2.3 STM and SFM

Scanning Tunneling Microscopy and Scanning Force Microscopy

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Contents

- Introduction
- Basic Principles and Instrumentation
- Common Modes of Analysis and Examples
- Sample Requirements
- Artifacts
- Conclusions

Introduction

Scanning Tunneling Microscopy (STM) and its offspring, Scanning Force Microscopy (SFM), are real-space imaging techniques that can produce topographic images of a surface with atomic resolution in all three dimensions. Almost any solid surface can be studied with STM or SFM: insulators, semiconductors, and conductors, transparent as well as opaque materials. Surfaces can be studied in air, in liquid, or in ultrahigh vacuum, with fields of view from atoms to greater than 250 × 250 μm. With this flexibility in both the operating environment and types of samples that can be studied, STM / SFM is a powerful imaging system.

The scanning tunneling microscope was invented at IBM, Zurich, by Gerd Binnig and Heinrich Rohrer in 1981.[1] In ultrahigh vacuum, they were able to resolve the atomic positions of atoms on the surface of Si (111) that had undergone a 7 × 7 reconstruction (Figure 1). With this historic image they solved the puzzle of the atomic structure of this well studied surface, thereby establishing firmly the credibility and importance of this form of microscopy. For the invention of STM, Binnig and Rohrer earned the Nobel Prize for Physics in 1986.

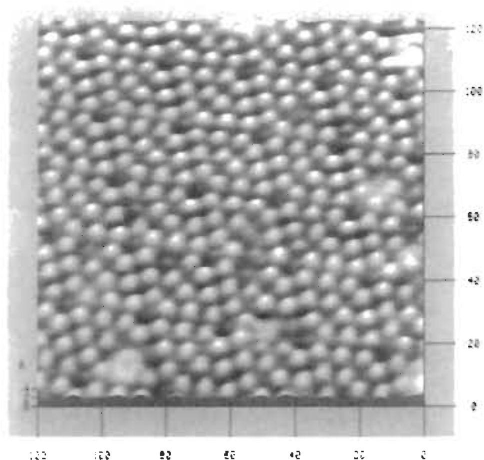

Figure 1 **Ultrahigh-vacuum STM image of Si (111) showing 7 × 7 reconstruction.**

Since then, STM has been established as an instrument for forefront research in surface physics. Atomic resolution work in ultrahigh vacuum includes studies of metals, semimetals and semiconductors. In particular, ultrahigh-vacuum STM has been used to elucidate the reconstructions that Si, as well as other semiconducting and metallic surfaces undergo when a submonolayer to a few monolayers of metals are adsorbed on the otherwise pristine surface.[2]

Because STM measures a quantum-mechanical tunneling current, the tip must be within a few Å of a conducting surface. Therefore any surface oxide or other contaminant will complicate operation under ambient conditions. Nevertheless, a great deal of work has been done in air, liquid, or at low temperatures on inert surfaces. Studies of adsorbed molecules on these surfaces (for example, liquid crystals on highly oriented, pyrolytic graphite[3]) have shown that STM is capable of even atomic resolution on organic materials.

The inability of STM to study insulators was addressed in 1985 when Binnig, Christoph Gerber and Calvin Quate invented a related instrument, the scanning force microscope.[4] Operation of SFM does not require a conducting surface; thus insulators can be studied without applying a destructive coating. Furthermore, studying surfaces in air is feasible, greatly simplifying sample preparation while reducing the cost and complexity of the microscope.

STM and SFM belong to an expanding family of instruments commonly termed Scanning Probe Microscopes (SPMs). Other common members include the magnetic force microscope, the scanning capacitance microscope, and the scanning acoustic microscope.[5]

Although the first six or seven years of scanning probe microscope history involved mostly atomic imaging, SPMs have evolved into tools complementary to Scanning and Transmission Electron Microscopes (SEMs and TEMs), and optical and stylus profilometers. The change was brought about chiefly by the introduction of the ambient SFM and by improvements in the range of the piezoelectric scanners that move the tip across the sample. With lateral scan ranges on the order of 250 μm, and vertical ranges of about 15 μm, STM and SFM can be used to address larger scale problems in surface science and engineering in addition to atomic-scale research. STM and SFM are commercially available, with several hundred units in place worldwide.

