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 as its versatility and high resolution continue to feed a dizzying variety of disciplines 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 which 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, STM and AFM formed the scanning probe microscopy (SPM) family, which includes other methods such as near-field scanning optical microscopy (NSOM).

The STM spawned the next 25 years of the continuously developing field of scanning probe microscopy and specifically atomic force microscopy, which now includes dozens of different methods under its name to probe various properties—including mechanical, electrical, magnetic, chemical, and optical—of materials and surfaces. Atomic force microscopy is a powerful tool in various research enterprises. It is found in practically any university characterization facility alongside optical and electron microscopes, and most undergraduates in science or engineering fields at this point are at least familiar with it, if not have performed a laboratory experiment in their undergraduate curriculum.

The focus of this book is to understand and appreciate the role this young technique has played in industrial research and development (R&D). AFM has penetrated into a variety of industrial research sectors as witnessed by the diverse set of applications described in this book. Alongside electron and optical microscopy, which have been
around for decades and have reached an impressive level of commercial maturity and ease of use, AFM has become a vital characterization method despite its youth and continued technical evolution. So, though AFM is still an active area of academic research as its capabilities continue to develop and be better understood, it has proven to be a useful microscopy to address industrial and commercial needs from quality control and assurance to product formulation and process monitoring.

The goal of this introductory chapter is to provide an overview of AFM to nonspecialists and introduce the various topics that are the subject of subsequent individual chapters. As such, this chapter will provide a brief review of the beginnings of AFM and special features that make it particularly suited for industrial research. Then a brief overview of AFM operation will be presented including the hardware, software, calibrations involved, and finally the different nanomechanical methods that will be described in detail both in theory and application. Entire books are written on AFM operation, and this chapter is not meant to be an exhaustive introduction to its operation, merely serving to provide enough information for the rest of the book to be followed intelligently. Readers interested in more detail about AFM operation can consult a number of excellent books on the topic [2–5].

1.1 A WORD ON NOMENCLATURE

Before the rest of this chapter continues, some definitions are in order. Similar to many other surface science techniques, AFM has succumbed to a somewhat unwieldy mess of abbreviations and jargon that has become a hard-to-navigate alphabet soup. SPM refers to an umbrella of a variety of methods. Methods that fall under SPM include perhaps its most famous member, atomic force microscopy (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. And then 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 properties. To date, there are dozens of SPM/AFM-based methods. It is beyond the scope of this book to list and/or define all the related methods. These methods characterize a huge variety of material properties. Some of the more common methods have been included in Figure 1.1 with the category of properties that they measure. The wide diversity of properties of materials that can be measured with AFM is clear. The focus of this book is on nanomechanical characterization, due to its broad appeal in industrial R&D. Again, Figure 1.1 is offered to demonstrate the variety and flavor of properties that can be probed with SPM.

1.2 ATOMIC FORCE MICROSCOPY—THE APPEAL TO INDUSTRIAL R&D

This book specifically focuses on AFM, which is the method that has most penetrated the general characterization and industrial research fields. AFM has key features that make it especially attractive to industrial research and development. First, its resolution,
the heart of its utility as a characterization tool, is indeed impressive with 5- to 10-nm lateral resolution and angstrom vertical resolution achieved routinely on commercial instruments with commercial cantilevers. The limits of lateral resolution continues to be pushed with specialized techniques and cantilevers, and it has reached true “atomic” resolution where point defects of certain materials can now be imaged under certain operating parameters and imaging modes, especially under liquid [6–8]. True atomic resolution is still currently mostly achieved through STM, which operates through an entirely different tip–sample interaction mechanism based on quantum mechanical tunneling. Many of these STM studies are conducted in ultrahigh vacuum (UHV), though some are performed in ambient and liquid conditions. It is fair to say that AFM is catching up, however, with recent groundbreaking work imaging cyclic aromatic molecules on Cu[111] [10, 11].

