# Imaging and Indenting:
## The Pros and Cons of Stretching Functionality

## Application Note

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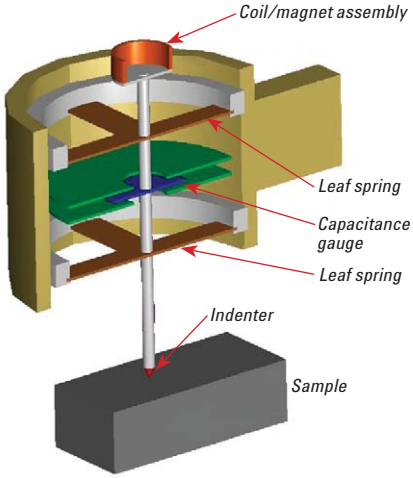


Figure 1. Actuation and sensing schematic for Agilent nanoindenters.

Atomic-force microscopes (AFMs) and nanoindenters (NIs) both work by probing a surface. AFMs are specially designed for high-resolution imaging, and NIs are specially designed for measuring mechanical properties. However, because AFMs and NIs share some operating principles, their functions have some degree of crossover. The principles of profilometry and nanoindentation are explained in detail elsewhere [1, 2]. The purpose of this note is to explain the design features that make each type of instrument well suited for its intended function. This note also addresses the pros and cons of using one type of instrument to perform the intended function of the other.

### Measuring Mechanical Properties with a Nanoindenter

Agilent NI systems have been specially designed for the purpose of deriving mechanical properties from the fundamental measurements of force and displacement. Broadly, accurate determination of mechanical properties requires that (1) the fundamental measurements of force and displacement be accurate, and (2) the models used to infer properties be appropriate. The design considerations relating to the first requirement are myriad. For example:

- Mechanisms for actuation and sensing must be easily calibrated, stable, and independent.
- Motion must be substantially one-dimensional.
- Frame must be very stiff, relative to contact stiffness.

The second requirement is met by using an oblique tip (a Berkovich pyramid) and by making dents that are large relative to surface roughness.

All Agilent NI systems use electromagnetic actuation. This means that force is imposed on the indenter shaft by passing current through a coil that sits within an annular magnet as illustrated in Figure 1. With this system, the electromagnetic force is directly proportional to the current in the coil. This actuating mechanism is easily calibrated against standard weights, and this calibration generally does not change over the course of years. During a test, the motion of the indenter has no influence on the applied force, except through the small and quantifiable change in force exerted by the springs

supporting the indenter shaft. Figure 2 shows the force on the test surface as a function of time over a 50 second period in which the indenter is held in contact with a sample of fused silica at 35.3 µN (10% of the peak force). The standard deviation in force during this period is 19 nN, and the trend is 0.254 nN/sec. These data were acquired with the standard XP-style actuator.

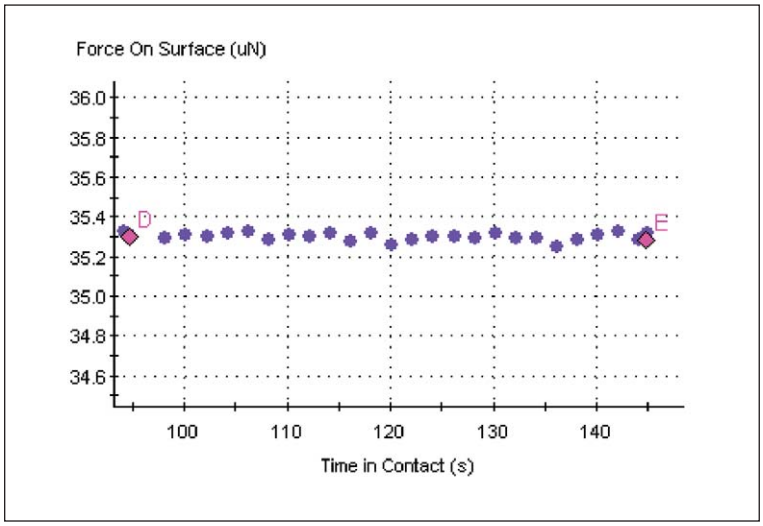


Figure 2. Contact force during a 50-second hold period on fused silica. Standard deviation is 19 nN; trend is 0.254 nN/sec.

