The Children of the STM

The Nobel Prize-winning scanning tunneling microscope has inspired a whole generation of imaging devices that use everything from magnetic forces to sound waves to examine samples

GARY McCLELLAND drags a sharp probe across a graphite surface and watches as it catches briefly, slips, and then catches again in a regular pattern. It seems like a straightforward—perhaps even dull—demonstration until McClelland, a physicist at IBM's Almaden Research Center in San Jose, California, explains that the slip-stick cycle is not created by some irregularity in the surface. The surface is as smooth as a surface gets. Instead, the probe is responding to a landscape of microscopic hills and valleys created by the surface atoms themselves. It is observing friction at the level of individual atoms.

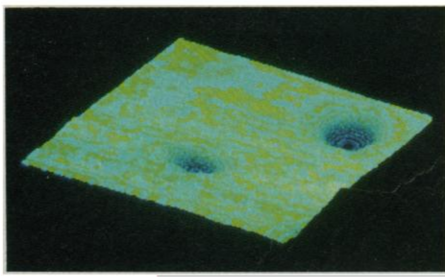
McClelland's friction force microscope is one of a growing family of devices that are changing the way scientists look at matter. Instead of focusing light, x-rays, or electrons on a sample, these scanning probe microscopes map out a surface by "feel," much as a blind man taps his cane to explore the ground in front of him.

In many cases, the new microscopes offer better resolution than was previously available, but perhaps more important is their ability to see a sample in new ways. One member of the family, for instance, is sensitive to magnetic fields and can be used to examine the magnetic patterns on a computer hard disk. A second was designed to monitor ion flow into and out of living cells. Others measure electric charge, temperature, optical spectra, and residual stress. The devices have opened a new window on the microscopic world.

The patriarch of the family is the scanning tunneling microscope (STM), invented in 1981 by Gerd Binnig and Heinrich Rohrer of IBM's Zurich Research Laboratory. The STM, which won the 1986 Nobel Prize in Physics for its creators, examines a sample by means of a tiny probe that is brought to within a nanometer (a billionth of a meter) of the surface. A voltage applied between the tip of the probe and the sample creates a small tunneling current—a flow of electrons that "leaks" or "tunnels" through the gap between tip and sample—whose magnitude depends sensitively on the width of the gap. As the probe is scanned across the sample, the tip is moved up and down to keep the tunneling current constant. By monitoring this up-and-down movement of the tip, a computer produces a topological map showing the bumps and valleys formed by the atoms on the sample's surface.

The STM is sensitive enough to show individual atoms, giving it better resolving power than the best electron microscopes. In addition, it does not demand special sample preparation and it does not damage a sample as do the high-energy electrons of electron microscopes. In less than 10 years, these assets have made the STM a mature technology. Several companies manufacture them for commercial sale, and there are hundreds of STMs around the world being used to look at metals, semiconductors, small molecules adsorbed onto surfaces, and, with some extra effort, even large biological molecules such as DNA.

Paul Hansma, University of California, Santa Barbara



Mathew Mate, IBM

Portrait of versatility. *Atomic force microscope images of a white blood cell (top) and microscopic concavities in a layer of liquid.*

But the STM has its limitations. Because it depends on a tunneling current, it does not work particularly well on nonconductors, such as biological molecules and cells. And even when the STM works perfectly, it provides only one detail about a sample—the shape of the electron cloud surrounding the atoms on its surface. The map it produces can therefore be difficult to interpret if there are several types of atoms present.

So in the past few years researchers have developed other scanning probe microscopes that scan a sample the same way STMs do but depend on different types of interactions between the tip and surface and thus provide different information about the sample.

These children of the STM include:

■ **Atomic force microscope.** In 1985, Binnig, along with Calvin Quate of Stanford University and Christoph Gerber, also from IBM's Zurich laboratory, modified the STM to avoid the need for a tunneling current. Instead of bringing the tip close to the sample, they pushed it right up against the surface. Keeping the force between tip and surface constant, the tip is scanned across a sample like a needle running along a groove in a phonograph record. Recording the tip's motion produces a topographic image of the sample similar to that from an STM.