SPMs are simpler to operate than electron microscopes. Because the instruments can operate under ambient conditions, the set-up time can be a matter of minutes. Sample preparation is minimal. SFM does not require a conducting path, so samples can be mounted with double-stick tape. STM can use a sample holder with conducting clips, similar to that used for SEM. An image can be acquired in less than a minute; in fact, "movies" of ten frames per second have been demonstrated.[6]

The three-dimensional, quantitative nature of STM and SFM data permit in-depth statistical analysis of the surface that can include contributions from features 10 nm across or smaller. By contrast, optical and stylus profilometers average over areas a few hundred Å across at best, and more typically a μm. Vertical resolution for SFM / STM is sub-Å, better than that of other profilometers. STM and SFM are excellent high-resolution profilometers.

STM and SFM are free from many of the artifacts that afflict other kinds of profilometers. Optical profilometers can experience complicated phase shifts when materials with different optical properties are encountered. The SFM is sensitive to topography only, independent of the optical properties of the surface. (STM may be sensitive to the optical properties of the material inasmuch as optical properties are related to electronic structure.) The tips of traditional stylus profilometers exert forces that can damage the surfaces of soft materials, whereas the force on SFM tips is many orders of magnitude lower. SFM can image even the tracks left by other stylus profilometers.

In summary, scanning probe microscopes are research tools of increasing importance for atomic-imaging applications in surface science. In addition, SFM and STM are now used in many applications as complementary techniques to SEM, TEM, and optical and stylus profilometry. They meet or exceed the performance of these instruments under most conditions, and have the advantage of operating in an ambient environment with little or no sample preparation. The utility of scanning probe microscopy to the magnetic disk, semiconductor, and the polymer industries is gaining recognition rapidly. Further industrial applications include the analysis of optical components, mechanical parts, biological samples, and other areas where quality control of surfaces is important.

Basic Principles

STM

Scanning tunneling microscopes use an atomically sharp tip, usually made of tungsten or Pt-Ir. When the tip is within a few Å of the sample's surface, and a bias voltage V_t is applied between the sample and the tip, quantum-mechanical tunneling takes place across the gap. This tunneling current I_t depends exponentially on the separation d between the tip and the sample, and linearly on the local density of states. The exponential dependence of the magnitude of I_t upon d means that, in most cases, a single atom on the tip will image the single nearest atom on the sample surface.

The quality of STM images depends critically on the mechanical and electronic structure of the tip. Tungsten tips are sharpened by electrochemical etching, and can be used for a few hours in air, until they oxidize. On the other hand, Pt-Ir tips can be made by stretching a wire and cutting it on an angle with wire cutters. These tips are easy to make and slow to oxidize, but the resulting tip does not have as high an aspect ratio as a tungsten tip. As a result, Pt-Ir tips are not as useful for imaging large structures.

In its most common mode of operation, STM employs a piezoelectric transducer to scan the tip across the sample (Figure 2a). A feedback loop operates on the scanner to maintain a constant separation between the tip and the sample. Monitoring the position of the scanner provides a precise measurement of the tip's position in three dimensions. The precision of the piezoelectric scanning elements, together with the exponential dependence of I_t upon d means that STM is able to provide images of individual atoms.

Because the tunneling current also depends on the local density of states, STM can be used for spatially resolved spectroscopic measurements. When the component atomic species are known, STM can differentiate among them by recording and comparing multiple images taken at different bias voltages. One can ramp the bias voltage between the tip and the sample and record the corresponding change in the tunneling current to measure I versus V or dI/dV versus V at specific sites on the image to learn directly about the electronic properties of the surface. Such measurements give direct information on the local density of electronic states. This technique was pioneered by Hamers, et al., who used tunneling spectroscopy to map the local variations in the bonding structure between Si atoms on a reconstructed surface.[7]

On the other hand, the sensitivity of STM to electronic structure can lead to undesired artifacts when the surface is composed of regions of varying conductivity. For example, an area of lower conductivity will be represented as a dip in the image. If the surface is not well known, separating topographic effects from electronic effects can be difficult.

