FEATURE

Tiny Tips Probe Nanotechnology

by Ineke Malsch

Images and properties emerge from a minute interface

Industry wants precise nanoscale measurements and materials characterization for the production and quality control of nanostructures. Attaining these measurements, however, has proved more difficult than designing and fabricating such structures. In recent years, advances in scanning probe microscopes (SPMs) have led some companies to install these devices in-line for repeatable, accurate measurements of structures in three dimensions.

Since the 1980s, researchers and companies have developed several SPM types and subtypes to image and manipulate nanostructures, including atoms and molecules. Scanning tunneling microscopes (STMs) can image the atomic structure of conducting crystalline surfaces. Scanning near-field optical microscopes (SNOMs) can create optical images of delicate structures—for example, biological molecules on surfaces. Atomic force microscopes (AFMs) can image and analyze a surface down to atomic resolution regardless of its electrical conductivity. Although these instruments are still mostly applied in research and product development, the semiconductor and several other industries are beginning to use AFMs in quality control.

Veeco instruments/R. M. Westervelt, Harvard University

Figure 1. Bubbles and stripes in a 100-μm atomic force microscope image of an 8-μm-thick magnetic garnet film originally developed for magnetic bubble memories.

SPMs use a sharp tip, the probe, which touches or nearly touches a sample's surface to create images or measure the properties of materials. The AFM, in particular, provides two major advantages over optical and scanning electron microscopes—nanometer resolution and three-dimensional images. Manual SPMs usually consist of four elements:

- a piezoelectric or flexure-stage scanner, which scans the sample surface with the probe;
- a detector to read the probe–sample interaction;
- a control station, which includes a computer and an SPM controller–electronics unit that controls the SPM's operation and generates and analyzes digital images; and
- a stage, the structure on which the probe, scanner, and accessories rest during imaging.

In many models, the scanner and detector are combined into one unit, usually called the SPM head.

In general, industry relies more on manual than automated SPMs, but automated units have become almost indispensable in the semiconductor and data-storage industries (Figure 1). Various levels of automation are available, including for the measurement of batch-produced samples, analysis of several images at once, and, at the high end, the interfacing of the microscope with other hardware to minimize human handling of semiconductor wafers during fabrication and to make hundreds of identical measurements in real time.

Origins

Heinrich Rohrer and Gerd Binnig of IBM's Zurich Research Laboratory received the 1987 Nobel Prize in Physics for inventing the STM, the first of the SPMs, in 1981. The STM functions by scanning a tip along a conducting surface and measuring the tunneling current between surface and tip (Figure 2). The result is an image of the electron cloud of the surface atoms (Figure 3). STMs can provide subatomic resolution in lateral and vertical directions. The STM allows the study of atomic structures on conducting or semiconducting surfaces, and it is widely used to study layer growth in the semiconductor industry.

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Veeco instruments/A. Olbrich, Infineon, Munich, Germany

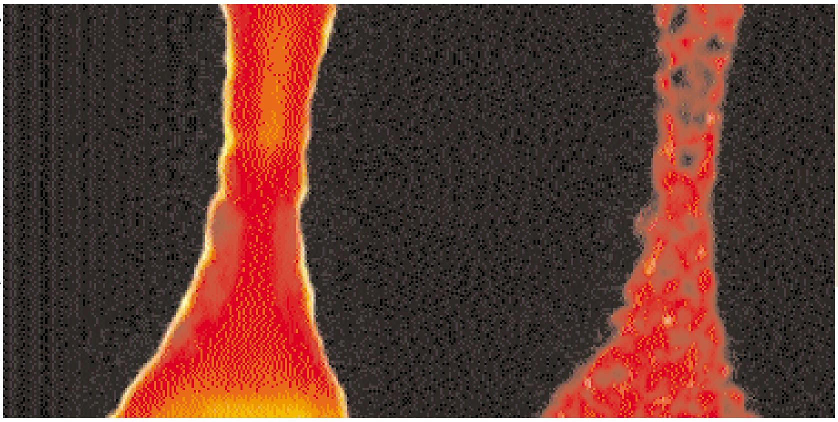


Figure 2. Topography (left) and tunneling current image (right) of an 8.5-nm-thick silicon dioxide tunnel oxide for an electrically erasable and programmable read-only memory device.

