Construction of a UHV scanning tunneling microscope

by S. Chiang
R. J. Wilson

In our laboratory, we have built an ultrahigh-vacuum scanning tunneling microscope (STM). The STM is mounted onto one flange in an ultrahigh-vacuum chamber which is connected by a transfer chamber to a surface-analysis system equipped with 500-Å-resolution SAM/SEM, XPS, LEED, and sample-heating and sample-cleaning facilities. Samples and tips can be moved throughout the combined vacuum system. We describe the design and performance of the instrument and show some preliminary data on Si(111). We also show some recent results of experiments on tip preparation.

Introduction

We are interested in using the scanning tunneling microscope for measuring the surface structure of well-characterized single-crystal metal or semiconductor samples with submonolayer coatings of evaporated metals or adsorbed molecules. Therefore, we have designed and built an STM which allows the movement of both samples and tips throughout an ultrahigh-vacuum surface-analysis system, allowing us to compare the STM results with those of more established surface-analysis techniques. The STM design follows the principles of the early successful designs of Binnig et al. [1, 2], including a spring suspension, a piezoelectric tripod for scanning the tunneling tip, and a piezoelectric walker to move the sample without coupling in external vibrations. Data acquisition is automated with an IBM Personal Computer. The design is described in more detail below, together with preliminary results on Si(111) showing atomic steps.

In order to make the STM a more reliable instrument and to help make STM measurements more amenable to theoretical interpretation, more information is clearly needed on the characteristics of the tunneling tips. Therefore, we have begun to measure the properties of tunneling tips and the effects of various tip-cleaning procedures. We discuss some recent measurements by SEM and Auger spectroscopy on the properties of ground and etched tips.

Instrumentation of the scanning tunneling microscope

We have added a sample-transfer chamber and the STM chamber to a Vacuum Generators Escalab surface-analysis system, which consists of a sample-preparation chamber and a surface-analysis chamber. 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 includes an airlock, an electron-beam heater, a high-energy argon ion gun for sputter-cleaning samples, rear-view LEED optics, and an evaporator for producing metal films. The sample-transfer chamber contains a rack and pinion mechanism which permits sample movement from this chamber into any other which is connected radially to it. One such chamber holds the STM, an optical microscope and television camera for observing the tip-to-sample approach, and flanges with mechanisms associated with the sample-movement facilities.

Like the earlier STM models in Zurich [1,2], the vibration isolation consists of two sets of spring stages which reduce most of the vibrations to frequencies <2 Hz. Viton spacers are used to damp the high-frequency vibrations propagating along the springs. Additional damping of the low-frequency vibrations is achieved by using SmCo permanent magnets mounted on the intermediate stage to induce magnetic eddy currents in pieces of copper on the support and on the inner stage. In order to try to limit the effects of thermal drift, most parts on our STM stage were made from invar or quartz.

To position the sample in our microscope, we have used a one-dimensional piezoelectric walker, like that developed by Binnig and Gerber [3], which we call a "micropede." The piezoelectric micropede body is divided into three electrodes which act as a body and a foot at each end. A piece of metal, currently aluminum, is machined so that the piezoelectric plate rests on a flat surface between two rails. The tension of the rails against the feet can be adjusted with two screws and springs. The feet of the micropede are lapped to fit the piece of metal. Application of voltage to a foot makes it expand, clamping that end of the micropede. Alternately clamping the feet and extending and contracting the body of the micropede cause it to advance along the railing. With a step size which is usually between 0.3 μm and 1.0 μm, the micropede can walk at a speed up to ~2 mm/min. The entire micropede is mounted onto a cross-slide which can be mechanically pushed to move the sample sideways.

In our instrument, the tip is scanned using small piezoelectric tubes, 6.35 mm in diameter and 25.4 mm long. The small wall thickness of 0.50 mm gives these tubes a sensitivity of ~100 Å/V. These tubes are repoled by ~500 V and can be used with voltages from −300 V to +800 V, giving a total range of motion of 11 μm. To make the piezoelectric scanning tripod, three orthogonal tubes are soldered to a piece of invar. On that piece is another invar block that is isolated electrically and holds the removable stub on which is mounted the tunneling tip.

