

ATOMIC FORCE MICROSCOPY

It is surprisingly easy to make a cantilever with a spring constant weaker than the equivalent spring between atoms, allowing a sharp tip to image both conducting and nonconducting samples at atomic resolution.

Daniel Rugar and Paul Hansma

In 1986 Gerd Binnig and Heinrich Rohrer shared the Nobel Prize in Physics for inventing the scanning tunneling microscope and discovering that it can image individual surface atoms with unprecedented resolution.¹ The success of the scanning tunneling microscope has led to the invention of a host of other "scanning probe" microscopes, which rely on mechanically scanning a sharp tip over a sample surface.² The atomic force microscope is one of the most successful of these new devices.³

Binnig got the idea for the atomic force microscope in 1985 while he was in California, on leave from IBM's research laboratory in Zurich, Switzerland. Prior to going on leave, Binnig had been so busy with his work on the scanning tunneling microscope and with helping others to get started in the field that he had found little time to think about new ideas. While in California, as a visitor at Stanford University and the IBM Almaden Research Center, he found more time to think. According to Binnig, the inspiration for the atomic force microscope came as he lay on the floor of his house. He looked up and noticed the subtle structure of the ceiling, which reminded him of the topography seen in scanning tunneling microscope images. He wondered why the surface must always be imaged with a current; why not a force? Among other things, using a force would have the advantage that

insulating samples could be imaged, unlike the case with the scanning tunneling microscope.

Binnig talked about his ideas with Christoph Gerber of IBM and Calvin Quate of Stanford, and when they calculated the forces between atoms, they were surprised to find that they could easily make a cantilever with a spring constant weaker than the equivalent spring between atoms. For example, the vibrational frequencies ω of atoms bound in a molecule or in a crystalline solid are typically 10^{13} Hz or higher. Combining this with the mass of the atoms, of order 10^{-25} kg, gives interatomic spring constants k , given by $\omega^2 m$, on the order of 10 N/m. For comparison, the spring constant of a piece of household aluminum foil that is 4 mm long and 1 mm wide is about 1 N/m. They believed that by sensing angstrom-size displacements of such a soft cantilever spring, one could image atomic-scale topography. Furthermore, the applied force would not be large enough to push the atoms out of their atomic sites.

As the ideas became more focused, Gerber dropped everything he was working on and was able to build the first prototype in just a few days. The results that Binnig, Quate and Gerber obtained with this prototype started the field of atomic force microscopy.³

A sensitive instrument

The concept of using a force to image a surface is a general one and can be applied to magnetic and electrostatic forces as well as to the interatomic interaction between the tip and the sample. Whatever the origin of the force, all force microscopes have five essential components:

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- ▷ A sharp tip mounted on a soft cantilever spring
- ▷ A way of sensing the cantilever's deflection
- ▷ A feedback system to monitor and control the deflection (and, hence, the interaction force)
- ▷ A mechanical scanning system (usually piezoelectric) that moves the sample with respect to the tip in a raster pattern
- ▷ A display system that converts the measured data into an image.

The scanning, feedback and display systems are very similar to those used for scanning tunneling microscopy.

Although the basic elements of all force microscopes are similar, the details of implementation vary. The original atomic force microscope, for example, used a handmade cantilever spring formed from a piece of gold foil approximately 1 mm long. A small diamond stylus glued to the foil served as the tip. Today, the most advanced AFM cantilevers are microfabricated from silicon, silicon oxide or silicon nitride using photolithographic techniques.⁴ Typical lateral dimensions are on the order of 100 microns, with thicknesses of order 1 micron. This geometry gives spring constants in the range of 0.1–1 N/m and resonant frequencies of 10–100 kHz. The cantilevers can be fabricated with integrated tips, or else small diamond chips can be glued on by hand.

