# **Asylum Research**

# Introduction

The world of two-dimensional (2D) materials has grown dramatically since graphene was first isolated in 2004.<sup>1</sup> In little more than a decade, the library of single- and fewlayer materials has expanded to over a dozen elemental and compound crystals. Synthesis techniques have also blossomed beyond the original "sticky tape" mechanical exfoliation to include chemical exfoliation and chemical vapor deposition (CVD, Figures 1 and 2), among many others. Yet this growth shows no sign of stopping. With predictions of up to 500 more 2D systems<sup>2</sup> and a shift in focus towards synthesis in commercial-scale quantities, new challenges and opportunities continue to flourish.<sup>3</sup>

This boom is fueled not just by fundamental interest in a completely new class of materials, but also by the massive potential of 2D materials for next-generation technology. Reduced dimensionality creates fascinating electrical, optical, mechanical, and related properties that could be exploited in myriad ways.<sup>3</sup> Although electronic and optoelectronic applications are prime targets, other possibilities abound everywhere from chemical sensing to water purification and tissue engineering.

Ever since the atomic force microscope (AFM) was used to confirm the first isolation of graphene,<sup>1</sup> it has played an essential role in 2D materials research. But just as the world of 2D materials has grown, so has the power of AFMs. Today's AFMs can image crystal lattice structure as well as nanoscale morphology and sense local electrical, mechanical, and functional response in more ways than ever before. Here, we describe these capabilities and discuss how AFMs contribute to continued progress in 2D materials research.



**Figure 1: CVD growth of molybdenum disulfide (MOS<sub>2</sub>) on graphene** – Three-dimensional topography image of MoS<sub>2</sub> (triangles) grown by CVD on epitaxial graphene. The terraces are attributed to miscut of the silicon carbide wafer substrate. Subsequent growth precipitates new nuclei on previously grown triangles, forming multilayered MoS<sub>2</sub> pyramids. Scan size 2 µm. Imaged on the Cypher AFM in tapping mode. Image courtesy of I. Balla, S. Kim and M. Hersam, Northwestern University.

# **Evaluating Nanoscale Morphology**

For both brand-new 2D materials and existing ones made in new ways, understanding basic morphology—shape and size—is essential. Simply detecting the material's presence may be sufficient in some cases, although not trivial. In other cases, measuring the dispersion of flakes, flake or device dimensions, or sample roughness and uniformity may be key. Data on layer thickness and the number of layers are also frequently needed, in part because these quantities can influence many sample properties.

With spatial resolution well below a nanometer, AFMs excel at tasks like these and have distinct advantages over optical tools. Any substrate may be used, unlike white light interferometry. A wider range of sample thickness and quality can be accommodated than with Raman techniques. AFMs also visualize the distribution in features on sub-µm length scales instead of giving a single, spatially-averaged value.

Information on morphology is obtained by imaging topography (height), typically in contact or tapping mode (Figures 1 and 2). Both methods produce accurate results when performed carefully.<sup>5</sup> For example, in tapping mode it is important to ensure the cantilever does not switch between the attractive and repulsive regimes.



**Figure 2: Controlling MoS**<sub>2</sub> **crystalline orientation in CVD processes** – "Bottom-up" strategies such as CVD have potential for commercial-scale synthesis of MoS<sub>2</sub> but need further refinement to enable growth with controlled crystalline orientation. (right) Topography images of a MoS<sub>2</sub> domain grown by CVD on annealed sapphire. (left) The height profile (blue line) reveals a domain thickness of <1 nm. Individual terraces in the atomically flat sapphire can also be seen with step height ~0.22 nm, which is sufficiently low to allow continuous, single-crystal MoS<sub>2</sub> growth. Imaged on the Cypher AFM in tapping mode. Adapted from Ref. 4.



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During topographic imaging or other AFM experiments, environmental control can be desirable or even critical. For instance, humidity control may improve day-to-day reproducibility of high-resolution thickness measurements. A more dramatic example is preventing irreversible changes from oxidation reactions with use of an inert gas environment. Specialized measurement cells are available to meet these and related needs. If even more stringent environmental control is required, the entire AFM can be enclosed in a glovebox.

