

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

Asylum Research

Asylum Research **Cypher™** and **MFP-3D™** atomic force microscopes (AFMs) provide valuable information for characterizing thin films and coatings. They quantify 3D roughness and texture with unmatched spatial resolution and measure nanoscale functionality including electrical, magnetic, and mechanical behavior.

Thin films and coatings play a critical role in everything from food containers to photovoltaics. To meet such varied needs, they are made from every class of material and by numerous processes including physical and chemical vapor deposition techniques, atomic layer deposition, and sol gel processing.¹ A key step in developing any new film is characterizing its surface structure and physical properties, whether in engineering commercial products (Figure 1) or pursuing fundamental materials science (Figure 2).

The intrinsic dimensions of films (thickness, grain and domain sizes, etc.) make it important to characterize them on sub-nanometer to micrometer length scales. The AFM is a powerful tool for this purpose for many reasons. For instance, it possesses much higher spatial resolution than other stylus or optical-based methods.⁴ Samples need not be optically reflective or electrically conducting, allowing access to virtually any film. AFMs also provide complementary information to electron microscopes, such as accurate 3D surface profiles, and offer a more flexible operating environment for work at both ambient and non-ambient atmospheres and temperatures.

Here we describe the extensive features of Asylum Research AFMs for thin film characterization and show examples over a range of applications. With today's AFMs, surface roughness can be measured more accurately, quickly, and easily than ever before. A wider array of built-in analysis tools and automated routines mean higher productivity and greater ease of use. Also, research and instrumentation advances have created a variety of AFM modes for measuring nanoscale film functionality including electrical, magnetic, and mechanical response.

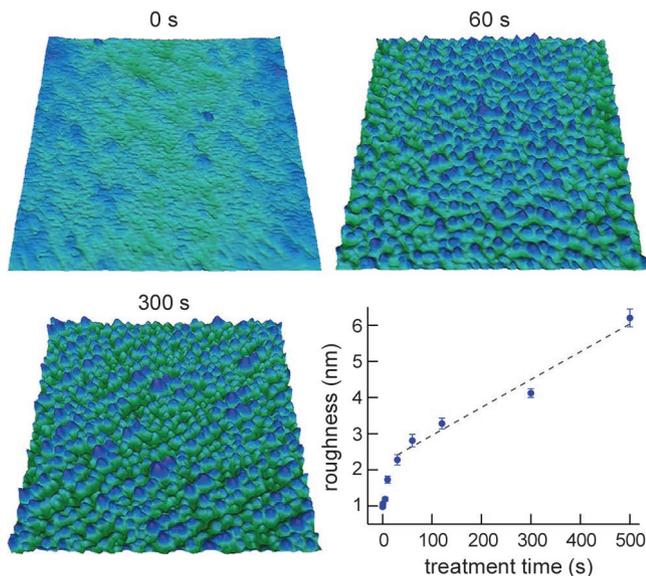


Figure 1: Oxygen plasma treatment of polyethylene terephthalate (PET) films. PET fibers coated with a conducting polymer such as polypyrrole could be used in “smart” electronic textiles. However, achieving good coating-to-fiber adhesion remains a key challenge. Images of PET films exposed to oxygen plasma show that RMS surface roughness increased with exposure time. Films processed longer than 60 s displayed surface etching and uniform nanoscale features. The graph reveals a linear dependence of roughness on treatment time after ~30 s. Combined with data on surface chemistry, the results can be used to optimize treatment parameters for improved coating adhesion and conductivity. Scan size 1 μm; height scale 35 nm. Imaged with the Cypher S AFM. Adapted from Ref. 2.

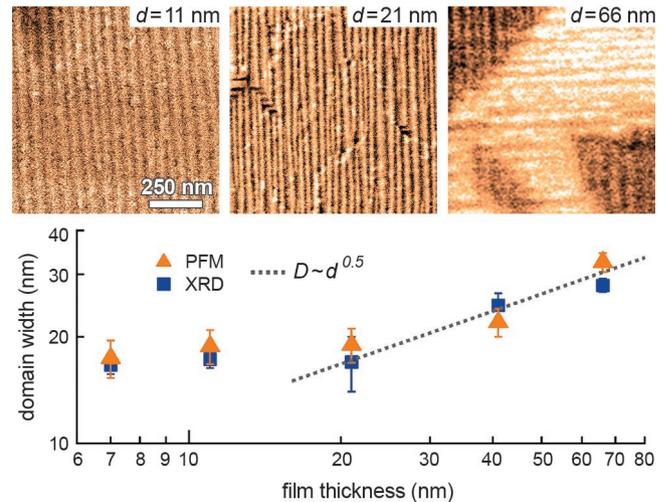


Figure 2: Strain effects in ferroelectric NaNbO_3 (NNO) films grown on TbScO_3 (TSO) substrates with metal organic chemical vapor deposition (MOCVD). Growth of epitaxial NNO on TSO results in significant anisotropic misfit strain. Understanding relations between strain, crystal structure, and ferroelectric response will enable fine-tuning of film properties. The lateral piezoresponse force microscopy (PFM) image on a film with thickness $d=11$ nm reveals a strong in-plane piezoresponse with highly ordered domains (vertical stripes). For a thicker film ($d=21$ nm), distortions in the alignment appear. For an even thicker film ($d=66$ nm), 90° domains (horizontally striped regions) are observed, indicating a 1D to 2D domain pattern transformation. The graph shows values for the lateral piezoelectric domain width D obtained by PFM and x-ray diffraction (XRD). The dependence of D on d changes from approximately constant to the predicted $D \propto d^{0.5}$ (dotted line) at $d \approx 20$ nm, where the 1D to 2D transformation occurs. Acquired on the MFP-3D AFM. Adapted from Ref. 3.