A second critical feature of AFM that makes it particularly amenable to industrial research is the flexibility of the environment in which it can operate. Despite being a high-resolution microscopy that rivals the resolution of electron microscopy, AFM can operate in an ambient or even liquid environment with minimal compromise to its resolution. The ability to work in “real-world” environments makes it a critical tool for many industrial R&D applications, where the research emphasis is consistently to understand mechanisms and materials in real-world situations as opposed to
idealized materials and conditions that typically exist in academic research endeavors. In many cases, the researcher may not want the sample to be forced into a pristine vacuum environment where perhaps key materials or components will be evacuated and thus missed in the characterization effort. In addition, the flexibility of an environment permits in situ characterization of processes. For example, processes such as corrosion, lubrication, catalyst dissolution, crystal growth (for active pharmaceutical ingredient, API) can all be studied in situ with AFM as a unique attribute of this microscopy. The power of in situ measurements for biological applications is simple as many biological processes and materials simple cannot survive ex situ.

A corollary of the utility of the AFM’s flexible environment to industrial R&D is the flexibility of type and shape of surface or material that AFM can image. There are virtually no restrictions on the size of a material that can be probed with AFM. Many commercial “large-sample” AFMs currently exist that can image any design or shape of a surface from engine parts to thin films. Indeed, one of the AFMs built in the early 1990s (Topometrix Explorer) was advertised as being able to be placed on top of jet wings to image fractures. In addition, there are practically no mechanical or electrical restrictions on the type of sample an AFM can image. Unlike electron microscopy or STM that require conducting or semiconducting surfaces to image or avoid artifacts [or in the case of insulating surface in scanning electron microscopy (SEM) that requires deposition of a thin conducting layer such as Cr], AFM can image insulating surfaces in addition to conducting or semiconducting surfaces with minimal sample prep. Typically, the most significant requirement on the sample for an AFM is that it be smooth. Commercial instruments will have a maximum z (vertical) range that can be imaged, dictated by the design and desired resolution of the particular instrument. The maximum z range is typically from 1 µm (for high-resolution studies) to several microns (for lower resolution studies), meaning that the sample cannot have features, a tilt, or overall roughness that exceed that limit in order for the AFM to image effectively. Smooth surfaces are often prepared with the use of an ultramicrotome. An ultramicrotome is simply a very controlled and precise way of cutting of thin (down to 100 nm) sections, at controlled speeds and in a controlled fashion to result in ultrasmooth surfaces of nanometer or less than nanometer roughness. The ultramicrotome typically employs the use of a glass knife for a coarse cut and then a diamond knife for a fine cut. Cryomicrotomy is simply ultramicrotomy at cold temperatures. Cryomicrotomy is typically used to prepare polymer surfaces to cut below the glass transition temperature of the material; depending on the instrument, cryomicrotomes can cool down to −180°C.

Finally, AFM is fundamentally a mechanical probe of the surface through actual contact between the tip and sample. So, in addition to the ability to provide high resolution on surface features, the goal of any form of microscopy is the unique tip–sample interaction mechanism enabling the probing of many properties, most obvious of which is mechanical properties. Mechanical properties covers a wide range of materials and properties. Entire textbooks and courses are devoted to the studies of mechanics, for example, by Callister and Rethwisch [12]. We very briefly review some of the main concepts below to provide enough of a background for the rest of the chapter.
1.3 MECHANICAL PROPERTIES

When one considers mechanical properties, a range of descriptors come to mind, including “stiffness,” “toughness,” “hardness,” “brittleness,” and “ductility” to name a few. Each of these properties has their own mathematical expressions with associated experimental methods of measuring them. An interaction between two materials can be typically categorized into either elastic (or recoverable) or dissipative or plastic (permanent or nonrecoverable) interaction. On the bulk scale, most mechanical analysis of materials relies on measuring their strain (material displacement) as a function of stress (pressure exerted on the material) or vica versa. The measurement and analysis of stress versus strain behavior of various materials is a rich and active field, and there are many excellent textbooks that describe it [12]. The description provided below is simplified to provide a context for these measurements ultimately described on the nanoscale.

Elastic deformation of a material is typically nonpermanent or reversible and exists in a regime where the strain (material displacement) is proportional to the stress (pressure exerted on the material) by a proportionality factor known as a modulus (though in reality, no material is truly only elastic). Elastic materials thus typically follow Hooke’s law. Figure 1.2 shows a representative stress versus strain curve for materials highlighting the elastic region (blue). Additionally, in elastic

![Figure 1.2](basic_stress_vs_strain_curve.png)

**Figure 1.2** Basic stress vs. strain curve showing three mechanical regimes of elastic deformation, plastic deformation, and fracture. For color details, please see color plate section.
materials, the loading curve (as force is applied) is identical to the unloading curve (as the force is removed.) Modulus is a key parameter used to describe elastic behavior. Depending on the direction in which a material is being stressed, the material will have different moduli (e.g., a tensile modulus, a shear modulus, etc.).

