Scanning Tunneling Microscopy Studies at UC Davis

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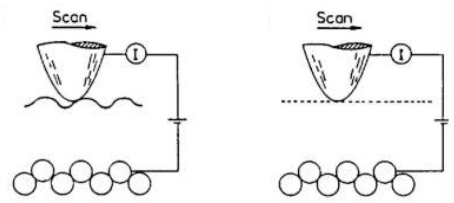
Abstract: This research project used two different scanning tunneling microscopes (STMs) to study crystalline semiconductor surfaces. Preliminary observations of silicon (111) were conducted in an attempt to calibrate the z-axis of one scanning tunneling microscope in preparation for a new experiment. Additionally, germanium (111) was studied with the aim of verifying periodicities in domain boundaries observed in a previous experiment at this facility. The Si(111) sample proved calibration proved impossible due to mechanical problems with the STM and the ultra-high vacuum (UHV) system containing it. The Ge(111) sample was successfully scanned and atomically flat terraces were observed, but more detailed observations of the surface were not possible. While both experiments experienced technical setbacks and mechanical problems, the principles they are based on are sound and only superficial changes should be necessary in both systems to achieve the desired results.

Introduction: Scanning Tunneling Microscopy has been successfully employed since the early 1980's to study both electronic and topographic characteristics of conducting and semi-conducting surfaces. One of the most important aspects of scanning tunneling microscopy is that it yields three-dimensional images of surfaces with resolution of individual atoms. To generate topographic images, a conducting tip is brought close enough to the surface for a tunneling current to be established[1]. The current varies exponentially with the separation between the surface and the tip so even a slight change in the distance between the two greatly effects the current. STM can be generated in two different modes, constant height mode and constant current mode. In constant height mode, the current, and therefore the separation between the sample and the scanning tip, is set at a fixed value and the displacement of the tip from some reference position is monitored as the surface is scanned. In constant height mode the tip position is fixed and changes in the current are recorded as the sample is scanned. The constant current mode was the first mode developed for scanning tunneling microscopy and allows observation of surfaces that are not atomically flat[1]. The constant height mode allows for much faster imaging of surfaces, as long as they are atomically flat[1]. Schematic views of both modes are depicted in Figure 1. All scans in the course of this research were conducted in constant current mode.



(a) (b)

Figure 1: Schematic representation of a scanning tunneling microscope tip in (a) constant current mode and (b) constant height mode.

Experiment: The first half of this research project focused on obtaining STM images of a Si(111) sample. The 7x7 reconstructed surface of Si(111), a surface state obtained by careful annealing of the silicon sample, has a widely-studied surface topography with periodic height variations that are known very accurately. This makes a Si(111) 7x7 sample ideal for calibrating the z-axis of an STM before a new experiment is run. Several mechanical malfunctions in the STM system, including the presence of high-frequency resonant noise in the scanning tip, caused this area of research to remain incomplete. The STM in which the silicon scans were conducted is depicted in Figure 2.

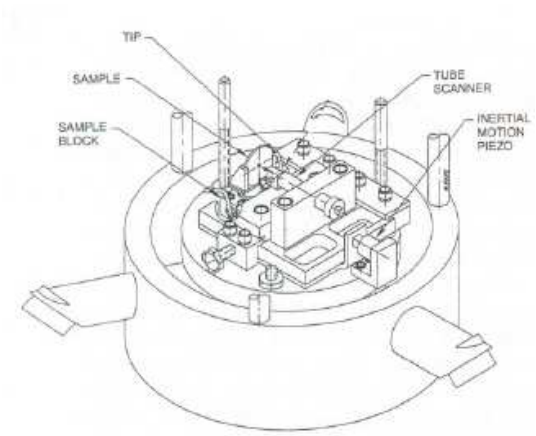


Figure 2: The Scanning Tunneling Microscope (STM) used for scanning Si(111).

The second half of this research project focused on preparing and scanning a germanium (111) sample to verify certain domain periodicities observed in an earlier experiment. Preparation of the germanium sample involved many cleaning cycles. The first step in the cleaning cycle is sputtering the sample with argon ions to remove surface impurities such as oxygen, nitrogen and carbon. The argon used for this purpose was leaked into the chamber at 1.0 x 10^{-5} Torr (the base pressure of this UHV chamber was ~10^{-9} – 10^{-10} Torr) and held at a beam energy of 0.25 keV for fifteen minutes. Germanium is particularly sensitive to ion bombardment and if the incident ions are too energetic the surface becomes so severely pitted that annealing is not possible and there no atomically flat areas remain. After sputtering the sample was annealed between 750 °C and 850 °C for fifteen minutes. The melting temperature of germanium is around 930 °C. During the course of this experiment fifty-three cleaning cycles were carried out.

The UHV system in which the scanning, as well as the sputtering and annealing, of the germanium sample was conducted is shown in Figure 3.