All Agilent NI systems sense displacement using the three-plate capacitive arrangement shown in Figure 1. All three plates are circular disks. The two outside plates are fixed to the head and have holes in the center just large enough to accommodate the indenter shaft. The center plate is fixed to the indenter shaft and is free to move vertically between the two outside plates. The position of the indenter column within the gap is determined by observing the voltage between the center plate and either of the two outside plates. This displacement sensor is calibrated using a laser interferometer. Although the relationship between voltage and displacement is not linear, the calibration is extremely stable. Like the calibration of force, the calibration of displacement is generally stable for years. During a test, changes in force have no influence on the measured displacement, except through the predictable influence of frame stiffness. Figure 3 shows displacement into the test surface as a function of time over a 50-second period in which the indenter is held in contact with a sample of fused silica at a constant force of 35.3 µN (10% of the peak force). The standard deviation in displacement over this period is 1.77Å. Prior to any correction for thermal drift, the trend in displacement is 0.024 Å/sec.

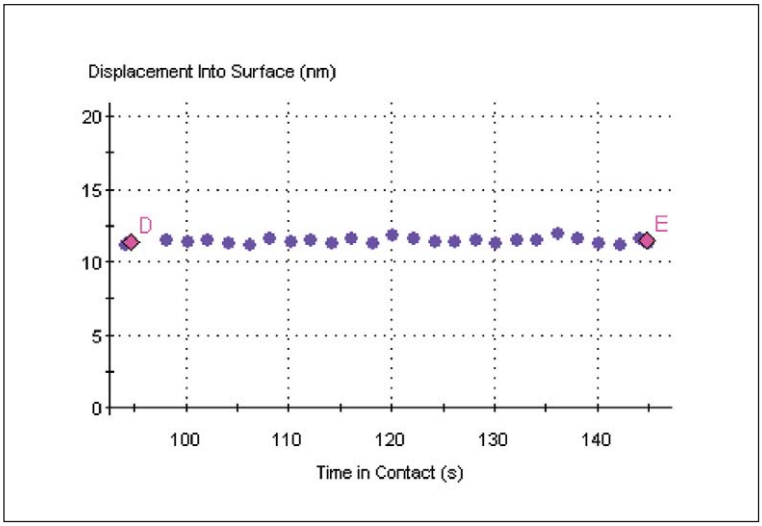


Figure 3. Displacement into surface during a 50-second hold period on fused silica. Standard deviation is 1.77Å; trend is 0.024 Å/sec.

The springs that support the indenter shaft are designed to be very compliant (80 N/m) in the direction of testing and very stiff in the lateral direction (~10,000 N/m). As a result the indenter moves only in one direction when actuated. Thus, no lateral forces are exerted on the indenter tip under normal operation. One-dimensional motion is critical for the successful operation of the continuous-stiffness-measurement (CSM) option, which is used to determined strength profiles with depth for thin coatings [3] and complex modulus of polymers [4, 5]. Because motion is one dimensional, the system can be modeled as a simple harmonic oscillator, so that the response of the sample can be isolated from the response of the equipment.

In any mechanical test (not just indentation), most of the sensed deformation occurs in the test sample, but some of the deformation inevitably occurs in the equipment. Equipment deformation may occur in the mounting, the probe, the gross positioning system, or other places. In a well-designed mechanical testing system, the test frame is very stiff relative to the expected stiffness of the test sample, and this is true of Agilent NI systems.

