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Scanning Tunneling Microscope Studies of Surface Defects

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1. Project Objective

Experiments proposed for our UHV Scanning Tunneling Microscope systems focus on the study of surface roughness and defect formation. Our objective is to study the formation of surface defects of the nature where surface crystalline structure is maintained but periodicity is not. Two complementary systems will be studied, one where defects are thermodynamically produced on stepped vicinal surfaces of silver, and a second system where epitaxial growth — surfaces grown using a commercial molecular beam epitaxy (MBE) apparatus — on GaAs substrates produces kinetically induced surface roughness. STM as a real space measure can probe defect type and distributions, information not available to surface diffraction techniques which measure a complementary value of averaged surface disorder. Our interest is the study of the energetics of these processes, both of which have important technological implications.

Presented in this report will be a description of the current state of development of our experimental instrumentation and its planned completion. Following that is a discussion of the results obtained so far in the development of our STM. We conclude with a short description of experiments we will be prepared to perform once our system is completed.

2. STM Instrumentation

We have constructed two complete UHV microscope systems. A single set of feedback / signal processing electronics and computer hardware / software has been designed and assembles to be shared between the two tunneling microscopes. Considerable development of vibration measurement and isolation has been necessary for the successful operation of our STM.

Our original UHV microscope system is shown in figure 1. This microscope has been operational over two years and all data presented in this report are from this system. The vibration isolation for this system is accomplished externally with pneumatic isolation legs, and internally with a single stage in vacuum isolated by three viton O-rings. This microscope is constructed to use mechanical motion for the coarse sample-tip approach and

a single tube piezo mounted vertically for scanning. The chamber has only limited sample processing and characterization and no provision for a sample load lock.

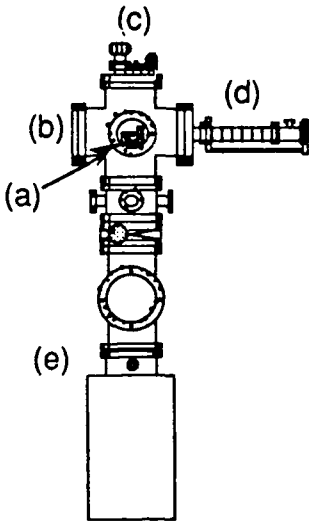

Figure 1. Schematic of Original Single Chamber STM Vacuum System. (a) Tunneling Microscope inside chamber. (b) Sample Processing: Temperature Measurement and Sputter. (c) Microscope Mechanical and Electrical Feedthroughs. (d) Sample Manipulator and e^- beam heater. (e) TSP and Ion Pumps.

Our second STM system which is almost completed is shown in figure 2. Here we have expanded on the original design to construct two separate and isolable chambers. The first chamber includes a load lock, sample processing station for heating and sputtering of samples, a rear view LEED, and a CMA for Auger spectroscopy. Samples are transfered to the second, STM chamber using a magnetically coupled transfer rod and wobble sticks. This microscope is also vibrationally isolated using external pneumatic legs for the entire system, however the internal isolation is an superior two spring stage system with eddy current damping. The coarse approach is accomplished here with a piezo inchworm motor and is controlled externally by computer. As with our first version microscope, scanning is accomplished with a single tube piezo mounted, however, horizontally in this design. Figure 3 shows the specifics of the microscope chamber design. This microscope and vacuum chamber

design has been carefully planned to allow use of laser surface heating of samples in the microscope assembly.