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

Imaging Topography

Surface structure such as roughness and texture is often closely related to a film's behavior. For instance, grain size affects electromigration in copper interconnects, while the microstructure of diamond-like carbon coatings affects friction in cutting tools. Roughness also determines wettability in biocompatibility coatings for medical implants, and nanopatterning to control roughness improves the efficiency of LEDs. Measuring surface roughness during the product development cycle can provide process control metrics and reveal structure-property relations. Measurements can also be used to understand the effects of post-deposition treatments (polishing, heat, plasma, etc.) and elucidate product failure modes.

Three-dimensional AFM images of topography (height) give nanoscale information on film structure including roughness, defects, amorphous and crystalline phases, and nucleation and growth modes. Topographic imaging is usually performed in tapping mode. Here, the AFM cantilever is oscillated near its resonance frequency, and the probe tip only contacts the sample during part of the oscillation cycle. Compared to contact mode, the much gentler lateral and vertical tip-sample forces in tapping mode reduce damage to both the sample and the tip. The high spatial resolution of tapping mode arises from ultrasharp tips and the small tip-sample contact area, so that even atomic and crystal structures can be resolved (Figures 3 and 4).

The accuracy of AFM topography measurements depends on the spatial resolution in both the vertical (Z) and lateral (XY) directions. In the Z direction, resolution is limited mainly by instrument noise. Factors such as mechanical vibrations and random electrical fluctuations create variations in the height signal. Height features smaller than these variations cannot be meaningfully resolved. Linearity of the Z piezo can also impact

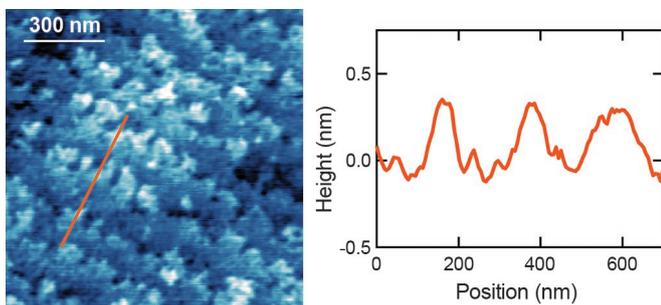


Figure 3: Topography imaging of atomically flat strontium vanadate (SVO). The epitaxial film was grown by pulsed electron-beam deposition and was ~6.5 nm thick. SVO has potential applications in electronic switching and sensing, and recent advances in synthesis techniques enable growth of low-dimensional, ultrathin films with improved functionality. The image was acquired with the Cypher S AFM and demonstrates its superb Z resolution. Height scale 0.9 nm. The section across the line in the image shows the atomically flat nature of the film, with step-terrace structures high corresponding to the lattice spacing of one unit cell. Adapted from Ref. 5.

Asylum AFMs for Topographic Imaging

- The [Cypher](#) and [MFP-3D Infinity™](#) AFMs feature the latest generation of position sensors with exceptionally low noise, as low as 35 pm in Z (Infinity) and 60 pm in X and Y (Cypher). All Asylum AFMs feature closed-loop scanners for accurate and repeatable scanning motion. This eliminates image distortions and enables high-precision offsets and zooms on specific scan areas.
- Small cantilevers (<10 μm long) make line scan rates up to 40 Hz routinely achievable on Cypher AFMs, so that a full 256×256 pixel image takes only 5-10 seconds.
- Cypher AFMs do not require vibration isolation equipment in most labs, even when imaging atomically flat samples. They were designed for maximum mechanical stability and include an integrated acoustic enclosure. These features make them nearly immune to normal environmental noise and reduce thermal drift by 10× compared to less advanced AFMs.
- [GetStarted™](#) makes tapping mode faster and simpler by automatically optimizing imaging parameters. Available on MFP-3D Infinity and Cypher AFMs, a predictive algorithm sets parameters before the tip even touches the sample, ensuring that high-quality data is acquired from the first scan line.
- [SpotOn™](#) automated laser alignment for Cypher AFMs greatly reduces setup time. With just a mouse click on the desired position, the fully motorized stages align the laser spot on the cantilever and center the photodetector.

Learn More

Webinar: [“Smaller and Quieter: Ultra-High Resolution AFM Imaging”](#)

Webinar: [“There’s No Other AFM Like Cypher: Recent Technological Advances”](#)

Application Note: [“Measuring Surface Roughness with AFM”](#)

accuracy, especially on samples with higher roughness. Lateral resolution is primarily determined by the size of the AFM cantilever tip and its impact on the tip-sample contact area. The tip radius is typically <10 nm when new, so that a tip-sample contact radius of <1 nm is common in tapping mode. However, gradual tip wear will affect both the achievable lateral resolution and measurement repeatability or precision. Tip blunting can be avoided if the AFM has a responsive mechanical Z-axis. By quickly adjusting the cantilever base in response to surface height variations, the Z axis prevents the tip from applying larger forces than intended, protecting the tip from damage.

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

Another issue in topography measurements is the potential for image drift, which affects fidelity and repeatability. Thermal variations are the main source of drift, for instance due to fluctuations in room temperature. These variations can cause the AFM components to expand and contract so that the relative position between the cantilever and the sample slowly changes. Thermal drift can be greatly reduced by careful instrument design or by actively controlling the AFM temperature.