Feedback circuitry is required to control the movement of the tip so as to keep the current constant as the tip is scanned laterally across the surface. This is accomplished by a piezoelectric element—"z-piezodrive"—which controls the movement of the tip in the z direction. Our circuit has a low-noise FET preamplifier which acts as a current-to-voltage converter. Since the tunneling current is an exponential function of the distance between the tip and sample, a logarithmic amplifier is the next stage. This is followed by a proportional amplifier and an integrator in parallel so that the frequency response of the circuitry can be adjusted. An additional integrator can be switched into the feedback loop to produce a slow "approach" of the tip to the sample when initially moving them close enough to measure a tunneling current. Finally, these outputs are summed and amplified by a high-voltage operational amplifier to produce the ± 150 V needed to drive the z-piezodrive over ± 1.5 μm.

An obvious problem is to prevent a collision as the micropede moves the sample towards the tip. We observe the motion of the micropede walking towards the tip with a Volpi TV-microprobe, which is an optical microscope with a working distance of 50 mm connected to a television camera so as to give an overall magnification of ~100. When observing the tip and its reflection in the sample surface, it is possible to observe a relative motion of the tip and sample as small as 2 μm with this system. Nevertheless, caution is still required because there are structures on the end of the tip which cannot be observed with this magnification. Therefore, when we perform an "approach," we first use the optical microscope and walk the sample within ~20 μm of the tip. The computer then switches the feedback circuit on, and the z-piezodrive is extended to test for a tunneling current. If no current is sensed, the computer retracts the z-piezodrive and steps the micropede. This process continues until tunneling current is obtained.

Analog data acquisition can be conveniently performed on a storage oscilloscope. In this case, two ramp generators, one about an order of magnitude faster than the other, control the motion of the x- and y-piezodrives which move the tip. We trigger the oscilloscope from the faster scan oscillator. The voltage on the z-piezodrive is then applied to the oscilloscope and added to the voltage from the slow ramp so that the traces from subsequent scans are displaced on the oscilloscope screen. The resulting image is then a topographic map of the surface under study. The STM image presented here was measured in this fashion.

Finally, we have automated our STM data acquisition using an IBM Personal Computer. This software uses two digital-to-analog converters to control the scanning piezodrives, and then measures the z-piezodrive voltage with an analog-to-digital converter. The scans are displayed in real time on an IBM Professional Graphics Display and plotted later. Data can be shipped to an IBM mainframe computer for more sophisticated image processing.

Preliminary studies of tunneling tips

Sudden changes of resolution due to changes in the tip condition have been seen in STM measurements [4]. It is possible that diffusion of atoms along the tip is responsible for such observations. The characterization, cleaning, and fabrication of tips, therefore, is an important problem if one wishes to make reproducible, high-resolution measurements with the STM. In Zurich, Binnig et al. [1,2] have successfully used very crudely ground tips which have a radius <1 μm. We have used this kind of ground tip in our STM, as well as etched field-emission tips.

We have characterized a ground tungsten tip in the Escalab using SEM and Auger spectroscopy. In **Figure 1**, we show two SEM photographs of a damaged ground tip which had been used to obtain STM images on a Au(100) surface. This tip was transferred in UHV from the STM to the Escalab analysis chamber. We see in the side view in Figure 1(a) that the ground tip has surface texture, with features which are 10 to 30 μm in size. In the higher-magnification picture of the same tip shown in Figure 1(b), we see several small structures on the end of the tip which are each about 0.1 μm across. Such structures may well act as mini-tips for the tunneling current. Auger spectroscopy measurements of this ground tip indicate that the surface is primarily composed of carbon and oxygen, and almost no tungsten signal is visible.

