An ultrahigh vacuum scanning tunneling microscope with interchangeable samples and tips

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Our ultrahigh vacuum scanning tunneling microscope (STM) is the first instrument in which both the samples and tips can be replaced *in situ* and analyzed by routine surface analysis techniques. The STM chamber is connected by a transfer chamber to a VG Escalab surface analysis system equipped with scanning Auger microscopy/scanning electron microscopy, x-ray photoemission spectroscopy, low-energy electron diffraction, and sample cleaning and preparation facilities. We describe the design of the instrument in detail and show high-resolution data which exemplify its performance and lead to determinations of the structure of several surfaces: $(\sqrt{3}\times\sqrt{3})R$ 30° Ag/Si(111), $(\sqrt{19}\times\sqrt{19})R$ 23.4° Ni/Si (111), and Au(111) thin films on mica.

I. INTRODUCTION

The scanning tunneling microscope (STM) is rapidly becoming an established technique for studying the atomic scale structure of solid surfaces.[1] Our research objective has been to use the STM to measure the surface structure of well-characterized single-crystal samples with submonolayer coverages of metal films or adsorbed molecules. For such studies, it is extremely desirable to have an ultrahigh vacuum (UHV) STM equipped with conventional surface analysis instruments which facilitate the sample preparation process. In our laboratory, we have built a STM which is mounted onto a single UHV flange in a vacuum chamber which is connected by a transfer chamber to a large surface analysis system with sample cleaning and preparation facilities. Our system is unique in that both samples and tips can be quickly moved throughout the combined vacuum system and analyzed.

Our STM design follows the basic principles of the early successful designs of Binnig and Rohrer,[2,3] including a double-spring suspension, a piezoelectric tripod for scanning the tunneling tip, and a piezoelectric louse walker to move the sample without coupling in external vibrations. An earlier version of our instrument has been previously described.[4] In this paper, we explain how improvements in the design of the current instrument lead to extremely high stability and low noise in the STM topographic images. We also describe in detail our sample and tip movement mechanisms. We have used this UHV STM to study several metal-on-semiconductor systems.[5,6] More recently, we have used this instrument to resolve single close-packed atoms on a Au(111) thin film.[7] We show some data here on these systems to demonstrate the performance of the instrument.

II. INSTRUMENTATION OF THE STM

The layout of our vacuum system is shown in Fig. 1. The left-hand side of the figure shows our VG Escalab Mark II. The surface analysis chamber is equipped with 500-Å resolution scanning Auger and scanning electron microscopy (SAM/SEM), a dual-anode Al/Mg x-ray source for x-ray photoemission spectroscopy (XPS), and an argon ion gun for depth profiling and ion scattering spectroscopy (ISS). The sample preparation chamber contains an airlock, an electron beam heater, a high-energy argon ion gun for sputter cleaning samples, rear view low-energy electron diffraction (LEED) optics, and an evaporator for producing submonolayer coverages of metal films on heated substrates. The right-hand side of Fig. 1 shows the sample transfer chamber and the STM chamber which we have connected onto the Escalab. The sample transfer chamber contains a rotatable rack and pinion mechanism which can move the sample from this chamber into any other which is connected radially to it. A 0.45-m-diam chamber to the right holds the STM, several large windows, a medium range optical telescope and a fiber optic bundle to carry light for observing the tip-to-sample approach, and several flanges with mechanisms associated with sample movement.

Our STM is also unique in combining the two common methods of vibration isolation. The STM, shown in Fig. 2, has its principal elements mounted onto the top plate of a stack of stainless-steel plates separated by Viton spacers, like the "pocket" STM design.[8] This stack is mounted onto double-spring stages with magnetic eddy current damping, like the earlier STM models in Zurich,[2,3] in order to reduce most of the vibrations to frequencies <2 Hz.