Besides improved technical specifications such as spatial resolution, recent years have seen other upgrades to Asylum Research AFMs. Automated setup tools and numerous data analysis routines have been added for streamlined operation. Also of note is the dramatic increase in image acquisition speed. Faster scanning increases throughput, for instance to assess film uniformity by imaging different sample regions. High scan rates are possible using AFMs like the Asylum Research Cypher that can use smaller cantilevers with higher resonant frequencies. Small cantilevers also provide noise and resolution benefits, and can better resolve and control very low (sub-piconewton) forces. These advantages mean that imaging features like single-point atomic defects and the DNA double helix is not just possible but commonplace on Cypher AFMs.

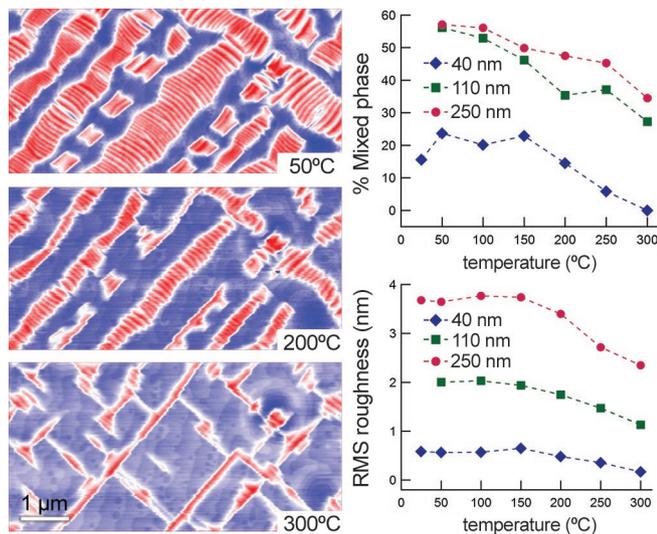


Figure 4: Temperature-dependent structural transformations in epitaxial BiFeO_3 (BFO) films. Highly strained BFO is a multiferroic material that exhibits complex structural changes near room temperature. Understanding how these changes affect the electromechanical response will hasten applications in sensors, actuators, and electronic memory. (left) Topography images for a 110 nm thick film reveal regions of atomically flat terraces (blue) and mixed-phase regions (red) that evolve with temperature. Height scale 8 nm. Acquired with the PolyHeater stage on the MFP-3D AFM. (right) Structural parameters as a function of sample temperature and film thickness. (top) The percentage of mixed phase was calculated by masking the topography images to determine the areal ratio of mixed-phase regions to the entire sample area. (bottom) RMS roughness is an indicator of the volume fraction of mixed-phase regions in the films. Adapted from Ref. 6.

Asylum AFMs for Roughness Analysis

- Roughness parameters calculated on Asylum Research AFMs include:

Root mean square (RMS)

$$R_{\text{RMS}} = \sqrt{\frac{1}{N} \sum_{i=1}^N z_i^2}$$

Average deviation

$$R_a = A_{\text{dev}} = \frac{1}{N} \sum_{i=1}^N |z_i - \bar{z}|$$

Standard deviation

$$R_q = S_{\text{dev}} = \sigma = \sqrt{\frac{1}{N-1} \sum_{i=1}^N (z_i - \bar{z})^2}$$

(S_{dev}^2 is the variance)

Skewness

$$R_{\text{sk}} = \frac{1}{N} \sum_{i=1}^N \left(\frac{z_i - \bar{z}}{\sigma} \right)^3$$

Kurtosis

$$R_{\text{ku}} = \frac{1}{N} \sum_{i=1}^N \left(\frac{z_i - \bar{z}}{\sigma} \right)^4 - 3$$

Here z_i is the height at a given pixel i , N is the total number of pixels in the image, and \bar{z} is the average height of the entire image. Some sources use the symbol S instead of R when referring to 2D image roughness.

- These and other surface parameters are easily calculated in the Asylum AFM software. All parameter calculations are user-accessible for validation or modification, and new parameters or algorithms can be defined if desired.
- The Asylum AFM software has many other built-in tools for analysis of topography. Masks are invaluable for including or excluding specific regions from analysis. Thresholding routines help generate regions of interest and masks. Histograms and sections show the distribution of height values. Other features include spatial Fourier transforms (power spectral density) and pore or grain analysis.

Analyzing Roughness

Roughness parameters summarize the complex 3D information of topography images, for instance to compare samples. There are many roughness parameters^{4,7} but just three main types: amplitude, spacing, and hybrid. Amplitude parameters characterize the surface based on its Z variations, while spacing parameters describe XY variations. Hybrid parameters such as slope and curvature describe variations in X, Y, and Z.

Surface roughness is most commonly described by amplitude parameters including the average deviation R_a or A_{dev} , the root mean square R_{RMS} , and the standard deviation R_q or S_{dev} (see sidebar above for definitions). These parameters have units of length, with higher values indicating greater height variation. Because they do not discriminate between peaks and valleys or the spacing of features, parameters can have the same value for samples with very different textures. R_{RMS} and S_{dev} are more

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

sensitive than A_{dev} to occasional highs and lows. Both R_{RMS} and S_{dev} describe the dispersion or spread in height variations, but S_{dev} is typically smaller than R_{RMS} because it does not include the average height. Figure 4 shows systematic analysis of topography images to characterize multiferroic films.