Other applications benefit from AFM experiments performed in a liquid environment. For instance, many "top-down" routes to facile synthesis of 2D materials involve liquid-phase reactions. Here, evaluating morphology *in situ* yields much richer information than simple post-process imaging in air. In the example in Figure 3, experiments were performed to monitor the spontaneous exfoliation of graphene in an ionic liquid. Sophisticated fully-sealed fluid cells are available to surround the entire cantilever and sample surface in a liquid solvent. Capabilities for operating under either static or perfusion conditions make these cells exceptionally versatile.



**Figure 3: Monitoring spontaneous graphene exfoliation in ionic liquids (ILs)** – Exfoliation methods show promise for large-scale production but are prone to aggregation. The tapping mode phase images were acquired on freshly-cleaved highly oriented pyrolytic graphite (HOPG) immersed in the IL 1-ethyl-3-methylimidazolium acetate (EMIm Ac) at 25°C. Distinct surface changes are observed as immersion time increases: HOPG's characteristic step edges gradually erode and are replaced by amorphous domains (see arrows). Analogous experiments were performed at other temperatures and in a second IL, 1-ethyl-3methylimidazolium trifluoromethansulfonate (EMIm TFMS). The graph shows that the time to exfoliation decreases sharply with increasing temperature. This Arrhenius-like behavior supports the hypothesis of spontaneous exfoliation via ionic intercalation between sheets. Imaged with the Cypher ES AFM. Adapted from Ref. 6.

#### **Evaluating Morphology with Asylum AFMs**

- SpotOn<sup>™</sup> automated laser alignment reduces setup time on Cypher<sup>™</sup> AFMs. Just click the mouse on the desired position, and the fully-motorized stages align the laser spot on the cantilever and center the photodetector.
- Setup for tapping mode is both faster and simpler on MFP-3D Infinity<sup>™</sup> and Cypher AFMs with GetStarted<sup>™</sup>. The predictive algorithm automatically optimizes imaging parameters before the tip ever touches the sample. Neither the tip nor sample is damaged by non-optimal settings, and high-quality data is obtained from the first scan line.
- GetReal<sup>™</sup> software simplifies accurate measurements of absolute force and stiffness on all Asylum AFMs. It automatically calibrates the cantilever spring constant and deflection sensitivity with a single click without touching the sample.
- Easily find single-layer flakes and other areas of interest with the high-resolution optics of Cypher family AFMs.
  Diffraction-limited optics with Köhler illumination provide sub-micrometer resolution for digital zooms and pans.

#### **Environmental Control with Asylum AFMs**

- For precise environmental control, the Cypher ES AFM contains a sealed cell with broad chemical compatibility for static or perfusion operation and relative humidity control. The Heater (ambient to 250°C) and CoolerHeater (0°C -120°C) stages give precise temperature control without extra electronics or cooling pumps.
- Environmental control options for MFP-3D family AFMs include the Closed Fluid Cell for static or perfusion operation in gases and liquids, the BioHeater for heating (ambient to 80°C), the Humidity Sensing Cell, and the MicroFlow Cell. For all MFP-3D AFMs except Origin, the PolyHeater (ambient to 300°C), PolyHeater+ (ambient to 400°C), and CoolerHeater (-30°C to +120°C) can be used for temperature control.
- Tapping mode in liquid is simpler, more stable, and more quantitative with blueDrive<sup>™</sup> Photothermal Excitation on Cypher family AFMs. Cantilever actuation with a blue laser produces exceptionally clean and stable signals.
- Turnkey glovebox solutions provide full environmental isolation for MFP-3D and Cypher AFMs, preventing irreversible surface chemical reactions with ambient oxygen or water vapor for more reliable measurements.

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- "AFM Imaging and Nanomechanics with blueDrive Photothermal Excitation"

However, tapping mode imaging in liquid has traditionally been challenged by the "forest of peaks," that is, spurious signals created by piezoacoustic excitation. This phenomenon, which can make tapping in liquid difficult and less stable, has been eliminated by new photothermal excitation techniques. Driving the cantilever oscillation via laser light results in a near-ideal response on any cantilever, in any environment.

