Scanning tunneling microscopy: new method for studying solid surfaces

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The recent intensification of research on solid surfaces has resulted in the development of new methods and instruments for studying surfaces. We wish to describe briefly a fundamentally new method for studying surfaces, "scanning tunneling microscopy," which has just appeared on the scene.[1]

The first scanning tunneling microscope (STM) was developed at the IBM Zurich Research Laboratory (Switzerland). The operation and capabilities of the apparatus have been tested on several materials. Even at this point it is clear that STM has several advantages over other methods. An STM can produce a three-dimensional picture of a surface on the atomic scale, with a resolution ~ 0.1–0.2 Å along the height of the microscopic surface relief; this is an order of magnitude better than that achievable with the scanning electron microscope. It should be noted that the STM uses weak electric fields (three orders of magnitude weaker than those in an ionization microscope; the energy of the "tunnel" beam ranges from ~ 1 meV to 4 eV), so that the surface under study is not damaged. Furthermore, the surface itself is freely accessible, so that physical and chemical processes taking place on it can be studied at the same time as the surface structure.

The STM operates by virtue of the tunneling of electrons under a vacuum barrier. It is a simple matter to show (see Ref. 2, for example) that the transmission coefficient D of a square potential barrier of height Δ and width l for particles of mass M and energy $E<\Delta$ is

$$D=\frac{4k^2\varkappa^2}{(k^2+\varkappa^2)\,\mathrm{sh}^2\, l\varkappa+4k^2\varkappa^2}, \tag{1}$$

where

$$\varkappa=\frac{1}{\hbar}\sqrt{2M(\Delta-E)}, \quad k=\frac{1}{\hbar}\sqrt{2ME}. \tag{2}$$

We then easily find that the tunnel current I across the vacuum gap of width l is given by[1]

$$I\propto \exp(-A\sqrt{\Delta}\,l), \tag{3}$$

where

$$A=\sqrt{\frac{8M}{\hbar^2}}=1.025\ \mathrm{eV}^{-1/2}\text{Å}^{-1}, \tag{4}$$

and Δ is the average work function of the two materials at the contact. It follows from (3) and (4) that if Δ is a few electron volts then a change in l by an amount as small as a single-atom step (a few angstroms) will change the tunnel current by three orders of magnitude. This sharp dependence of the tunnel current on the size of the vacuum gap is the key to the extremely high resolution of the STM.

For a long time, attempts to observe experimentally and make practical use of the vacuum tunneling of electrons went without success, primarily because of vibrations, which cause uncontrollable changes in the tunnel current by changing the width of the vacuum gap. Contamination of the surface under study by foreign atoms as the result of adsorption and oxidatin also causes uncontrollable changes in the tunnel current.

The basic part of the STM is a measurement cell consisting of a vacuum chamber which houses a plate with electrodes and apparatus for moving them. In all models of the STM, the electrodes are a tungsten tip and a plate made of the material under study.

Protection from external vibrations is provided by mounting the entire measurement cell on a massive stone bench, which itself floats on inflated rubber tubes. Protection from internal vibrations of the apparatus is provided by magnetic levitation of the plate and the electrodes. It is worthy of note that the STM operates at room temperature, except for a superconducting lead block, which is used for the magnetic levitation of the plate and the electrodes. Vibrations of the electrodes themselves have been suppressed by the use of a new technique for mounting and moving the electrodes. This new technique essentially eliminates mechanical coupling of the electrodes witht he surroundings.[3]

The basic device used to move the electrodes is a standard Philips piezoelectric drive. It can move the electrodes more than 1000 Å in three mutually perpendicular directions with a sensitivity of 0.2 A. Independently regulated voltages are applied to the three piezoelectric crystals of the drive to cause the motion. The voltage applied to the drive to move a tungsten tip along the direction normal to the surface is supplied from a control cell. This voltage is varied in accordance with the height of the microscopic surface relief in such a manner that the tunnel current is held constant at a constant voltage applied to the vacuum gap. The magnitudes of the voltages controlling the motion of the piezoelectric crystals determine the topographic pattern of the surface at a constant work function [see (3)]. In an effort to determine which changes in the control voltages are caused by changes

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in the gap width l and which are caused by changes in the work function Δ, the size of the gap is changed by an amount Δl. The relation

$$\Delta^{1/2} = \frac{\Delta (\ln I)}{\Delta l} \tag{5}$$

is then used to determine the change in the work function, through measurements of the change in the logarithm of the tunnel current, $\Delta (\ln I)$.

The STM can determine not only the height of details of the microsocpic surface relief but also their width. The tangential resolution of the STM depends on the dimensions of the tip used for the scanning.