The coarse positioning of the sample is accomplished in our instrument by using a piezoelectric louse walker,[2] as shown in Fig. 2. Our louse has a 3-mm-thick piezoelectric body and anodized aluminum feet, and is similar to ones which have been described previously.[3] Our sample is mounted onto a cylindrical tantalum stub, described in more detail below, which sits in the center of the top plate on the louse. A small screw can be tightened to hold the sample holder in place.

In our instrument, the tip is scanned using small piezoelectric tubes, 3.17-mm diameter and 25.4 mm long. The small wall thickness of 0.50 mm gives these tubes a sensitivity of ~ 100 Å/V. Since these tubes are repoled by ~ 500 V, they could be used with voltages from -300 V to $+800$ V, giving a possible total motion of 11 μ, although the usual working range with our electronics is 3 μ.[4] To make the piezoelectric scanning tripod shown in Fig. 2, three orthogo-

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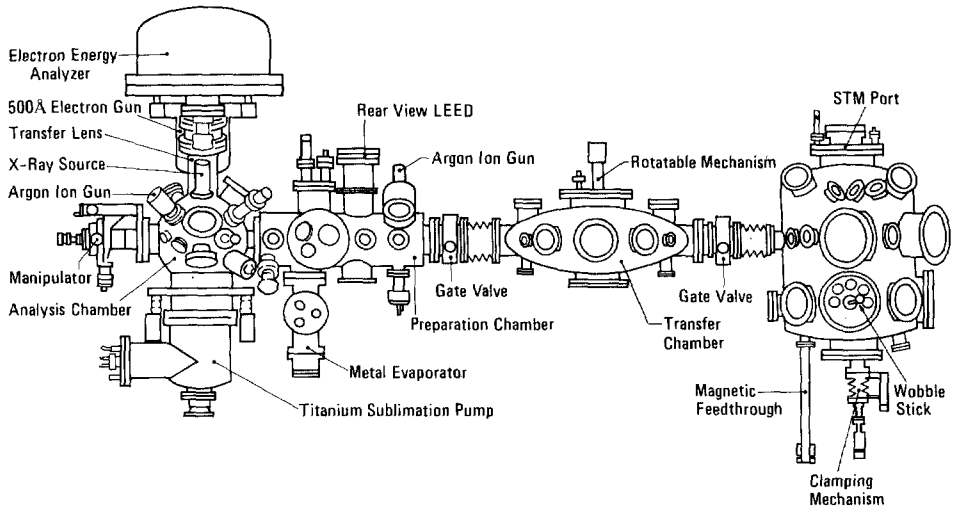

FIG. 1. Layout of our UHV surface analysis and STM system, with VG Escalab analysis and preparation chambers on the left, and transfer chamber and STM chamber on the right.

nal tubes have been soldered to a small cube of stainless steel using a silver–tin alloy solder with a melting point of 200 °C and a water soluble acid flux. To that cube is soldered a metallized aluminum oxide insulator and then another stainless-steel cube containing a slot for the tip.

We note that our STM and surface analysis system are extremely reliable. Using the airlock with our interchangeable samples and tips, our instrument has been operational and able to take high-resolution data on a series of different samples for one and one-half years, with the STM chamber opened to atmosphere only once during that time period.