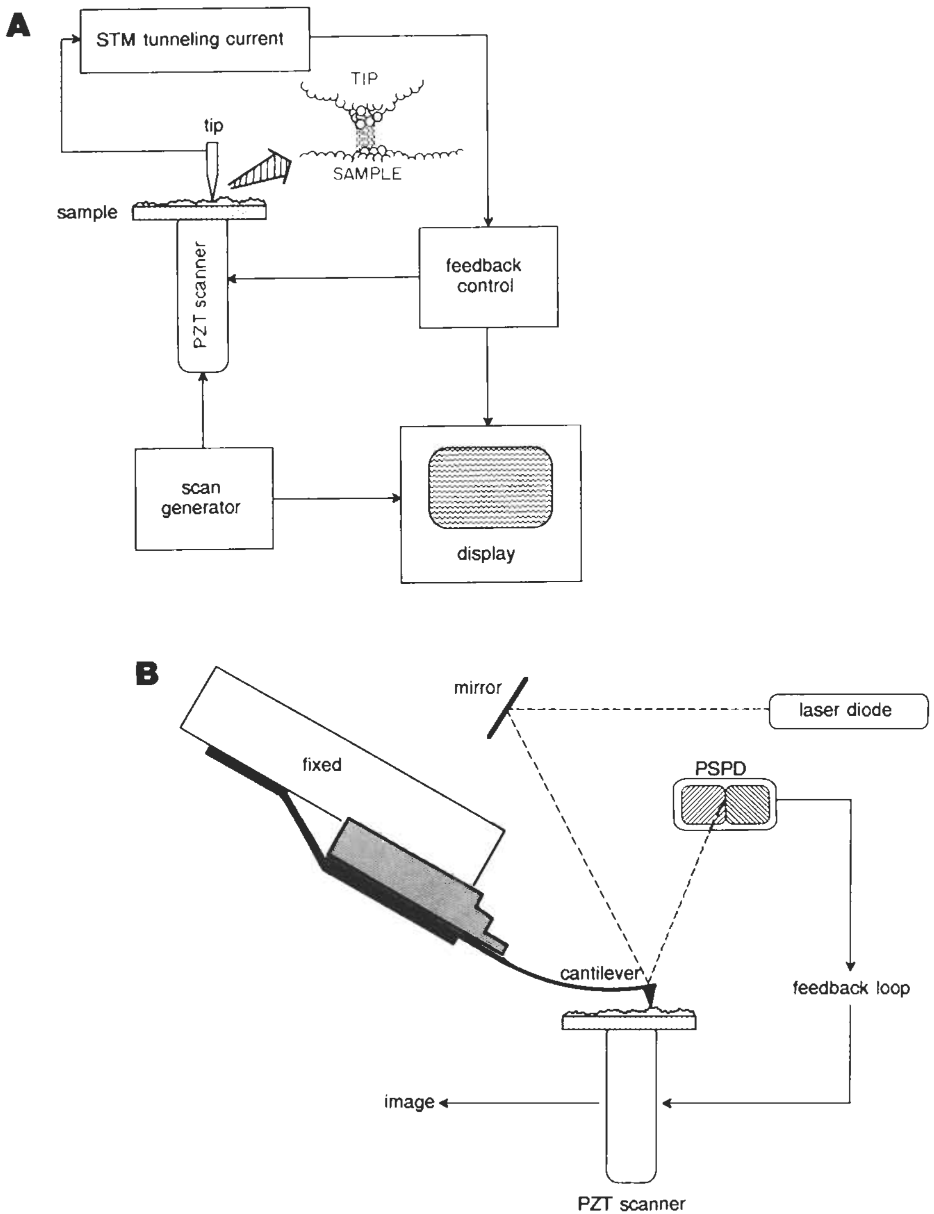

Figure 2 **Schematic of STM (a) and SFM (b).**

SFM

Scanning force microscopes use a sharp tip mounted on a flexible cantilever. When the tip comes within a few Å of the sample's surface, repulsive van der Waals forces

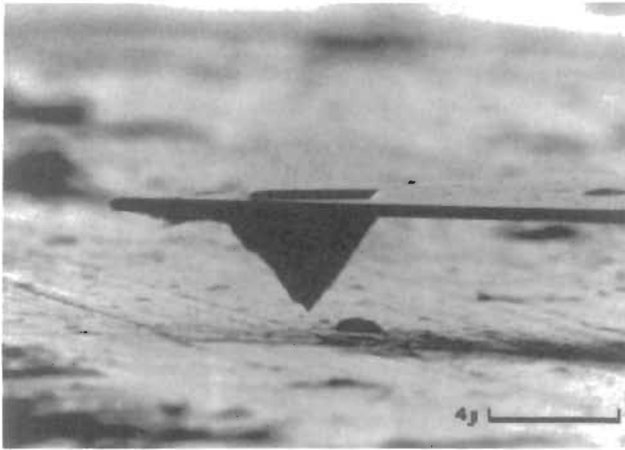

Figure 3 SEM image of SFM cantilever showing pyramidal tip.

