

A scanning tunneling microscope for the investigation of the growth of metal films on semiconductor surfaces

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We describe a scanning tunneling microscope which is part of an apparatus designed for the investigation of metal-semiconductor surfaces. The main parts of the tunneling unit are the piezoelectric walker ("louse") carrying the sample for the coarse approach and a piezoelectric xyz system for movement of the tip. The xyz system is self-compensating with regard to uniform thermal expansion. Our first measurements have been obtained on a Au(110) surface. The spatial resolution allows the observation of monoatomic steps. Corrugation perpendicular to the rows and troughs of the (110) surface with an amplitude around 1 Å is also visible. The noise in the z direction under optimum conditions is smaller than 0.2 Å rms.

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1. Introduction

The metal-semiconductor interface and, in particular, the growth behavior of metal films on semiconductor surfaces are of considerable physical and technical interest. It is not surprising that during the last one or two decades many surface-physical methods have been applied to such systems, leading to an insight into the metal-semiconductor interface from an atomic point of view [1]. Especially important were combined LEED-AES measurements [2], by which information on structural aspects of the growth mode could be obtained, and electron microscopy [3]. Although the resolution of the otherwise very suitable scanning electron microscope is only of the order of 5–10 nm, it could in some cases successfully be applied even in the submonolayer range [4].

It is obvious that the scanning tunneling microscope (STM) might be a very valuable supplementary tool for studying metal-semiconductor systems. The STM would not only allow the direct observation of surface structures in the ordered state, such as the case of a well-defined interface layer, but could also give insight into the interface formation during the initial stages of metal atom condensation.

Moreover, the STM can be used for spectroscopy of surface states and may therefore contribute to a better understanding of the thermodynamics and driving forces of the different modes of growth of the metal overlayers.

The STM has been used very successfully for imaging of semiconductor surfaces, where several beautiful examples demonstrate the possibilities of this instrument [5-8]. But the spectroscopical applications for Au on Si(111) [9, 10] also look very promising. The purpose of this paper is to describe an STM especially constructed for the study of metal-semiconductor interfaces. In addition to design details, we present our first results obtained on a Au(110) surface.

2. Experiment

It has recently been demonstrated that an STM capable of atomic resolution can be built in various ways [5-8, 11-13]. The main differences are found in the realization of the coarse approach between sample and tunneling tip, while the fine positioning during the measurements is always accomplished by means of a piezoelectric drive of the tip. All instruments have shown that the attainment of atomic resolution is dependent on an absence of mechanical vibrations and noise in the tunneling unit. Low-frequency variations such as temperature drifts or creeping of the piezoelectric material cause fewer problems, if one is not interested in the observation of a specific location on the sample surface.

The minimization of vibrations is usually not obtained in the first version of a design. Many changes and improvements are necessary until one is able to observe atomic corrugations in the range of a few tenths of Å. The purpose of our report is a description of an STM setup where atomic resolution has just been achieved, although more work will be needed to reach the ultimate state of the art. In addition to details of our tunneling unit, we briefly describe the combination with the rest of the surface-physical equipment which is necessary for our studies of metal-semiconductor interfaces.

The principle of our tunneling unit is illustrated in **Figure 1**. The main components are the piezoelectric walker ("louse"), which we built according to the idea and realization of Binnig et al. [5], and the xyz tip-positioning system. Both components are mounted on a stable base plate. The louse-feet are manufactured from Al and the dielectric layer consists of an approximately 25- μm -thick Al_2O_3 film produced by anodizing the polished Al disks. By using a voltage of 200 V between the louse-foot and the support (a hardened and polished steel disk), clamping forces of about 1 N were reached. The minimum step length in the single-step mode is 50 nm at present. The sample is located directly on top of a louse-foot in order to minimize the effect of vibrations and thermal expansion.

