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In the Classroom

Atomic Force Microscopy

CHERYL R. BLANCHARD
Southwest Research Institute
San Antonio, TX 78238
cblanchard@swri.edu

Atomic force microscopy is a powerful tool allowing a variety of surfaces to be imaged and characterized at the atomic level.

Scanning probe microscopes (SPM) define a broad group of instruments used to image and measure properties of material, chemical, and biological surfaces. SPM images are obtained by scanning a sharp probe across a surface while monitoring and compiling the tip–sample interactions to provide an image. The two primary forms of SPM are scanning tunneling microscopy (STM) and atomic force microscopy (AFM). STM was first developed in 1982 at IBM in Zurich by Binnig, et al. [1]. The invention of the scanning tunneling microsope (for which Binnig and Rohrer were awarded the Nobel Prize in Physics in 1986) has had a great impact on the technical community by providing a new and unique tool to advance fundamental science and technology. Although the ability of the STM to image and measure material surface morphology with atomic resolution has been well documented, only good electrical conductors are candidates for this technique. This significantly limits the materials that can be

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In the past 20 years, the number of instrumental techniques available to the chemist has grown exponentially. In order to help our readers keep up with this rapidly growing field, tutorial articles on chemical instrumentation will be a regular feature of The Chemical Educator. The articles are designed to serve as a brief introduction to emerging instrumental techniques, with an outline of the underlying principles and major applications.

—Martin Schimpf, Series Editor

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studied using STM and led to the development, in 1986, of the atomic force microscope by Binnig, Quate, and Gerber [2]. This enabled the detection of atomic scale features on a wide range of insulating surfaces that include ceramic materials, biological samples, and polymers.

Prior to the invention and commercial availability of SPMs, researchers traditionally used (and still use) a variety of microscopes to image surfaces and measure surface morphology on a microscale. Optical microscopes are the most common instrument available to image any sample that is not completely optically transparent. Resolution is limited to about 1µm and only images and size measurements from features lying in the surface (*x*-*y*) plane are obtainable. Also, optical microscopy has a relatively small depth of field. A more advanced technique, scanning electron microscopy (SEM), has been widely used, since its inception in the mid-1900s, to image microscopic features on sample surfaces. SEMs provide much greater resolution (~5nm) than optical microscopes and have a relatively large depth of field. Because a beam of electrons must travel to the sample to provide an image, the samples must be vacuum compatible and either electrically conductive or coated with a conductive layer to avoid charge buildup.

AFM provides a number of advantages over conventional microscopy techniques. AFMs probe the sample and make measurements in three dimensions, *x*, *y*, and *z* (normal to the sample surface), thus enabling the presentation of three-dimensional images of a sample surface. This provides a great advantage over any microscope available previously. With good samples (clean, with no excessively large surface features), resolution in the *x*-*y* plane ranges from 0.1 to 1.0 nm and in the *z* direction is 0.01 nm (atomic resolution). AFMs require neither a vacuum environment nor any special sample preparation, and they can be used in either an ambient or liquid environment. With these advantages AFM has significantly impacted the fields of materials science, chemistry, biology, physics, and the specialized field of semiconductors.

Contact mode AFM is one of the more widely used scanning probe modes, and operates by rastering a sharp tip (made either of silicon or Si_3N_4 attached to a low spring constant cantilever) across the sample. An extremely low force (~10^{-9} N, interatomic force range) is maintained on the cantilever, thereby pushing the tip against the sample as it rasters. Either the repulsive force between the tip and sample or the actual tip deflection is recorded relative to spatial variation and then converted into an analogue image of the sample surface.