between the atoms on the tip and those on the sample cause the cantilever to deflect. The magnitude of the deflection depends on the tip-to-sample distance *d*. However, this dependence is a power law, that is not as strong as the exponential dependence of the tunneling current upon *d* employed by STM. Thus several atoms on an SFM tip will interact with several atoms on the surface. Only with an unusually sharp tip and flat sample is the lateral resolution truly atomic; normally the lateral resolution of SFM is about 1 nm.

Like STM, SFM employs a piezoelectric transducer to scan the tip across the sample (Figure 2b), and a feedback loop operates on the scanner to maintain a constant separation between the tip and the sample. As with STM, the image is generated by monitoring the position of the scanner in three dimensions.

For SFM, maintaining a constant separation between the tip and the sample means that the deflection of the cantilever must be measured accurately. The first SFM used an STM tip to tunnel to the back of the cantilever to measure its vertical deflection. However, this technique was sensitive to contaminants on the cantilever.[4] Optical methods proved more reliable. The most common method for monitoring the defection is with an optical-lever or beam-bounce detection system.[8] In this scheme, light from a laser diode is reflected from the back of the cantilever into a position-sensitive photodiode. A given cantilever deflection will then correspond to a specific position of the laser beam on the position-sensitive photodiode. Because the position-sensitive photodiode is very sensitive (about 0.1 Å), the vertical resolution of SFM is sub-Å.

Figure 3 shows an SEM micrograph of a typical SFM cantilever. The cantilevers are 100–200 μm long and 0.6 μm thick, microfabricated from low-stress Si_3N_4 with an integrated, pyramidal tip. Despite a minimal tip radius of about 400 Å,

