample was moved into the STM and annealed by running current through the filament in the sample holder. There was uncertainty in the temperature measurements due to a broken thermocouple in the sample holder. All annealing temperatures (except those for the 4.5 ML and 4.75 ML experiments which took place after we repaired the thermocouple) are approximated from other measurements correlating the applied current and resulting sample temperature. The sample remained at the maximum annealing temperature for five minutes and then was allowed to cool down to approximately room temperature (305 K or below) before imaging.

Measurement Techniques

We used a scanning tunneling microscope (STM) to investigate the surface. Our STM is suspended in a ultrahigh vacuum (UHV) chamber from three springs to reduce vibrations. The STM itself comprises an atomically sharp scanning tip that can be moved by three piezoelectric feet. A bias voltage is applied and causes a tunneling current of electrons from the tip to unoccupied states in the surface (or vice versa, depending on the direction of the bias voltage). The tunneling current I is related to the bias voltage V and height z above the surface by

$$I = Ve^{-A\sqrt{\Phi z}},$$

where A is a constant and Φ is the average work function of the tip and the sample that describes the difference in energy between an electron at rest just outside the surface and the most loosely bound electrons in the solid. Thus, we may obtain a real-space image of the surface structure by keeping either the height or the tunneling current constant. In our

experiment, we held the bias voltage at 2.0 V and tunneling current at approximately 0.5 nA, letting the height change.

The certainty in our results is limited by the fact that we only used an STM for measurement. In an ideal environment, the STM can obtain resolutions of 0.01 Å vertically and 2 Å horizontally. However, since the STM is dependent on the electronic structure of the surface, we may not be completely certain that the heights it measures are due to physical atoms in the surface. Furthermore, the way in which the STM was used in this experiment did not include spectroscopy of the atoms. Thus, although we assumed that certain surface features were composed only of Iridium atoms, this was not proven via other experimental measurements.

Certain measures were taken, both in the experimental set-up and procedure, to optimize the STM's performance. To minimize vibrational effects, the STM platform has magnets placed such that movements of the platform will induce eddy current damping. Furthermore, the entire vacuum chamber is mounted on three Nitrogen stabilizers, and the loudest vacuum pumps (our turbo and roughing pumps) were turned off for the duration of the imaging. In our preparation to use the STM, we made our own tips by chemically etching Tungsten wire with a 3-molar KOH solution. Since the tip quality may greatly impact the STM's imaging abilities, we were very selective with the tips, only using those which appeared under a microscope to have a nearly atomic point. We also made sure to replace the STM tip when the image quality decreased. Finally, we limited thermal drift effects in our images by allowing the sample to cool following annealing.

Results

We observed the effects of changing the amount of Iridium dosed onto the Ge(110) sample at different temperatures. For comparison, we examined the surface structure of clean Ge(110). The first trial examined 0.5 ML Iridium dosed at an estimated annealing temperature of 550 K. The next set of experiments were at approximately 650 K, where we compared the effects of dosing the sample with 0.5 ML Ir and 2 ML Ir. In the last two trials we observed a dosage of 4.5 ML Ir at annealing temperatures of 750 K and 850 K. Representative images of each trial are presented below in Fig. 1-4.

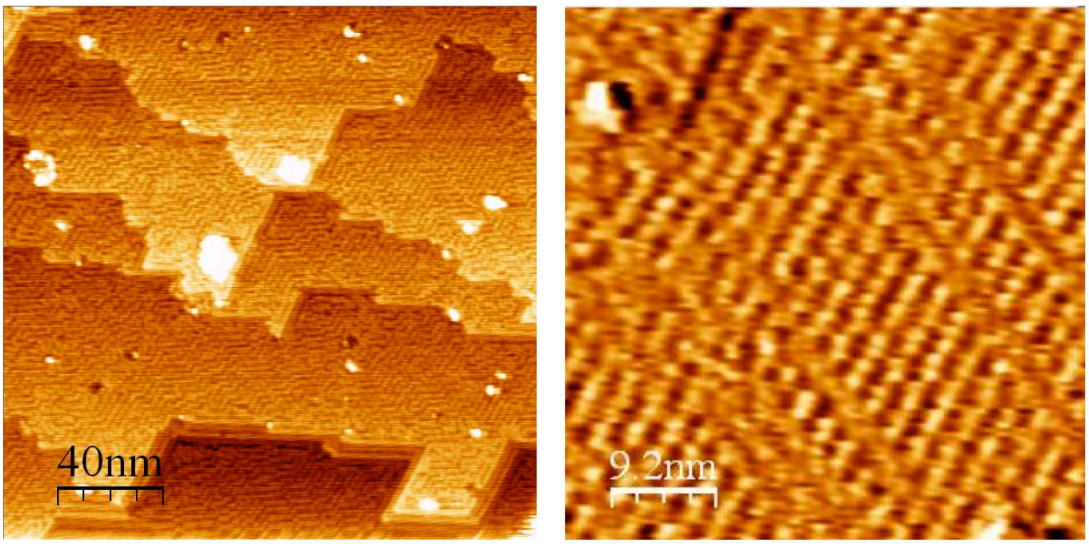

Fig. 1. The surface of clean Ge(110) demonstrates step edges (left) and a c(8 x 10) reconstruction with lines about 2.77 nm apart, as found by Mullet (2012). Each visible bright spot on the right is a five-adatom cluster.