Because the atomic force microscope does not depend on a current, it can look at nonconductors as easily as conductors. Paul Hansma, a physicist at the University of California, Santa Barbara, says it can examine hard surfaces with a resolution of 1 to 2 angstroms—good enough to resolve individual atoms. It doesn't work as well with soft materials, such as living cells, he says, because the tip tends to distort their surfaces. So far his sharpest pictures of proteins show no details smaller than about 50 angstroms, good enough to see overall molecular shapes but not adequate to trace the positions of individual atoms in proteins. Hansma has also imaged blood cells, but with a resolution of only 100 to 200 angstroms. To get more detail, he is building a low-temperature atomic force microscope in which samples will be firmed up by freezing them in liquid nitrogen.

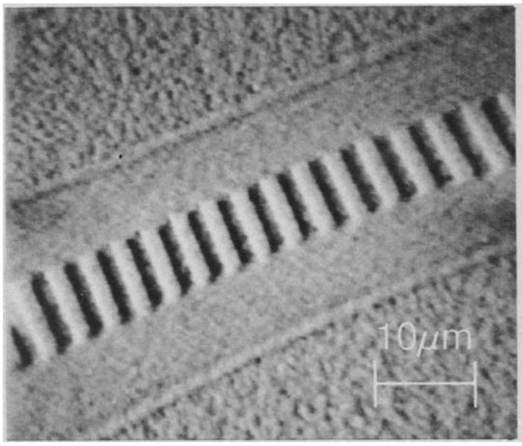
Like the STM, the atomic force microscope has caught on quickly with researchers. At least one company, Digital Instruments in Santa Barbara, has started to make atomic force microscopes commercially and others have announced production plans.

■ **Friction force microscope.** In 1987, McClelland and IBM-Almaden colleagues Mathew Mate, Ragnar Erlandsson, and Shirley Chiang modified an atomic force microscope so that they could measure how much the tip drags as it is moved across a surface. The goal, McClelland says, was to investigate friction at an atomic scale. It is nearly impossible to see directly what happens when two surfaces rub against each other, he notes, because their interface is hidden and because the frictional force is not spread evenly over each surface but is concentrated where bumps in the surfaces catch against each other. Using the tip of the atomic force microscope as one of the two surfaces gets around these difficulties and "in principle allows one to measure the friction of a single atom," McClelland says.

Practice has not quite caught up with principle yet, however. The IBM group has used downward pressures on the tip that range from 10 micrograms up to several milligrams. These pressures create a contact region several nanometers or more across, McClelland says, while a single atom measures 0.2 to 0.3 nanometer. Although this means that the researchers are not seeing the friction of just a single atom, they are seeing the next best thing.

As the probe drags across the graphite surface, the friction increases and decreases at regular intervals of about 2.5 angstroms—a distance equal to the spacing between atoms on a graphite surface. This implies that although the contact area is much larger than a single atom across, the microscope is still able to distinguish atom-sized variations of frictional force. No one knows exactly why this is so, McClelland says, and he is trying to improve the microscope in order to take a closer look at how the frictional forces arise. Understanding friction on the atomic level is important for designing lubricants and materials that will stand up to wear.

■ **Magnetic force microscope.** The success of the atomic force microscope pointed the way to analogous scanning microscopes that depend on other types of forces acting between the tip and the sample. One of the first was developed in 1987 by Yves Martin and Kumar Wickramasinghe at IBM's T. J. Watson Research Center in Yorktown Heights, New York. By using a magnetized tip on a probe that was scanned a few tens of nanometers over the surface, the researchers found they could map out magnetic fields along a sample with a resolution of 100 nanometers. That number has since been improved to 25 nanometers, Wickramasinghe says.



John Mamin, IBM

Magnetic bits. *A magnetic force microscope images test pattern on a computer hard disk.*

In the magnetic force microscope, the probe is vibrated up and down at close to its resonance, or "natural," frequency, so that a small applied movement generates a large, easily observable motion at the end of the probe. When the tip of the probe is brought close to the sample—typically to within 50 to 100 nanometers—the force of the sample's magnetic field changes the probe's resonance frequency, which in turn produces detectable changes in the amplitude of the probe's oscillations. A laser beam reflected off the probe detects the amplitude change, and a computer transforms the amplitude measurements into a reading of the magnetic field over the sample.

The magnetic force microscope has been particularly useful in studying magnetic recording devices, such as computer hard disks and the recording heads that store information on them, says Dan Rugar, a physicist at IBM-Almaden. It proved its worth when another IBM researcher came to him with an experimental hard disk that was not performing up to design. Teaming with co-worker John Mamin, Rugar imaged the pattern of magnetic fields on the disk and found the problem was self-erasure—some of the closely packed bits of information laid down by the recording head were being spontaneously transformed by the magnetic fields on the disk. This knowledge of exactly how the disk was failing was critical for successful redesign of the system, Rugar says.