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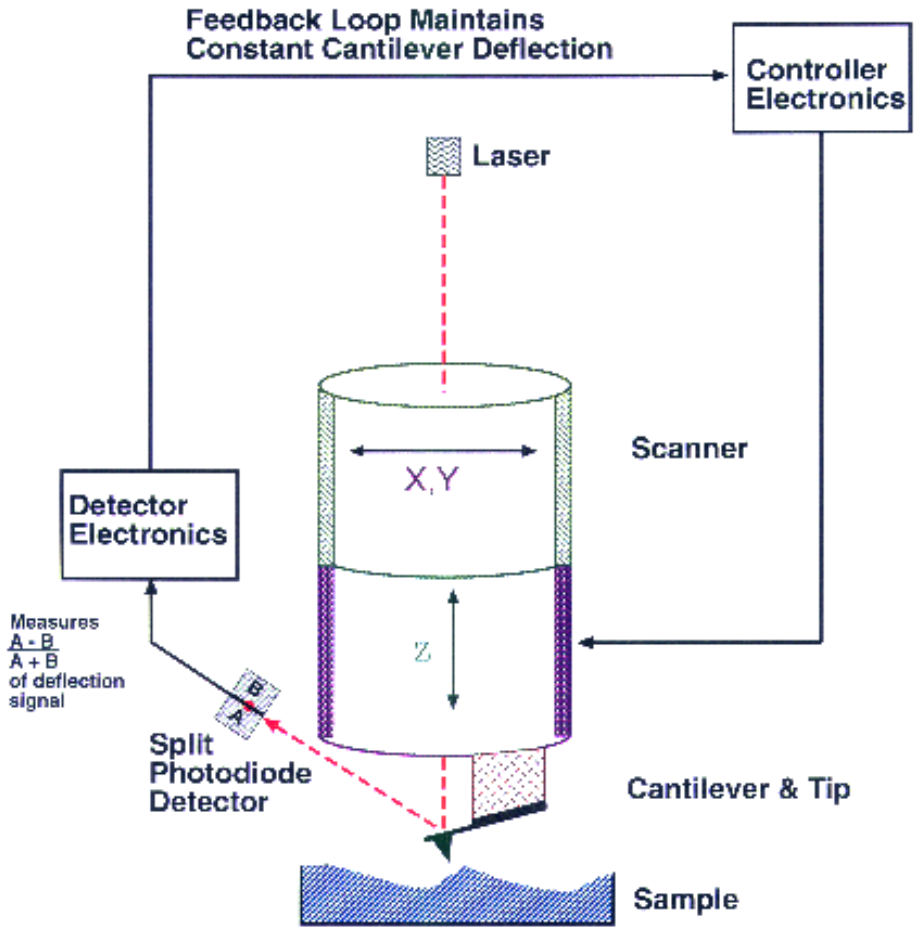

FIGURE 1. SCHEMATIC DIAGRAM SHOWING THE OPERATING PRINCIPLES OF THE AFM IN THE CONTACT MODE (COURTESY OF DIGITAL INSTRUMENTS, SANTA BARBARA, CA).

The principal behind the operation of an AFM in the contact mode is shown in Figure 1. The AFM tip is first brought (manually) close to the sample surface, and then the scanner makes a final adjustment in tip–sample distance based on a setpoint determined by the user. The tip, now in contact with the sample surface through any adsorbed gas layer, is then scanned across the sample under the action of a piezoelectric actuator, either by moving the sample or the tip relative to the other. A laser beam aimed at the back of the cantilever–tip assembly reflects off the cantilever surface to a split photodiode, which detects the small cantilever deflections. A feedback loop, shown schematically in Figure 1, maintains constant tip–sample separation by moving the scanner in the *z* direction to maintain the setpoint deflection. Without this feedback loop, the tip would “crash” into a sample with even small topographic features (although this

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FIGURE 2. LABORATORY SETUP OF THE DIMENSION 3000 SPM INCLUDING THE SCANNER, SAMPLE DISPLAY SCREEN, CONTROL SCREEN, PROCESSOR, AND IMAGE DISPLAY SCREEN (PHOTO COURTESY DIGITAL INSTRUMENTS, SANTA BARBARA, CA).

phenomenon can happen even with careful AFM operation). By maintaining a constant tip-sample separation and using Hooke's Law ($F = -kx$ where F is force, k is the spring constant, and x is the cantilever deflection), the force between the tip and the sample is calculated. Finally, the distance the scanner moves in the z direction is stored in the computer relative to spatial variation in the x-y plane to generate the topographic image of the sample surface.