We have also used sharp field-emission (FEM) tips [5,6] in our tunneling microscope. These tips are electrochemically etched from tungsten wire 0.13 mm in diameter in 2N NaOH solution. Since the etching occurs preferentially at the meniscus of the solution, a special controller circuit is used to stop the etching process when the etching current suddenly decreases as the bottom part of the wire drops off. Using this method, we can reproducibly fabricate tips with a radius of ~0.1 μm.

Sharper tips are important for measuring rough surfaces. The lateral resolution of the STM depends not only on the radius of curvature and the included angle of the end of the tip, but also on the size of the structures on the surface [7]. If the surface is flat on an atomic scale, the lateral resolution of the instrument will depend on the size of the single atom or cluster of atoms at the end of the tip. If the surface topography is very rough, the tip dimensions must be small compared to the surface structure in order to follow the contours of the surface.

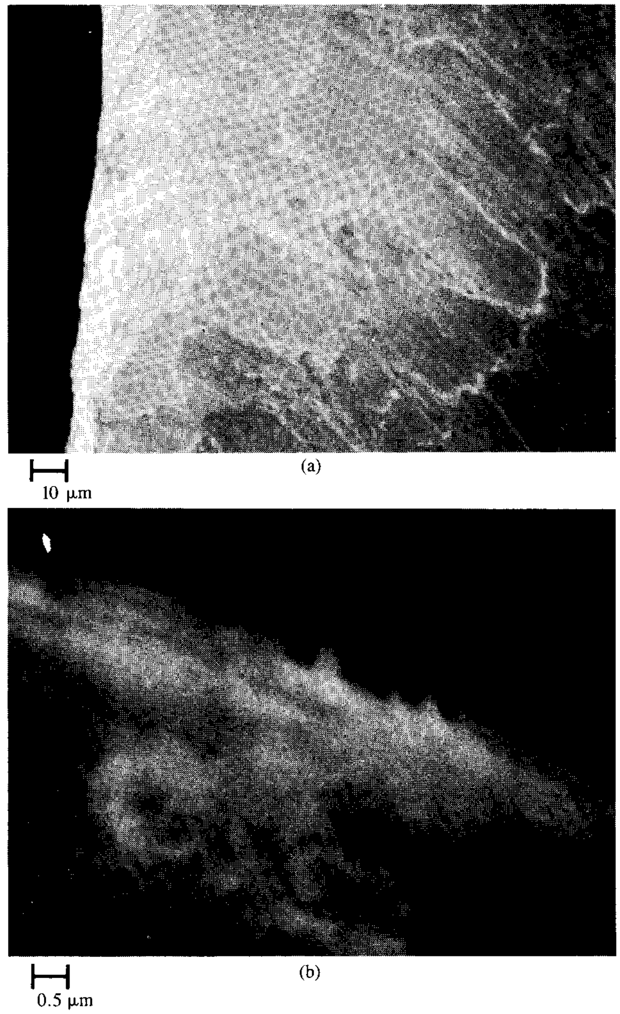

Figure 1

SEM photographs of a ground tungsten tip at two different magnifications. This tip had been used for STM measurements on a Au(100) surface before being transferred in UHV for the SEM measurements. (a) We see features 10 to 30 μm in size. (b) At higher magnification, we see structures on the end of the tip which are 0.1 μm across.

We have done some preliminary measurements with SEM and Auger spectroscopy to test the effects of different cleaning procedures on the surface composition of FEM tips. We have not yet tested the effects of these cleaning procedures on STM performance. In **Figure 2**, we show SEM pictures of two field-emission tips. Figures 2(a) and 2(b) show an FEM tip at two different magnifications before any cleaning procedure. In the higher-magnification picture, the end of the tip has a radius of about 1000 Å. Auger spectroscopy measurements of this tip show primarily carbon and oxygen signals from contamination, with very little tungsten signal. We have used an electron-beam heater to heat this tip and expect that the sharp end will be much hotter than the shank because of the high electric fields there. After heating so that the end of the shank reaches a temperature of ~900°C, we obtained the SEM photograph shown in Figure 2(c). The heating procedure has clearly melted and blunted the end of the tip. After the heating, Auger spectroscopy shows that the surface is primarily composed of oxygen and tungsten, with no discernible carbon contamination.