The SNOM functions by tunneling light through a small hole, which enables the measurement of structures that are smaller than the wavelength of the light itself. D. W. Pohl, now at the University of Basel in Switzerland, and colleagues invented the SNOM in 1984. Even very small structures on a surface, such as a biological molecule, reflect light in the visible spectrum. However, biological molecules are smaller than the wavelengths of the light used in optical microscopes. Hence, the contribution of a single molecule is too small for a normal optical microscope to distinguish.

In a SNOM, the probe consists of an optical fiber with a sharp tip covered with a nonoptical coating that has a small opening at the end. The tip is brought in contact with the sample and moved across it to illuminate the surface. The light does not directly image the sample, but as the tip moves, a tiny dithering motion occurs in the X–Y plane. The microscope relies on this contact and motion to generate the topographical image. The advantage of the SNOM is that it generates an optical image alongside a topographical image. SNOMs are used to study living cells and biomolecules; for characterization and analysis applications in optoelectronics and telecommunication; and in imaging structures on transparent surfaces.

Atomic Forces

Gerd Binnig and Christoph Gerber of IBM Zurich and Cal Quate of Stanford University invented the AFM in 1986. This instrument measures atomic forces on a surface by scanning a sharp tip attached to a flexible cantilever across the sample. The tip diameter can be as small as 5 nm (10^{-9} m). An optical readout or a piezoelectric crystal translates the motion of the cantilever into an electronic signal. The outcome is a three-dimensional image of the surface structure displayed on a screen. Maximum resolution is typically on the atomic scale in the lateral and vertical dimensions, and the AFM enables the measurement of forces as small as 10^{-12} N. “This allows atomic resolution and interacting with the short-range forces of the chemical bonds,” Gerber says. “With these characteristics, the instrument has surpassed the resolution of the STM on distinct surfaces.”

Over the years, researchers have adapted the AFM and its functioning to suit different applications. In the contact mode, the cantilever lightly drags the tip across the surface and follows the surface structure. Contact is necessary for applications such as scratch tests. In the tapping or noncontact mode, the AFM cantilever–tip assembly oscillates up and down as it scans across the surface. This mode is much less disruptive to soft samples such as delicate biological molecules or certain surface features (Figure 4).

AFM tips play a key role in performance. Their useful lifetime varies with the type of sample, its topography, and the imaging mode. Some tips operating on automated systems will endure for days without breaking, while others may last for only a few uses. Tips are usually made of silicon or silicon nitride, and they may be uncoated or coated with materials such as aluminum, a cobalt alloy, or diamond, depending on their intended use. Sometimes, tiny spheres are used instead of tips, not for imaging but for various force measurements.

Researchers continue to adapt the AFM to measure other properties, including chemical, mechanical, electrical, and magnetic. The friction force microscope (FFM) moves a sharp tip across a surface in the contact mode. The friction between the surface and tip twists the cantilever. A laser beam, reflected by the cantilever, hits an optical detector screen. The location of the detected light is a measure of the cantilever’s torsion and, hence, of friction. Thus, FFMs can acquire chemical contrast images, which show different chemical compositions of distinct surface areas and have many applications, including in polymer blends, polymer wetting, and thin-film technology.