Plastic deformation is, on the other end, permanent and nonrecoverable. Plastic deformation does not follow Hooke’s law and so stress is not proportional to strain. In Figure 1.2, the region of plastic deformation is shown in green. In contrast to elastic deformation, the loading curve is not identical to the unloading curve, and, in fact, the unloading curve returns to a nonzero strain at a zero stress level. There are many common properties associated with plastic deformation including yielding (onset of plastic deformation), ductility (plastic deformation at a fracture), and hardness (resistance to localized plastic deformation).

Finally, there is another very important class of materials called viscoelastic materials, where the elastic properties are frequency dependent. For example, when a stress such as pulling is exerted on the material, the material’s response is rate dependent. Perhaps the most familiar viscoelastic material to most readers from childhood is Silly Putty, where its response to pulling depends on how fast it is pulled. If Silly Putty is pulled very slowly, the material stretches out very nicely. If Silly Putty is pulled very fast, it will quickly break. Polymers and rubbers, two materials that play a significant role in consumer products, and which are characterized quite heavily with AFM, are also viscoelastic materials.

1.4 OVERVIEW OF AFM OPERATION

1.4.1 AFM Hardware

A schematic of AFM hardware is shown in Figure 1.3. The main components of the AFM are (1) the cantilever, (2) optical detection system, (3) x-y-z scanner, and (4) feedback loop.

1.4.2 Cantilevers and Probes

The heart of the AFM lies in its cantilever/probe assembly that interacts or probes 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 out of silicon. Thus, today AFM cantilevers are typically made by micromachining silicon (single-crystal Si) or silicon nitride (Si$_3$N$_4$). The dimensions of a cantilever vary and dictate the stiffness, or spring constant, of the cantilever. Depending on the material that needs to be probed and the mode of operation, one might pick a lever with a stiffer (higher) or softer (lower) spring constant. Cantilevers are generally either rectangular in geometry (Si or Si$_3$N$_4$) or triangular (Si$_3$N$_4$), with now hybrid cantilevers being manufactured (e.g., Si tips on SiN cantilevers). Generally, the Si$_3$N$_4$ probes have lower force constants and might be used for imaging in liquid, among other applications, whereas the Si probes can be brittle and tend to break or chip in contact when scanning a surface. Typically, the
cantilevers are hundreds of microns in length, tens of microns in width, and a few microns in thickness; for reference, the diameter of a human hair is approximately 100 µm. Cantilevers can be coated with gold or aluminum to provide high reflectivity. Commercial cantilevers are also available with other thin metal coatings for magnetic or electrical imaging. Cantilevers and probes can also be functionalized chemically or biologically for specific interactions.

Probes are grown at the end of the lever and vary in shape and sharpness. A sharper probe can often improve the lateral resolution of the AFM. Probe design is a continuously evolving field as manufacturers strive for the most robust, reproducible, and sharp tips. Cantilever/probe assemblies are mounted into a cantilever holder that typically includes a piezoceramic element (described below) that can vibrate the cantilever at a given frequency—either above, at, or below its resonance—depending on the desired tip–sample interaction or AFM mode.

1.4.3 Optical Detection System

In order 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 the back side of the cantilever and directed toward a position sensitive detector.
(PSD) as seen in Figure 1.3. Optical detection systems are the most common way to track cantilever motion in commercial AFM systems. However, other methods exist to bypass optical detection systems (in, e.g., a dark medium where the laser light would be absorbed) with self-actuated cantilevers where their motion is read by piezos integrated into the levers. The laser is typically a visible photodiode, though some commercial instruments have implemented superluminescent diode (SLD) laser. The laser is detected by a four-segment position-sensitive detector (PSD), which can track the vertical and lateral motion of the cantilever accurately, which can be converted to units of nanometers or displacement through careful calibration described below.