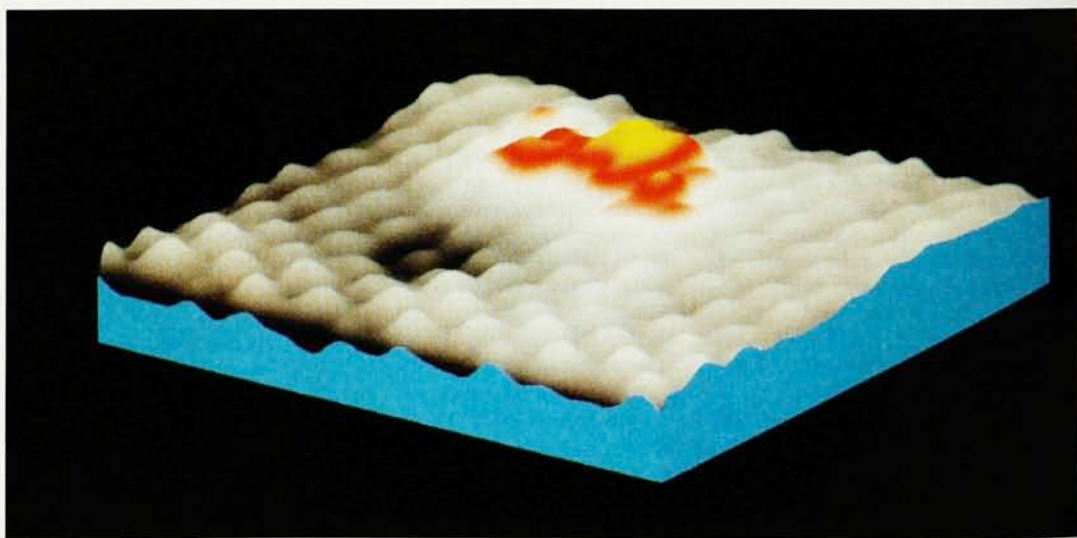
Some of the best force sensors for magnetic and electrostatic imaging have been made from fine, electrochemically etched wires. The etched wires have a tapered geometry that varies from about 10 microns in diameter at the point of attachment to less than 500 Å at the end. The end of the wire may be bent with a knife edge to form a tip.

The other critical component of the AFM is the sensor

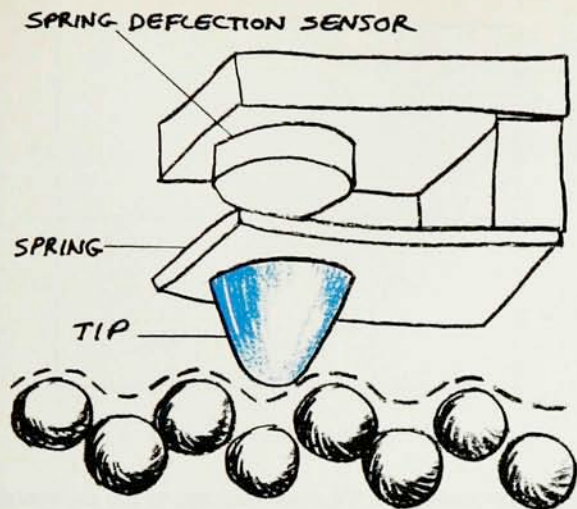
that detects the cantilever's deflection. Ideally, the sensor should have subangstrom sensitivity and should exert negligible force on the cantilever. Electron tunneling, which was the method Binnig used, has the virtue of being extremely sensitive: The tunneling current between two conducting surfaces changes exponentially with distance, typically by a factor of 10 per angstrom of displacement. Although excellent results have been achieved using tunneling detection, it has the disadvantage that its performance can be degraded if the tunneling surfaces become contaminated. For this reason, among others, most AFMs being designed now use some form of optical detection.

Optical detection schemes are divided into two basic types: interferometry⁵ and beam deflection.⁶ Both of these methods are capable of measuring cantilever deflections on the order of 0.1 Å with a detection bandwidth of dc to 10 kHz. In a typical beam-deflection AFM, light from a diode laser is specularly reflected from a mirror-like cantilever surface. The direction of the reflected light beam is sensed with a position-sensitive (two-element) photodetector.

Interferometer systems used for atomic force microscopy have taken many different forms, and some of the more recent methods are almost as simple to implement as beam deflection.⁷ One of the main advantages of interferometry is that it can be used with cantilevers that don't have a mirror-like reflecting surface. This is especially important for magnetic and electrostatic imaging, which often use fine wire cantilevers. Some of the early AFM interferometers had rather poor performance at low frequencies due to the large physical path difference between the reference beam and the light reflected from



Individual molecule of sorbic acid on a well-ordered graphite surface, imaged by an atomic force microscope. The protruding ridge that is highlighted in color is attributed to the 8-angstrom-long physisorbed molecule. The atomic corrugations on the graphite surface are also visible in the image. The entire image is 25×25 angstroms. (Image by Thomas R. Albrecht, Douglas P. E. Smith and Calvin F. Quate, Stanford University.)