Temperature is another important experimental variable. For instance, it enables fine-tuning of liquid-phase processes and allows studies of long-term reliability under realistic conditions. Temperature control in AFM experiments is usually achieved using specialized sample stages. Current AFMs feature sample stages that provide highly stable, precise temperature control as high as 400°C.

# **Imaging Lattice Structure**

In many cases, characterizing the crystalline nature of 2D materials is fundamental. Knowing lattice structure verifies that the desired crystal was produced and helps evaluate the synthesis process. Sometimes the relative crystalline orientation of different layers may affect overall device performance.

Measurements of lattice structure have traditionally been considered the domain of high-resolution transmission electron microscopy (HRTEM) or low-temperature, ultra-high-vacuum scanning tunneling microscopy (LT-UHV STM). Both techniques provide sufficient resolution to visualize the crystal structure and evaluate characteristics such as symmetry, quality, and orientation. Measurements of lattice constants can be obtained from either an image directly or its spatial Fourier transform.

However, imaging the atomic lattice of 2D crystals is also possible on some of today's commercial AFMs such as the Cypher. Instrumentation improvements mean spatial resolution comparable to—or even better than—HRTEM's current limit<sup>7</sup> of ~50 pm can now be achieved routinely. Regions of 2D materials with local atomic flatness present optimal conditions for highresolution AFM imaging, since they allow fewer atoms (or even a single atom) in the tip to interact with the surface.

AFM techniques also have several practical advantages over HRTEM and LT-UHV STM for lattice imaging. AFMs normally operate under ambient conditions, without vacuum or cryogenic equipment. Samples and substrates may be conducting, insulating, or semiconducting. Sample preparation is usually minimal, significantly shortening the time before the first image is acquired. AFM images represent the surface topography and are thus more directly interpreted than images of phase interference (HRTEM) or tunneling current (LT-UHV STM).

These concepts are demonstrated in Figures 4 and 5, which show high-resolution AFM images of 2D materials acquired in ambient conditions. The figures also show that imaging can be performed in either tapping mode (Figure 4) or contact mode (Figure 5). In both tapping mode and contact mode, the height (Z-sensor) signal can be used for imaging; in tapping mode, the amplitude signal can also be used.



Figure 4: Measuring lattice constants of dioctylbenzothienobenzothiophene ( $C_g$ -BTBT) molecular crystals – Solution processing of 2D single-crystal organic semiconductors could enable highthroughput manufacturing of novel optoelectronic devices. In this work, single- and few-layer sheets of  $C_g$ -BTBT were formed by floating-coffeering-driven assembly on a silicon/silicon oxide substrate. The image of a  $C_g$ -BTBT bilayer (left) was acquired with the amplitude signal in tapping mode. The 2D Fourier transform (right) reveals peaks corresponding to the crystal lattice. Vectors indicate the oblique unit cell with lattice constants a and b with angle  $\theta$ . The histograms of a, b, and  $\theta$  were obtained from analysis of 10 images acquired at different places on a bilayer region with total area >5×10<sup>4</sup> µm<sup>2</sup>. The narrow distributions indicate the bilayer is composed of a single-crystal phase. Imaged with the Cypher AFM in air. Adapted from Ref. 8.

#### **Imaging Atomic Lattices with Asylum AFMs**

- 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 with position sensors to eliminate image distortions for high-precision offsets and zooms.
- Even when imaging atomically flat samples, Cypher AFMs do not need add-on vibration isolation in most labs. An integrated enclosure and super-stable mechanical design makes them inherently immune to normal environmental vibration. Thermal drift is also lower by 10× than in less advanced AFMs.
- Small cantilevers (<10 µm long) and small laser spot sizes (3×9 µm<sup>2</sup>) make line scan rates up to 40 Hz routinely achievable on Cypher AFMs. This means it takes only a few seconds to acquire complete 256×256 pixel images.

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The impressive spatial resolution in these figures has been achieved by careful instrument design. In the lateral direction, resolution depends on XY scanner performance and tip sharpness. Closed-loop control provides more accurate XY scanning than the open-loop operation of older AFMs. Use of symmetric design, more thermally stable materials, and smaller enclosures reduces additional scanning distortion due to thermal drift. Because newer AFMs are more stable, transient noise that might break sharp tips is also minimized.