The xyz system is constructed following an idea of van de Walle et al. [11]. Movement of the tip for scanning parallel

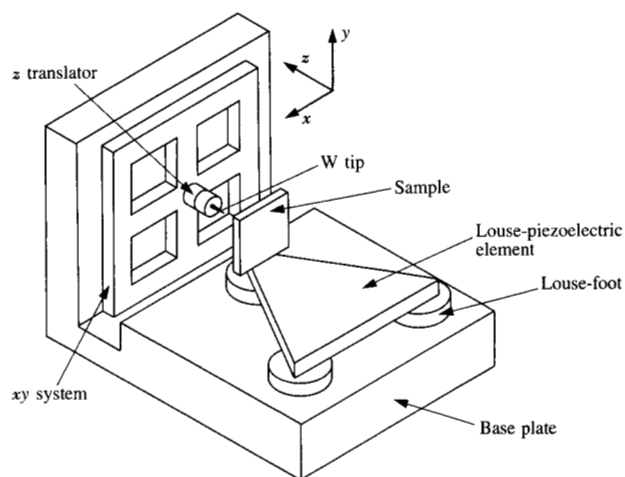


Figure 1

Schematic view of the tunneling unit. The temperature compensation in the z direction is not shown; its realization is similar to that described by van de Walle et al. [11]; see text.

to the sample surface is realized by a window-like frame made from one piezoelectric ceramic disk. By applying voltages to appropriate parts of the frame and considering the fact that the corners of the xy system are rigidly fixed to the base plate, a movement of the midpoint of the frame parallel to the xy plane is achieved. This construction has the advantage that uniform thermal expansions of the xy system ideally do not change the position of the midpoint and are therefore self-compensating. The movement of the tip normal to the surface is realized by a stack translator, which is mounted in the middle of the xy system. Since the same stacks are used for connection of the frame with the base plate, thermal compensation has also been achieved for the z axis. We used piezoelectric material with high sensitivity (P1-60, manufactured by Quartz and Silice: The longitudinal and transverse sensitivities are $d_{33} = 400 \times 10^{-12}$ m/V and $d_{31} = -120 \times 10^{-12}$ m/V, respectively), which, however, has the disadvantage that the voltage sources must be extremely stable and noiseless. The scanning range is approximately $2 \times 2 \mu\text{m}^2$ and the height of the tip can be changed by 1.6 μm .

As a tunneling tip we used a W field-emission cathode prepared by electrolytic etching of a 0.2-mm-diameter W wire. Since the mean radius of the tip during the beginning of the experiment was considerably smaller than 1 μm , it could not be resolved with visible-light microscopy. Similar tips which we studied in a separate vacuum chamber equipped with a fluorescence screen showed a field-emission pattern according to a [110] orientation of the axis. The radii of these tips could be estimated to be about 50 nm. More systematic studies are planned in the future to obtain tips

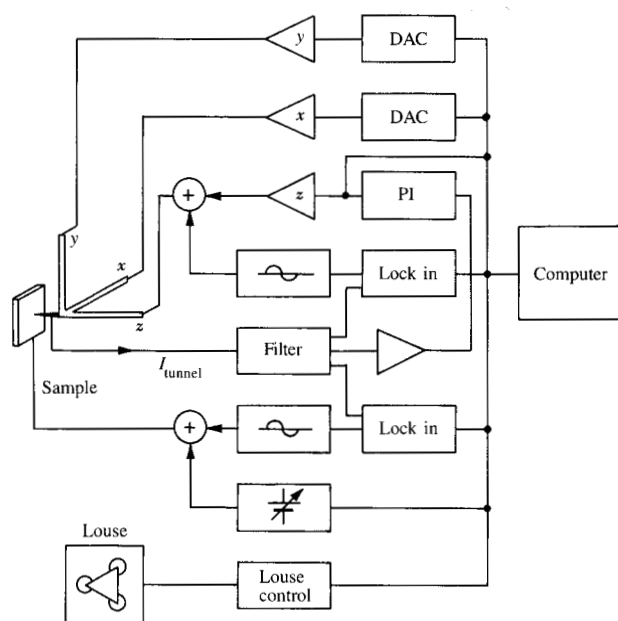


Figure 2

Electric circuit of the STM. The control of the tunneling current (I_{tunnel}) is achieved by means of a proportional-integral regulator (PI).