### 1.4.4 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 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). Piezoelectric materials can be made in several shapes including tubes, disks, and bimorphs, depending on the range and geometry of motion required. Although piezoelectric materials are very effective at moving 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. Artifacts induced by piezoelectric materials are reviewed in detail in Chapter 6.

Piezoelectric scanners can be programmed to move with either open-loop or closed-loop feedback circuits. In open-loop scanners, the piezo is moved by programs to ramp $x$ and $y$ bias voltages to move the prescribed amounts. So in open-loop scanners, if the piezo has to move a large distance or move around on the sample, there can be considerable hysteresis of creep with the best recourse of patience or moving/zooming in and out in small increments since hysteresis/creep is proportional to the displacement or motion the scanner is trying to execute.

One solution to this problem is to use closed-loop scanners that use additional $x$-$y$-$z$ sensors to independently measure and then correct for scanner movement. In closed-loop operation, feedback circuits are continually adjusted to the applied scanner biases to correct for the $x$-$y$ motion and to achieve the desired motion. It is important to note that correction of $x$-$y$ motion in an AFM is slightly different than correction of $z$ motion. This is due to the fact that $x$-$y$ motion can be predicted, whereas $z$ motion cannot as it depends on the surface topography of the sample being scanned. Therefore, typically there are active feedback circuits to control $x$ and $y$
motion as corrected by the motion detected by the external sensors. Closed-loop $z$ operation monitors $z$ motion with a passive feedback circuit to obtain an image of the $z$ sensor signal, in addition to the $z$ piezo signal. The $z$ sensor signal will be a more accurate representation of the surface topography, though it also is usually noisier as well, which could affect topographic measurements on the subnanometer length scale. Finally, it is important to note that the piezo/other electromechanical 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. The piezo used to vibrate the cantilever is often referred to as a shake or dither piezo and is integrated into the cantilever holder and is specific to oscillating the cantilever.

### 1.4.5 AFM Software

Atomic force microscopy software is primarily used for controlling the AFM stage and then doing subsequent data/image analysis to extract the information of interest. The software interface with the electronics controller is critical and responsible for setting motion of the $x$-$y$ stage, controlling the probe approach to the surface, setting feedback parameters to optimize the image quality. Image processing is an integral part of effective AFM characterization and analysis. Many image processing tools are integrated into commercial AFM software, though several third-party software packages specific to AFM also exist including SPIP, Gwyddion, and WSxM among others.

### 1.4.6 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. So this system requires several different calibrations to accurately know the actual force between the tip and sample. First, one has to start with a calibration of the cantilever stiffness or spring constant (N/m). Then, one has to measure the optical lever sensitivity or conversion of volts to nanometers on the photodetector (though the order of these two measurements can be reversed depending on the method being used). This calibration factor is specific to cantilever and alignment. Only then can the conversion be done of measured volts on the PSD to units of force, or Newtons, that the cantilever exerts on the surface. We now briefly go through each of these calibrations.

### 1.4.7 Cantilever Spring Constant

For accurate mechanical measurements of tip–sample interactions, precise calibration of the cantilever spring constant is required in order to measure the force exerted on your sample. Cantilevers have spring constants associated with all degrees of motion, for example, normal and lateral. Typically, it is the normal spring constant...
that requires calibrating for most force measurements; lateral or torsional spring constants are required for lateral force or frictional force microscopy. We discuss here calibration of normal spring constants.

The normal spring constant of a cantilever can be measured in several ways. For a perfectly rectangular cantilever, the spring constant, $k_L$ (L for lever), is given by

$$k_L = \frac{E \omega l^3}{4l^3}$$

where $E$ is the Young’s modulus of the material, $\omega$ is the width, $l$ is the length, and $t$ is the thickness, all of the cantilever. While width and length of the cantilevers can be fairly easily measured by SEM, the accurate measurement of thickness ($t$) is more difficult. With the value of $k_L$ depending on the cube of the thickness, a small inaccuracy in the value of $t$ will lead to great uncertainty in the value of $k_L$. Therefore, for more accurate measurements of $k_L$, other methods exist. A commonly used method is the Sader method [13], which requires only measurements of length, width, resonance frequency, and quality factor of the cantilever. Based on the viscous damping of the cantilever, the spring constant can then be evaluated [14]. Another method involves pressing the lever against a reference cantilever with known spring constant [15]. A common method implemented on several commercial instruments is known as the thermal noise method where the thermal noise spectrum is measured [16, 17]. Other methods for normal spring constant calibration include the somewhat tedious but nondestructive method of the added mass method [18], where the change in frequency is measured as known added masses are added to the cantilever. With all these methods present, practically it is reasonable to measure a spring constant to within 20% error. Getting below that level of error is experimentally challenging and has to include among other things accounting of the exact geometry of the tip, how far from the end of the tip the spot is from the lever, and the tilt of the cantilever. For a detailed discussion see chapter in Haugstad [3].