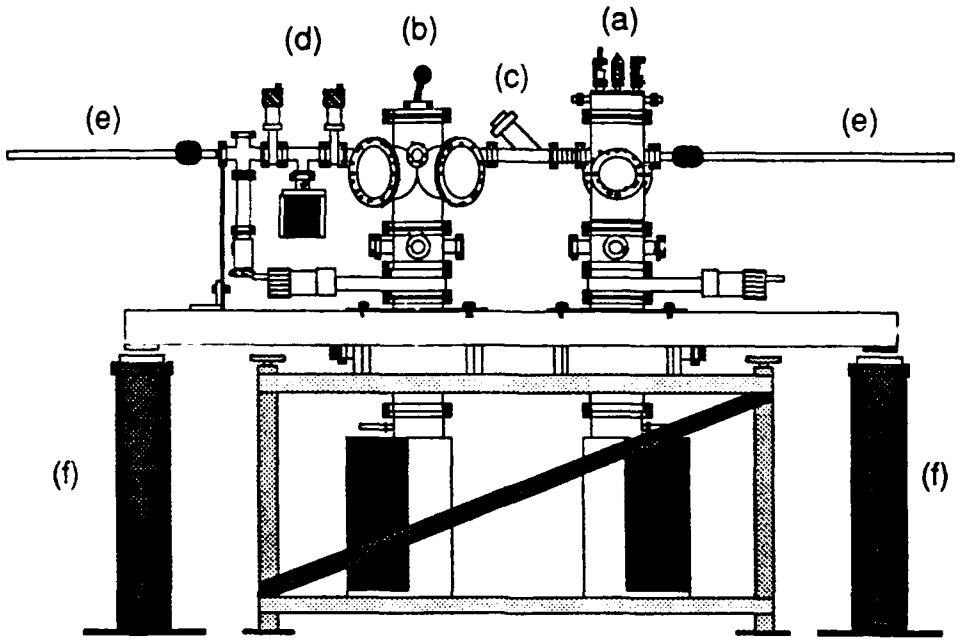

Figure 2. Schematic of second double chamber STM Vacuum System. (a) STM chamber. (b) Process Chamber. (c) Isolation Valve. (d) Load Lock. (e) Linear Sample Transfer. (f) Pneumatic Vibration Isolation.

As stated this system is nearly complete. Construction of the processing / characterization chamber with load lock is finished with the exception of the sample heating and sputtering stage. The microscope chamber is complete and the microscope unit it self has been constructed and is currently being tested and refined. It will be ready for use shortly.

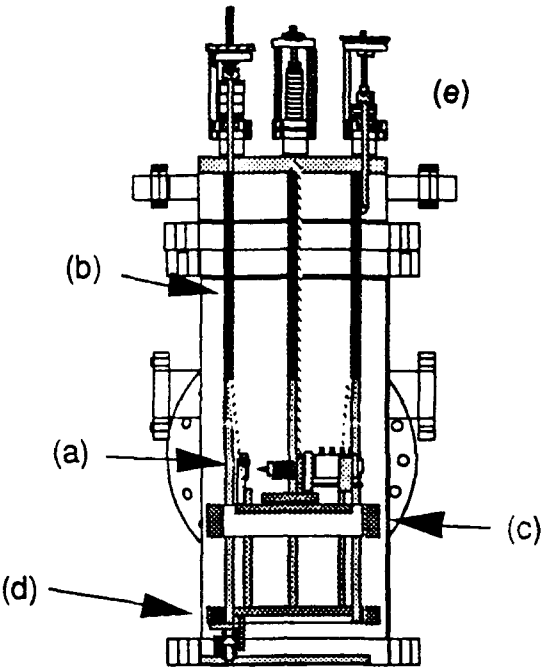

Figure 3. Schematic of New Microscope Chamber with Vibration Isolation. (a) Tunneling Microscope Sample, Tip, and Approach Motor. (b) Vibration Isolation Springs, and Support Rods. (c) Two Stage Vibration Isolation. (d) Eddy Current Damping. (e) Microscope Mechanical and Electrical Feedthroughs.

Temperature measurement for sample processing in both systems requires a non-contact method because of the need to transfer the sample between different stages in the vacuum chamber. An optical pyrometer has been purchased and successfully used in initial experiments on silicon surfaces. This equipment will allow us to do programmed temperature processing of all our samples in either chamber.

We have designed, tested and modified all our feedback and sample processing electronics. Current measurement is accomplished with a preamplifier with noise <50pa, and selectable gains of 10^8, 10^9, and 10^{10} V/A. Tunneling bias is selectable from ±15V to ±1mV. Current measure and control uses an absolute value circuit with linear or logarithmic input to the integration stage. Gain and time constant of the integrator is selectable, and a computer controlled hold and sample circuit is included. Piezo control is accomplished with four high voltage channels mixing two input channels into four output

channels, Z+X, Z-X, Z+Y, and Z-Y. Special Z position control circuitry allows a 'fast withdraw' for controlled, safe coarse sample tip approach. A signal amplifier conditions the Z position signal for measurement by computer hardware.