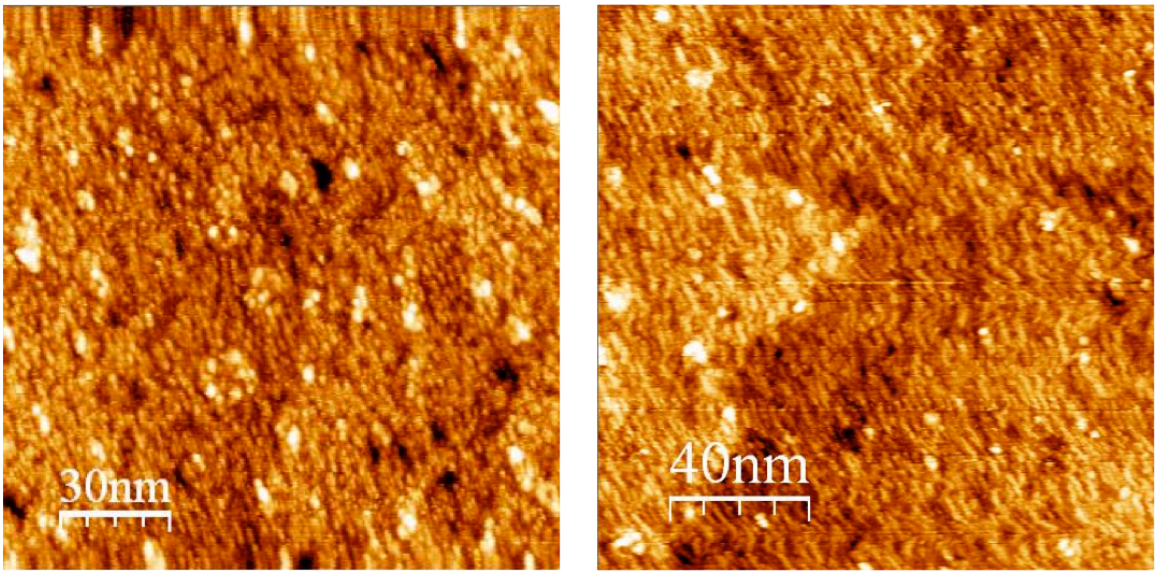

Fig. 2. Ge(110) dosed with 0.5 ML Iridium and annealed at 550 K (left) and 650 K (right). The higher temperature surface appears to have slightly longer islands that are better aligned.