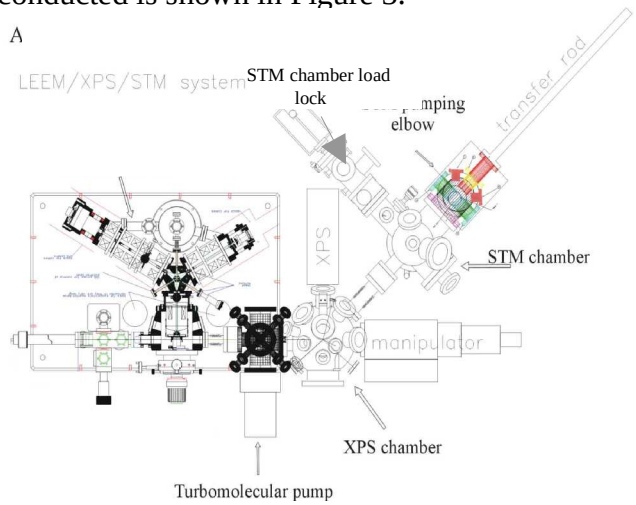


Figure 3: The UHV system in which the Ge(111) sample was prepared and scanned. The X-Ray Photon Spectroscopy (XPS) chamber was where the sputtering and annealing took place. XPS analysis was used to determine if the sample was free of contaminants. This chamber system is also equipped with a Low Energy Electron Microscope (LEEM) which is able to analyze the crystal pattern of a surface by measurement of low-energy electron diffraction.

Results: None of the silicon scans that were taken yielded repeatable results. The resonant noise present in the tip made observation of surface features impossible and several times the pressure in the chamber got too high (~10^{-8} Torr) to keep the silicon clean.

The germanium scans gave preliminary results that showed atomically flat terraces on the surface. These preliminary observations are shown in Figure 4.

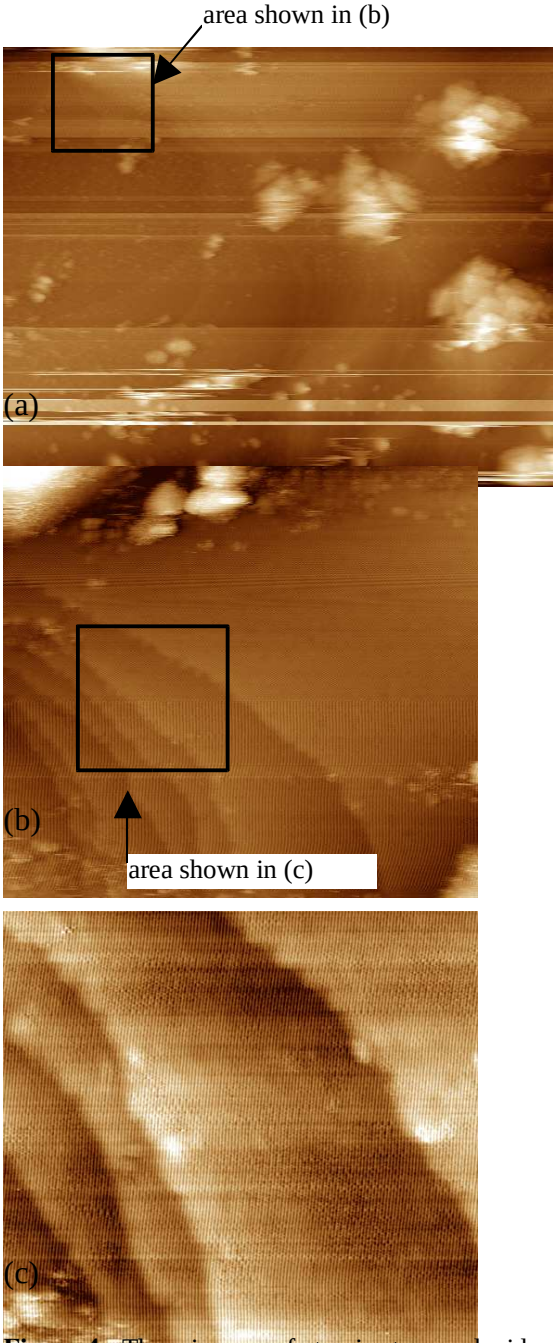


Figure 4: Three images of atomic steps and wide, atomically flat terraces on Ge(111). The images are (a) 3000 Å square, (b) 1000 Å square, and (c) 500 Å square. The white spots visible all through (a), as well as at the top left of (b) and the far right of (c) are imperfections in the surface. The horizontal streaks in (a) are due to problems with the tip scanning large areas and not features of the sample surface.

Taking more detailed scans (the goal was resolution on the order of a few Å) was impossible because the scanning tip began to resonate too much to distinguish real surface features from the noise.. The scanning tip in the STM was replaced as soon as possible, however, time was needed to transfer the scanning head from the STM chamber into the load lock (see Figure 3) and then allow the load lock chamber to reach atmospheric pressure before venting. The tungsten scanning tip was replaced and the entire scanning head was back in the STM chamber within a matter of hours. No further evidence of terraces was found and it was soon determined that the sample itself was damaged beyond repair and a completely new germanium sample would be needed to generate more detailed images.

Conclusions: The specific experiments conducted during this research program failed because of technical difficulties, but they are still valid learning experiences and necessary steps to more in-depth study of surfaces and molecules adsorbed on them. The next step in the silicon experiment is to find and eliminate the source of the resonance in the STM, this involves checking the electrical connections for any strain on the wires between the tip and the feedback system, as well as making sure the piezo tube scanner (see Figure 1) is situated properly in the STM. Finally, once the adjustments are made, the z-axis calibration can be completed and the actual experiment (observation of the step-wise formation of benzene on a Palladium (111) surface) can begin.

The only way to complete the germanium experiment is to prepare an

entirely new sample and go through the preparation process all over again. While atomically flat terraces were observed, a cleaner and undamaged sample is needed to detect the desired phenomenon.

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Sources:

[1]P. Hansma, J. Tersoff, J. Appl. Phys. 61, R1- R23 (1986).