Analytical Series

Overview of Atomic Force Microscopy

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Atomic force microscopy (AFM) is a family of nanoscale characterization techniques that has exploded onto the overall characterization and nanotechnology field. Its versatility and high resolution continue to contribute to a variety of fields, from biology to physics and chemistry to engineering. AFM entered the scientific arena in 1981 with the now-famous invention of its older sibling in the scanning probe microscopy family, the scanning tunneling microscope (STM), in the IBM Zurich labs of Gerd Binnig and Heinrich Rohrer. For this invention, they received the Nobel Prize in Physics in 1986. The AFM was then invented in 1986 by Gerd Binnig, Cal Quate, and Christoph Gerber.[1] Together, the STM and AFM formed the scanning probe microscopy (SPM) family, which includes other methods such as near field scanning optical microscopy (NSOM).

Similar to many other surface science techniques, atomic force microscopy (AFM) has succumbed to a somewhat unwieldy jumble of abbreviations and jargon that has become hard to navigate. Scanning probe microscopy (SPM) refers to an umbrella of a variety of methods, including perhaps its most famous member, AFM, in addition to others such as scanning tunneling microscopy (STM), near-field scanning optical microscopy (NSOM or SNOM, depending on the continent), and other lithographic methods. Within AFM, there are dozens of methods that rely on the AFM probe-sample interactions to provide a variety of material properties, including electrical, optical, magnetic, and mechanical. To date, there are dozens of SPM/AFM-based methods. Furthermore, AFM's flexibility is difficult to surpass because it can operate in nearly any environment (liquid, gas, ambient, vacuum) and on any surface. There are no constraints on the sample except that it fit inside the AFM instrument and be smooth enough for the scanner to handle (described in detail below.) It is beyond the scope of this article to list and/or define all of the AFM-based methods, but the wide diversity of properties of materials and diversity of samples that can be measured with AFM is clear.

OVERVIEW OF AFM OPERATION

AFM Hardware

A schematic of AFM hardware is shown in *Figure* 1. The main components of the AFM are the (1) cantilever; (2) optical detection system; (3) x-y-z scanner; and (4) feedback loop.

Cantilevers and Probes

The heart of the AFM lies in its cantilever/probe assembly that interacts or probes the materials to provide the information of interest. The technology to fabricate AFM cantilevers takes advantage of technology developed for the semiconductor industry to make similar scale devices and features from single crystal silicon or silicon nitride (Si_3N_4). The dimensions of a cantilever vary and dictate the stiffness, or spring constant, of the cantilever. Cantilevers are generally either rectangular in geometry (Si or Si_3N_4) or triangular (Si_3N_4), and now hybrid cantilevers are being manufactured (e.g., Si tips on SiN cantilevers). Typically, the cantilevers are hundreds of microns in length, tens of microns in width, and a few microns in thickness. An SEM image of an AFM cantilever/tip assembly is shown in *Figure* 2.

Cantilevers can be coated with gold or aluminum to provide high reflectivity or other thin metal coatings for magnetic or electrical imaging. Cantilevers and probes can also be functionalized chemically or biologically for specific interactions.

Optical Detection System

To track the motion of the cantilever/probe assembly as it scans a surface, an AFM typically includes an optical detection system that consists of a laser reflected off of the back side of the cantilever and directed towards a position sensitive detector (PSD), as shown in *Figure* 1. Optical detection systems are the most common way to track cantilever motion in commercial AFM systems. However, other methods exist with self-actuated cantilevers where their motion is read by piezos integrated into the levers. The laser is typically a visible photodiode, although some commercial instruments have implemented a superluminescent diode laser (SLD). The laser is detected by a four-segment PSD that can track the vertical and lateral motion of the cantilever accurately. This motion can then be converted to units of nanometers or displacement through careful calibration, as described below.

x-y-z Scanner

An AFM can operate either by scanning the sample relative to a stationary tip (sample-scanning) or scanning the tip relative to a stationary sample (tip-scanning.) Each has its own advantages in terms of the size of sample it can accommodate (tip-scanning—more flexible), ability to accessorize (tip-scanning—again more flexible), and stability and signal to noise (sample-scanning—easier to build with better specifications.) Each shares the requirement to move the tip relative to the sample in a highly accurate way with minimal noise.