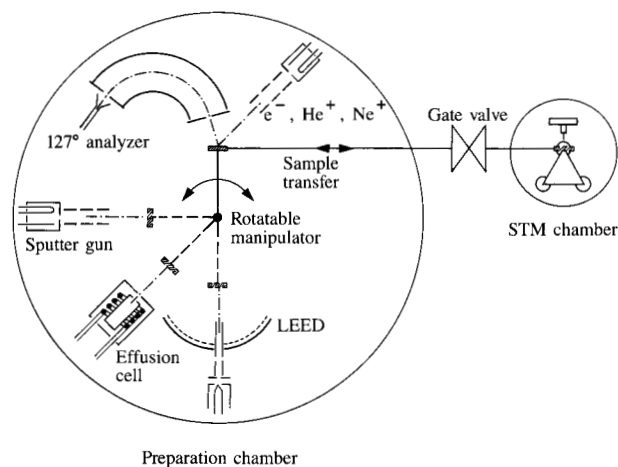


Figure 3

Principal arrangement of the STM with the main part of the surface-physical equipment for measurements on metal-semiconductor surfaces.

with even smaller radii and sufficient rigidity. The actual measurements, however, were probably performed with a less ideal tip geometry due to tip crashes in the test phase.

To decouple acoustic noise and mechanical vibrations from the tunneling unit, it is suspended with a double spring

system. Damping of oscillations is realized by a system of additional viton pieces.

The electric circuit for the STM is shown schematically in **Figure 2**. Facilities are provided for recording dI/dz and dI/dU curves using lock-in techniques. Information on the work function and the spectroscopy of electronic surface states can therefore be obtained. The entire system is computer-controlled. For our first experiments we used a small personal computer (Commodore CBM 8296 D) connected to an interface consisting of a timer, a counter, and 16-bit analog-to-digital and digital-to-analog converters (ADC, DAC). Acquisition and evaluation of data with the present system are extremely time-consuming and allow only a provisional operation of the STM. These limitations will be removed in the future by a more powerful computer system which is especially suitable for image processing.

The combination of the STM with the rest of the surface-physical equipment is illustrated in **Figure 3**. Not every component is operative at present. The main idea of the apparatus is a separation of sample preparation and tunneling measurements in different UHV chambers. This has the advantage that the tunneling equipment remains permanently under the same vacuum and temperature conditions, but has the disadvantage of somewhat more complicated sample handling via a transfer line.

In the analysis chamber, the sample may be transferred to a rotatable sample manipulator, which can be adjusted to several positions, namely in front of a reverse-view LEED system, a quartz-controlled effusion cell for metal evaporation, a sputter ion gun for sample cleaning, and a rotatable 127° cylindrical condenser energy analyzer, which by means of an ion/electron gun can be used for ion scattering and angle-resolved Auger electron spectroscopy (ISS and AES).

Our first tunneling experiments were performed on a Au(110) single crystal. For technical reasons, no particular surface-cleaning procedure and surface-analytical measurements could be carried out during these tests. From photoemission experiments, however, it was known that the surface, which was previously prepared by spark erosion and subsequent annealing and noble-gas ion bombardment cycles, was well oriented and ordered. The sample was contaminated in these experiments by at most one or two atomic layers of hydrocarbons. The quality of the surface certainly became worse during the test phase of the experiment due to touching of the tip to the sample, which could be avoided later by improving the approach electronics.

The results which are discussed here were obtained in the following way. After the distance between sample and tip had reached a few tenths of mm, the final approach was performed in the single-step mode, where between single steps of the piezoelectric louse it was always checked whether the z piezoelectric element could approach the tip closely

enough to obtain 0.2 nA tunneling current. After tunneling current could be obtained, the tip was withdrawn for about 0.5 μm and treated in the field-emission mode for a few minutes. During this procedure the current showed a drastic noise decrease. The tunneling current was usually set to a value of 0.2–1.0 nA with voltages between tip and surface of 1–100 mV. The scanning of the surface was realized in a stepwise motion of the tip in the xy plane. The voltage which must be applied to the z piezoelectric element in order to obtain a constant tunneling current was read by an ADC and stored in the memory and floppy disk of the computer.