Scheme for an atomic force microscope. The tip is fastened to a cantilever spring that has a lower spring constant than the effective spring between two atoms. With sufficient sensitivity in the spring deflection sensor, the tip can reveal surface profiles with atomic resolution.

the cantilever. This problem has now been solved by using either a fiberoptic technique that places a reference reflector within microns of the cantilever or by using a two-beam differential technique.

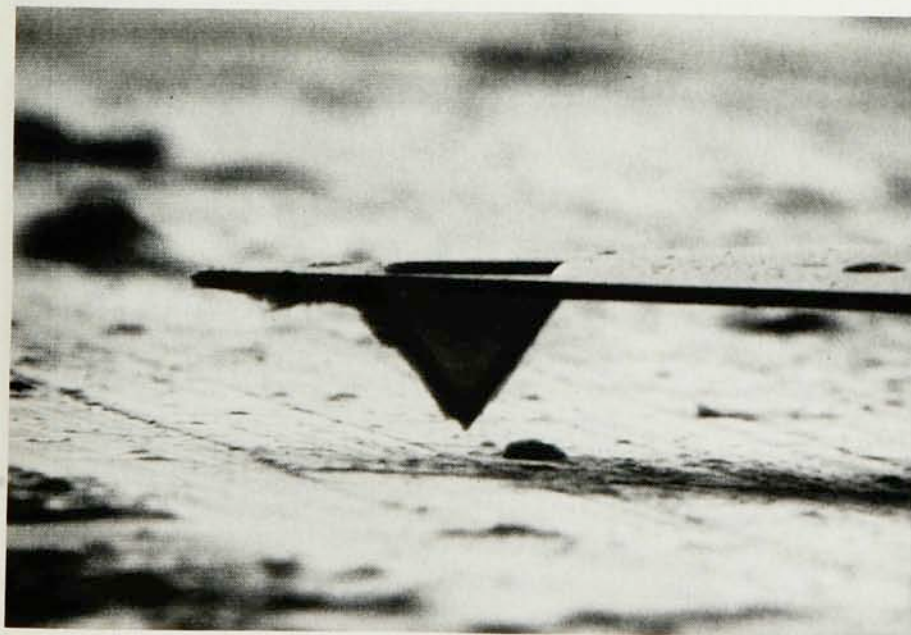
Because the AFM is such a sensitive instrument, care must be taken to ensure that external vibrations, such as from the laboratory building, do not limit its performance. The effect of external vibration is to cause unwanted motion of the tip with respect to the sample and the deflection sensor. The immunity of the AFM to external vibration depends on the frequency ν of the vibration relative to the lowest resonant frequency ν_0 of the mechanical system. The mechanical system includes both the cantilever and the rest of the AFM. The amplitude of relative tip motion is attenuated by a factor $(\nu/\nu_0)^2$ in the limit $\nu \ll \nu_0$. Thus, if the lowest resonant frequency is greater than 20 kHz, a typical 20-Hz building vibration of amplitude 1 micron results in relative tip motion of less than 0.01 Å, a nearly harmless level. Because cantilevers can be readily made with high resonant frequencies, the limiting factor is usually the rest of the AFM. Good AFM designers focus on making the mechanical components of

the AFM rigid and compact, especially in the path from the cantilever to the sample.

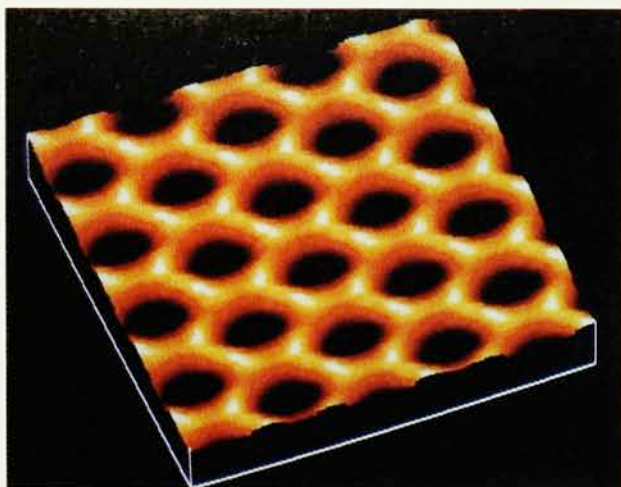
Atomic resolution

One of the most exciting results from atomic force microscopy has been the discovery that atomic resolution can be achieved when the tip is in contact with the sample while scanning.⁸ Conceptually, this "contact mode" of imaging is like using a stylus profilometer to measure the topography of surface atoms. At first, this idea may seem implausible, especially when one considers the macroscopic size of typical AFM tips. The AFM achieves such high resolution by using a very small loading force on the tip—typically 10^{-7} to 10^{-11} N—which makes the area of contact between the tip and sample exceedingly small. This small and well-controlled loading force is the essential difference between the contact-mode AFM and earlier stylus profilometers,⁹ which typically used loading forces on the order of 10^{-4} N.