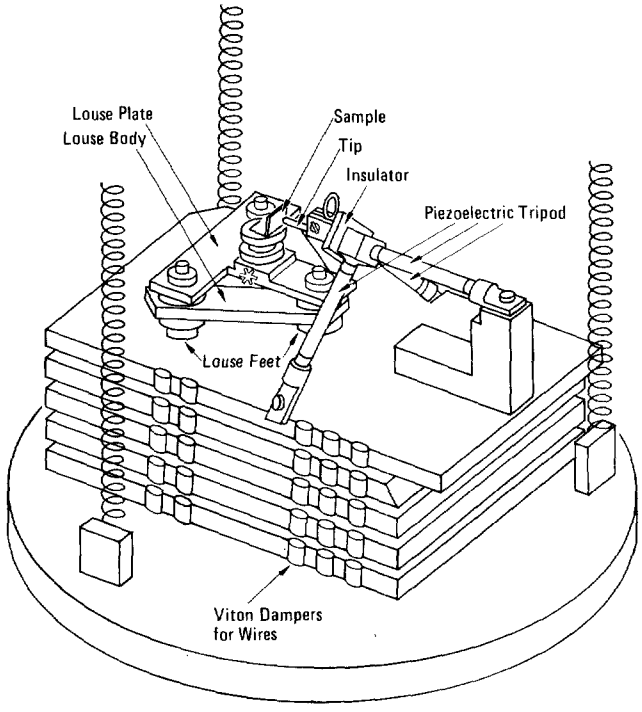

FIG. 2. Schematic diagram of our STM, showing pocket STM hung on double-spring stages. The louse piezoelectric walker carrying the sample is on the left, and the piezoelectric tube tripod for scanning the tip is shown on the right.

III. METHODS OF SAMPLE AND TIP MOVEMENT

Both samples and tips are mounted on the standard VG Escalab cylindrical stubs and moved throughout our vacuum system. The long motions of 0.5 to 1 m are all accomplished with mechanical mechanisms. A trolley on a wire and track moves the sample from one end of the preparation chamber to the analysis chamber. The rotatable rack and pinion mechanism in the transfer chamber moves samples from the preparation chamber to the STM chamber. In the STM chamber the sample rides an elevator, composed of a magnetic motion feedthrough, from the top level to the bottom one where the STM is suspended. VG wobble sticks are used to move the sample from any one of these mechanical mechanisms to a station for analysis. A number of sample parking places are also situated in every chamber.

A typical sample holder for our instrument is shown in Fig. 3. Tantulum stubs are used for samples which require annealing for cleaning. The sample rests against Ta legs, which are spot welded to the stub, and is clamped down by Ta clips. These samples can be heated from the rear by an

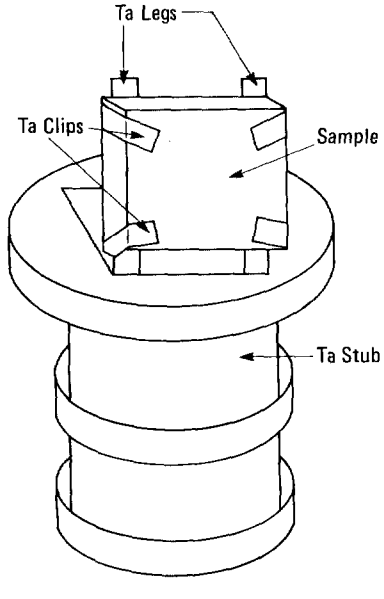

FIG. 3. Sample holder, composed of cylindrical tantalum stub, with Ta legs and clips for holding the sample vertically.

electron beam heater with a barium oxide cathode. Typically, about 3.8 kV and 4 mA are sufficient to heat a Si crystal to 1030 °C for cleaning. Since thermocouple wire leads are not compatible with our sample geometry and motion, the sample temperature is measured with a Wahl DHS-52 infrared pyrometer.

The most complex part of the sample and tip movement process occurs during the actual transfer onto the STM stage. Before transferring samples or tips into the STM, we clamp down the spring stages with a mechanism incorporating a frictional clamp. The wobble stick at the lower level of the STM chamber has a wire mounted onto its end. To pick up a sample from a parking place, one puts the wire through a hole in the sample stub which is above the center of gravity of the stub and lifts it up. The sample can then be gently lowered into the louse. Then the wobble stick wire can be used to turn the screw on the louse to hold the sample firmly in place.

Our interchangeable tips are very simple. The actual tip wire is bent so as to have a hook at one end, as shown in Fig. 4. The wobble stick wire can go through this hook and carefully carry the tip from a special stub with a frictional clamp to the slot in the tip block on the STM. Then the clamping screw, which has a wire spot welded across its head, is turned to lock the tip into place. This design has the great advantage that only the tip wire itself is being moved, as compared to our earlier design in which an entire stub holding a tip was moved onto the piezoelectric tripod, with the consequent lowering of the resonant frequency of the tripod. In this instrument, the pieces which are actually scanned consist of just two-stainless steel cubes, 3 mm on a side, which have an extremely small mass.