The results shown in Figures 4(a) and 4(b) are plots of these voltage values. In Figure 4(a) they are connected by lines in the scanning direction and in Figure 4(b) by lines perpendicular to the scanning direction. Except for subtraction of a linear varying background in the x and y direction (by choosing an appropriate plane), no corrections were applied to the data.

3. Results and discussion

It cannot be the purpose of our paper to discuss details of the topology of the Au(110) surface on the basis of our data, since details of the chemical constitution and of surface order and reconstruction could not be determined in the present setup. Therefore, our results can only be used for a demonstration of the attainable resolution of the instrument.

The example shown in Figure 4 is one of our first results in which atom-like features are unambiguously resolved. In particular, at A, B, and C steps were recorded with heights lying between 2.1 $\text{\AA}$ and 2.6 $\text{\AA}$. The atomic radius of Au is 1.45 $\text{\AA}$, according to the lattice parameter [14]. We therefore believe that the observed steps at A, B, and C limit a terrace and an island, respectively, of monoatomic height. The discrepancy of our measurement from the theoretical value of 2.89 $\text{\AA}$ probably depends on the fact that the length scales used in the figure are derived from the piezoelectric sensitivity as given by the manufacturer. The fluctuations in the position of the z piezoelectric element due to mechanical vibrations and electronic ripple and noise appear to be less than 0.2 $\text{\AA}$, but irregularities are observed which are larger than this value and are probably related to sudden changes of the atomic structure of the tunneling tip.

The sample was oriented in such a way that the scans followed a $[110]$ -like direction. The corrugation along the scan lines is expected to be very small because of the fact that neighboring atoms touch each other. Accordingly, no corrugation is visible in the data as shown in Figure 4(a). On the other hand, perpendicular to these lines a much stronger corrugation resulting from the row structure of the (110) surface and from a possible 2×1 or 3×1 reconstruction could be expected. Actually, the image as plotted in Figure 4(b) clearly reveals areas of periodic structures in some parts (for example, area E) which are qualitatively similar to the results of Binnig et al. [15, 16]. The amplitude of the

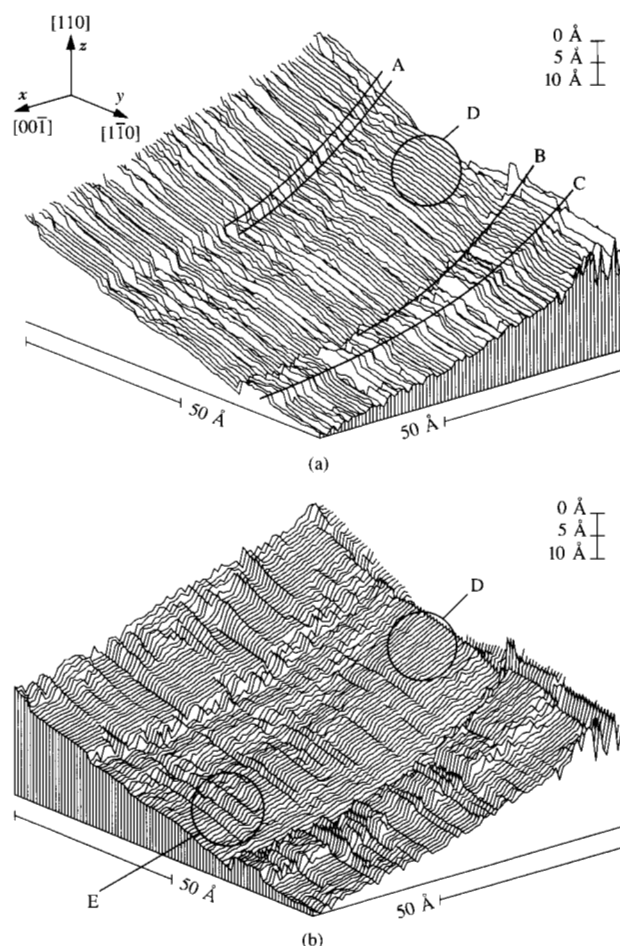


Figure 4

(a) STM image of a Au(110) surface. The lines connect the data points in the scanning direction (along the $[110]$ line). (b) The same data points connected by lines perpendicular to the scanning direction.