For experiment control, data acquisition, storage and display we are using an IBM AT system. Experiment control and acquisition uses and IBM data acquisition board. The display is a high resolution graphics board (1280x1024 pixels with 256 displayable colors) and a matching monitor. Data storage is accomplished using Bernoulli Box technology and two exchangeable 20 megabyte disk drives. Software developed for this system is menu driven with easy entry of experimental parameters. The data is acquired in sizes up to 256x256 points with 12 bit resolution and up to 16 samples per point for signal averaging. Data is displayed real time and 'leveled' before storage in one of 10 RAM image storage locations. Quick file storage and retrieval is also included. All software for this experiment has been developed on the AT using C programming language and assembly code programming.

Vibration proved to be an early problem in our development of an STM and one which required considerable development to measure, understand, and solve. We have used a borrowed accelerometer and software developed to take the fourier transform of acceleration data to measure the frequency spectrum of vibration. With those results we determined that pneumatic optical table legs are a necessary first stage of isolation for our vacuum chambers. We have also tested and refined our two isolation systems for the in vacuum stages supporting the tunneling microscopes. We plan to purchase our own accelerometer in the next year to continue improving our vibration isolation.

To accomplish the study of thermodynamically induced surface defects we plan to use pulsed laser surface heating to induce kinetically frozen high temperature surface configurations. We have purchased and received a Nd:YAG laser with variable pulse lengths. The laser is ready for use with the new microscope and all the optical components necessary have been purchased.

3. Current Results

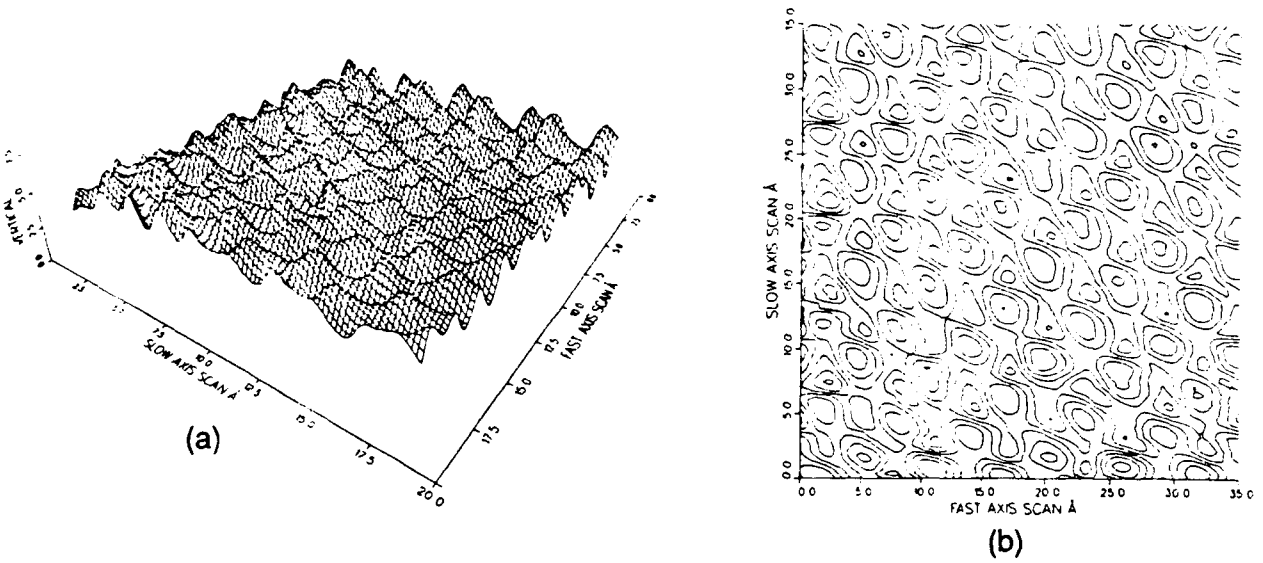

Figure 4. Highly Oriented Pyrolytic Graphite. Constant Current Image. a) Line Scan representation, b) Top Down Topographical View.

Our current microscope has been successfully used on four surfaces. Initially we tested the microscope on highly oriented pyrolytic graphite samples. Figure 4 shows the hexagonal structure observed on this surface. Later we observed the silicon (111) 7x7 reconstructed surface after careful anneal cycling. Initial images of As capped GaAs surfaces were obtained on test samples. And initial work on the Ag(115) and (117) surfaces has indicated the need for LEED and AES characterization of metal surfaces to insure that they are free of contamination and annealed showing well ordered diffraction patterns.