The laboratory setup for an AFM (specifically the Dimension[1] 3000 SPM, shown in Figure 2, includes the scanner, sample display screen, control screen, processor, and image display screen. A special table to isolate mechanical and acoustical vibrations is also usually necessary to perform high resolution (atomic scale) work.

[1] Dimension and Bioscope are trademarks and Nanoscope is a registered trademark of Digital Instruments, Santa Barbara, CA.

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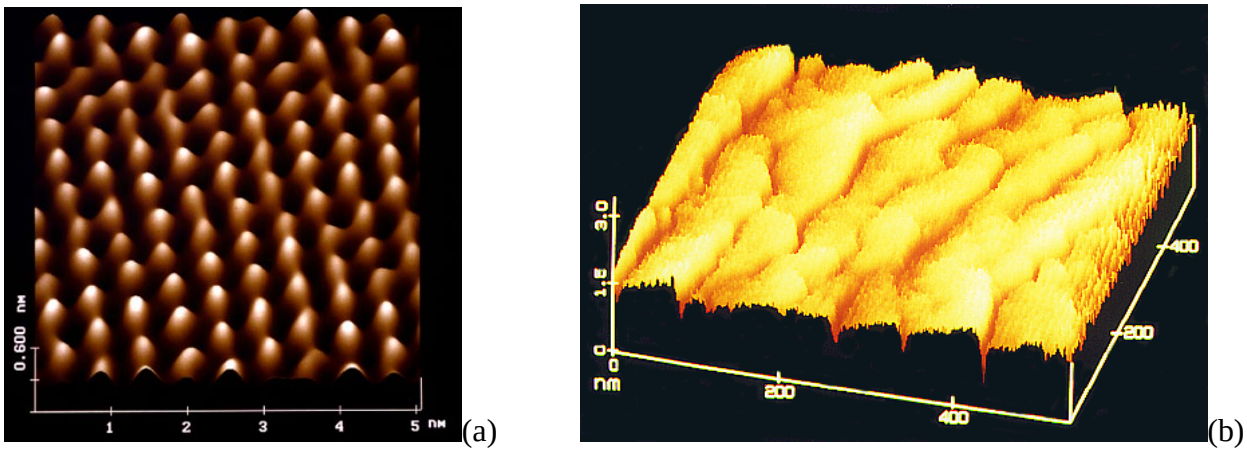

(a) (b)

FIGURE 3. IMAGES GENERATED USING CONTACT MODE AFM. (A) A 5nm SCAN ATOMIC SCALE IMAGE SHOWING SURFACE ATOMS ON FRESHLY CLEAVED MICA. IMAGE (A) WAS TAKEN WITH A NANOSCOPE SPM (COURTESY DIGITAL INSTRUMENTS, SANTA BARBARA, CA), AND (B) AN IMAGE OF BOVINE BONE OBTAINED IN THE WET CELL.

Examples of images generated using contact mode AFM are shown in Figure 3. A freshly cleaved surface of mica was imaged with a BioScope[1] AFM to reveal the atomic structure of mica in Figure 3a. Figure 3b illustrates the use of the wet cell in contact mode (Imaged with a NanoScope[1] SPM), which is a great advantage when studying biological materials, to image bovine bone. The hard hydroxyapatite mineral phase appears to be raised above the interconnecting soft collagen phase.