Tip-sample motion is often accomplished in commercial instruments by piezoelectric materials, which are materials that respond either by expansion or contraction in response to an applied voltage. These kinds of materials provide the ability for very fine motion (nanometers to microns). Although piezoelectric materials are very effective at moving the tip or sample, they are plagued by nonlinear behavior, such as hysteresis and creep, that has serious consequences for accurate AFM imaging and interpretation of data.

Finally, it is important to note that the piezo method used to move the AFM tip or sample in x, y, and z is independent from the piezo used to actuate or vibrate the cantilever, often referred to as a "shake" or "dither" piezo.

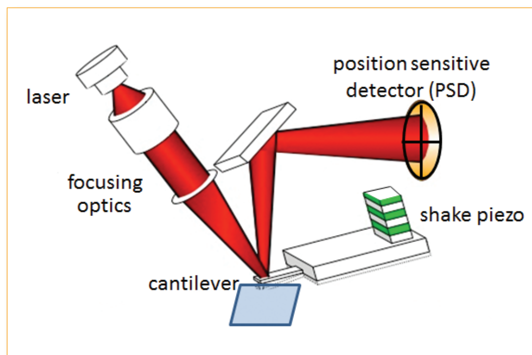


Figure 1—Schematic of basic operating principles of AFM. A laser is focused and directed off the back end of a cantilever and directed towards a PSD to monitor its vertical and lateral motion as it interacts with the surface. Depending on the mode used to image the sample, the cantilever can be vibrated with a shake piezo for dynamic imaging modes that involve the resonance of the cantilever.

AFM Software

AFM software is primarily used for controlling the AFM stage and then performing subsequent data/image analysis to extract the information of interest. The software interface with the electronics controller is critical and responsible for setting the motion of the x–y stage, controlling the probe approach to the surface, and setting feedback parameters to optimize the image quality. Image processing is an integral part of effective AFM characterization and analysis.

Calibrations

Often, the AFM measures the force exerted onto the material with a cantilever/tip assembly. The challenge is that what is actually being measured is the cantilever deflection in volts by the PSD, which requires calibration of the cantilever spring constant

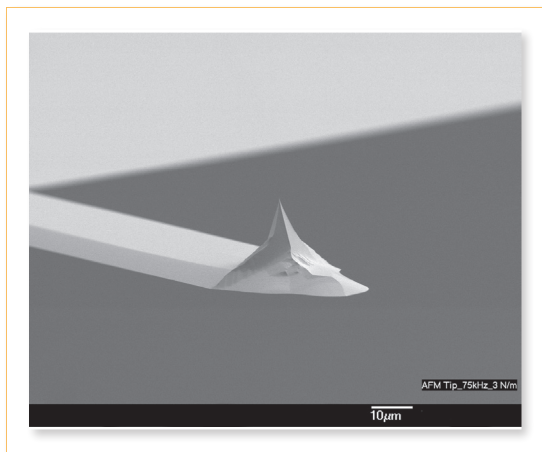


Figure 2—SEM image of an AFM cantilever/tip assembly.

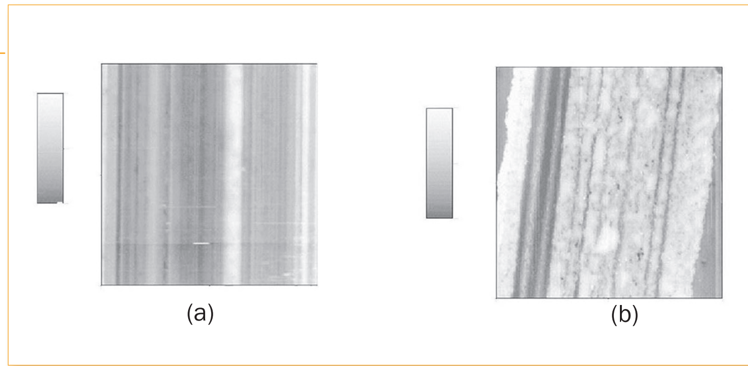


Figure 3—AFM images of the topography of a lubricant film collected in contact mode showing (a) striations in the direction of rubbing for a lubricant with just base stock and (b) formation of a patchy antiwear film in a lubricant containing antiwear additive.