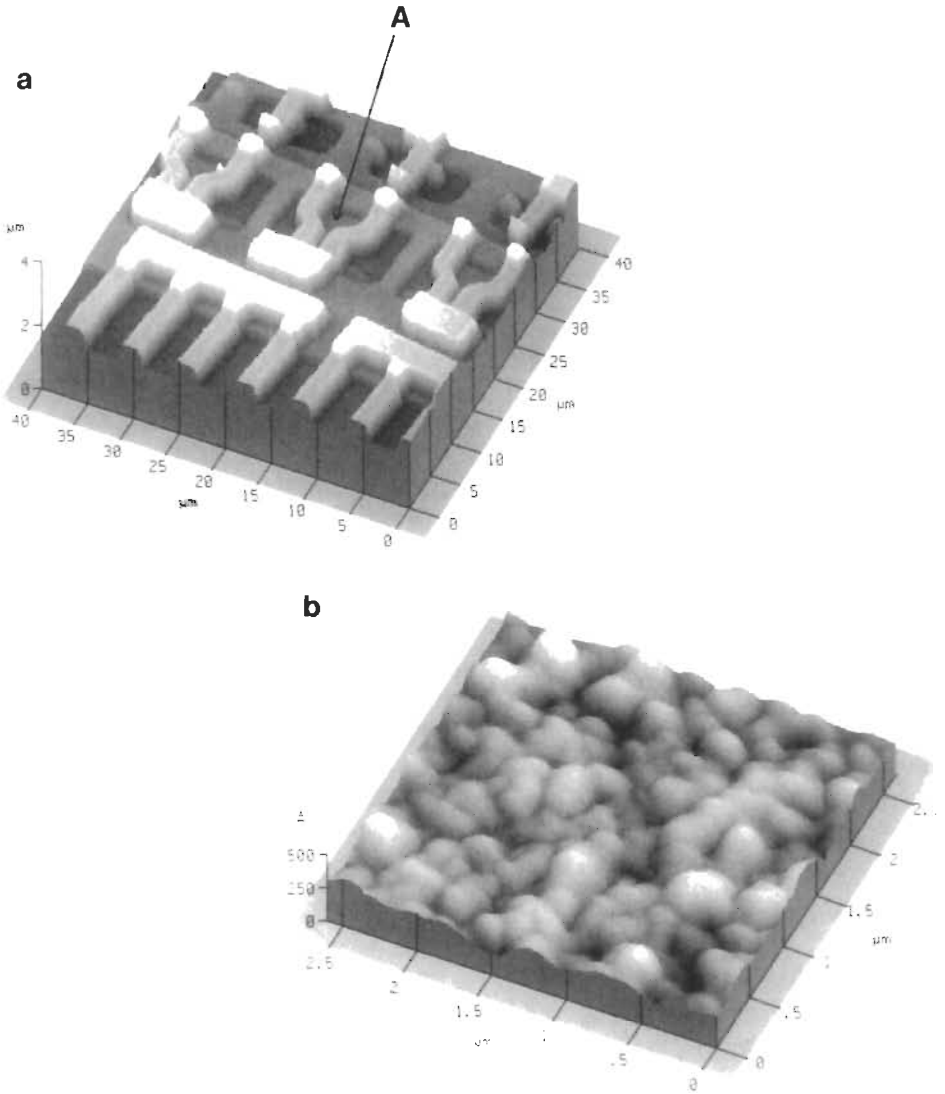

Figure 4 **SFM image of an integrated circuit (a) and close-up of silicon oxide on its surface (b).**

which is needed to achieve high lateral resolution, the pressure exerted on the sample surface is small because of the low force constant of the cantilever (typically 0.2 N / m), and the high sensitivity of the position-sensitive photodiode to cantilever deflection. The back of the cantilever may be coated with gold or another metal to enhance the reflectance of the laser beam into the detector.

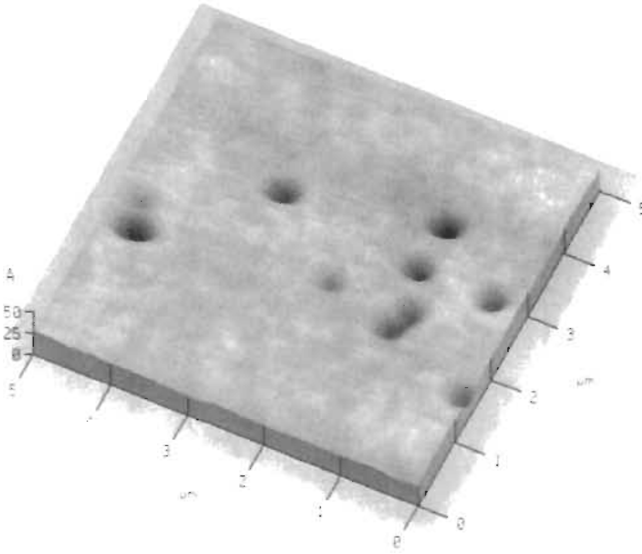

Figure 5 **SFM image of oxidized Si wafer showing pinhole defects 20 Å deep.**

Common Modes of Analysis and Examples

STM and SFM are most commonly used for topographic imaging, three-dimensional profilometry and spectroscopy (STM only).

Topography

Unlike optical or electron microscopes, which rely on shadowing to produce contrast that is related to height, STM and SFM provide topographic information that is truly three-dimensional. The data are digitally stored, allowing the computer to manipulate and display the data as a three-dimensional rendition, viewed from any altitude and azimuth. For example, Figure 4a shows an SFM image of an integrated circuit; Figure 4b is a close-up of the oxide on the surface of the chip in the region marked *A* in Figure 4a. In a similar application, Figure 5 is an SFM image of a Si wafer with pinholes, 20 Å deep. Easily imaged with SFM, these pinholes cannot be detected with SEM.

Profilometry

The three-dimensional, digital nature of SFM and STM data makes the instruments excellent high-resolution profilometers. Like traditional stylus or optical profilometers, scanning probe microscopes provide reliable height information. However, traditional profilometers scan in one dimension only and cannot match SPM's height and lateral resolution.