Our tips are usually fabricated from 0.5-mm tungsten wire. These are bent into the proper shape and then etched like field emission tips.[9] Our usual etching procedure involves the application of 12 V ac to the tip, producing tips which are ~1000 Å in radius. These tips are loaded into the vacuum system through the airlock and then heated with the electron beam heater until the shaft is at ~1000 °C. This heating procedure melts the end of the tip and reduces the carbon contamination[4]; empirically, previously heated tips are more stable when used in the STM.

IV. DATA ACQUISITION AND ANALYSIS

Analog data acquisition can be conveniently performed on a storage oscilloscope, with two ramp generators controlling the motion of the x and y piezodrives which move the tip. Digital data are acquired using an IBM personal computer/XT (PC/XT). The computer sends trigger pulses to the ramp generators and then measures the z piezodrive voltage with an analog-to-digital converter. After background subtraction, top view color images are displayed on an IBM Professional Graphics Display. Although some data analysis is done on the PC, more sophisticated image processing, including digital filtering, statistical differencing, registration of STM images with a bulk lattice, and three-dimensional perspective views, is performed on an IBM 4381 mainframe computer with an IBM 7350 display using the IBM programs HLIPS and IAX.

V. USE OF STM IMAGES FOR STRUCTURAL DETERMINATION

Now we briefly show some high-resolution STM images measured with our instrument and describe our structural determinations. All images shown here are taken in the constant current feedback mode, with an image acquisition time of ~5 min. Our sample preparation procedures for Si(111) are described in Ref. 5. Figure 5 shows the STM topographic image obtained for the $(\sqrt{3}\times\sqrt{3})R\,30°$ Ag/Si(111) reconstruction, hereafter called $\sqrt{3}$. A honeycomb of bright protrusions is evident, with a corrugation of about 1 Å.[5,10] By carefully checking the registration of these features with the bulk silicon lattice for STM images which simultaneously show the $\sqrt{3}$ Ag structure and the Si(111) 7×7 structure

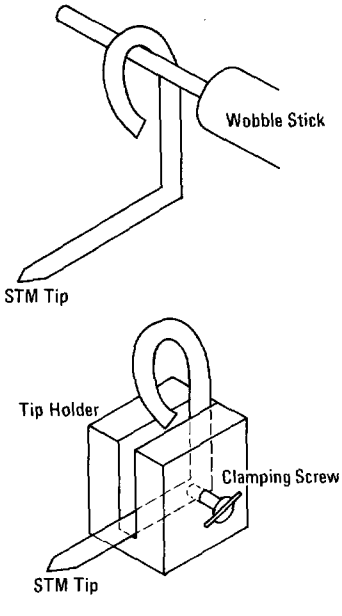

FIG. 4. Schematic diagram showing how we remove the STM tip using a wire mounted on the end of a wobble stick.

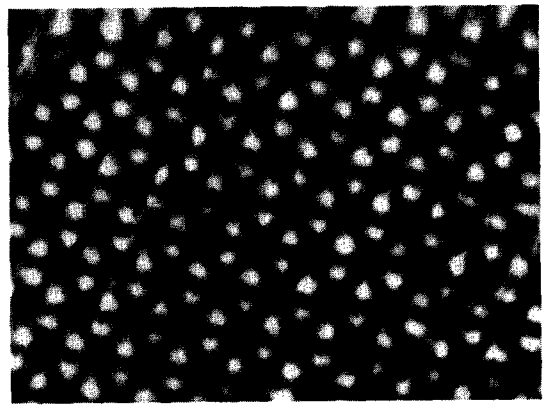

FIG. 5. STM image of ~70×50 Å² region of the $(\sqrt{3}\times\sqrt{3})R\,30°$ Ag/Si(111) surface, with corrugation of about 1 Å at a tip bias of −0.39 V and a constant tunnel current of 0.2 nA. White represents elevated features. This figure has been corrected for thermal drift and piezoelectric creep.