Vertical resolution depends on the noise floor, below which real features cannot be distinguished from random mechanical and electrical fluctuations. The noise floor can be lowered by making the mechanical loop between the tip and sample as short and stiff as possible. Combined with improved acoustic isolation, this minimizes disruptions from environmental vibrations.

An additional factor in attaining higher spatial resolution is use of smaller cantilevers. With lengths ten times smaller than their conventional counterparts, smaller cantilevers have intrinsically lower thermal noise for the same spring constant and much higher resonant frequencies. To accommodate small cantilevers and enable faster scanning, AFMs must have smaller laser spot sizes, faster photodiodes and control electronics, and higher instrument resonances than older AFMs.

Images resolving the atomic lattice can also be acquired by measuring the cantilever's lateral deflection in contact mode. This approach is called lateral force microscopy (LFM), or friction force microscopy if the difference between trace and retrace signals is used. Image contrast in both cases arises from periodic slip-stick of atomic-scale frictional forces. (The actual tipsample contact area is much larger, typically ~100 nm<sup>2</sup>.) These techniques are particularly useful for samples fabricated on rougher substrates, because the contrast is largely independent of topography. Figure 6 shows an example of LFM imaging of the graphene lattice.

# **Probing Electrical and Functional Response**

Ranging from insulating, to semiconducting, to highly conductive, the electrical properties of 2D materials could enable many types of disruptive technology.<sup>11</sup> They may hold the key to extremely small, fast transistors with superior performance at minimal power; other possibilities include new flexible displays, photovoltaic devices, and light-emitting diodes (LEDs). However, realization of such next-generation devices requires knowledge of 2D materials beyond simply topography. Information on relevant physical behavior, especially electrical and related functional response, is paramount.

Several AFM modes interrogate electrical and optoelectronic behavior with nanoscale spatial resolution.<sup>12</sup> For instance, nanoscale current mapping is performed with conductive AFM (CAFM), providing complementary information to macroscale methods like four-point probes that test a whole device.



**Figure 5: Engineering strain in epitaxial graphene** – Understanding the strain created during graphene deposition will facilitate control of its electronic properties. (a) Topography image of graphene grown on hexagonal boron nitride (hBN) by high-temperature molecular beam epitaxy (MBE). Two regions of graphene, separated by a central crack, display hexagonal moiré patterns that arise from a lattice mismatch between the graphene and the hBN substrate. Defects and variable periodicity in the moiré patterns represent strain-induced spatial variations in lattice constant. (b)-(d) Topography images acquired within the square regions indicated in (a). The scan size of each image is 5 nm [see scale bar in (b)]. The vectors indicate that the graphene regions in (b) and (d) have the same lattice orientation and are aligned with the hBN substrate in (c), confirming epitaxial growth. Imaged in air with contact mode on the Cypher AFM. Adapted from Ref. 9.



**Figure 6: Mapping graphene grain orientation with fast, high-resolution scanning** – Low-pressure CVD growth of graphene on copper foil is an attractive option for large-area synthesis. This image of CVD graphene on copper was acquired with the lateral deflection signal in contact mode (LFM) and is part of a larger image (scan size 20 nm). The 2D Fourier transform of the larger image displays the hexagonal pattern of peaks indicative of crystalline graphene (dashed lines) and enables the local lattice orientation to be determined. By collecting more than 1000 such images unattended over 7 h (<30 s per 512×512 pixel image), the grain orientation over a 25×25 µm<sup>2</sup> region was mapped. Imaged on the Cypher AFM in air. Adapted from Ref. 10.

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Moreover, CAFM current images are acquired simultaneously with topography images. As seen in Figure 7, this facilitates correlations between local structure and characteristics such as charge distribution and transport. CAFM can also be used to obtain I-V curves at user-defined device locations for deeper analysis. When performed on optically responsive systems with an illumination source, CAFM is called photoconductive AFM.