or stiffness [N/m] and the optical lever sensitivity on the photodetector [V] to ultimately convert to force. Many different methods exist to calibrate the normal spring constant on the cantilever, including a strict geometrical measurement of the cantilever to derive *k,* the spring constant given by

$$k_L = (E\omega t^3)/(4l^3)$$

where E = Young's modulus of the material, ω = width, l = length, and t = thickness, all of the cantilever. Other methods include the Sader method,[2,3] pressing the lever against a reference cantilever with known spring constant,[4] the thermal noise method,[5,6] and the added mass method.[7] With all these methods, it is reasonable to measure a spring constant to within 20% error.

Tip-Shape Calibration

The AFM tip shape is a critical parameter for many measurements. The tip shape (and diameter) plays a significant role in defining the resolution. The tip shape also is a critical parameter that needs to be well-known in order to extract any quantitative information from the tip-sample interaction, like material properties (e.g., modulus) of the surface. Knowing the shape of the AFM probe is a "moving target," since the shape is most likely constantly changing during the course of imaging, either through contamination or wear. This problem is compounded by the fact that, realistically, AFM probes are not necessarily manufactured in the cone geometry that is commonly idealized, and manufacturing is not wholly reproducible, so there is variety between batches. Direct imaging of the AFM tips by high resolution techniques, such as SEM or transmission electron microscopy (TEM),[8,9] can provide true 3-D information on the tip. However, such techniques tend to damage the tips when trying to obtain the highest resolution images necessary. Other common ways to calibrate tip shapes are by reverse imaging of the tip[10] or blind reconstruction.[11]

Readers interested in more detail about AFM operation can consult a number of excellent books on the topic, including ones by Eaton,[12] Haugstad,[13] Magonov,[14] Meyer,[15] and Bonnell.[16]

Operation Modes

Contact Mode

Contact mode is conceptually the simplest mode where the tip maintains contact with the sample throughout the experiment. In contact mode, the tip maintains a constant deflection through the microscope feedback system as it raster-scans along the material. The user can control the load at which this deflection is maintained. The user can allow for either more aggressive contact resulting from a higher load and therefore more cantilever deflection, or a less aggressive contact resulting from a lower load and therefore less cantilever deflection. Another term to describe this imaging mode is "constant force" mode. This mode is typically conducted with softer levers that can respond with deflection to materials. It is also a fairly aggressive tip-sample interaction compared to the other modes, and can damage softer samples (e.g., polymers) or brush away loosely bound material as it scans along the surface. By scanning in contact mode perpendicular to the cantilever axis in a mode called frictional force microscopy, lateral forces or friction by a surface exerted onto a tip can be measured.[17,18]

Tapping Mode

With the popular tapping mode, the cantilever is oscillated at its resonant frequency by a shake piezo (described previously in the section on x–y–z scanner). The cantilever resonances are a function of their stiffness and geometry, and are typically in the 10s or 100s of kHz. The amplitude of oscillation is dictated by the user-inputted drive voltage and can range from very low (a few nm) to high (over 100 nm) depending on the application. As the cantilever is oscillated, it interacts with the surface in intermittent contact (as opposed to constant contact, as in contact mode) where it "taps" along the surface. The feedback in this mode is via the amplitude of oscillation during the *intermittent*

contact, also known as the setpoint. The setpoint can be set as high (i.e., a high percentage—e.g., 90%—of the free air oscillation for a very gentle tip-sample interaction) or as low (i.e., a low percentage—e.g., 50%—of the free air oscillation for a more aggressive tip-sample interaction) as the user desires. Other names for this mode include amplitude modulation mode and intermittent contact mode.