near step edges, we have recently established that these protrusions are silver atoms with a silver coverage of $\frac{2}{3}$.[11]

Figure 6 shows an example of the STM topographic image for the $(\sqrt{19}\times\sqrt{19})R$ 23.4° Ni/Si(111), hereafter called $\sqrt{19}$, a surface for which only a few percent coverage of Ni completely changes the reconstruction. The many bright spots observed in the STM image must be silicon atoms because the low nickel coverage precludes their being nickel atoms. By considering the Ni binding site determined by surface extended x-ray absorption fine structure [12](SEXAFS) and the known dimer–adatom–stacking fault model of the Si(111) 7×7 surface,[13] we made a new model of the $\sqrt{19}$ surface in which each subsurface nickel atom in a unit cell is bonded to six silicon adatoms.[6] This model successfully explains all of the features observed in our STM images, including surface dislocations, extra adatoms in a unit cell, and anomalous cells.

Figure 7 shows a three-dimensional perspective view of a topographic STM image of a 2500-Å-thick Au(111) film epitaxially deposited on mica. The sample was transferred through air, loaded into our UHV system, and then cleaned by sputtering and annealing until Auger spectroscopy revealed that contamination levels were $<1\%$ of a monolayer. In contrast to previous work on Au(111) which observed only monatomic steps separated by wide terraces,[14,15] here we have resolved the individual close-packed metal atoms on this surface with the near neighbor distance of 3.0 ± 0.3 Å, as calibrated against Si(111) 7×7 images. We have also obtained very similar results on similar Au(111) thin films on mica measured in another STM operating in air, both with and without organic monolayer films of cadmium arachidate and of octadecyltrichlorosilane (OTS). The measurements in air were performed in the constant height mode, where the observed current modulation was $\sim10\%$ of the dc tunneling current of 2 nA at a tip bias of 50 mV. Since very similar atomic resolution images are obtained both in air with organic monolayers and in UHV on clean samples, the Au(111) surface may be useful for instrumental calibration, as an alternative to highly oriented pyrolytic graphite.

VI. CONCLUSIONS

We have described the design of our STM and UHV system in which both samples and tips are removable for further study by other surface analysis techniques. Our data on metals on semiconductors and on Au(111) thin films clearly show that our STM is an extremely stable and reliable instrument for surface science studies.

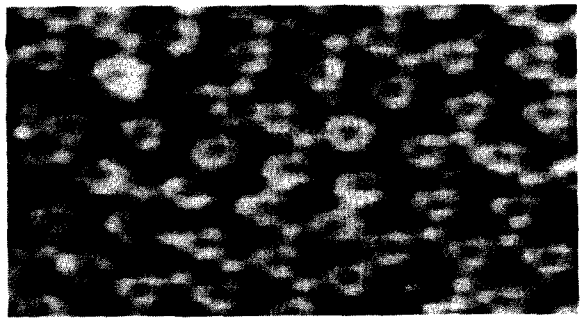

FIG. 6. STM image of $\sim180\times100$ Å^2 region of $(\sqrt{19}\times\sqrt{19})R$ 23.4° Ni/Si(111) surface, recorded at tip voltage of $+1.20$ V and tunneling current of 0.2 nA, showing corrugation of ~3 Å.

FIG. 7. Three-dimensional perspective view of STM topographic image of $\sim12\times14$ Å^2 region of Au(111) thin film epitaxially deposited on mica and then sputtered and annealed in UHV. The corrugation is 0.3 Å at a tip bias of 30 mV and a tunneling current of 3 nA. The separation of the close-packed atoms is ~3 Å. This figure has been corrected for thermal drift and piezoelectric creep. We attribute the elongation of the observed atoms in the slow-scan direction to an asymmetric tip.

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