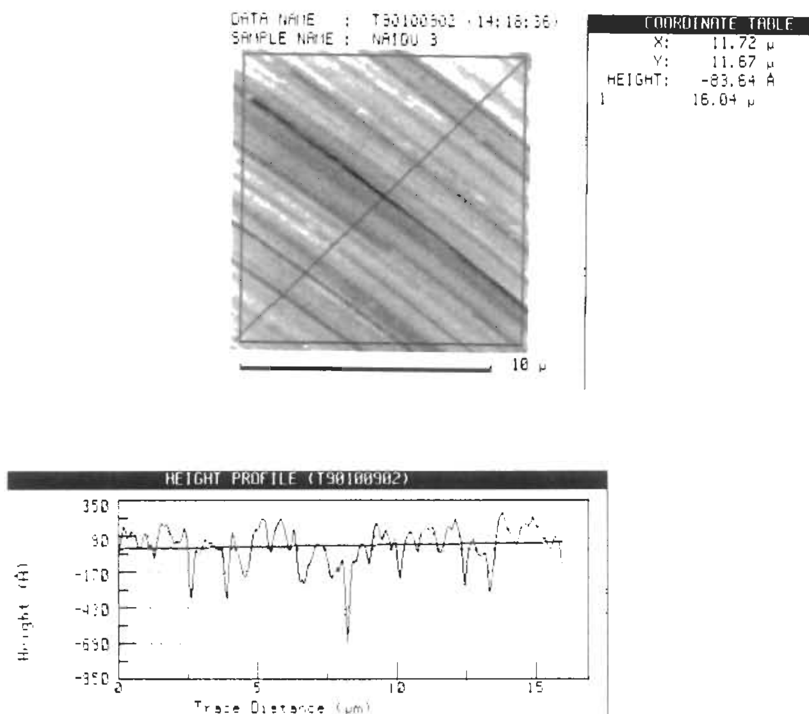

Figure 6 **SFM image of a magnetic storage disk demonstrating roughness analysis.**

In the magnetic storage disk industry, the technology has advanced to the point where surface roughness differences on the order of a few Å have become important. Optical and stylus profilometers, while still preferable for scanning very large distances, cannot measure contributions from small features. Figure 6 is an SFM image of a thin-film storage disk (top), shown top-down, with heights displayed in a linear intensity scale ("gray scale"). Using the mouse, the height profile of any cross section can be displayed and analyzed (bottom). Figure 7 shows a thin-film read–write head. The magnetic poles are recessed about 200 Å; their roughness is comparable to that of the surrounding medium. Note the textural difference between the glass embedding medium and the ceramic. SFM is not affected by differences in optical properties when it scans composite materials.

Profilometry of softer materials, such as polymers, is also possible with SFM, and with STM if the sample is conducting. Low forces on the SFM tip allow imaging of materials whose surfaces are degraded by traditional stylus profilometry. However, when the surface is soft enough that it deforms under pressure from the SFM tip, resolution will be degraded and topography may not be representative of the true

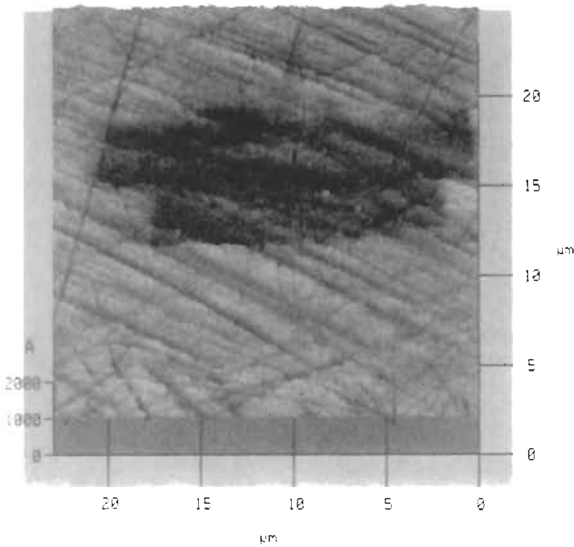

Figure 7 **SFM image of a thin-film read–write head showing magnetic poles (dark rectangles) recessed 200 Å.**

surface. One can investigate the reproducibility of the image by scanning the sample in different directions at various scan rates and image sizes.

Spectroscopy

The preceding topography and profilometry examples have focused on the scanning force microscope. STM also can be used for topographic imaging and profilometry, but the images will be convolutions of the topographic and electronic structure of the surface. A similar effect is seen with SEM, arising from differences in secondary electron coefficients among different materials.

Taking advantage of the sensitivity of the tunneling current to local electronic structure, the STM can be used to measure the spectra of surface-state densities directly. This can be accomplished by measuring the tunneling current as a function of the bias voltage between the tip and sample, or the conductivity, dI/dV, versus the bias voltage, at specific spatial locati ›ns on the surface. Figure 8 is a spectroscopic study of GaAs(110). The image on the left was taken with negative bias voltage on the STM tip, which allows tunneling into unoccupied states, thereby revealing the Ga atoms. Taken simultaneously but with a positive tip bias voltage, the image on the right results from tunneling out occupied states, and shows the positions of the As atoms.