Skewness and kurtosis are dimensionless roughness parameters that describe the spatial distribution of height variations. If the variations follow a normal distribution, the skewness R_{sk} and the kurtosis R_{ku} are both equal to 0. Skewness characterizes the symmetry of the height variation about its mean line, with $R_{\text{sk}} > 0$ for surfaces with high peaks or spikes and $R_{\text{sk}} < 0$ for ones with deep valleys or pits. Kurtosis describes the uniformity of height variations, with $R_{\text{ku}} > 0$ for surfaces with many peaks and valleys and $R_{\text{ku}} < 0$ for those with fewer excursions.

As with any instrument, surface roughness measured by the AFM depends on experimental variables such as tip radius and scan size.⁴ Features smaller than the tip diameter, particularly pits or valleys, cannot be sensed. Therefore, measurements of parameters like A_{dev} tend to decrease with increasing tip size. Likewise, features larger than the scan size cannot be detected. Thus images with larger scan sizes tend to give higher roughness values because features with both short and long spatial wavelengths are detected. When comparing roughness values, it is good practice to keep tip and imaging conditions as similar as possible.

Studying Solvent and Thermal Effects

During the device integration process or during product use, a film can be exposed to non-ambient conditions such as liquid or vapor solvents or high or low temperatures. Mimicking these conditions during AFM measurements makes results more meaningful and allows studies of long-term durability and reliability. Measuring how topography, functionality, or mechanical properties change with temperature or solvent concentration also provides fundamental materials insight.

Asylum AFMs have many capabilities for operating in controlled environments. Highly stable, precise temperatures and heating rates can be achieved. For instance, Figure 4 shows topography measurements up to 300°C on multiferroic films, and Figure 5 tracks the morphology of a block copolymer film during exposure to toluene vapor. AFM measurements in liquid or gas solvents often involve specialized cells that surround both the cantilever and sample. Cells can either provide a static environment or allow perfusion of liquids and gases.

For some film applications, corrosion and other electrochemical reactions are an important consideration. Electrochemistry cells for the AFM allow in-situ investigation of processes such as film deposition or electroplating, oxidation, corrosion, and mass transfer. With an electrochemistry cell, topographic imaging can precisely monitor nanoscale structural changes induced by electrochemical reactions (Figure 6).

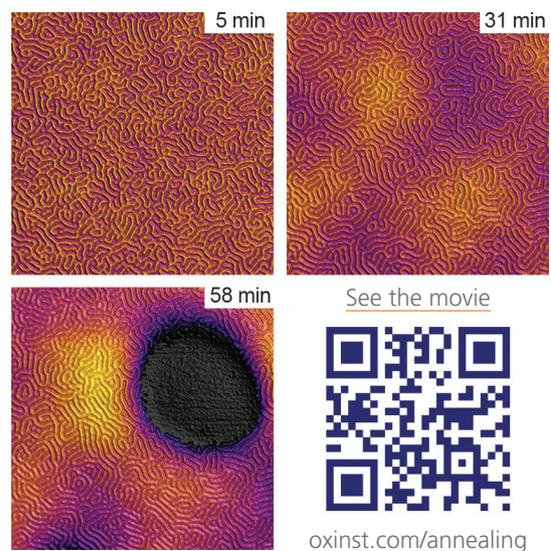


Figure 5: Solvent vapor annealing of a polystyrene-polybutadiene-polystyrene (PS-PB-PS) triblock copolymer film. Films formed by spin casting organize into microphase morphologies that do not necessarily represent the lowest energy state. Annealing often results in restructuring, and solvent vapor techniques offer advantages over standard thermal annealing. Topography images were acquired continuously as the film was exposed to toluene vapor in a closed sample cell. Scan size 2 μm , line scan rate 10 Hz. Imaged with the Cypher ES.

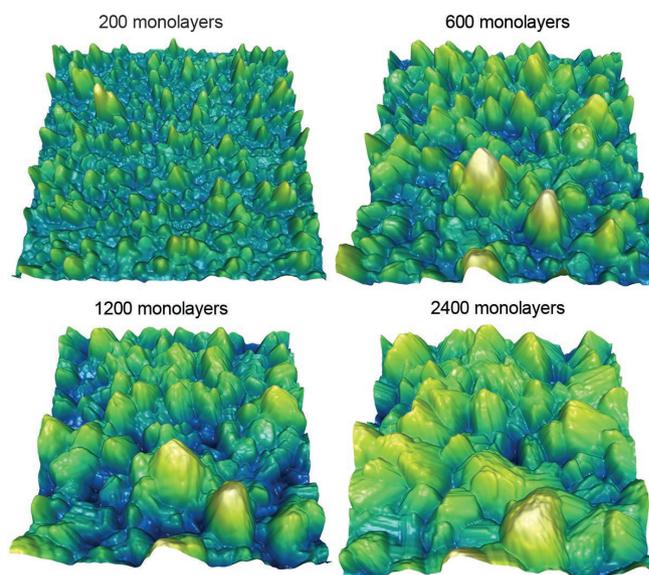


Figure 6: In-situ monitoring of zinc electrodeposition in an ionic liquid electrolyte. Zinc is an attractive material for electrodes in rechargeable batteries, but it can form detrimental morphologies that short circuit the cell. Uniform surface roughness that does not change with time reduces the probability of forming such features. These topography images show the morphology of a film grown at 325 mV deposition overpotential versus increasing film thickness (or equivalently, time). Surface roughness initially increased with thickness and time but eventually became constant (not shown). In contrast, the roughness always increased for films deposited at higher and lower overpotentials. Imaged with the MFP-3D AFM and the Electrochemistry Cell. Scan size 2 μm , height scale 200 nm. Adapted from Ref. 8.