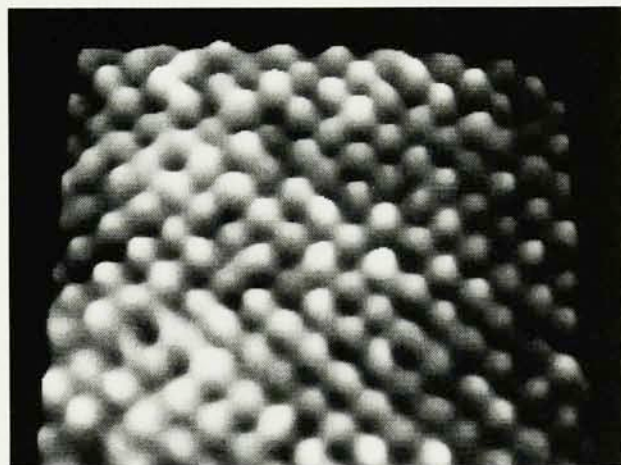
Atomic resolution has been achieved on a number of different materials, including both insulators and conductors. The first materials that showed atomic corrugations



Microcantilever with microfabricated tip for a contact-mode atomic force microscope. This silicon nitride cantilever was manufactured by Park Scientific Instruments, Mountain View, California. (Photograph by Greg Kelderman.)



Individual carbon atoms in graphite. The atoms are only 1.5 Å apart, but can be imaged with an unusually good tip. (From Srinivas Manne and Scot Gould, University of California, Santa Barbara.)



Sodium chloride crystal surface, at atomic resolution. (From G. Meyer and N. M. Amer, reference 10).

were layered materials, such as graphite,⁸ molybdenum sulfide and boron nitride.⁴ More recently, atomic structures on gold and on the ionic crystals sodium chloride and lithium fluoride have been observed.¹⁰

The interpretation of atomic resolution AFM images depends critically on the details of the tip-sample interaction, a topic of ongoing theoretical study.¹¹ Ideally, the interaction between tip and sample would be via a single atom at the end of the tip. However, according to theoretical calculations assuming common interaction forces, it appears that this ideal is not realized in practice. Instead, the force is probably transmitted through a small irregularity on the tip, on the order of a few atoms in size. Because the atomic configuration at the end of the tip depends on the particular tip used, the structure seen within the unit cell of a crystal may be subject to some variability.

In addition to observing structure on atomically smooth surfaces, the AFM has successfully imaged surfaces with adsorbed organic molecules, such as sorbic acid,

DNA and proteins.¹² To avoid damage to the molecules, the interaction force must be kept below 10^{-8} N. This is difficult to achieve in air due to meniscus forces arising from capillary condensation—that is, due to the thin water and hydrocarbon films that are ubiquitous on surfaces in air. One way to overcome this problem is to operate the AFM with the sample, tip and cantilever immersed in a liquid such as water or ethanol.¹³

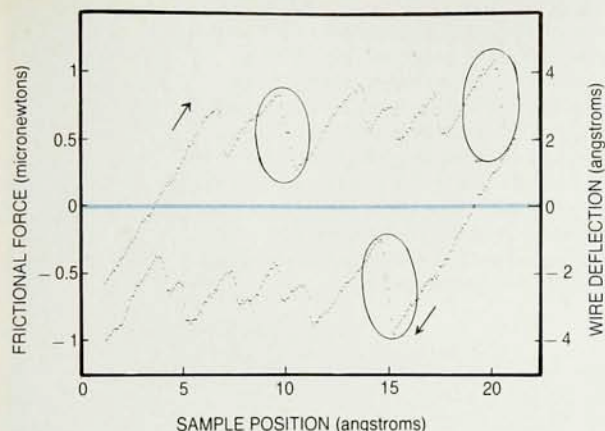
Besides allowing smaller interaction forces, operation under water also opens the possibility of observing biological samples in a physiologically relevant environment. For example, the AFM has imaged, in real time, the polymerization of fibrin, a protein important in blood clotting. The images reveal that in the initial stages, polymer chain growth occurs not by the addition of monomers to a few long chains, but by the fusion of many short chains to form progressively longer chains.¹²