CHARACTERIZATION WITH AFM

The most useful measurements AFM can perform for coatings materials are topography and mechanical measurements. As noted, these measurements can be performed in practically any environment and on any surface (with the constraints that the sample be smooth and that it fits into the instrument). This is in contrast to STM, which requires an electrically conducting or semiconducting material, or electron microscopy based-methods, which require a vacuum or very low pressure environment and a conducting sample (or at least one that is then coated with a thin conducting layer).

The very low loads of AFM (nanoNewtons of force) and very small deformation into the sample (on the order of nanometers) are especially suited to characterize coatings mechanically, where other methods may typically suffer from the "substrate effect" in which substrate properties are convoluted into the measurement of the substrate properties. A commonly reported rule is the so-called "10% rule" where coating-only hardness can be measured if the depth of the indent is less than 10% of the coating thickness. Fortunately, the substrate effect is not typically a problem for AFM measurements, unless the coating itself is incredibly thin like, for example, single atom thickness. Elastic mechanical property measurements of coatings with AFM is still an area of active research, since cantilevers are typically made of Si. Plastic measurements are often not possible, although Si cantilevers with diamond tips for this purpose are manufactured. Nanoindentation perhaps remains the gold standard for measuring elastic and plastic properties of coatings, albeit not with the spatial resolution of an AFM, and for thin coatings, subject to "substrate effect" problems. Nanoindentation is not discussed in this tutorial, but its application to study coatings can be found in more detail,[19,20] specifically regarding characterization of scratch resistance of polymeric coatings.[21]

Topography

AFM is renowned for its ability to map out true three-dimensional topography of a surface. Vertical resolution in AFM is typically very high and on the order of an angstrom (0.1 nm). Lateral resolution is governed by the operation mode and the quality and sharpness of the AFM probe where a sharper AFM probe can improve the resolution. Topography is collected in practically every AFM mode and can be imaged in either tapping or contact mode as described previously.

AFM has been used to characterize the topography of thin lubricant boundary films on steel generated under the presence of different additives.[22,23] As seen in *Figure* 3, the morphology of the film is a strong function of the lubricant composition. In *Figure* 3a, the lubricant contains only basestock, resulting in grooves in the direction of rubbing. In *Figure* 3b, when the lubricant contains an antiwear additive, an antiwear film forms with the characteristic patchy morphology to protect the surface.

Atomic resolution with AFM has been touted since the 1990s. However, it is important to differentiate between true atomic resolution where defects are imaged[24] and "lattice imaging" where the periodic lattice of a very flat surface is imaged,[25,26] often in lateral force mode. True atomic imaging can be challenging to accomplish and is typically achieved in advanced modes such as frequency modulation, low amplitude imaging in liquids,[27] or ultra-high vacuum. "Routine" atomic imaging on a wide variety of materials is still an active area of research.

One key limitation in topography imaging with AFM is the x, y, and z maximum length scales that can be imaged. Depending on the type of scanner used, the maximum x–y range of AFMs is typically in the 100–125 μm range, and the maximum vertical range, again depending on the scanner, is typically up to 5 μm. This limitation can be a problem in cases where a large field of view is necessary to find a particular feature of interest.

Mechanical Measurements

Measuring mechanical properties with AFM has been a "holy grail" of the field since its inception 25 years ago, and it is still a very robust research area. There are entire books devoted to the topic[20,28] and a thorough discussion is beyond of the scope of this article. We review the main methods available for obtaining contrast based on mechanical properties.