Veeco Instruments/Y. Kim and M. Soriaga, Texas A&M University, College Station

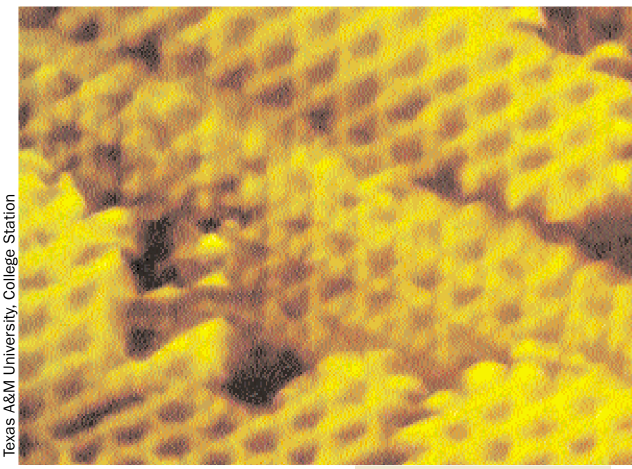
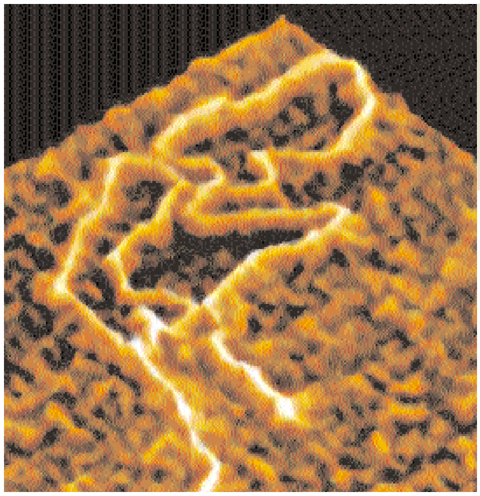


Figure 3. A 15-nm electrochemical scanning tunneling microscope image of a (3 × 3) iodine lattice on a palladium monolayer electrodeposited on a platinum (111) surface.

FFMs allow the study of the basic mechanisms of sliding under various environmental conditions. “The company IAVF in Karlsruhe, Germany, investigates surfaces of machine parts, applying FFM,” says Ernst

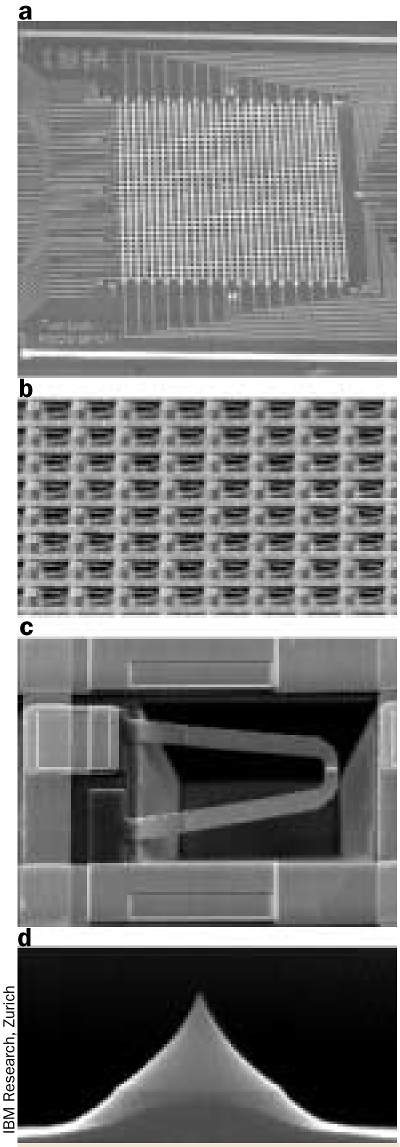


Veeco Instruments/W. Blaine Stine (stineb@stineb.pprd.abbott.com)

Figure 4. Tapping-mode atomic force microscope provides clear and reproducible resolution of DNA on mica.

Meyer of the Institute of Physics at the University of Basel. "They characterize the tribomutated layer that forms after the running in of a machine. This layer is only 10 to 50 nm thick, but it determines the long-term performance of the motor." Other companies use FFMs in research on friction and rheology between two machine parts. "In our own institution, we have proved that one monolayer of lubricant is enough to reduce friction by a factor of 10," Meyer says.

The magnetic force microscope (MFM) is a noncontact AFM. It can measure the stray field of ferromagnetic and superconducting samples, which indicates the location of ferromagnetic domain walls. And it can locate vortices in superconductors, which result from surface defects. The MFM tip also can change the magnetic state of a sample. Widely used for quality control in the data-storage industry, it can determine the sensitivity and response of magnetic recording heads and storage media.



IBM Research, Zurich

Figure 5. IBM's millipede chip (a), only 7 × 14 mm, consists of an array (b) of addressable cantilever (c) silicon tips (d), and operates as a nanomechanical storage device.