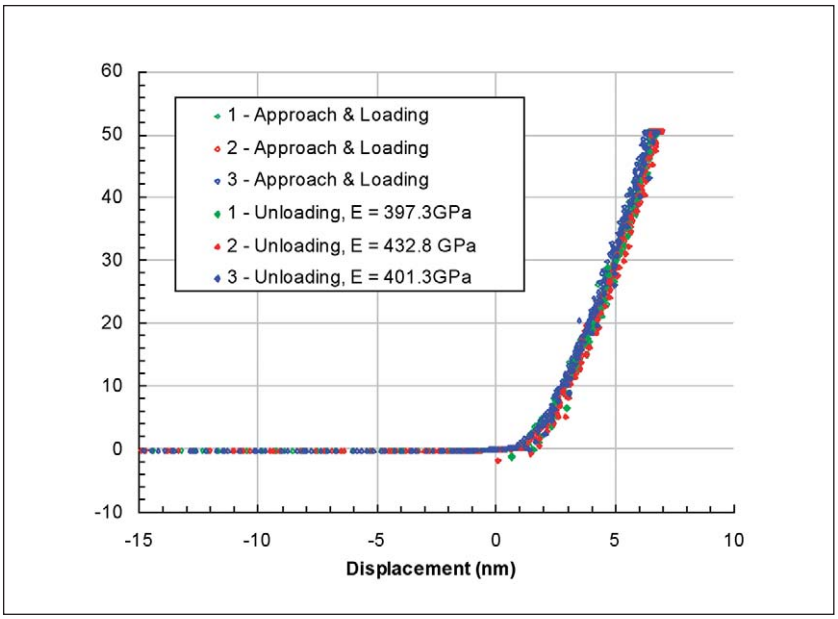


Figure 4. Three consecutive indents on sapphire ($E$ = 400 GPa). Loading and unloading curves coincide, indicating that the indents are completely elastic.

Further, Agilent NI systems have a means for determining frame stiffness and accounting for it during testing.

The most common probe used in NI systems is a Berkovich diamond; Berkovich is the geometry and diamond is the material. The Berkovich geometry is a three-sided pyramid. This indenter shape is oblique: a cone with similar aspect ratio would have an included angle of 140.6°. This indenter is ideal for measuring mechanical properties for a number of reasons. First, because it is diamond, it is very rigid, and so deformation in the indenter is small and predictable. Also, because the material is very hard and the shape is oblique, the apical shape of the diamond is not altered by normal used. This is important because standard NI analysis requires that contact area be inferred *from the known geometry of the probe.* In fact, the apical shape of the probe is precisely determined in a calibration wherein a material of known elastic modulus is probed. If the apical shape changes in any way, then this calibration is no longer valid and must be re-determined before testing can continue.

The analytic models that are commonly used to infer properties from force-displacement data assume that the contacting bodies are semi-infinite with frictionless surfaces [6]. Results are compromised to the extent that these conditions are not met in the physical test. Friction can have a significant and deleterious effect on results [7]. The oblique shape of the Berkovich diamond mitigates the effect of friction by reducing the resolved force acting along the interface between the indenter and the sample. For a given applied force, $F_A$, the force acting along the interface is $F_T = F_A \cos\theta$, where $\theta$ is the half-included angle of the indenter. For a Berkovich indenter, with an effective half-angle of 70.3°, the tangential force is approximately $F_T = 0.34\ F_A$. If a sharper indenter were used, say with an included angle of 30°, the tangential force would be $F_T = 0.86\ F_A$! Of course, if the surfaces are truly frictionless, then this difference is of no significance, but when the potential for friction exists, the Berkovich shape mitigates the consequences. Even when friction is not an issue, sharper tips are undesirable, because they do not manifest the assumption of a semi-infinite body. Various researchers have attempted to derive a correction to the expression for calculating elastic modulus [8-10], but experimental verification has yielded conflicting results [11]. Analysis shows that the magnitude of the correction depends on tip shape and Poisson's ratio, and it may be as high as 50% for indenters with a half-included angle of 15° [8]. To summarize, indenters with small apical angle are to be avoided when probing for mechanical properties.

With all of these design considerations, Agilent NI systems allow the user to measure mechanical properties at the scale of nanometers. Figure 4 shows three successive indents to 50 µN on sapphire, performed with a DCM II actuator. The elastic moduli derived from these curves are also shown to allow comparison with the nominal value for this material: 400GPa.