Some old subject areas, such as friction and elasticity, have been found to yield new phenomena when studied with atomic or molecular resolution. For example, an atomic scale stick-slip phenomenon was discovered by measuring the lateral force on the cantilever while scanning the tip across a graphite surface.¹⁴ This is a manifestation of friction on an atomic scale and provides a new perspective in tribology. Another new research area is the distribution and wetting behavior of liquid films, such as lubricants, when they are only a few nanometers thick.¹⁵

The contact-mode AFM also excels at topographic imaging of larger scale objects. Whole blood cells, integrated circuits and recording pits in optical disks are just a few of the samples that have been studied. Indeed, technological applications appear to be abundant because the AFM can make three-dimensional quantitative measurements with higher resolution and on a wider variety of samples than virtually any other technique.

Long-range forces

When the tip is in contact with the sample, the interaction is dominated by relatively short-range interatomic forces. By moving the tip away from the sample by 10–100 nm, longer range forces, such as magnetic, electrostatic and attractive van der Waals forces, become accessible. This type of noncontact imaging is usually performed using a different method of force detection. Instead of measuring static cantilever deflections, as in contact-mode atomic force microscopy, the cantilever is driven to vibrate near its resonant frequency by a small piezoelectric element.³ The presence of a force, or more precisely the gradient of the force, $\partial F_z / \partial z$, acts to modify the effective spring constant C of the cantilever according to $C = C_0 + \partial F_z / \partial z$,



Atomic-scale friction measured by observing the transverse deflection of a tungsten cantilever scanning a graphite sample. A stick-slip effect is observed: The tip moves with the surface until the force on the cantilever exceeds the frictional force; the cantilever then slips one or two lattice spacings. The circled sections indicate double slips. The wire spring constant is 2500 N/m; the load is 56 μN . (From reference 14.)

where C_0 is the spring constant of the isolated cantilever. For example, if the sample exerts an attractive force on the cantilever, the spring constant will effectively soften. As a result, the resonant frequency will decrease, and this decrease is then detected by measuring the amplitude, phase or frequency change of the vibration.

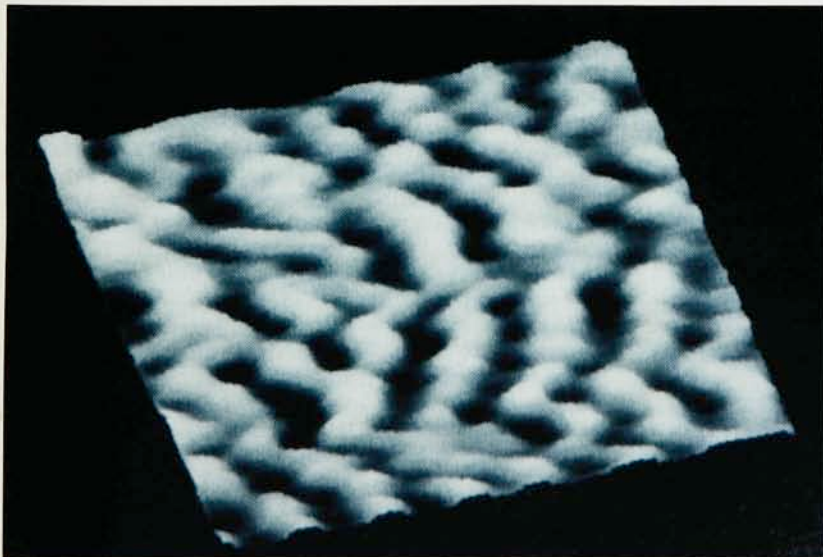
The force sensitivity of this method of resonance shift detection is set in part by the finite Q of the resonance (typically about 100) and by thermal vibration of the cantilever, which is the dominant noise source. The thermal vibration amplitude is determined by the equipartition theorem and is given by $\frac{1}{2} C_0 \langle (\Delta z)^2 \rangle = \frac{1}{2} kT$. For a 1 N/m cantilever at room temperature, the rms amplitude is 0.6 Å. Under typical operating conditions, the thermal noise limits the detectable force gradient to about 10^{-5} N/m. To see how this force gradient corresponds to force, consider a typical $1/r^2$ type force. With a tip-to-sample spacing of 100 nm, this 10^{-5} N/m force gradient corresponds to a force of 5×10^{-13} N, a value that would be difficult to measure from the static deflection of the cantilever. One could expect to measure even smaller forces by using cantilevers with higher Q values or by operating the AFM at lower temperatures.