In addition to the spring constant of the lever, which is in units of newtons/meter and ultimately gives the newtons, or force, exerted by the cantilever, the optical lever sensitivity of the photodetector needs to be calibrated to convert photodetector voltage to meters. This is typically accomplished by conducting a static force curve to measure the deflection of the cantilever against a very stiff surface. With such a measurement, it is assumed that the deflection of the cantilever follows a 1:1 relationship with the piezo pushing down on the surface, so that by measuring the slope of the repulsive wall of the force curve, an accurate nanometer/Volt calibration of the photodetector is obtained. With the calibration of the cantilever spring constant (N/m) and optical lever sensitivity (m/V), a calibration of newtons of force per voltage on the photodetector is obtained.

Note that calibration of the fundamental or first eigenmode cantilever spring constants is discussed here. As multifrequency methods are developed further, calibration protocols for higher order eigenmode spring constants are needed and are currently an active area of research. For more detail and definition on cantilever eigenmodes, see Chapter 3.
1.4.8 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 such as material properties (e.g., modulus) of the surface. Knowing the shape of the AFM probe is a moving target, as the shape is most likely changing during the course of imaging either through contamination or wear. This problem is on top of the fact that AFM probes are realistically not necessarily manufactured in the cone geometry that is commonly idealized, and manufacturing is not wholly reproducible so that there is variety between batches. Direct imaging of the AFM tips by high-resolution techniques such as SEM or transmission electron microscopy (TEM) \[19, 20\] do not provide true three-dimensional (3D) information of the tip to fully characterize the entire probe. Also, 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 \[21\] or blind reconstruction \[22\].

1.5 Nanomechanical Methods Surveyed in Book

As the reader will observe, many different nanomechanical AFM modes are implemented into industrial R&D. These modes range from the “simple” modes such as force curves, nanoindentation, and phase imaging to more sophisticated modes such as contact resonance, Harmonix™, dynamic indentation, and bimodal imaging as well as many of these methods conducted at environmental conditions such as temperature and humidity taking advantage of the flexible environment in which the AFM can operate. Below, the different nanomechanical methods implemented in the industrial R&D applications described in this book are listed. Note that this is not an exhaustive list of all AFM-based nanomechanical methods, but rather the nanomechanical methods that are somewhat well established and have entered the industrial R&D market surveyed here. Most methods have a dedicated chapter (Chapters 3–7) that describes the underlying theory and experimental practice of each technique. Note that Chapter 2 on contact mechanics provides the background theory that underpins all these methods.

**Force Curves (Chapter 3)** Single-point measurements where the AFM tip is directed in and out of a surface while the cantilever deflection is measured as a function of tip–sample separation. Force curves can be conducted in static mode or in dynamic mode. In the latter, the tip is oscillated at its resonant frequency as it approaches and retracts from the surface. Mapping of force curves exists in various forms such as force volume imaging, pulsed force mode, and peak force QNM.
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**Contact Mode** In this mode, the tip is in constant contact with the surface at a load (force) set by the user. The tip is then raster scanned while in contact the whole time. This mode is useful for imaging topography but can be harsh or abrasive on the surface, depending on the material. If the tip is scanned in a direction perpendicular to the cantilever axis, the side-to-side force exerted on the cantilever can be measured in a mode called lateral force microscopy, which can provide information on friction of the surface [23].