Semiconductor uses

Although there are at least 20 SPM suppliers and manufacturers worldwide, Veeco Instruments (Santa Barbara, CA) is the market leader for research and industrial SPMs. In 2001, Veeco Instruments' sales increased to $134 million from $98 million the year before, a 37% increase, according to Veeco's annual report. After research, the semiconductor industry is the main market for AFMs. Several semiconductor companies use Veeco's AFMs on pilot production lines for semiconductor wafers to measure circuit features, including the topography of side walls and surface roughness. Infineon Technologies AG (Munich, Germany), formerly Siemens Semiconductors, "is applying the AFM as a metrology tool in production processes," says Jim Flach of Thermo Instruments BV (Breda, The Netherlands), a Veeco distributor in the Benelux countries. "They fabricate small structures and use the AFM to measure them. Several hard-disk manufacturers, such as Seagate, apply it to measure magnetic read–write heads."

Other companies use a technique called conducting atomic force microscopy as a small volt-and-ampere meter to measure miniaturized electronic elements such as transistors. The advantage of the system is that electronic information can be achieved on a nanometer scale, which reveals crucial information for the developers of semiconductor production processes. As the dimensions of structures shrink below 100 nm, the materials no longer behave the same way as they do in bulk. Conducting AFM has been most useful for assessing the uniformity of dielectric thin films such as the gate oxide of a transistor.

The industrial market for AFMs is slowly emerging. Veeco Instruments has steadily increased its market share, in part by buying or merging with competitors. Other companies have withdrawn themselves from the market. In the late 1990s, Zygo Corp. (Middlefield, CT) distributed AFMs made by IBM, but in March 2000, it stopped. The Zygo/IBM instrument is a type of AFM that is apparently difficult to manufacture. It can measure not only the sides of a nanostructure but also the height, which is an important attribute for future chip production. Veeco has just released a new, automated AFM, the Dimension X3D, for measuring in-line semiconductor structures in three dimensions.

Not everyone is scared off by the market leader in industrial SPMs, however. For example, Nanosurf AG (Liestal, Switzerland) produces portable AFMs and STMs that are inexpensive and easy to handle. They are about 10 cm in diameter and can be powered and controlled by a laptop computer. Nanosurf's main market is secondary schools and universities, but the company also focuses on industrial quality control.

SPM prices are not the main barrier to industrial sales. Indeed, the instruments manufactured by Veeco, the market leader, are the most expensive. SPMs are relatively slow because one tip must scan across a surface line-by-line. To address this problem, groups such as IBM's Zurich Research Laboratory are working on arrays of tips, including a millipede read–write head for polymeric data-storage disks. This 7 × 14-mm chip may find future use in mobile telephones, digital cameras, and other portable electronic systems (Figure 5).

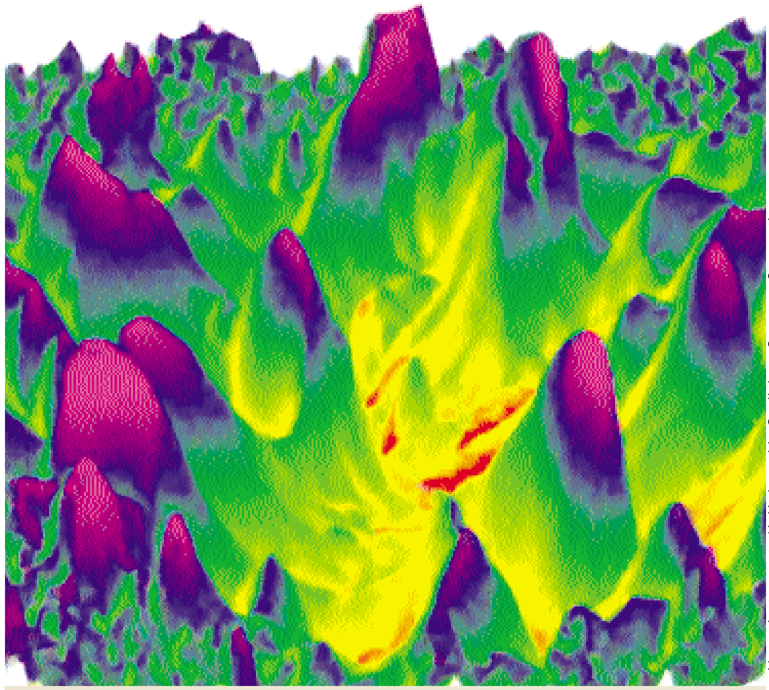
Joost Frenken of the Kamerlingh Onnes Laboratory at

Leiden University in The Netherlands has proposed an in vivo STM that would produce moving images. This instrument would be especially useful for studying chemical reactions or even processes in living cells. “Frenken was one of the first researchers in the world working on this,” comments Flach. The potential industrial applications in the pharmaceutical and fine-chemical industries are easy to imagine.