We have also tried applying high voltage to a tungsten FEM tip to clean it. Figure 2(d) shows an SEM picture of a tip after −5 kV has been applied to it. The high voltage has

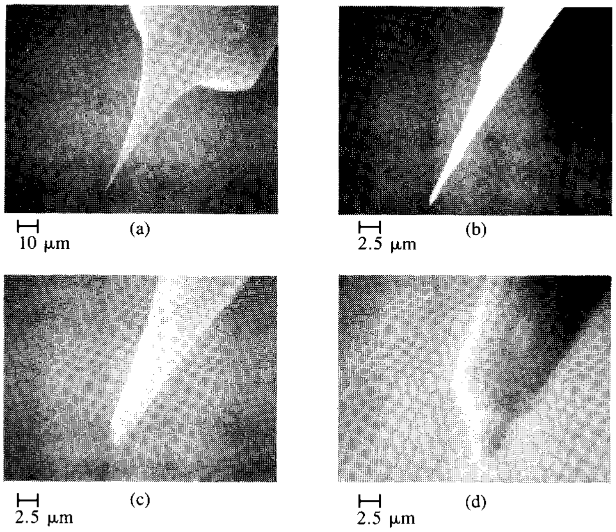

Figure 2

SEM photographs of two different field-emission tips made from etched tungsten wire. (a) Low-magnification picture taken before cleaning. (b) Higher-magnification picture taken before cleaning, showing a tip radius of ~0.1 μm. (c) Same tip after heating end of shank to ~900°C. (d) A second tip after applying −5 kV to it.

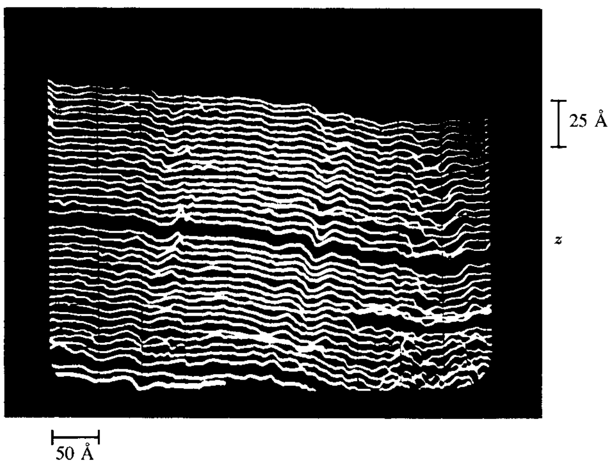

Figure 3

STM image from clean Si(111) sample, measured on roughened part of surface. The spacing between scan lines is due to thermal drift and is uncalibrated. See text for measurement parameters.

blunted the tip severely. Auger spectroscopy after the high-voltage treatment shows that the surface is primarily composed of carbon and tungsten, with no oxygen signal. A common tip-sharpening procedure for the STM is to apply high voltage to the tip in the field-emission mode until there is a drastic change in the current from the tip to the sample [8], presumably corresponding to burning the end of the tip. Such field desorption cleaning can be carried out away from the sample so that contamination is not spread onto the surface under study.

We have also used such sharp tips to operate our instrument as a field-emission scanning microscope, like that of Young et al. [9]. In this mode, we apply 20 V to the tip, and it retracts about 1000 Å farther from the surface than it is in the tunneling mode. This mode of operation thus gives us the possibility of measuring surface topography while the tip is several thousand Å from the surface, with concomitant degradation of lateral resolution. But aside from possible problems of field-desorbing contamination from the tip onto the surface, field-emitted electrons from the tip may cause electron-stimulated desorption on some surfaces.

STM results on Si(111)

Using the STM instrument which we described above, we have been able to observe atomic steps on a clean Si(111) surface. The surface was prepared by heating it to about 800°C. No Auger signals from carbon or oxygen contamination were observed after the cleaning procedure. Parts of the Si surface were heated excessively so that the surface roughened; unfortunately the STM measurements were done on such a rough area.