Phase Imaging

Phase imaging is arguably the best known method for obtaining contrast based on mechanical properties. Phase imaging is obtained in tapping mode of operation, where the phase lag between the cantilever and its response are mapped as a function of the topography.[29,30] It is very useful for obtaining contrast, but quantitative interpretation is very challenging since both elastic and

inelastic interactions between the tip and sample affect the phase and can often lead to contrast reversals, poor resolution, and other artifacts for the unskilled user.[31-34] Phase imaging is often used to discriminate materials with different elastic moduli. For example, a phase image of a polypropylene with a rubber is shown in *Figure* 4 where the rubber domain clearly demonstrates a higher phase (more dissipative) than the surrounding polypropylene, resulting in a very quick discrimination of the two materials. For a thorough treatise of phase imaging, the reader is referred to the literature.[13,28]

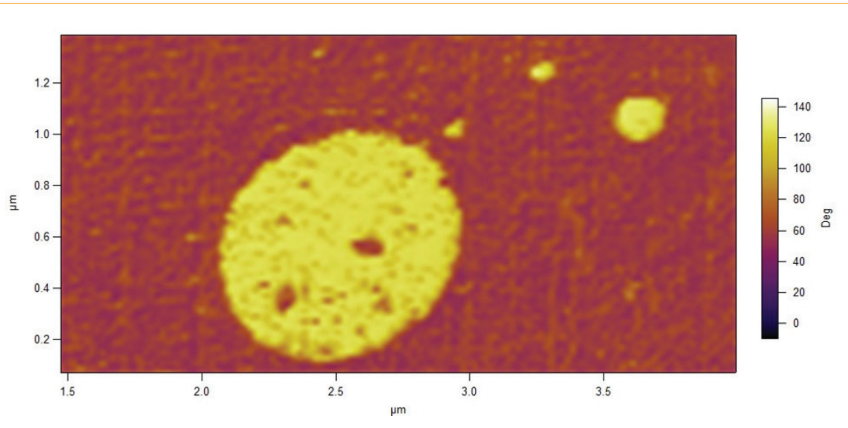
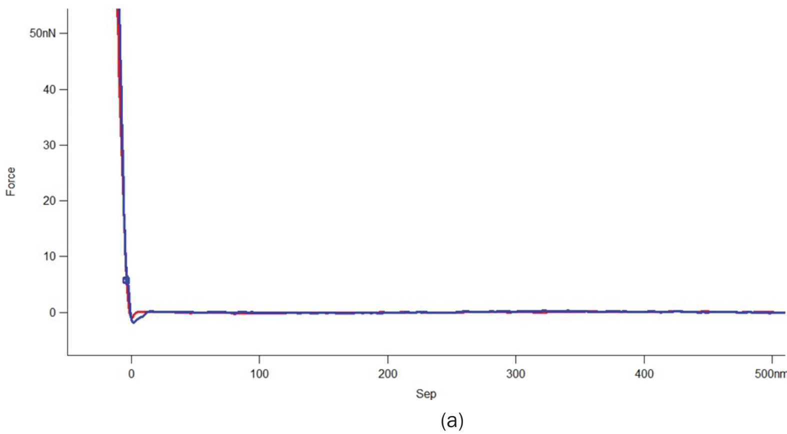


Figure 4—Phase image of a blend of polypropylene with rubber easily differentiating the rubber domain in the middle from the surrounding PP matrix.



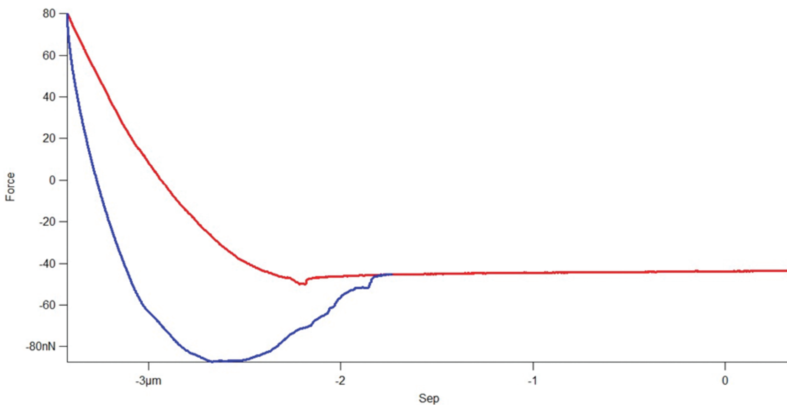


Figure 5—Example of a static force curve measuring force (nN) vs tip-sample separation (µm) on sapphire (a) and rubber (b).