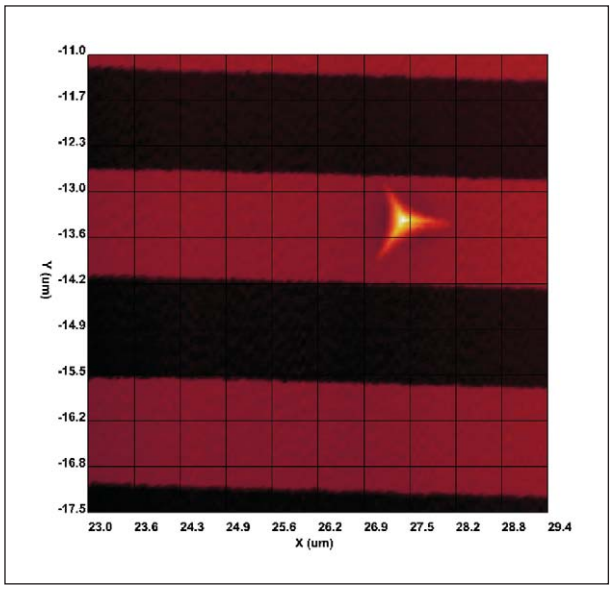


Figure 5. NanoIndenter DCM/NanoVision scan of a square area (6.5 µm on a side) on an AFM-verification grid. Grid steps are nominally 19 nm in height with a periodicity of 3 microns. Image size is 100 x 250. Image generation time is 6.7 minutes.

## Imaging with a Nanoindenter

NI systems can also be used to generate topographic images. Figure 5 shows an image of a silicon grid used for AFM verification, obtained with a DCM II actuator. The grid has periodic steps; the steps have a height of 19 nm and a period of 3 microns. A square area of 6.5 µm on a side was scanned using a scanning force of 1.0 µN; the resulting scan was used to select a site for an indentation test. Following this test, the same area was scanned again to reveal the residual indentation impression.

As a complement to nanoindentation, imaging with a nanoindenter has its advantages. First and foremost, the topographic image can be used for selecting sites for nanoindentation. Imaging can also be used to assess the shape of a residual indentation impression for unusual phenomena such as pileup. If aliasing (explained below) is not a problem, then dimensional measurements obtained from these scans are very reliable. Figure 6 shows the cross profiles through the indent of Figure 5—spatial and topological accuracy are easily verified.

However, an NI system cannot match the imaging capability of an AFM. The most obvious problem is that the oblique tip shape that is ideal for obtaining mechanical properties is not ideal for imaging. "Aliasing" occurs when the indenter passes over a feature sharper than its own slope. The side of the indenter comes into contact with the feature first, and the feature effectively "images" the indenter, rather than the other way around. If the surface is very rough, the resulting image can appear like a field of Berkovich pyramids! Fortunately, this problem does not arise when imaging an impression made with the same indenter, because the hole relaxes more in the normal direction than in the lateral direction. Thus, the residual hole is always more oblique than the indenter that made it. The sharp probe of an AFM is much better for image generation. Although it is easy enough to change the indenter tip on an NI system, doing so usually negates the advantages gained by using the system as both an indenter and a profilometer.

The moving mass of an NI system is *orders of magnitude* larger than the moving mass on an AFM. The moving mass of our smallest actuator, the DCM II, is about 130 mg. This higher mass (relative to the moving mass of an AFM) gains definite advantages for mechanical property measurement: higher frame stiffness, higher force capacity, and a substantial anchor for interchangeable indenter tips. But the inertia of this larger mass makes the NI system less responsive dynamically, thus limiting scan speed. An AFM can generate images much more quickly than an NI system.