Figure 3 shows an STM image which we obtained with a voltage of +1.9 V on the sample and a tunneling current of 0.25 nA. Each scan line took 10 s. The spacing between the scan lines is caused by thermal drift and is uncalibrated. We see two well-ordered features reproducing in subsequent scan lines, which we interpret to be atomic steps about 150 Å apart. There is some uncertainty in the calibration of the *z*-piezodrive, due to a lack of rigidity in the scanning tripod. Thus, the vertical bar in the figure may correspond to a motion of the *z*-piezodrive which is as much as a factor of two smaller than that indicated.

Conclusions

Although this STM is able to measure atomic steps on Si(111), there are some difficulties with its design. The connections of the tip holder to the scanning piezodrives may not be sufficiently rigid. Also, the pieces on which the tip is mounted are quite massive relative to the piezodrives. These two problems limit the scanning speed of the instrument to 0.1 Hz per line. We are currently improving our instrument to correct these problems. Nevertheless, this STM is the first one in which both samples and tips have been removable for further study by other surface-analysis techniques. To continue our study of tunneling tips, we are in the process of building a field-ionization microscope (FIM). We hope to be able to make very reproducible tunneling tips by using the FIM to characterize and prepare them on an atomic scale using the methods which have recently been shown by Fink [10]. We plan to use our STM to measure overlayers of thin metal films on semiconductors in the near future.

Acknowledgments

We would like to thank G. Binnig and Ch. Gerber for helpful and stimulating discussions and technical advice. We would also like to thank Ch. Gerber for expert technical assistance in the latter stages of assembling the instrument.

References

1. G. Binnig and H. Rohrer, *Helv. Phys. Acta* **55,** 726 (1982).
2. G. Binnig and H. Rohrer, *Surf. Sci.* **126,** 236 (1983).
3. G. Binnig and C. Gerber, *IBM Tech. Disclosure Bull.* **22,** 2897 (1979).
4. A. M. Baro, G. Binnig, H. Rohrer, Ch. Gerber, E. Stoll, A. Baratoff, and F. Sylvan, *Phys. Rev. Lett.* **52,** 1304 (1982).
5. R. Gomer, *Field Emission and Field Ionization*, Harvard University Press, Cambridge, MA, 1961.
6. E. W. Müller and T. T. Tsong, *Field Ion Microscopy, Principles and Applications*, Elsevier, New York, 1969.
7. E. Stoll, *Surf. Sci.* **143,** L411 (1984).
8. G. Binnig and Ch. Gerber, IBM Zurich Research Laboratory, Rüschlikon, Switzerland, private communication.
9. Russell Young, John Ward, and Fredric Scire, *Rev. Sci. Instrum.* **43,** 999 (1972).
10. Hans-Werner Fink, *IBM J. Res. Develop.* **30,** No. 5, 460–465 (1986, this issue).

Received September 16, 1985; accepted for publication October 14, 1985

Shirley Chiang *IBM Almaden Research Center, 650 Harry Road, San Jose, California 95120.* Dr. Chiang is a Research staff member in the Surface, Thin Film, and Plasma Science Department at the IBM Almaden Research Center. She joined IBM at San Jose in 1983 after receiving her Ph.D. degree in physics from the University of California at Berkeley. Her current research interests include the use of the scanning tunneling microscope to study the relationship of surface structure to the chemical properties of reconstructed surfaces and adsorbates on metals.

Robert J. Wilson *IBM Almaden Research Center, 650 Harry Road, San Jose, California 95120.* Dr. Wilson has been a Research staff member in the Surface, Thin Film, and Plasma Science Department at the IBM Almaden Research Laboratory since 1983. Prior to joining IBM, he was involved in postdoctoral research at Bell Laboratories. He obtained his Ph.D. degree in physics from the University of California at Berkeley. His research interests include the application of scanning tunneling microscopy and other surface analysis techniques to studies of nucleation and growth phenomena and adsorbate structure.