The data above were collected in UHV environment to achieve the most pristine surface. Spectroscopy in air is usually more difficult to interpret due to contamination with oxides and other species, as is the case with all surface-sensitive spectroscopies.

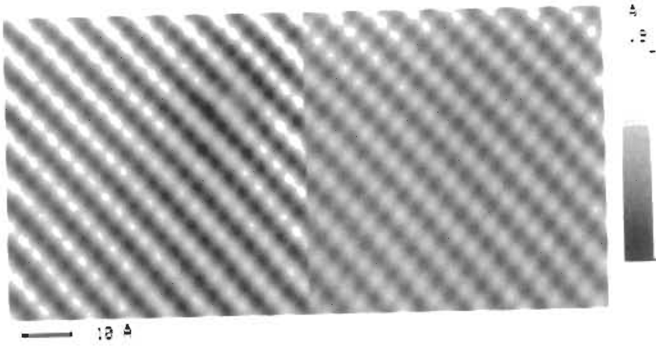

Figure 8 Spectroscopic study of GaAs(110). With a positive voltage on the STM tip, the left-hand image represents As atoms, while the corresponding negative tip voltage on the right shows Ga atoms. (Courtesy of Y. Yang and J.H. Weaver, University of Minnesota)

Sample Requirements

For atomic resolution an atomically flat sample is required to avoid tip imaging (see below). STM requires a conducting surface to establish the tunneling current. Doped Si has sufficient conductivity to enable STM imaging, but surfaces of lower conductivity may require a conductive coating. SFM can image surfaces of any conductivity. Both STM and SFM require solid surfaces that are somewhat rigid; otherwise the probes will deform the surfaces while scanning. Such deformation is easily diagnosed by repeatedly scanning the same area and noting changes.

The deformation of soft surfaces can be minimized with SFM by selecting cantilevers having a low force constant or by operating in an aqueous environment. The latter eliminates the viscous force that arises from the thin film of water that coats most surfaces in ambient environments. This viscous force is a large contributor to the total force on the tip. Its elimination means that the operating force in liquid can be reduced to the order of 10^{-9} N.

An example, Figure 9 is an SFM image of a Langmuir-Blodgett film. This film was polymerized with ultraviolet light, giving a periodicity of 200 Å, which is seen in the associated Fourier transform. The low forces exerted by the SFM tip are essential for imaging such soft polymer surfaces.

Poorly cleaned surfaces may not image well. While ordinary dry dust will be brushed aside by the tip and will not affect the image, oily or partially anchored dirt will deflect the SFM tip or interfere with the conductivity in STM. The result is usually a line smeared in the scan direction, exactly as one would expect if the tip began scanning something which moved as it was scanned. If the sample cannot be cleaned, the best procedure is to search for a clean area.

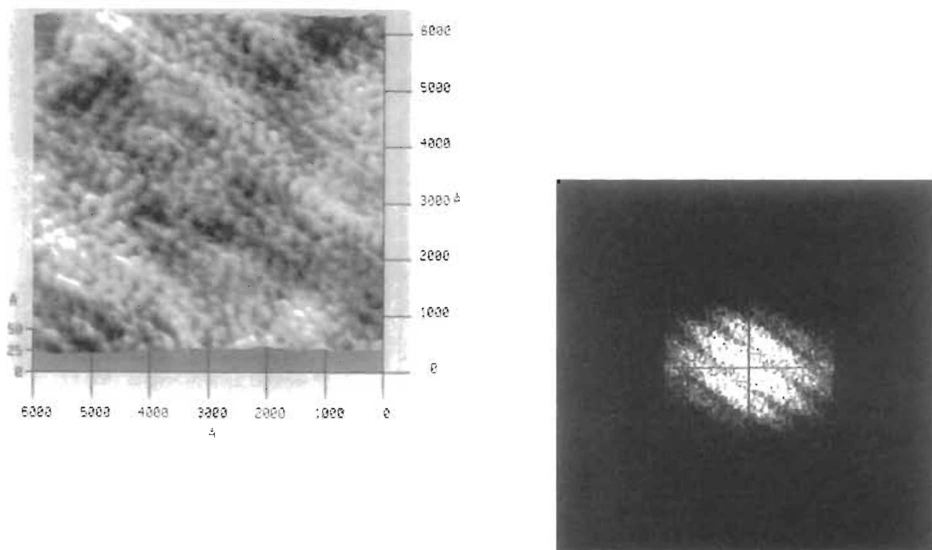

Figure 9 **SFM image of Langmuir-Blodgett film (top) and associated Fourier transform (bottom). (Courtesy of T. Kato, Utsunomiya University)**

Maximum sample sizes that can be accommodated by SFM or STM vary. Current systems can scan a 8-inch Si wafer without cutting it. When industry calls for the capability to scan larger samples, the SPM manufacturers are likely to respond.