Recent advances in the development of AFM technology have led to a number of useful imaging modes including TappingMode[2] and LiftMode[2] AFM. Although operating in the contact mode has proven successful, it suffers from a number of drawbacks that limit its use on a number of sample types. First, the constant downward force on the tip often damages (and thus changes) many softer surfaces (polymers and biological samples) and even some hard surfaces such as silicon. Also, many samples, such as small particles or biological samples like DNA and cells, must be placed on a substrate for imaging purposes. In contact mode, the sample is often destroyed or even pushed out of the field of view by the rastering tip. These complications have been addressed through the development of TappingMode AFM. In the TappingMode, the AFM tip–cantilever assembly oscillates at the sample surface while the tip is scanned; thus, the tip lightly

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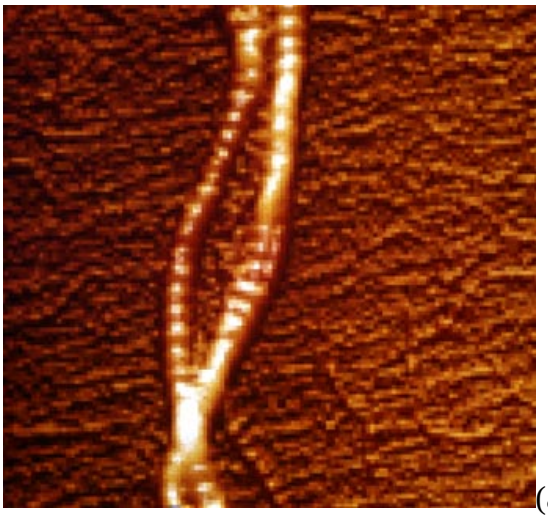
(a)

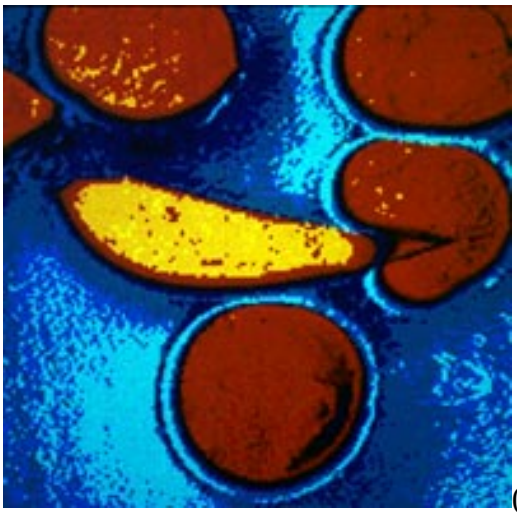
(b)

FIGURE 4. AFM IMAGES ACQUIRED IN THE TAPPINGMODE. (A) A 2µm SCAN OF FIBRILLAR COLLAGEN AND (B) NORMAL AND SICKLED HUMAN RED BLOOD CELLS. IMAGES TAKEN WITH A NANOSCOPE SPM (COURTESY OF DIGITAL INSTRUMENTS, SANTA BARBARA, CA).

taps the sample surface while rastering and only touches the sample at the bottom of each oscillation. This prevents damage to soft specimens and avoids the "pushing" of specimens around on the substrate. By using a constant oscillation amplitude, a constant tip–sample distance is maintained until the scan is complete. TappingMode AFM can be performed on both wet and dry sample surfaces.

Examples of images acquired by TappingMode are shown in Figure 4. A 2µm TappingMode image of fibrillar collagen is shown in Figure 4a. Resolution of a *d*-spacing (the spacing between crystalline lattice planes) of 70 nm confirms measurements made with a transmission electron microscope; the latter requires tedious and time-consuming specimen preparation versus the minimal sample preparation for the AFM. Figure 4b is an image of both normal and sickled human red blood cells. Sample preparation consisted solely of a simple smear on a glass slide. Note the sickled cell has indented a normal (softer) red blood cell.

LiftMode AFM provides the operator with a tool to record dual information about a sample surface at one location, such as topography and magnetic gradients (obtained in the magnetic force microscopy or MFM mode), thereby allowing the useful association of the two images. LiftMode AFM operates by first scanning a line on the sample