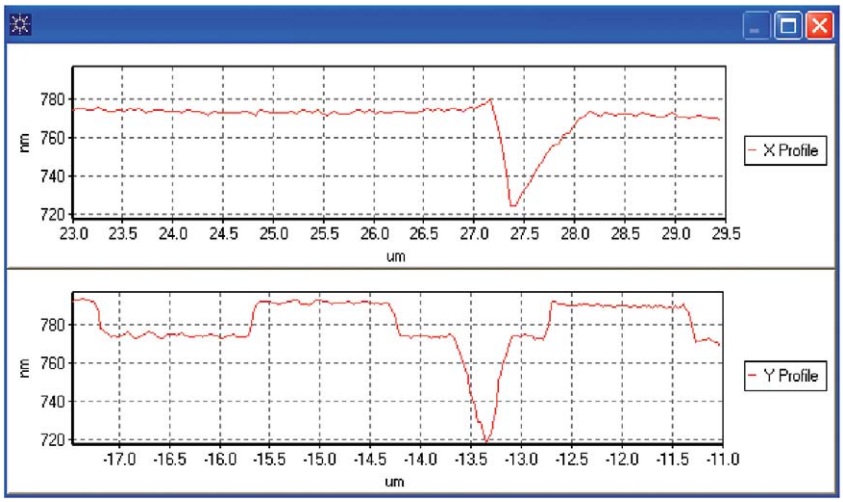


Figure 6. Line profiles through the center of the residual impression. Step height of 19nm and periodicity of 3 µm are easily verified. Residual depth of indentation is about 53 nm.

## Imaging with an Atomic-Force Microscope

Atomic Force Microscopy (AFM) has developed and is widely used primarily as an imaging tool upon its invention in 1986. Goals for AFM include: 1) imaging with ultra-high resolution, 2) identifying different materials, 3) probing a wide range of surface properties either qualitatively or quantitatively, and 4) extending its application to various environments. Much effort has been invested in turning AFM into a comprehensive surface tool. Continuous progress in AFM instrumentation and emergence of advanced imaging modes dramatically broaden AFM's scope of enabling aspects, which further facilitates its extensive application.

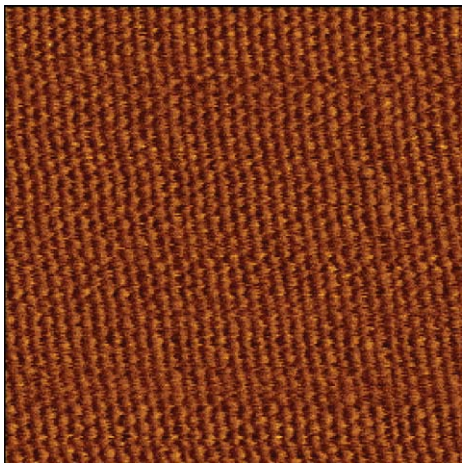

Figure 7. An atomic-resolution AFM topographic image of graphite. Scan size: 8 nm × 8 nm.

In brief, the working principle of AFM technique can be explained here using acquisition of the topography image as an example: A sharp tip is positioned at the free end of a cantilever while a diode laser is focused onto the back of the cantilever and reflected towards a four-segment photosensitive detector (PSD). When the tip rasters against a sample, this laser-beam deflection configuration allows for sensitive detection of either the variation of reflected laser location on PSD (contact mode case) or the variation of the amplitude (oscillation mode case) due to tip-sample interactions. At every point of a scanned area, the tip is under the control with a feedback loop to adjust its position in vertical direction in order to maintain the tip-sample force at a set-point level. Therefore, those Z height adjustments at each pixel can be recorded to obtain a mapping of the sample topography. High-resolution imaging is definitely a key feature associated with the AFM functions. It has proven that AFM offers the capability to visualize a sample's surface morphology in situ and in real time with an unprecedented spatial resolution since atomic or molecular-level structural information of various types of materials including metal, semiconductor, organic thin films or even some biological systems has been resolved successfully. Such achievements, to some extent, can be attributed to the incorporation of piezoelectric materials into the scanner construction. The piezoelectric effect, i.e. the ability of some materials (such as certain ceramics) to generate an electric field or electric potential in response to applied mechanical stress even at nanometer-scale, allows both the tip scan in X and Y direction and adjustment in Z direction are controlled accurately at sub-nanometer level. For example, the lateral and vertical resolutions of AFM for a crystal surface can reach as high as 0.1 and 0.01 Å, respectively [12, 13]. An example of high-resolution AFM imaging is shown in figure 7, in which the crystalline structure of graphite is clearly resolved.