Force Curves

Force curves are single point measurements where the AFM tip is lowered into contact with the surface and then withdrawn. The deflection on the cantilever is then graphed with the piezo motion of the cantilever. This is similar to *Figure* 5a, which is a sample force curve conducted on sapphire, where the red curve is the tip approaching the surface and the blue curve is the tip withdrawing or retracting from the surface. Interpretation of the force curves is as follows: The tip approaching the surface (straight section on right of graph) is followed by a small dip in the red curve referred to the "snap into contact," where the tip literally snaps into contact with the sample. Then, as the tip continues to approach the sample, the tip-sample interaction becomes repulsive and is in contact along the vertical portion of the curve. As the tip withdraws back following the blue curve, there is a slight adhesion dip from which the cantilever snaps back out and retracts to a position far from the surface. *Figure* 5b is a force curve of the identical cantilever, except on a rubber sample. Theoretically, the components of the force curve are the same as in *Figure* 5a. They start with attractive force, show a small snap into contact, followed by a repulsive wall in contact, and then, upon withdrawal, there is adhesion until the tip retracts further from the sample. There are a couple of key differences between the force curves in *Figures* 5a and 5b. In *Figure* 5b, the adhesion dip in the blue retract curve is very large due to the large adhesion of the material. In addition, there is significant hysteresis (separation) between the approach and retract curve in the repulsive (vertical) section, which is consistent with a highly dissipative, highly adhesive rubber. With such an analysis, force curves can be utilized to learn useful qualitative information about the sample.

Force curves can be fit with a number of contact mechanics models including Hertz (no adhesion),[35] DMT model (adhesion in the contact region between the tip and sample),[36] and JKR (adhesion outside of contact region)[37] in order to extract mechanical properties such as modulus. For any meaningful information to be extracted from force curves, the experimentally obtained deflection vs piezo motion must be converted to force vs tip-sample separation. Interpretation of the force curve to extract mechanical properties inherently contains assumptions with whatever contact mechanics model is used, as well as the tip shape

and geometry—all of which greatly affect the accuracy of the quantitative number measured.

Advanced Methods

There are a number of emerging advanced methods to measure mechanical properties or get contrast based on mechanical properties. These methods are briefly referenced here for the interested reader. Multifrequency methods, where the cantilever is excited at multiple eigenmodes (not just the first eigenmode, as is done in conventional tapping AFM), has shown great promise in enhanced contrast based on material properties.[38,39] These multifrequency-based methods are also the basis for some recent methods to measure modulus in the higher order eigenmode.[40] Finally, contact resonance and force modulation methods are dynamic contact methods where the tip in contact with the sample is in resonance[41-44] for contact resonance and off-resonance for force modulation; these methods are used to extract the elastic and viscoelastic properties of materials with a wide range of moduli up to 200 GPa or higher, including metals, glasses, and alloys and down to polymers.

Applications to Coatings

Due its wide range of capabilities of imaging topography and simultaneously mechanical properties such as stiffness, dissipation, and friction, by the methods described above and under a number of environmental conditions, AFM has been successfully applied to a wide variety of coatings research. Recently, degradation mechanisms and the durability of wood coatings were probed with AFM to reveal information on the morphology and microstructure of the coatings with artificial aging.[45] Tapping mode AFM, described previously, was used to image the topography of polyester films to investigate their microstructure as a function of degradation.[46] Finally, AFM to measure both topography and mechanical properties through the variety of methods described here (force modulation, friction force mapping, and phase contrast) was used to examine latex films and the effect of solvent and temperature on such systems.[47,48]

CONCLUSIONS

Atomic force microscopy is a powerful characterization tool with many applications to coatings. Among the most useful is that AFM can accurately measure topography and mechanical properties with nanometer lateral and angstrom vertical resolution in a variety of environments (fluid, vacuum, etc.). Its most common modes, contact mode and tapping mode, as well as some advanced multifrequency modes, are described. Key features of AFM hardware and operation are discussed to familiarize the basic user, with references for further detail provided for the interested and/or more advanced reader. CT

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