By sensing Coulomb forces, an AFM with an etched wire tip can nondestructively image the distribution of electrical charge on a surface.¹⁶ Insulating samples can be locally charged *in situ*, either by applying a voltage pulse

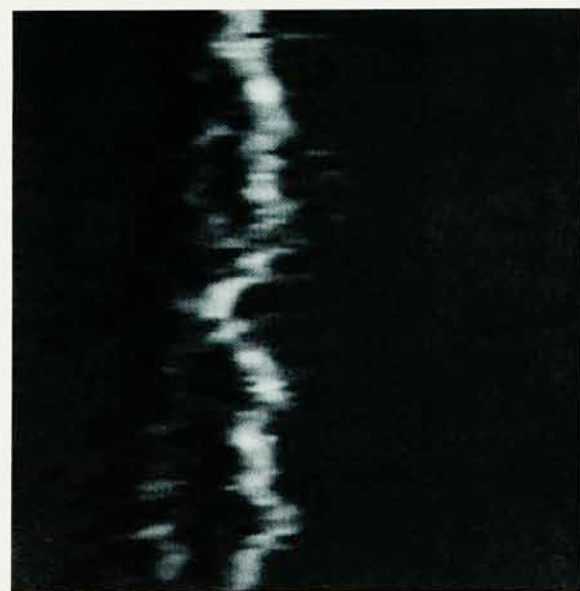
to the tip (creating a localized discharge) or by contact electrification (simply touching the tip to the insulator surface with no voltage applied). The resulting charge patterns can be imaged with 100-nm resolution, which is at least an order of magnitude better than previous nondestructive charge imaging techniques. In an initial contact electrification study, a polymer surface was contact charged with a nickel tip and found to contain regions of both negative and positive charge. This bipolar charge transfer was unexpected, as was the fact that the charged region was significantly larger than the area of contact. In other electrostatic force applications, the AFM has been used to perform potentiometry on a microscopic scale¹⁷ and to image domain walls in ferroelectrics.¹⁸

In principle, the force microscope should be sensitive enough to detect single electrons. For an electron-to-tip separation of 10 nm, the force gradient due to the interaction of an electron with its image charge in the tip is 6×10^{-5} N/m. Although this force gradient is in principle a detectable value, more work will be necessary to demonstrate conclusively the microscope's ability to image a single electron.

If the tip has a magnetic moment, then magnetic fields that emanate from magnetic samples can be sensed due to the force $\nabla(\mathbf{m} \cdot \mathbf{B})$. This is the basis of magnetic force microscopy.¹⁹ The first magnetic force imaging experiments used tips made from iron or nickel wire. More



Polyaniline, a biopolymer, at high resolution. The center-to-center spacing of the adjacent polymer chains is only 5 Å. This contact-mode atomic force microscope image was made under water. (From reference 12.)



Polymerization of the blood protein fibrin from an aqueous solution into a net that is the molecular fabric of blood clots. This sequence of contact-mode atomic force microscope images is from a video tape that captured the polymerization in real time. Gaps visible in the first image are filled in by the second image. The third image shows the net widening. After about 30 minutes from the initiation of the process, the fibrin net nearly covered the 450×450 nm field of view. (From reference 12.)

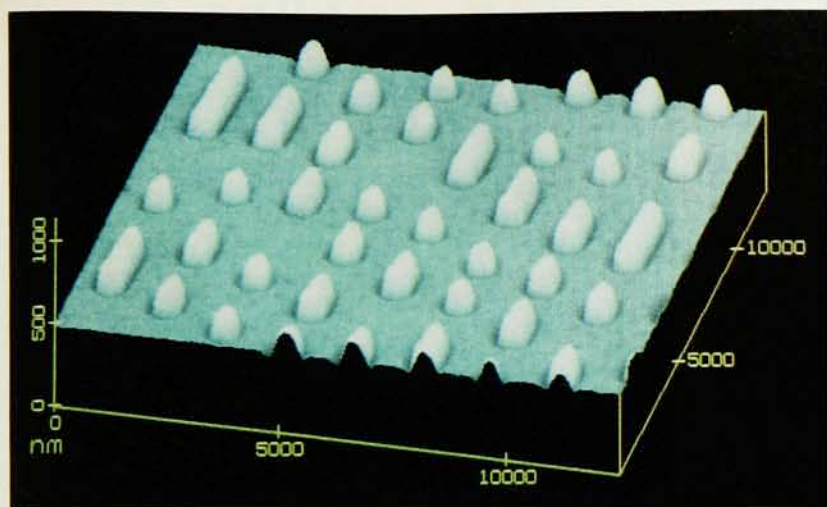
recently, silicon microcantilevers with thin-film magnetic coatings have been successfully demonstrated. The lateral resolution of magnetic imaging is determined by the overall tip shape and the spacing between tip and sample. Resolution on the order of 20 nm has been reported.²⁰