Because CAFM scans in contact mode, lateral forces arise that could potentially damage the tip or sample. The resulting wear may affect the measured current and complicate image interpretation. To avoid such issues, fast current mapping techniques have been recently developed. Here current is measured while acquiring a high-speed array of force curves instead of scanning in contact. Fast current mapping is based on a fast-force-curve approach, in which the cantilever is moved vertically in a continuous sinusoidal motion while it is also scanned laterally. As in CAFM, fast current mapping methods yield parallel topography and current data to elucidate local structure-property relations. They also offer a wealth of data analysis options, as long as complete curves of current and deflection versus time are stored.

Electrostatic force microscopy (EFM) and Kelvin probe force microscopy (KPFM) are other modes to evaluate local electrical behavior on both sheet materials and operating devices, either with or without illumination. EFM senses electric field variations due to long-range electrostatic force gradients and yields useful images of qualitative contrast with minimal setup. In comparison, KPFM quantitatively measures the contact potential difference between the tip and sample. This supplies valuable contrast even when topography cannot: for instance, to detect single layers on rough substrates, differentiate single- and multiple-layer regions, or distinguish crystalline grain boundaries and orientation.

#### **Electrical and Functional Response with Asylum AFMs**

- Use the ORCA module for CAFM experiments on Cypher S and MFP-3D AFMs. Its cantilever holder features a sensitive, low-noise transimpedance amplifier operable over a wide current range (~1 pA to 20 nA) and is available in a variety of gain options. The Dual Gain ORCA option contains two separate amplifiers for an even wider current range. The outputs from both amplifiers can be measured simultaneously for high sensitivity and high resolution from ~1 pA to 10 µA.
- Improve photocurrent measurements on all Asylum AFMs with the dual-pass approach of Eclipse<sup>™</sup> mode. Topography is measured in contact mode in the first scan pass. In the second pass, the laser is turned off and CAFM measurements are performed.
- Acquire current and force curve arrays simultaneously with Fast Current Mapping Mode on Cypher and MFP-3D Infinity AFMs. With pixel rates up to 300 Hz on Infinity AFMs and 1 kHz on Cypher AFMs, a 256×256 pixel array can take less than 10 min to acquire. Use of ORCA and Dual Gain ORCA amplifiers ensure high measurement sensitivity over a wide current range.
- Asylum Research offers the only commercial high-voltage PFM mode (up to ±220 V for MFP-3D Origin AFMs; up to ±150 V for Cypher and MFP-3D Infinity AFMs). Highsensitivity measurements with resonance-enhanced PFM are integrated into software on using Dual AC Resonance Tracking (DART) mode or the Band Excitation option.

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	2D Materials with the AFM"
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Figure 7: Creating a photoswitchable diode with MoS, - Optically controlling the electronic response of 2D materials will facilitate development of next-generation photodetectors and LEDs. Here, MoS, flakes were exfoliated on mixed-self-assembled monolayers (mSAMs) of azobenzene chemisorbed on gold (Au). As a photochromic material, azobenzene molecules switch from a stable trans-configuration to a metastable cis-configuration when exposed to UV light. The CAFM current-voltage curves were acquired before illumination (trans 1, blue, after exposure to UV light (cis, red), and after exposure to white light (trans 2, green). Current rectification occurs for the trans- but not the cis-mSAM. Rectification is further demonstrated in the image of CAFM current for one, two, and three layers of MoS<sub>2</sub> (1L, 2L, and 3L, respectively) on a trans-mSAM. With a bias voltage (0.1 V) below the heterostructure turn-on voltage (0.5 V), the image and its histogram indicate lower conductance in the MoS, flakes than the bare mSAM. Scan size 1 µm; acquired on the MFP-3D AFM with the ORCA module. Adapted from Ref. 13.

#### AFM Characterization of 2D Materials: New Directions, Future Applications

Another use of KPFM is to examine band bending in semiconductors, as described in Figure 8. Furthermore, proper calibration of KPFM experiments allows quantitative measurements of the work function in conductors and semiconductors.

Characterization of electrical response at microwave frequencies is possible with a near-field technique called scanning microwave impedance microscopy (sMIM). sMIM detects local changes in sample conductivity and permittivity using a microwave source coupled to a shielded probe. Applications include sensing buried conducting layers, analyzing electric defects, and measuring type and concentration of semiconductor dopants with high sensitivity.