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

Asylum AFMs for Solvent and Thermal Effects

- The Cypher ES AFM was specifically designed for precise environmental control and contains a sealed cell compatible with even the harshest solvents. It enables static or perfusion operation, including relative humidity control of gases. The Heater (ambient to 250°C) and CoolerHeater (0°C to 120°C) sample stages provide precise temperature control.
- For MFP-3D family AFMs, the Closed Fluid Cell enables static or perfusion operation in gas and liquid environments, with temperature control added by the BioHeater™ (ambient to 80°C). A Humidity Sensing Cell is also available for MFP-3D family AFMs.
- Temperature control is available for MFP-3D AFMs with the PolyHeater (ambient to 300°C), PolyHeater+ (ambient to 400°C), and CoolerHeater (-30 to +120°C) sample stages.
- The Electrochemistry Cell for MFP-3D family AFMs is fabricated from chemically inert polyether ether ketone (PEEK) for robust electrochemical capabilities.

Learn More

Webinar: [“There’s No Other AFM Like Cypher: Recent Technological Advances”](#)

Measuring Electrical, Electromechanical, and Magnetic Functionality

Many applications exploit the functional properties of a thin film—its electric, magnetic, or other response—to achieve the desired performance. Examples include piezoelectric actuators, pyroelectric sensors, and ferroelectric nonvolatile memory. Topographic measurements on these films lend insight but do not directly assess functionality. Therefore, a number of AFM techniques have been developed to measure functional properties (Table 1).⁹⁻¹² These modes leverage the AFM’s exquisite force sensitivity to probe magnetic, electrostatic, and other interactions between the tip and the sample. The AFM’s nanoscale spatial resolution means that measurements can be made on both uniform (blanket) films and patterned ones, for instance for failure analysis of thin-film devices. Simultaneous imaging of topography and functionality also enables deep insight into local structure-property relations.

AFM modes to characterize electrical properties include Conductive AFM (CAFM), Electrostatic Force Microscopy (EFM), and Kelvin Probe Force Microscopy (KPFM).⁹ As seen in Figure 7, CAFM can easily detect conductivity variations at terrace steps in graphene. Electrical modes are also valuable for assessing film coverage and uniformity. A related technique, Scanning Microwave Impedance Microscopy (sMIM),¹⁰ enables permittivity and conductivity measurements. An example is given in Figure 8, which shows electrical permittivity imaging of buried structures.

Piezoresponse force microscopy (PFM) has powerful and versatile capabilities for characterizing piezoelectric, ferroelectric, and multiferroic films.¹¹ It provides nanoscale information on both static and dynamic electromechanical response such as domain structure, growth, and polarization reversal (Figure 2). When measuring thin films with conventional PFM techniques, it can be challenging to get good signal-to-noise ratio without using drive voltages high enough to cause polarization switching or even sample breakdown and damage. One option for attaining higher sensitivity at lower drive voltage is to operate near the cantilever’s contact resonance frequency.

Mode	What is sensed	Information obtained
CAFM	current	conductivity, film uniformity and defects, dielectric breakdown, dopant distribution
EFM	electrostatic forces	electrostatic gradients, capacitance variations, embedded conductors
KPFM	electric potential	surface potential, work function, film uniformity and coverage
sMIM	capacitance and resistance	film thickness, dielectric constant, permittivity or conductivity variations, buried charge
PFM	electromechanical response	piezoelectric domains, polarization vector and switching, ferroelectric coercive field
MFM	magnetostatic forces	magnetic domains, magnetization hysteresis, magnetic coercive field

Table 1: AFM modes for functional properties. For each mode is shown its acronym as defined in the text, the physical quantity measured, and examples of information that can be acquired.

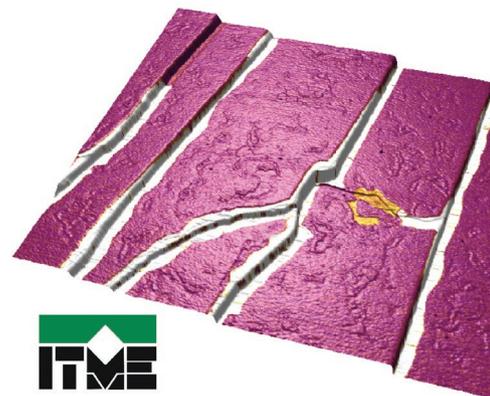


Figure 7: Electrical characterization of graphene with CAFM. The sample was a quasi-freestanding bilayer formed by chemical vapor deposition on a semi-insulating 4H-SiC (0001) (silicon carbide) substrate. The image shows CAFM current for a bias voltage of -1.5 V DC overlaid on topography. Areas with three regimes of electrical conductance can be identified: low (purple), intermediate (gold), and high (white). The step edges of the underlying substrate induce a resistance anisotropy that results in high conductance channels along the terrace steps. Scan size 10 μm, current scale 120 pA. Acquired with the Cypher S AFM and the ORCA™ CAFM module. Sample courtesy Institute of Electronic Materials Technology (ITME, Warsaw, Poland).