**Tapping Mode or Intermittent Contact Mode or Amplitude Modulated AFM (Chapter 4)** One of the most common modes, the tip is oscillated at or near a resonant frequency as it raster scans along the surface. Either the amplitude is held constant by the feedback loop (amplitude modulated) or the frequency is held constant (frequency modulation.) This mode provides a more gentle interaction with the surface than contact mode and thus is preferred for soft materials such as polymers or biological materials. Topography is imaged as well as phase imaging, described below. Recently, multifrequency/higher mode tapping mode has become an object of much research where the tip is oscillated at a higher eigenmode than the tapping mode that occurs at the conventional first eigenmode [24–26].

**Phase Imaging (Chapter 4)** In tapping mode, the cantilever is oscillated or driven at a particular frequency and amplitude. When the cantilever interacts with the surface, the oscillation amplitude is reduced and a phase shift occurs between the drive and response. This phase shift can be plotted as the tip is raster scanned over the surface. Meaning and interpretation of phase shift remains a very active area of research, but it is sensitive to mechanical properties of the material.

**Contact Resonance and Force Modulation (Chapter 5)** In this dynamic contact method, a tip is in contact with the surface, and the tip–surface system is oscillated either below resonance (force modulation) or at a resonant frequency, that is typically much higher than the resonant frequency of the free cantilever (contact resonance). The advantage of this method is that the tip–sample system is now a linear system (as opposed to a nonlinear system such as in tapping mode where the tip is “striking” the surface as it taps) and so is easier to model mathematically to extract mechanical properties.

**Best Practices (Chapter 6)** This chapter outlines experimental best practices to maximize on the information obtained from AFM images. It is intended for an AFM user and outlines some of the common pitfalls and “things to watch out for” in the quest to attain repeatable, accurate, and quantitative measurements.

**Nanoindentation (Chapter 7)** Indentation is a classical method that has been around for over a century, starting with a test that used a 10-mm diameter steel ball to indent and was [27, 28] invented by J.A. Brinell. In the early 1920s, the now popular macroindentation test, the Vickers test, was invented using a pyramidal tip instead of a spherical tip, allowing hardness measurements independent of indenter
1.6 Industries Represented

Size [29]. Approximately 20 years later, indenting with diamond pyramidal shapes was developed [30]. Thus the indentation world had quickly moved from macro- to microindentation and nanoindentation was not far behind. In nanoindentation, Young’s modulus and hardness are measured in the few to 100s or 1000s of micronewtons with very low displacement of several to hundreds of nanometers.

The nanoindentor’s fundamental measurement is a force versus displacement curve as a diamond tip indents into a material resulting in elastic and/or plastic deformation, not unlike the AFM force curve. The size of residual impressions or deformation is not typically measured. Tip–sample contact areas are instead calculated from the depth measurement together with a knowledge of the actual shape of the indentor (which is calibrated before). Nanoindentation can be performed with a diamond AFM tip (AFM-based nanoindentor) or an instrumented nanoindentor. In an instrumented nanoindentor, the vertical motion of the diamond tip is restricted in the z direction and thus is more accurate than an AFM-based nanoindentor where all the cantilever’s degrees of freedom can convolute the measurement. Complexities and best practices for nanoindentation are covered thoroughly in Chapter 7.

**Dynamic Nanoindentation (Chapter 7)** Dynamic nanoindentation has an oscillation imposed onto the tip as it penetrates into the surface. This enables viscoelastic properties to be probed such as storage modulus (E’), loss modulus (E’’), and loss tangent. It is also referred to as “continuous stiffness measurement” by some vendors.

**1.6 INDUSTRIES REPRESENTED**

**Semiconductors** AFM as a key metrology tool for semiconductor devices and to probe mechanical properties of key industrial materials (Chapters 8 and 14).

**Chemicals** Learn how AFM is used to characterize polymer materials, blends, and composites (Chapter 9).

**Food Science** Use of AFM to investigate the molecular nanostructures formed through food processing or those that are naturally present in foods (Chapter 10).

**Petroleum (downstream)** Physical and chemical sensing in hydrocarbon environments (Chapter 11).

**Personal Care Products** Tribology of hair and skin (Chapter 12).

**Pharmaceuticals** Study the effect of single-particle properties on bulk processing behavior of pharmaceutical powders and the role in developing new types of formulations (Chapter 13).

**Biomaterials** Use AFM to study morphology, temperature, and humidity effects of drug eluting biodegradable coatings (Chapter 15).
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REFERENCES