One of the key advantages of magnetic force microscopy over competing electron-beam imaging techniques is that no special sample preparation is required. Even samples with thin, nonmagnetic overcoats are acceptable because magnetic fields from the sample will penetrate through the overcoat. This capability is especially important when examining certain technological samples, such as magnetic and magneto-optical recording disks. Images of bit patterns on magnetic recording materials can give important information about the writing process, such as the size and shape of the bits, as well as information about media noise, overwrite characteristics and the material's ability to sustain high data densities. Magnetic force microscopy has also been applied to soft magnetic materials, such as Permalloy and iron single crystals.²¹ Among other things, the technique can sense the direction in which the magnetization rotates within Bloch walls and can see features such as Bloch lines.

The future

Part of the future for atomic force microscopy is easy to predict. Because of its great versatility as a metrology tool, the AFM will find numerous applications in product development and quality control in the optical, semiconductor and magnetic recording industries. Engineers and scientists will make quantitative measurements of things ranging from surface roughness to magnetic bit shapes and from integrated circuit topography to lubricant thickness. The ability to measure three-dimensional profiles with nanometer resolution should make the AFM an important tool for inspecting optical disk stampers, measuring linewidths on integrated circuit masks, and other applications. The AFM is also well suited for visualizing thin-film growth morphology and grain size. This large base of technological applications will stimulate the development of instruments that are easy to use and that can handle large samples such as whole magnetic disks and silicon wafers. It will also stimulate innovations such as the recent measurement of surface conductance using a contact-mode AFM with a conducting tip.²²

In the physical sciences, the AFM will contribute new knowledge by allowing familiar phenomena such as friction, contact electrification, elasticity and wetting to be



Stamper used to mold compact disks. The data bumps on this metallic mold leave 60-nm-deep pits in amorphous polyolefin optical disks. This contact-mode atomic force microscope image shows the bumps lined up in tracks that are 1.6 microns apart. (Digital Instruments photograph.)

studied on a smaller scale than previously possible. It is not yet known whether the AFM will make significant contributions to traditional surface science problems. Only a few microscopes have been operated under ultrahigh vacuum conditions with atomically clean surfaces. One interesting application would be to use the AFM to observe surface reconstructions on both insulators and conductors. More work will be necessary, however, to prove that the AFM tips have sufficient resolution and stability to make a significant contribution in this area. A good test case would be the imaging of the silicon 7×7 surface reconstruction, which is the surface reconstruction that made the scanning tunneling microscope famous.

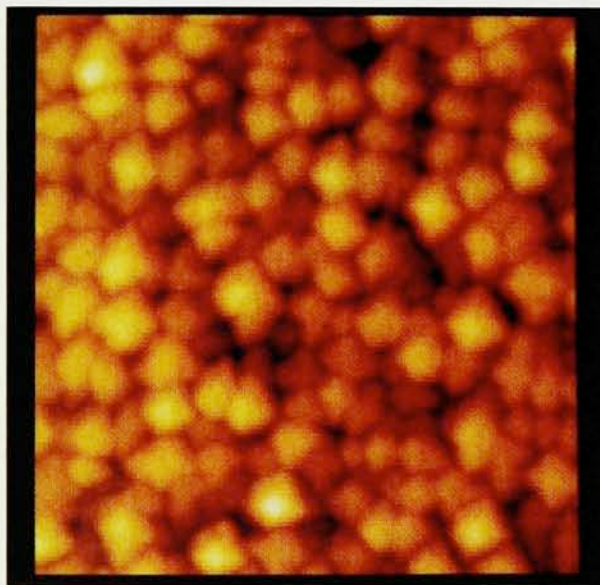
Biological applications of atomic force microscopy, though technically challenging, are destined to be of great importance. Already the AFM has imaged individual biological molecules such as amino acids, biopolymers such as DNA, macromolecules such as proteins, and even entire cells. In some cases it has even been possible to watch biological processes as they occur, as in the polymerization of the blood clotting protein fibrin, which was described above. We can expect to see significant benefits if this real-time imaging capability can be extended to include processes on cell surfaces. It may be possible, for example, to observe the process by which viral particles, such as the AIDS virus, attach onto cell membranes. Perhaps the most exciting potential application of the AFM is the sequencing of DNA. Several groups working with AFMs and other scanning probe microscopes are learning how to hold DNA molecules fixed on a substrate and are attempting to distinguish individual bases. Although this is a very challenging goal, there is no fundamental reason why the technical problems cannot be solved. When they are solved, it may be possible to reduce the time necessary to sequence DNA, such as that of the human genome, by orders of magnitude.