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

For nanoscale measurements of magnetic behavior, magnetic force microscopy (MFM)¹² makes use of the interaction forces between a magnetic sample and a magnetized tip. It sensitively probes functional properties of ferromagnetic and multiferroic films such as those used in read/write heads, spintronic devices, and high-density data storage. MFM can provide information on magnetic domain patterns and walls, magnetic vortices, and even flux lines in superconductors. Like other AFM methods, MFM is an attractive characterization technique because it provides high-resolution images, needs little or no sample preparation, and can be used in a range of environmental conditions. Figure 9 shows MFM studies to understand the effect of film deposition variables on magnetic domains.

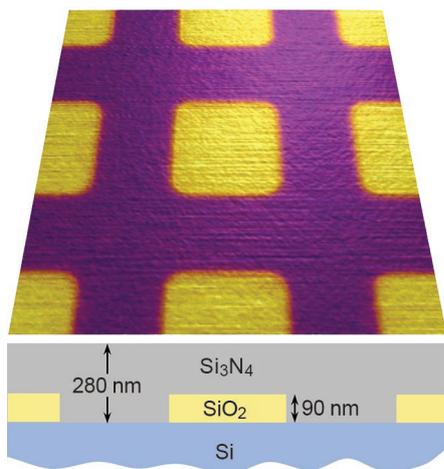


Figure 8: sMIM imaging of subsurface permittivity variations. The schematic diagram shows that the sample contained silica (SiO_2) squares 90 nm thick buried under a thicker silicon nitride (Si_3N_4) film. After deposition of the Si_3N_4 film, the sample was polished so that topography variations were less than 0.5 nm. The image contains the relative capacitance signal obtained with sMIM overlaid on topography. The buried structures are clearly detected, despite little or no difference in topography. Scan size 20 μm . Acquired on the MFP-3D AFM. Image courtesy PrimeNano, Inc.

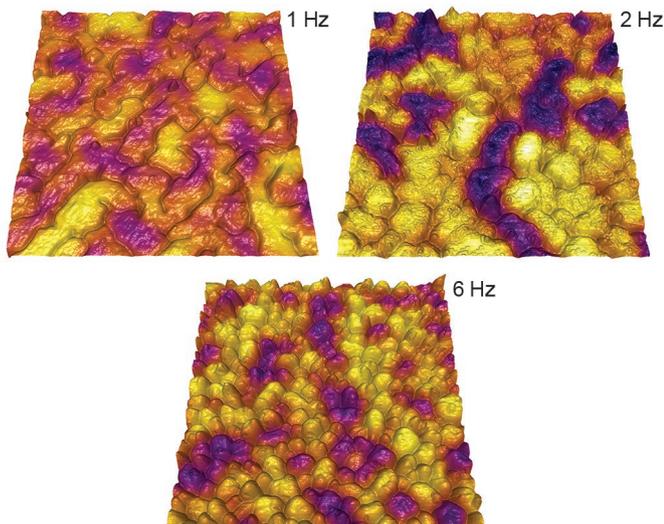


Figure 9: MFM evaluation of ferromagnetic FePt films grown by pulsed laser deposition (PLD). Understanding how magnetic behavior depends on PLD laser frequency could enable better control of film properties for applications such as data storage and recording. In these images, light (yellow) and dark (purple) regions represent domains polarized in the up and down directions, respectively. Overlaying the MFM phase signal on topography shows that the films deposited at 2 Hz and 6 Hz form single domain structures that correlate exactly with FePt islands. The 1 Hz film has a percolated morphology and forms multiple magnetic domains. Scan size 1 μm . Acquired with the MFP-3D AFM. Adapted from Ref. 13.

Asylum AFMs for Electrical, Electromechanical, and Magnetic Functionality

- The **ORCA** module provides superb capabilities for CAFM on Cypher and MFP-3D family AFMs. It features an exclusive low-noise amplifier for sensitive measurements over a wide current range (~1 pA-20 nA). The Dual Gain ORCA option enables an even wider current range (~1 pA-10 μA).
- NanoTDDDB is an exclusive Asylum mode to measure time-dependent dielectric breakdown on much smaller length scales (~20 nm) than possible with conventional probe stations. Based on CAFM, NanoTDDDB involves applying a tip-sample voltage until a breakdown event is detected. The voltage can be held constant or ramped up to ± 220 V or ± 150 V (system dependent).
- Asylum provides the only commercial high-voltage PFM mode (up to ± 220 V or ± 150 V depending on system). Resonance-enhanced PFM for high sensitivity is fully integrated into software on all Asylum AFMs with the patented Dual AC™ Resonance Tracking (DART) method or the exclusive Band Excitation option.
- sMIM is a mode for scanning microwave impedance microscopy that is available integrated exclusively with MFP-3D family and Cypher S AFMs. It enables imaging of variations in electrical permittivity and resistivity on insulators, semiconductors, and conductors.
- The **Variable Field Module 3** for all MFP-3D family AFMs except Origin offers further versatility for studies of magnetic field dependence. It creates adjustable fields as high as ± 0.75 T in plane. The unique design based on permanent magnets avoids heating and associated drift.