■ **Electrostatic force microscope.** A force microscope with an electrically charged tip will respond to electric charges on a surface. Early in 1988, Martin and Wickramasinghe along with David Abraham, also of IBM–T. J. Watson, reported using such a microscope to study the electronic properties of a silicon sample. Wickramasinghe says the device should be particularly useful for studying the distribution and concentration of dopant ions in semiconductors, information that is important for understanding the performance of integrated circuit chips.

More recently, Bruce Terris and co-workers at IBM-Almaden have used an electrostatic force microscope to study contact electrification—the static electric charge created when two surfaces touch or rub against each other. Understanding how these static charges behave is important to such things as xerography, Terris notes—the toner particles used in copy machines are charged by contact electrification so their placement on a piece of paper can be controlled electrically. The IBM group deposits charges on a surface by touching the tip of the microscope to the sample and then pulls the tip back to scan the surface and see how the charge distributes itself. So far, they have a resolution of about 200 nanometers and a theoretical sensitivity of three electronic charges, but Terris says they hope to improve to the point where they can see a single electron.

■ **Attractive mode force microscope.** Another IBM–Yorktown Heights product, this device depends on the attractive forces between molecules. When the probe tip is brought within 2 to 20 nanometers of the sample, the two are pulled together by van der Waals forces and by the surface tension created by water molecules that condense out of the air between the tip and the sample.

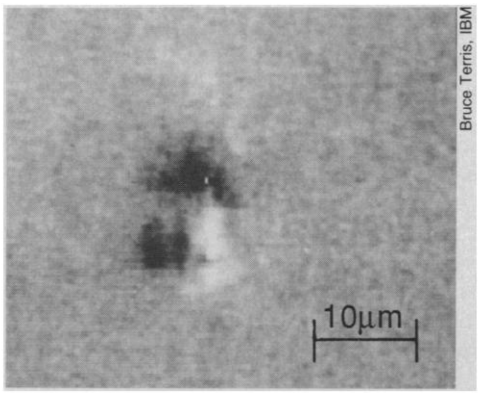
Monitoring this attractive force with the same type of vibrating probe used in the magnetic force microscope produces a topographic map of the sample's surface. The information is similar to that provided by the atomic force microscope but the laser force microscope has the advantage that there is no physical contact between its tip and the sample—an important consideration for applications in semiconductor manufacturing, since contact with the tip can damage delicate circuits.

■ **Scanning thermal microscope.** It is the world's smallest thermometer, Wickramasinghe says. He and Clayton Williams of IBM–Yorktown Heights put a tiny thermocouple on a scanning probe. Since the voltage induced across the thermocouple is proportional to its temperature, they can scan the probe across a surface and record temperature changes. The microscope, which has a resolution of tens of nanometers, could be used to monitor temperature variations in living cells, Wickramasinghe suggests, or to measure flow speeds in a stream of gas or liquid by watching how fast the tip cools off when immersed in the stream.

■ **Optical absorption microscope.** Wickramasinghe also applies the scanning thermal microscope to determine the chemical makeup of a surface by using absorption spectroscopy. The technique depends on the fact that elements vary in the efficiency with which they absorb light of different wavelengths. Thus shining a laser on a sample heats up some atoms more than others, and the thermal microscope can detect these temperature differences. Varying the wavelength of the laser light gives an absorption spectrum of the surface, which in turn gives its chemical composition.

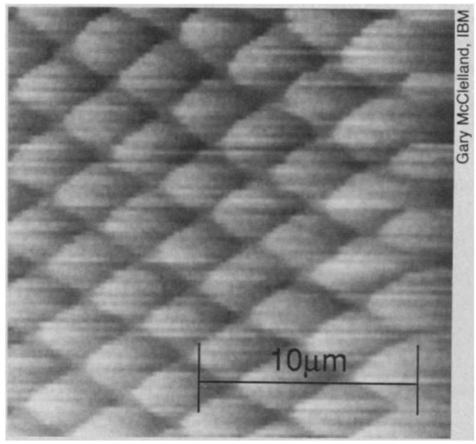
Recently Wickramasinghe realized that he did not have to put a thermocouple on the tip of the probe to get an absorption spectrum. Since the microscope tip and the surface are composed of different conductors, they will themselves form a thermocouple when brought close together. Shining a laser on the surface as before, Wickramasinghe can thus monitor the variation in voltage between tip and surface and calculate surface temperature from that. This method generates an absorption spectrum of the surface with a resolution of 1 nanometer. "We should be able to record the spectrum of a single molecule," he says.