Other than direct visualization, AFM is also capable of sensing materials, allowing various materials or even different states of the same material in a heterogeneous or complex sample to be effectively differentiated. This important characteristic results from the fact that AFM instrument features an easy multi-channel data acquisition so the impact of the tip-samples interactions on the tip during the scanning can be monitored from different perspectives simultaneously. In contact mode, the tip will exhibit changes not only in vertical deflection due to the variations in sample morphology but also in lateral twisting caused by the friction force when tip keeps in contact and moves along the sample surface. Although those combined tip-sample interactions lead the deflected laser to shift its location on PSD, the four quadrants of the PSD enable the separation of the total signal and derive the tip-sample interactions at each direction. While the signal difference between the top and bottom half of the PSD is served as an input signal for the feedback system to maintain a constant tip-sample interaction in Z direction and thus to produce a topography image, the signal difference between the left and right half of the PSD will be used directly to translate into a friction (or lateral force) image. Because factors of the sample such as hardness, hydrophilicity or terminal chemical functionality can have an effect on friction force, the tip lateral twisting is highly dependent on natures of materials. Therefore, the contrast variations in friction images can be used to detect different materials. In oscillation mode, tip-sample interactions may cause changes in amplitude, phase and the resonance frequency of the vibrating cantilever. The spatial variation of the change can be presented in height (topography) or interaction (amplitude or phase) images that can be collected simultaneously. While the motion of the scanner at each probe location that try to maintain the oscillation

amplitude as a set value (set-point) is used to generate a topographic data set, the actual oscillation amplitudes and the phase lag between the AC drive input and the cantilever oscillation output at each probe location can be collected to produce the amplitude and phase image, respectively. It has been demonstrated by many research groups that phase contrast is very sensitive to the differences in material properties such as variation of the mechanical (stiffness, friction) and adhesive properties. The materials sensing capability of AFM is demonstrated in figure 8 using semicrystalline polymer linear low density polyethylene (LLDPE) as an example. Visualization of amorphous and crystalline components can be achieved because phase images revealed lamellar structures, which are embedded in amorphous surrounding.

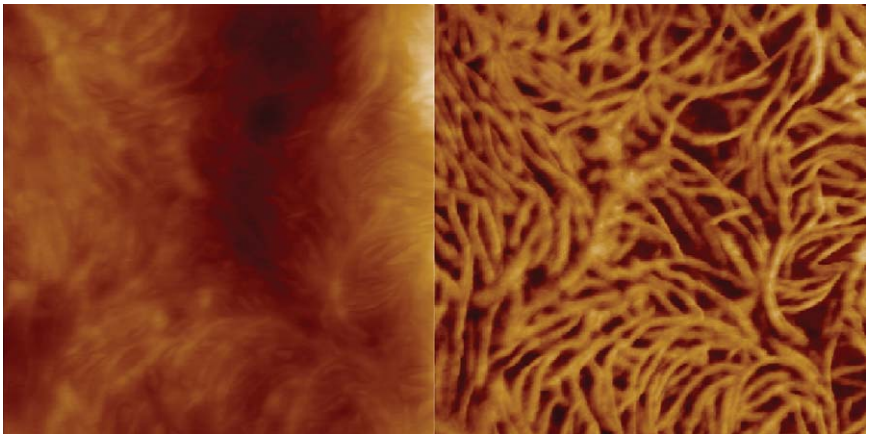

Figure 8. AFM topographic (left) and phase (right) images of polymer LLDPE. Scan size: 2 µm × 2 µm.