Artifacts

The main body of artifacts in STM and SFM arises from a phenomenon known as *tip imaging.*[9] Every data point in a scan represents a convolution of the shape of the tip and the shape of the feature imaged, but as long as the tip is much sharper than the feature, the true edge profile of the feature is represented. However, when the feature is sharper than the tip, the image will be dominated by the edges of the tip. Fortunately, this kind of artifact is usually easy to identify.

Other artifacts that have been mentioned arise from the sensitivity of STM to local electronic structure, and the sensitivity of SFM to the rigidity of the sample's surface. Regions of variable conductivity will be convolved with topographic features in STM, and soft surfaces can deform under the pressure of the SFM tip. The latter can be addressed by operating SFM in the attractive mode, at some sacrifice in the lateral resolution. A limitation of both techniques is their inability to distinguish among atomic species, except in a limited number of circumstances with STM microscopy.

STM

In STM, the tip is formed by an atom or cluster of atoms at the end of a long wire. Because the dependence of the tunneling current upon the tip-to-sample distance is exponential, the closest atom on the tip will image the closest atom on the sample. If two atoms are equidistant from the surface, all of the features in the image will appear doubled. This is an example of multiple tip imaging. The best way to alleviate this problem is to collide the tip gently with the sample, to form a new tip and take another image. Alternatively, a voltage pulse can be applied to change the tip configuration by field emission.

STM tips will last for a day or so in ultrahigh vacuum. Most ultrahigh-vacuum STM systems provide storage for several tips so the chamber does not have to be vented just to change tips. In air, tips will oxidize more rapidly, but changing tips is a simple process.

SFM

At present, all commercial SFM tips are square pyramids, formed by CVD deposition of Si_3N_4 on an etch pit in (100) Si. The etch pit is bounded by (111) faces, which means that the resulting tip has an included angle of about 55°. Therefore the edge profiles of all features with sides steeper than 55° will be dominated by the profile of the tip.

Because many kinds of features have steep sides, tip imaging is a common plague of SFM images. One consolation is that the height of the feature will be reproduced accurately as long as the tip touches bottom between features. Thus the roughness statistics remain fairly accurate. The lateral dimensions, on the other hand, can provide the user with only an upper bound.

Another class of artifacts occurs when scanning vertical or undercut features. As the tip approaches a vertical surface, the side wall may encounter the feature before the end of the tip does. The resulting image will appear to contain a discontinuous shift. Changing the angle of the tip with respect to the sample's surface can minimize the problem. Side wall imaging also occurs in STM, but less frequently since an STM tip has a higher aspect ratio than that of an SFM tip.

Improving the aspect ratio of SFM tips is an area of active research. A major difficulty is that the durability of the tip likely will be compromised as aspect ratios are increased.

Conclusions

Scanning probe microscopy is a forefront technology that is well established for research in surface physics. STM and SFM are now emerging from university laboratories and gaining acceptance in several industrial markets. For topographic analysis and profilometry, the resolution and three-dimensional nature of the data is

unequalled by other techniques. The ease of use and nondestructive nature of the imaging are notable.

The main difficulty with STM and SFM techniques is the problem of tip imaging. Neither technique is recommended for obtaining accurate measurements of edge profiles of vertical or undercut surfaces. In addition, SFM tips cannot accurately image the lateral dimensions of features with sides steeper than 55° at present. Obtaining SFM tips with more suitable aspect ratios is an area of active research.

Scanning tunneling and scanning force microscopes are only two members of the family of scanning probe microscopes. Other types of scanning probe microscopes may become widely used in the near future. The magnetic force microscope, for example, may one day be used routinely to study magnetic domains in storage media.

Related Articles in the Encyclopedia

Light Microscopy, SEM, TEM, STEM, and Surface Roughness

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