corrugation is about 1 $\text{\AA}$ and therefore consistent with the height difference of 1.44 $\text{\AA}$ between row and trough atoms of a nonreconstructed Au(110) surface. The lateral distance of the maxima of the corrugation in the $[001]$ direction measures between 4 $\text{\AA}$ and 4.5 $\text{\AA}$ and is therefore also consistent with an expected value of 4.08 $\text{\AA}$ for the Au lattice parameter. In some parts of the image we observe periodic structures whose distances are twice as large which could be associated with a 2×1 reconstruction.

We again emphasize that these interpretations are of a tentative character. More information is needed with regard to the characterization of the surface to provide a convincing interpretation of the observed data.

Although atomic-scale resolution has been obtained with the present setup, a number of improvements are possible in the future. The problem of sample handling and

characterization is not of principal difficulty and will soon be adequately solved. A more serious difficulty concerns the characterization and stability of the tunneling tip. In our opinion, some of the structures and part of the noise visible in Figure 4 are probably related to sudden changes of the atomic arrangement of the tunneling tip. More experience with the tip preparation and systematic field-emission studies are therefore needed. Finally, the noise caused to an equal extent by mechanical vibrations and by the imperfections of the electronic circuit will be reduced in the future by a more stable and rigid construction of the tunneling unit and improvements to the electronic supply.

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References

1. L. J. Brillson, *Surf. Sci. Repts.* **2**, 123 (1982).
2. G. Le Lay, *Surf. Sci.* **132**, 169 (1983).
3. J. A. Venables, G. D. T. Spiller, and M. Hanbücken, *Repts. Progr. Phys.* **47**, 399 (1984).
4. M. Futamoto, M. Hanbücken, C. J. Harland, G. W. Jones, and J. A. Venables, *Surf. Sci.* **150**, 430 (1985).
5. G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel, *Phys. Rev. Lett.* **50**, 120 (1983).
6. R. S. Becker, J. A. Golovchenko, E. G. McRae, and B. S. Swartzentruber, *Phys. Rev. Lett.* **55**, 2028 (1985).
7. R. M. Feenstra, W. A. Thompson, and A. P. Fein, *Phys. Rev. Lett.* **56**, 608 (1986).
8. J. E. Demuth, R. J. Hamers, R. M. Tromp, and M. E. Welland, *IBM J. Res. Develop.* **30**, No. 4, 396-402 (1986).
9. G. Binnig, K. H. Frank, H. Fuchs, N. Garcia, B. Reihl, H. Rohrer, F. Salvan, and A. R. Williams, *Phys. Rev. Lett.* **55**, 991 (1985).
10. F. Salvan, G. Binnig, and H. Fuchs, *Europhys. Conf. Abstr. (ECOSS 7)* **9c**, 3, CO7 (1985); to be published in *Surf. Sci.*
11. G. F. A. van de Walle, J. W. Gerritsen, H. van Kempen, and P. Wyder, *Rev. Sci. Instrum.* **56**, 1573 (1985).
12. R. J. Behm, W. Hösler, E. Ritter, and G. Binnig, *Phys. Rev. Lett.* **56**, 228 (1986).
13. J. Moreland and P. K. Hansma, *Rev. Sci. Instrum.* **55**, 399 (1984).
14. Ch. Kittel, *Introduction to Solid State Physics*, John Wiley & Sons, Inc., New York, 1976.
15. G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel, *Phys. Rev. Lett.* **49**, 57 (1982).
16. G. Binnig, H. Rohrer, Ch. Gerber, and E. Weibel, *Surf. Sci.* **131**, L279 (1983).

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