Learn More

Application Note: [“AFM Tools for Electrical Characterization”](#)

Application Note: [“Piezoresponse Force Microscopy with Asylum Research AFMs”](#)

Webinar: [“Piezoresponse Force Microscopy: From Theory to Advanced Applications”](#)

AFM Characterization of Thin Films: High-Resolution Topography and Functional Properties

Measuring Mechanical Properties

In other applications, mechanical properties such as modulus, hardness, and friction are critical to the function of the film. For example, wear-resistant films improve performance in products from razor blades to hip replacements. Mechanical properties are also important simply to achieve overall product durability and reliability. For instance, low modulus and fracture toughness in low-k dielectric films can lead to fracture and delamination during integration with copper interconnects.

With its high spatial resolution and force sensitivity, the AFM has powerful capabilities for measuring nanoscale mechanical properties. AFM-based techniques can apply much lower forces compared to conventional nanoindentation, resulting in smaller indentation depths. This allows much thinner films to be probed without artifacts due to the substrate. In addition, the AFM's high lateral spatial resolution means that heterogeneous and patterned films can be evaluated directly.

Force curves are a well-known method for measuring elastic modulus.¹⁴ However, many film materials are relatively stiff (modulus >10 GPa), and so it can be difficult or impossible to generate high enough forces with standard cantilevers to sufficiently indent them. Conventional force curve mapping is also very slow, though the recent introduction of Fast Force Mapping allows much faster operation. Two recently developed AFM modes, AM-FM Viscoelastic Mapping Mode and Contact Resonance Viscoelastic Mapping Mode, offer both higher speed and quantitative nanomechanical measurements on higher-modulus materials (see sidebar). These modes can be used for both fast, qualitative contrast imaging as well as more sophisticated quantitative mapping (Figure 10).

Characterizing nanoscale tribology¹⁵ is paramount for many film applications. Force curves can be used to measure surface adhesion, while techniques such as lateral force microscopy (LFM) yield information on frictional forces. LFM involves moving the tip sideways while in contact with the sample and measuring the cantilever deflection in the horizontal photodiode channel. LFM can be used to image spatial variations in the lateral force or to acquire friction loops of the lateral force versus sliding distance (Figure 11). Calibrated measurements at different applied loads can be used to determine the coefficient of friction.

Get Results with Asylum AFMs

This note has only “scratched the surface,” but it highlights the power of AFMs for characterizing thin films and coatings. AFMs can quantify 3D surface roughness with unparalleled spatial resolution and map nanoscale functionality in remarkably many ways. Asylum AFMs have both impressive technical capabilities, such as high spatial resolution and sensitivity, and features for greater productivity and ease of use, like fast scanning and automated routines. To discover how AFMs can enrich your work, contact Asylum Research.

Asylum AFMs for Nanomechanical Properties

Asylum offers a wide range of techniques for nanomechanical measurements, so you can choose the most suitable one for a given application.

- The MFP-3D NanoIndenter™ option enables hardness and true ISO-compliant elastic modulus measurements on films thicker than a few hundred nanometers.
- Fast Force Mapping Mode on the MFP-3D Infinity and Cypher AFMs removes the frustration from force curve mapping by acquiring complete images in minutes, not hours (<10 min for 256x256 pixels).
- Asylum's exclusive AM-FM Viscoelastic Mapping Mode enables high-speed mapping of elastic modulus E' and viscoelastic loss tangent using familiar tapping-mode principles. Best for $100 \text{ kPa} < E' < 100+ \text{ GPa}$.
- Contact Resonance Viscoelastic Mapping Mode provides additional capabilities for quantitative mapping of elastic storage modulus and viscous loss modulus on all Asylum AFMs. Best for $1 \text{ GPa} < E' < 100+ \text{ GPa}$.

Learn More

Data sheet: [“The NanomechPro Toolkit: Nanomechanical Techniques for Diverse Materials”](#)

Webinar: [“Introduction and Innovations in High Speed Quantitative Nanomechanical Imaging”](#)

Webinar: [“Contact Resonance Tools for AFM Nanomechanics”](#)

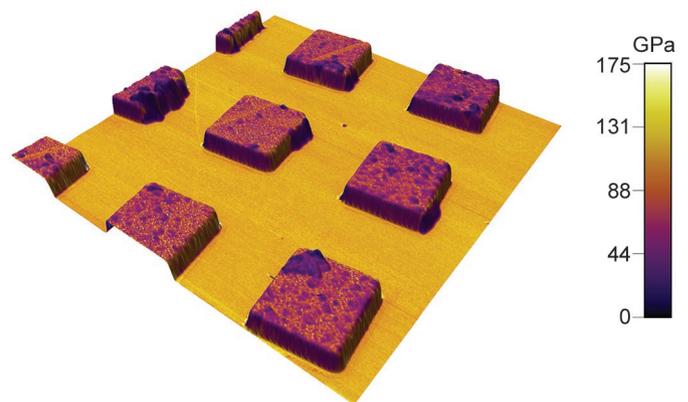


Figure 10: Elastic modulus image of a patterned titanium film on silicon, overlaid on topography. blueDrive photothermal excitation on the Cypher AFM was used for DART contact resonance imaging. Note the strong contrast between these two very high modulus materials. Scan size 25 μm .