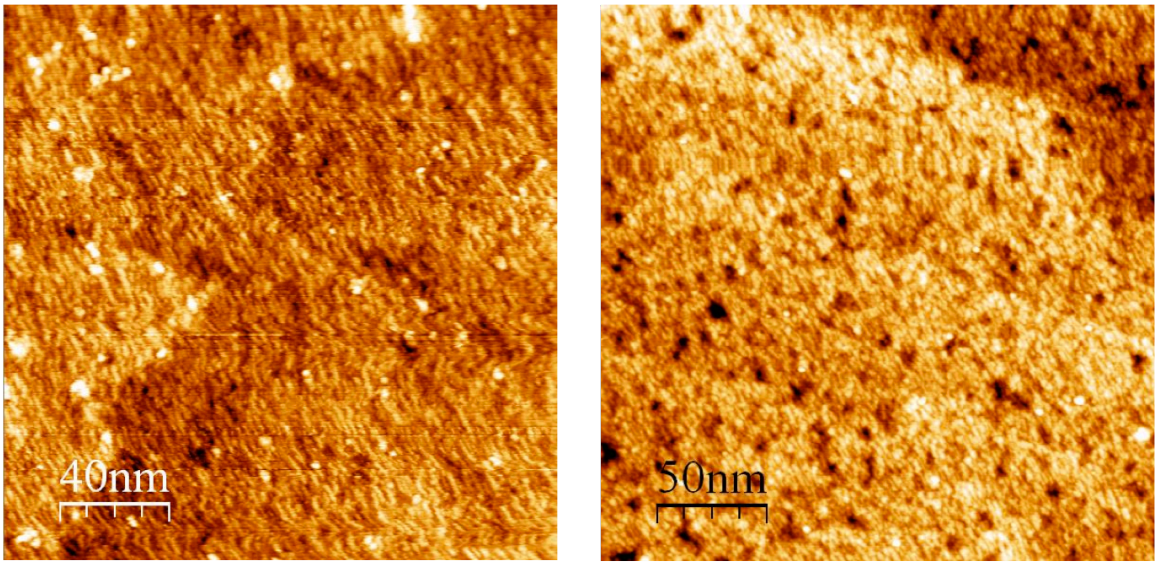

Fig. 3. Ge(110) dosed with 0.5 ML Ir (left) and 2.0 ML Ir (right) at approximately 650 K. Both dosages have small islands oriented in one direction with the distances between their peaks on average 2.7 nm or 4.2 nm apart. The higher dosage image shows more dark spots (holes) in the Iridium on the surface than the lower dosage.

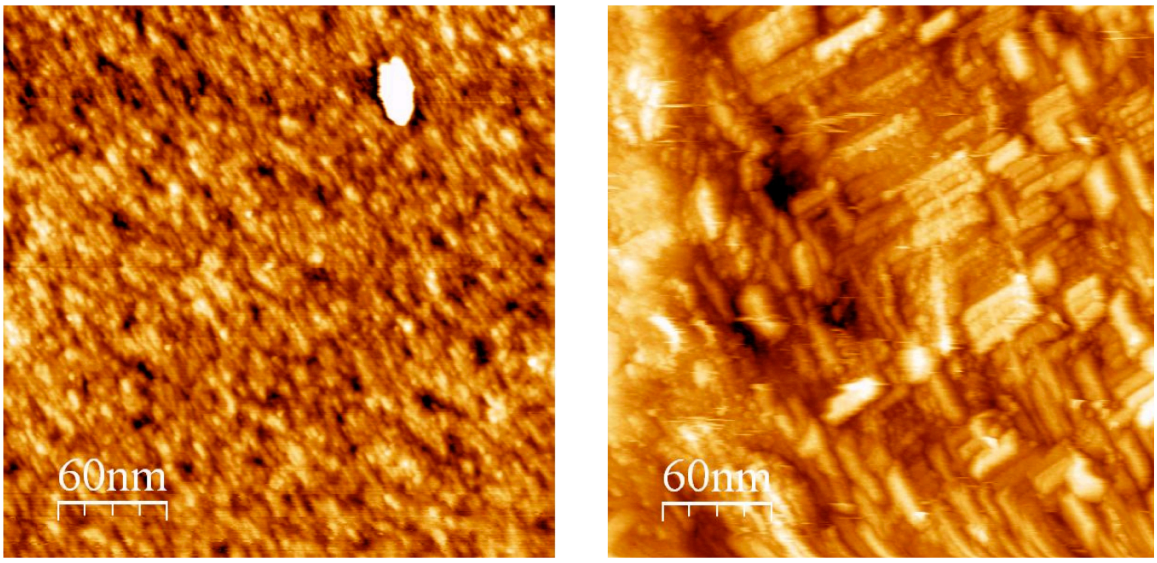

Fig. 4. 4.5 ML Iridium annealed at 750 K (left) and 850 K (right). The lower annealing temperature yielded small islands about 3.3 nm or 4.5 nm apart, while the higher temperature created islands around 7-9 nm apart. The higher temperature surface also had islands oriented in two directions perpendicular to each other. Larger islands were observed in both of these high-temperature high-dosage trials: the island in the top right of the 750 K image is 27.5 nm long and 15 nm wide, while a huge 938 nm long island (not shown) was observed on the 850 K surface.

Conclusion and further work

From the results shown above, we were able to draw three preliminary conclusions about the behavior of Ge(110) surfaces when dosed with Iridium. First, Iridium in small amounts at lower temperatures forms very small one-dimensional islands in one direction. Higher annealing temperatures at low Iridium coverages yield longer islands that are somewhat more aligned. At high coverage, annealing at higher temperature results in one-dimensional islands aligned in two perpendicular directions. Therefore, there were visible trends associated with changing both the amount of Iridium dosed onto a surface and the temperature at which the dosed surface was annealed.

Unfortunately, the breakdown of our STM limited our data to that described above. For more concrete conclusions we should expand the range of our trials to cover more high temperature anneals. For instance, would low coverages also exhibit islands oriented in two directions if they were annealed at 850 K or higher? With this sort of trial, we could attempt to distinguish which effects are due to temperature change and which are due to more monolayers of Iridium. In this particular experiment, we could also change the annealing time to see whether it impacts the surface organization.

Further research may also investigate epitaxial growth related to that investigated in this experiment. One avenue of research would be the effect of dosing with other materials, such as silver which was previously used in our lab. Other Germanium surfaces, such as Ge(100) could also be experimented upon.

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