If a needle with a spherical tip of radius **r** is used, the tangential resolution is given by[1]

$$\delta (r) = 3 \sqrt{\frac{2r}{A \sqrt{\Delta}}} \approx 3 \sqrt{r (\text{Å})}. \tag{6}$$

It can be seen from this expression that a tip radius $r \lesssim 100$ Å is required to achieve $\delta \lesssim 100$ Å. Needles of this type are common in emission microscopes, but the particular needles used in emission microscopes cannot be used in the STM because of their sensitivity to vibrations, which is a consequence of their considerable length. The inventors of the STM[1] accordingly developed a new procedure for fabricating needles. A tungsten bar 1 mm in diameter is ground at an angle of 90° in such a manner that minitips with radii from ~ 1000 Å to 1 μm are formed at the end of the needle. Because of the extreme sensitivity of the tunnel resistance to the gap width, only the current from the longest of these minitips is detected. The tip can be sharpened even further by touching it against the surface under study a few times. This procedure has yielded value $\delta \sim 10$ Å.

The first experiments in the first-generation STMs were carried out in a vacuum of 10^{-6} torr. The tunnel resistance was measured as a function of the vacuum gap between the tungsten tip and a platinum plate. The gap size was originally 10 Å. Measurements were taken at a constant voltage of 60 mV of both polarities; at this voltage, no field emission of electrons from the end of the tungsten tip was observed.

From (3) we find an exponential dependence of the tunnel resistance R on the gap size:

$$R (l) \sim \exp (A \sqrt{\Delta} l). \tag{7}$$

This dependence of R was observed, however, only after special cleaning of the electrode surfaces, since a vacuum of 10^{-6} torr is not high enough to avoid contamination of the electrodes. The cleaning procedure[3] involved bringing the tips into contact with the surface of the plate and then applying a 10-kHz voltage, which caused an ultrasonic cleaning of the electrodes. After the first cleaning session, the height of the tunnel barrier was 3.2 eV, and after several sessions it approached 5 eV, which corresponds to the average work function of platinum and tungsten, Δ.

The first-generation microscopes were also used to obtain three-dimensional topographic patterns of the Ir(110) surface of $CaIrSn_4$ single crystals.[4] Single-atom, two-atom, and three-atom steps were observed as well as the initial stages of the formation of screw dislocations.

The height of a single-atom step found with the STM, 6.7 Å, agrees well with the value found by an x-ray method, 6.87 Å.

In the microscopes of the second generation, the measurements were carried out in a vacuum of $\sim 10^{-10}$ torr. In such a high vacuum, there is no surface contamination, as is shown by the constant height of the tunneling barrier. This particular apparatus is described in detail in Ref. 4. Topographic pictures were recorded by scanning the tip along the surface at a constant tunnel current.

In a study of gold,[4] the surface was treated by the standard procedure which causes structural changes on the (110) surface: bombardment with argon atoms followed by annealing in a vacuum of $\sim 10^{-10}$ torr. Before processing, the (110) surface of gold was essentially atomically smooth. After the processing, the microscopic surface relief was studied at room temperature and at 300 °C. Corrugation of the (110) surface of gold along the [001] direction was observed at room temperature. The corrugation period ranged from 20 to 100 Å, and the corrugation amplitude from 0.1 to 2 Å. Only a very slight change in the number of steps of height 6 Å along the $[1\bar{1}0]$ direction was observed. At 300°C, a high density of single-atom and two-atom steps along the $[1\bar{1}0]$ direction was observed. Corresponding data demonstrating an increase in the density of atomic steps with increasing temperature were obtained for the Ni(100) surface through an analysis of the shape of helium diffraction lines.

Since there is no difficulty in reaching current densities of $\sim 10^3$–10^4 A/cm^2 in an STM, this instrument can be used to study the surfaces not only of metals but also of lightly doped semiconductors.

Scanning tunneling microscopes of the second generation were in fact used to observe a reconstruction of a Si(111) surface.[5] The formation of a 7×7 surface superlattice had also been observed previously, by low-energy electron diffraciton, for example, but the STM provided the first observations in coordinate space, rather than momentum space, substantially simplifying the interpretation of results.

A sample was prepared for study by heating it to 900°C in a vacuum of $\sim 10^{-10}$ torr directly in the test chamber. This procedure made it possible to remove the oxide film which appeared on the surface when the sample was transferred to the test chamber after chemical etching. The surface microrelief was measured at a voltage of 2.9 V (the tip was the positive electrode), because voltages below 2.5 V resulted in direct contact of the tip with the sample. A picture of the arrangement of atoms in two 7×7 unit cells with 9 minima and 12 maxima was obtained; this picture was used to draw conclusions about the arrangement of atoms on the Si(111) surface. The dimensions of the diagonals of the 7×7 cell were found to be 46 ± 1 Å and 29 ± 4 Å, in good agreement with the data from x-ray measurements: 46.56 Å and 26.88 Å.

The high spatial resolution makes scanning tunneling microscopy useful for studying the adsorption of atoms and molecules on solid surfaces, for monitoring crystal growth, and for studying ultrathin films of insulators on the surfaces of semiconductors.

Plans call for the development of a third-generation in-

strument to operate at liquid-nitrogen temperature, where even better mechanical stability can be achieved.

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