Standards

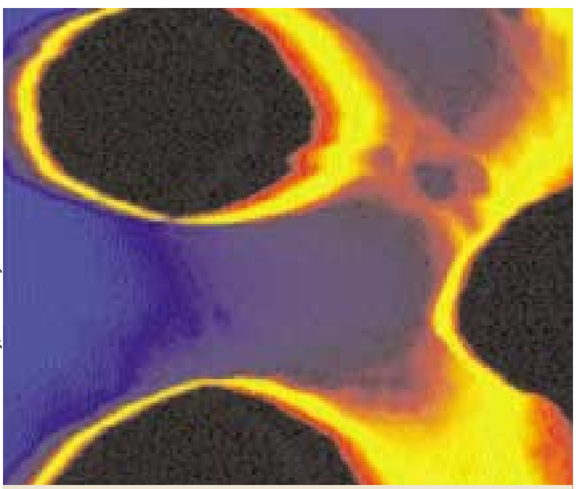
The National Measurement Institute of Germany’s Physical Technical Federal Institute recently did an interlaboratory comparison of the step-height determination of AFMs and found a wide spread in measurement results. Standardization organizations including CEN/STAR and VAMAS worry that a lack of standards hampers the greater use of SPMs by industry. CEN-STAR is the European Committee for Standardization/Standardization and Research group, an organization that deals with new technologies. VAMAS, the Versailles Project on Advanced Materials and Standards, was conceived by the world’s leading economic nations, including the G7 countries and the European Union, to support world trade in products dependent on advanced materials technologies through international projects aimed at providing the technical basis for harmonized measurements, testing, specifications, and standards.

In June, standards experts from the United States, Europe, and Japan began discussing a strategy for new working groups on nanotechnology at a workshop at the National Physical Laboratory in Teddington, England. They proposed developing standards guidelines for SPM operations and tip calibration, and carrying out more interlaboratory comparisons of SPM measurements to calibrate instruments.



Veeco Instruments/J. Hyde and L. Guglielmo, Quantum Corporation

Figure 6. A 20-µm atomic force microscope image of a laser-induced feature on a polymer substrate.



Veeco Instruments/I. Lee, J. Lee, and E. Greenbaum, Oak Ridge National Laboratory, University of Tennessee

Figure 7. A 3-µm atomic force microscope image of minor surface defects caused by an etching process in a microchannel plate.

The development and evolution of SPMs has had a great impact on scientific and industrial research, and industry is beginning to explore and exploit the capabilities of these microscopes in quality control and production. The acceptance of SPMs for industrial uses should be further spurred with the development of SPM standards.

Further reading

Stephan Altmann of the European Molecular Biology Laboratory (Heidelberg, Germany) lists SPM research groups and companies worldwide at http://www.embl-heidelberg.de/~altmann.

Other sites: www.veeco.com; http://www.nanotribo.org; http://www.zurich.ibm.com/st/nanoscience/cantilever.html.

Benatar, L. E.; Park, S. I. SPMs Step from Laboratory to Industry. *The Industrial Physicist*, June 1998, pp. 34–37.

Gnecco, E.; Bennewitz, R.; Meyer. E. Abrasive Wear on the Atomic Scale. *Phys. Rev. Lett*. **2002**, *88* (21), 215501.

Hegner, M.; Güntherodt, H.-J. Nanometer Scale Science and Technology—The impact of STM and AFM 2001; http://monet.physik.unibas.ch/~hegner/chapter.htm.

Meyer, E.; Overney, R. M.; Dransfeld, K.; Gyalog, T. *Nanoscience: Friction and Rheology on the Nanometer Scale*; World Scientific Publishing: Singapore, 1998; 392 pp.

van Hulst, N. F. Scanning Near-Field Optical Microscopy.

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