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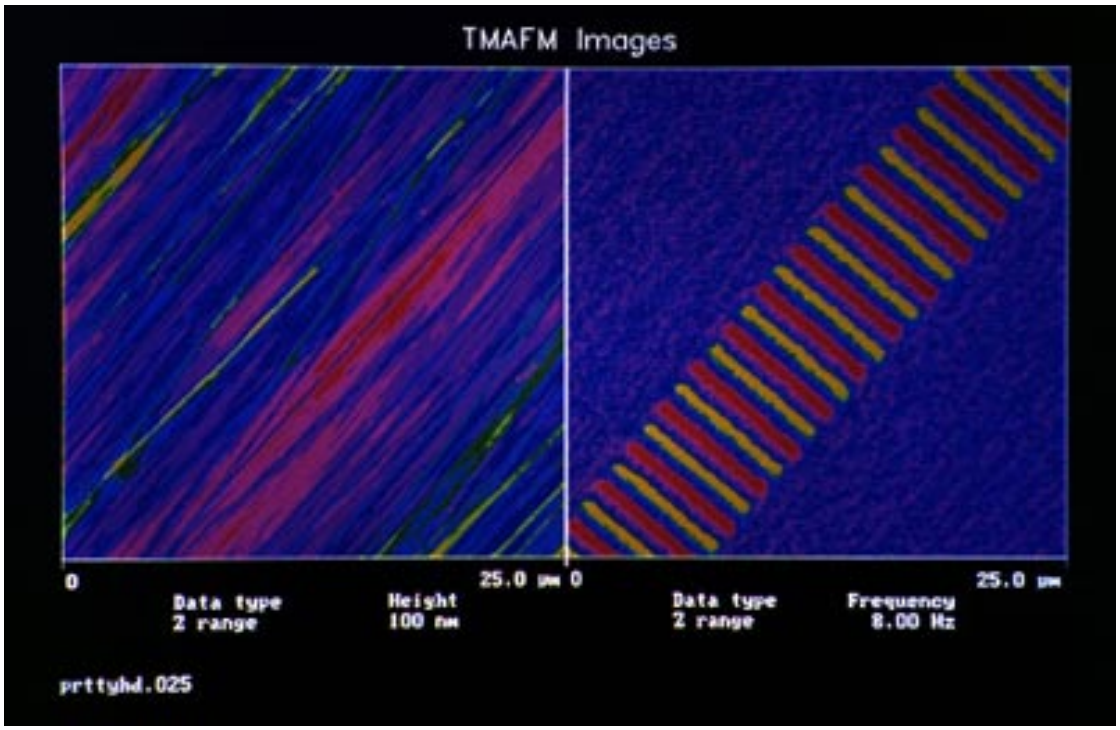

FIGURE 5. AFM IMAGES (25 µm SCANS) OBTAINED USING LIFTMODE AFM. THE IMAGE AT THE LEFT SHOWS THE SURFACE TOPOGRAPHY AND THE IMAGE AT THE RIGHT REVEALS THE CORRESPONDING MAGNETIC FORCE GRADIENT MAP OF A HARD DISK AT THAT SAME POSITION. IMAGES TAKEN WITH A NANOSCOPE SPM (COURTESY OF DIGITAL INSTRUMENTS, SANTA BARBARA, CA).

surface in TappingMode to obtain the topographical information. Then, the tip is lifted to a distance above the sample set by the operator and the same line retraced in a noncontact mode to obtain (for example) near surface magnetic field information. The process is repeated until the scan is complete and both images are saved. To perform MFM, a ferromagnetic tip and a ferromagnetic or paramagnetic sample are required. An example of coordinating images generated using LiftMode AFM is shown in Figure 5. The image on the left shows the surface topography of a hard disc with an 800 bit test track while the image on the right is an MFM image of the same area showing the magnetic force gradients. Note the MFM image shows clear resolution of the bit transition gradients without contamination from the surface topography. LiftMode AFM can also be used to record topography and electric fields or phase imaging data.

In summary, the continuing development of AFM technology provides scientists with a powerful tool to characterize a variety of sample surfaces. Minimal sample preparation, use in ambient conditions, and the ability to image nonconducting specimens at the

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atomic scale (in some cases) makes AFM an extremely versatile and useful form of microscopy. Recent advances in AFM have allowed the successful imaging of soft polymeric and biological samples and the imaging of magnetic microstructures.

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