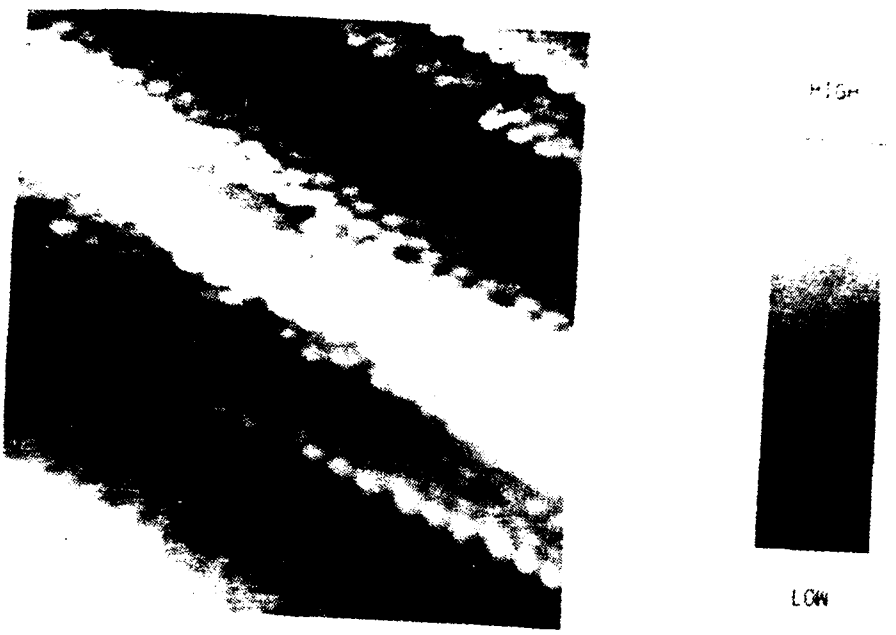

Figure 5. Si(111) 7x7 surface with steps and terraces. Image size 130Å horizontal and 240Å vertical. V_{sample} +1.5V and I_{tunnel} = 0.5nA.

Figure 5 shows a typical scan of Si(111) 7x7. Using such images we have calibrated the lateral and vertical motion of our piezo positioner. From this we have determined the scan to scan vertical stability of our microscope to be better than 0.1Å and the lateral drift at ≈ 2.5Å / min. Figure 5 is an example of the scan to scan stability of our present microscope. Because the lines represent points acquired on two separate scans the smoothness of the lines indicates the scan to scan stability. Two adjacent lines show the measured variation of the surface structure from scan to scan.

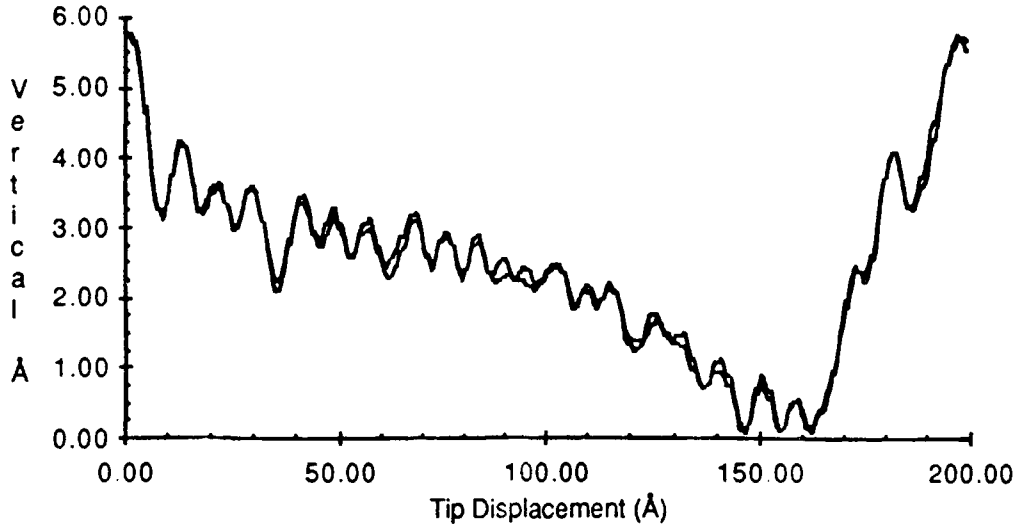

Figure 5. Single scan lines from a Si(111) 7x7 surface. Two adjacent lines in direction perpendicular to the scanning direction.