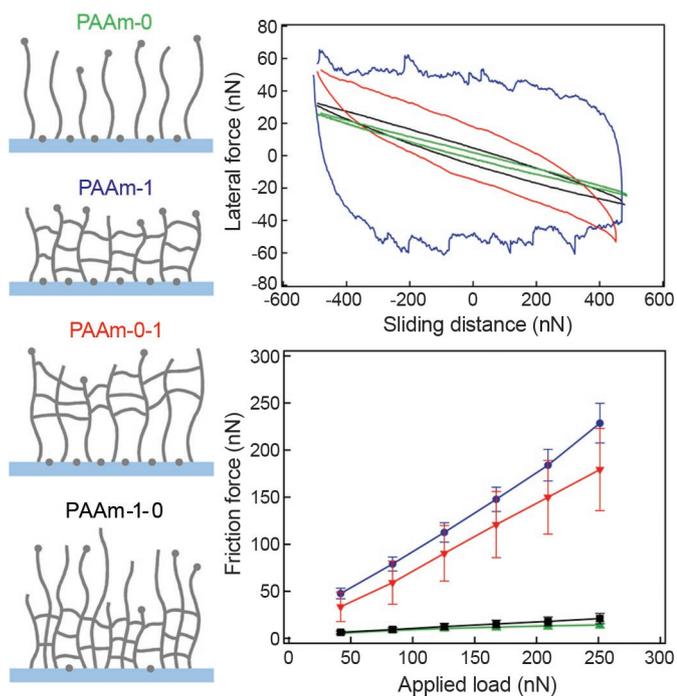


Figure 11: Nanotribology of layered polymer brush-gel films. Mechanically-graded layers with both high modulus and toughness occur frequently in nature, but synthesis of an all-polymer counterpart remains challenging. As shown in the simplified schematics, two-layer films of polyacrylamide (PAAm) on silicon were created with either a brush supporting a crosslinked brush-hydrogel ("gel") or a gel supporting a brush. LFM experiments were performed in water with different film configurations: pure brush (PAAm-0), pure gel (PAAm-1), brush-gel (PAAm-0-1), and gel-brush (PAAm-1-0). The top graph shows friction loops of lateral force versus sliding distance and the bottom, friction force versus applied load. (Curves are color-coded to film type.) The films displayed different tribological behavior, but the response was always determined primarily by the structure of the film's outer layer where the sliding occurred. Acquired on the MFP-3D AFM with the Closed Fluid Cell. Adapted from Ref. 16.

References

1. M. Ohring, *Materials Science of Thin Films: Deposition and Structure*, 2nd ed. (Academic Press, San Diego, CA, 2002).
2. T. Mehmood, A. Kaynak, X.J. Dai, A. Kouzani, K. Magniez, D.R. de Celis, C.J. Hurren, and J. du Plessis, *Mater. Chem. Phys.* **143**, 668 (2014).
3. A. Duk, M. Schmidbauer, and J. Schwarzkopf, *Appl. Phys. Lett.* **102**, 091903 (2013).
4. B. Bhushan, "Surface Roughness Analysis and Measurement Techniques," in *Modern Tribology Handbook Vol. 1* (CRC Press, Boca Raton, USA, 2001), Chap. 2.
5. M. Gu, S.A. Wolf, and J. Lu, *Adv. Mater. Interfaces* **1**, 1300126 (2014).
6. A.R. Damodaran, S. Lee, K. Jambunathan, S. Maclaren, and L.W. Martin, *Phys. Rev. B* **85**, 024113 (2012).
7. E.S. Gadelmawla, M.M. Koura, T.M.A. Maksoud, I.M. Elewa, and H.H. Soliman, *J. Mater. Process. Technol.* **123**, 133 (2002).
8. J.S. Keist, C.A. Orme, P.K. Wright, and J.W. Evans, *Electrochim. Acta* **152**, 116 (2015).
9. R. Berger, H.-J. Butt, M.B. Retschke, and S.A.L. Weber, *Macromol. Rapid Commun.* **30**, 1167 (2009).
10. K. Lai, W. Kundhikanjana, M.A. Kelly, and Z.X. Shen, *Appl. Nanosci.* **1**, 13 (2011).
11. S.V. Kalinin, B.J. Rodriguez, S. Jesse, E. Karapetian, B. Mirman, E.A. Eliseev, and A.N. Morozovska, *Annu. Rev. Mater. Res.* **37**, 189 (2007).
12. U. Hartmann, *Annu. Rev. Mater. Res.* **29**, 53 (1999).
13. I.A. Golovchanskiy, A.V. Pan, S.A. Fedoseev, and M. Higgins, *Appl. Surf. Sci.* **311**, 549 (2014).
14. H.J. Butt, B. Cappella, and M. Kappl, *Surf. Sci. Rep.* **59**, 1 (2005).
15. R.W. Carpick and M. Salmeron, *Chem. Rev.* **97**, 1163 (1997).
16. A. Li, S.N. Ramakrishna, P.C. Nalam, E.M. Benetti, and N.D. Spencer, *Adv. Mater. Interfaces* **1**, 1300007 (2014).

Visit www.oxford-instruments.com/thin-films to learn more

The foregoing application note is copyrighted by Oxford Instruments Asylum Research, Inc. Oxford Instruments Asylum Research, Inc. does not intend the application note or any part thereof to form part of any order or contract or regarded as a representation relating to the products or service concerned, but it may, with acknowledgement to Oxford Instruments Asylum Research, Inc., be used, applied or reproduced for any purpose. Oxford Instruments Asylum Research, Inc. reserves the right to alter, without notice the specification, design or conditions of supply of any product or service. Application Note 31 – 8/2015.

6310 Hollister Avenue
Santa Barbara, CA 93117
Voice +1 (805) 696-6466
Toll free +1 (888) 472-2795
Fax +1 (805) 696-6444

www.AsylumResearch.com
info@AsylumResearch.com
sales@AsylumResearch.com



The Business of Science®