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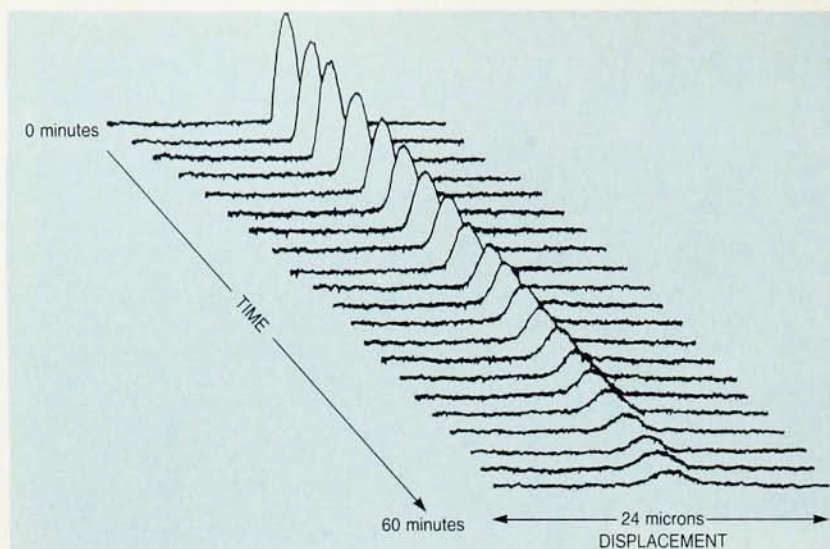
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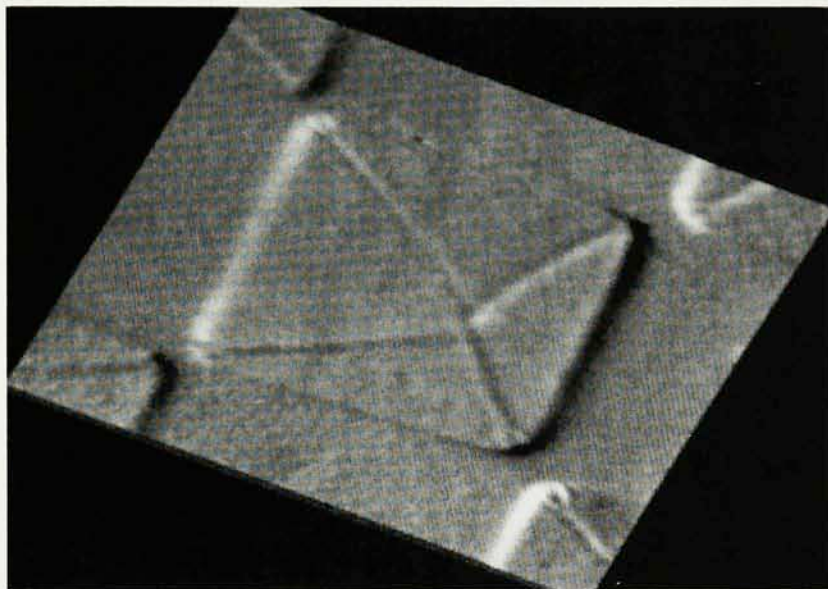
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Superconductor film, 200-nm thick. The yttrium barium copper oxide film was sputter-deposited by Chang-Beom Eom of Stanford University and imaged with an atomic force microscope. Note the remarkably square grains in this c -axis oriented film. The imaged area is 2 microns on an edge. (Park Scientific Instruments photograph.)



Discharge of an electrostatically charged region on the surface of an insulator. Plotted are contours of constant electrostatic force gradient, as measured by an atomic force microscope over the charged region. The area scanned is 24 microns by 5 microns. (From J. E. Stern, *et al.*, reference 16.)



Magnetic force image of a 30-nm-thick $\text{Ni}_{80}\text{Fe}_{20}$ (Permalloy) square. Naturally occurring domain walls can be seen criss-crossing the central square, which is 20 microns wide. (Image by Peter Grütter, IBM.)

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