Preliminary STM images have also been obtained on MBE GaAs and silver surfaces. The GaAs results showed that we could successfully image surfaces grown in a MBE apparatus and protected for transfer in air to our STM chamber by a passivating arsenic layer deposited after epitaxial growth. A line scan representation of these results is shown in figure 6. In the foreground of this image are several well resolved steps on the surface. We plan to pursue this technique to study the effect of varying growth process parameters on surface quality.

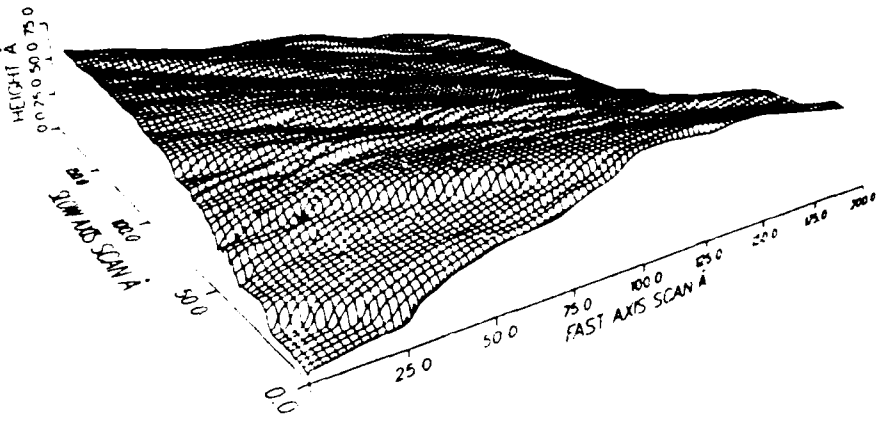

Figure 6. Line scan of MBE GaAs(001) test sample after removal of As passivation layer.

Preliminary images of the Ag(115) and (117) surfaces showed that we were able to achieve well resolved images but without the availability of LEED and AES we were unable to otherwise characterize the surface. We show on image of the Ag(117) surface in figure 7. The surface is not well ordered in this image. The top band represents a lower resolution state of the tip which changed during scanning. The vertical rows represent corrugation observed in the horizontal direction with alternating phase respective to the next underlying layer. No corrugation is observed vertically.

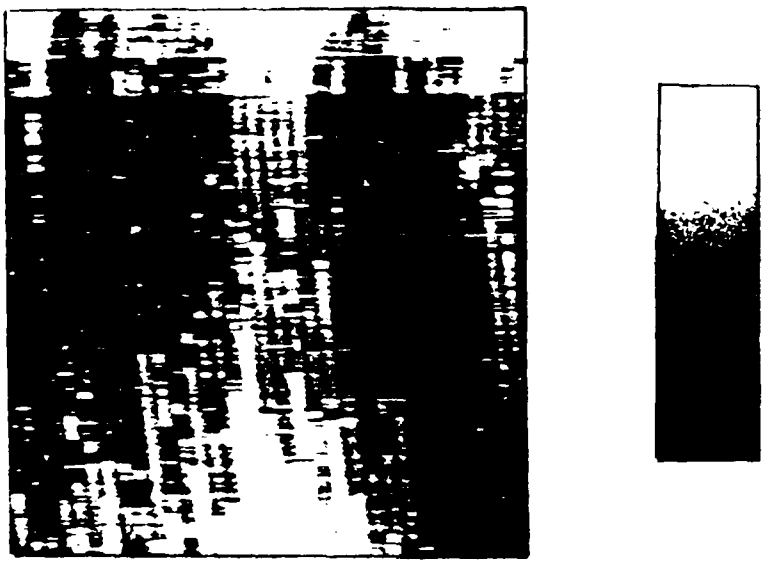

Figure 7. Grey scale image of Ag(117). Image size is approximately 750Å x 750Å with the grey scale representing 3Å change in height (black is low).

4. Planned Experiments

We have two STM systems, one now operational which has produced images of silicon, and a second close to completion for use in our studies of metal surfaces. The first system is appropriate for STM investigations of surfaces otherwise not requiring extensive characterization. Such experiments include the As capping procedure on GaAs samples, investigations of cleaved GaAs surfaces and investigations of the early stages of metal deposition on silicon. Once operational the second system, utilizing the same electronics and computer control as the first system, will allow us to better characterize the metal surfaces and perform the pulsed laser heating experiments necessary to obser . the high temperature defect distributions on vicinal metal surfaces. In short the development of our STM measurement system is nearly complete, and with the experience gained so far we plan to shortly begin the scientific investigations described in our proposals.