■ **Scanning ion-conductance microscope.** UC Santa Barbara's Hansma makes probes from glass micropipettes with tips having an inner diameter of 50 to 100 nanometers (see *Science*, 3 February 1989, p. 609). When a probe tip is placed in a salt [sodium chloride] solution covering a sample, the sodium and chlorine ions will carry a current through the pipette until the pipette comes in contact with the sample, which blocks the ion flow. The current flowing through the pipette thus provides a sensitive indication of how close the tip is to the sample surface. By scanning the tip in the normal way, Hansma can map out the surface topography. He envisions the microscope's most effective use to be mapping ion flows through the membranes of living cells, which can provide useful information about how excitable cells, such as nerve and muscle cells, respond to stimulation.



Charged surface. *Regions of negative (black) and positive (white) charge imaged by an electrostatic force microscope.*

■ **Scanning near-field optical microscope.** With a normal-lensed microscope that uses visible light, it is not possible to get resolution better than about 200 nanometers, or approximately half the wavelength of the light. But the normal limit can be circumvented by using a near-field technique, where either the light source or the detector is smaller than the light's wavelength and is brought up very close to the subject (see *Science*, 1 July 1988, p. 25). Dieter Pohl of IBM-Zurich calls this "optical stethoscopy" in reference to a physician's stethoscope: Although the sound of a heartbeat has a wavelength of many meters, a doctor can locate the heart to within a few centimeters by placing a stethoscope close to the source of the sound.



Atomic friction on graphite. *Friction varies from high (bright) to low with a periodicity of 2.5 angstroms; tip was dragged from left to right.*

In an analogous fashion, Pohl, Michael Isaacson at Cornell University in Ithaca, New York, and several other researchers have pieced together visible light pictures of samples, pixel by pixel, by scanning them with probes that have apertures 50 nanometers or less in diameter. The resolution is now about 30 to 50 nanometers, Isaacson says, and he expects to improve that to 10 nanometers and perhaps as little at 1 nanometer with very fine micropipettes. Because visible light does not damage samples and can be used in air or water, the near-field microscopes should be valuable in examining biological structures, such as viruses or chromosomes, in vivo.

■ **Scanning acoustic microscopes.** At Stanford, Quate's program to develop a scanning acoustic microscope actually predates the invention of the STM. Since the mid-1970s, he and various students have been imaging samples with sound, much like sonar except on a much smaller scale. IBM's Rugar and John Foster, who were both Quate students, were able to get 30- to 50-nanometer resolution by using sound with a frequency of 8 gigahertz and cooling samples to less than 0.5 K in liquid helium. At colder temperatures, the speed of sound is lower and the wavelength correspondingly shorter, making the resolution better.

More recently, Steven Meeks at IBM-Almaden has built a scanning phase-measuring acoustic microscope to examine residual stress in semiconductors. Unlike Quate's liquid helium acoustic microscope, which maps out details of the surface, Meeks' device can peer 5 micrometers down into a sample to detect stresses that can lead to cracking. It works by measuring the change in the velocity of sound from one part of the sample to another. Residual stresses, which are internal stresses left over from the manufacturing process, alter the speed of sound in the material.

■ **Molecular dip-stick microscope.** A clever application of the atomic force microscope rather than a distinct device, the molecular dip stick illustrates the versatility of the new of microscopes. To measure the thickness of lubricant films, such as used to protect computer hard disks, Mate of IBM-Almaden lowers the tip of an atomic force microscope into the lubricant like a dip stick probing a car's oil reservoir. The tip feels a strong attractive force from surface tension when it touches the lubricant and then a repulsive force when it reaches the underlying surface, so Mate can calculate the thickness of the film. The molecular dip stick can map out the depth of lubricant to an accuracy of 5 angstroms.

What's next? Molecular air pressure gauges? Or perhaps atomic radar microscopes? The only thing that seems certain is that the family of scanning probe microscopes will continue to proliferate.

■ ROBERT POOL

ADDITIONAL READING

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