Beyond topography and materials identification, investigations of sample surface properties such as electric, magnetic, thermal or mechanical ones are highly desired. These needs stimulate the generation of many derivatives of AFM. For example, conductive or current sensing atomic force microscopy (CSAFM), electric force microscopy (EFM) and Kelvin force microscopy (KFM) can be used to probe conductive domains, surface charges and potentials. Magnetic force microscopy (MFM) can provide the mapping of magnetic domains on the surface.

It occurs quite often that AFM measurements of some specific samples need to be performed under particular conditions, integrating AFM with comprehensive environmental control is always an attractive area for the instrument development. A sample plate with either heating or cooling capabilities can provide an easy control of the sample temperature. Another useful auxiliary part is the liquid cell, which allows the samples to be imaged within a liquid medium including a buffer solution. Extension of AFM in biological studies benefits a lot from those mentioned hardware because physiological conditions (related to a particular range for pH, temperature, glucose concentration and atmosphere pressure, etc.) are critically required for most biosamples such as DNA, protein, bacteria and cell to maintain their bioactivities. As a result, AFM studies of a biological process under near-physiological conditions can shed insights on how things may proceed *in vivo.* Further environmental controls are available in some commercial AFM. For example, most of Agilent AFM setups include an environmental chamber and the scanner can be hermetically sealed with this chamber, leaving the sample in a closed system and separated from the outside. The chamber also features eight inlet/outlet gas/electrical ports to offer total control of the sample environments such as temperature, humidity, gas atmosphere, etc.

It is worthwhile to mention that AFM technique is closely tied to the development of microfabricated probes. Taking the advantage of the maturity of technologies in semiconductor industry, manufacturing of AFM probes with a control on their stiffness, resonance frequency and tip sharpness is quite achievable. Probes with different geometry, aspect ratio, coating layer, orientation or made of special materials are also commercially available to serve some particular applications.

Although there is still room to be improved, AFM is a powerful imaging tool for materials characterization, capable of direct and three dimensional visualization of surface morphology with high-resolution, differentiation of different materials, probing a wide range of surface properties, and providing in situ and in real time monitoring of surface-initiated reactions.

## Measuring Mechanical Properties with an Atomic-Force Microscope

Although a nanoindenter has some use as a profilometer, the converse is not true. AFMs make very poor nanoindenters for a number of reasons.

The tip shape that is ideal for imaging is not ideal for measuring mechanical properties as previously explained. AFM tips wear and break easily, so calculating contact area from the known geometry of the tip is not practical. Even if the tip shape were to remain stable, the tip is too sharp to meet the assumptions underlying standard nanoindentation analysis.

The second problem with using AFMs to measure mechanical properties is the means by which contact force is determined. In most commercially available AFMs, contact force is inferred from the deflection of the cantilever and the stiffness of the cantilever. Therefore, any error in the stiffness of

the cantilever manifests directly as an error in force. It is a real challenge to determine cantilever stiffness to better than +20%, and this stiffness can vary significantly from beam to beam. Finally, the force capacity of an AFM is *very* limited, relative to that of a NI system.

Some may contend that AFMs can measure mechanical properties *qualitatively,* but even this claim is dubious, if tip shape changes. Slight changes in tip shape manifest as significant changes in the force-displacement relationship. In other words, if the same material is tested with two different tips (or one tip that has changed shape), the force-displacement curves will be different. If the same tip shape is assumed for both curves, the properties obtained from those curves will be different. Precise knowledge of the tip shape is necessary, in each case, to extract mechanical properties accurately.

## Conclusion

The intended purpose of each type of instrument dictates many aspects of design, and some of these aspects preclude extended functionality. The extent of cross-functionality is asymmetric: Although a nanoindenter can be used as a profilometer under certain circumstances, an AFM cannot be used to measure mechanical properties, either quantitatively or qualitatively, because the tip shape is not ideal and because contact force is not sufficiently well known. When vendors present indentation force-displacement curves acquired with an AFM, they rarely cite properties derived from those curves, or if properties are cited, the test material is not one for which properties are verifiable.

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