Other types of functional response in 2D materials could also lead to technology breakthroughs. For instance, ferroelectric and piezoelectric behavior could bring innovations in memory and logic devices, sensors and actuators, and other products. Piezoresponse force microscopy (PFM) is a powerful tool for characterizing piezoelectric, ferroelectric, and multiferroic materials on the nanoscale. It provides information on both static and dynamic electromechanical properties including domain structure, growth, and polarization reversal. Figure 9 shows an example of PFM imaging on ultrathin CuInP<sub>2</sub>S<sub>6</sub> (CIPS).

Ferromagnetic response in 2D materials could also be exploited for technology advances such as higher storage capacity, faster data transfer, and new spintronic devices. Magnetic force microscopy (MFM) uses the interaction forces between a magnetic sample and a magnetized tip to sensitively probe ferromagnetic and multiferroic materials on the nanoscale. With MFM, magnetic features such as vortices, and domain patterns and walls can be imaged.

# **Measuring Mechanical Properties**

Although most proposed uses of 2D materials exploit their electrical and functional response, their mechanical properties can also have a profound impact. For example, the high modulus of MoS<sub>2</sub> facilitates strain engineering of nanoelectronic heterostructures, while the high tensile strength of graphene affects its performance in flexible displays. More experimental data on mechanical properties are currently needed to both confirm theoretical predictions and hasten development of commercial products.

To assist in this need, a range of AFM techniques have been developed to provide unique information on nanoscale mechanical properties. A familiar example is the force curve method. This well-established approach for measuring elastic modulus was used to obtain the first result on graphene of 1 TPa.<sup>16</sup> However, these measurements required a specialized cantilever with extremely high spring constant and diamond tip to ensure sufficient sensitivity on such a stiff material. Conventional force curve mapping is also very slow, although recently introduced fast force curve techniques allow much faster operation.



**Figure 8: Tuning electrical properties of tungsten diselenide (WSe**<sub>2</sub>) – The ability to tailor band gaps in transition metal dichalcogenides (TMDs) would enhance the performance of 2D semiconductor devices. In this work, a transistor was created with WSe<sub>2</sub> as the channel, and defects were selectively induced by irradiation with a focused helium-ion (He<sup>+</sup>) beam. The images show topography (gray) and KPFM surface potential (color) after half the channel was exposed to a dose of  $5 \times 10^{14}$  He<sup>+</sup>/cm<sup>2</sup>. The surface potential profile across the dotted line in the KPFM image shows a sharp interface. The results indicate band bending due to Se vacancies, which act as electron donors in the area exposed to He<sup>+</sup>. Imaged on the Cypher AFM. Adapted from Ref. 14.



**Figure 9: Evaluating room-temperature ferroelectricity in CuInP<sub>2</sub>S<sub>6</sub> (CIPS)** – Ferroelectricity in 2D materials could be exploited for nonvolatile memory and other devices but remains relatively unexplored. Here, CIPS flakes were mechanically exfoliated onto heavily-doped silicon containing an oxide layer and a gold topcoat. The images of topography (gray) and PFM amplitude (color) contain flakes with two, three, and four layers (2L, 3L, and 4L, respectively). The corresponding sections across the dashed lines reveal ferroelectric behavior in flakes only a few nanometers thick, although the magnitude of response decreases with decreasing thickness. Imaged on the MFP-3D AFM. Adapted from Ref. 15.

To increase imaging speed and expedite measurements on higher-modulus materials, other AFM nanomechanical techniques have been developed. Two of these are AM-FM mode and contact resonance AFM (CR-AFM). Both modes enable fast, qualitative contrast imaging as well as quantitative modulus mapping, even on very stiff materials (to 100+ GPa). Additional information on damping and viscoelastic response can be obtained by AFM loss tangent imaging, which is performed either in standard tapping mode or as part of AM-FM mode. Figure 10 shows AFM loss tangent results to evaluate surface water layers on graphene. In Figure 11, CR-AFM was used to detect subsurface structural variations in oxygenintercalated graphene.

Tribological properties of 2D materials also influence performance in many cases. Nanoscale surface adhesion, which gives insight into wetting and reactivity effects in chemical sensors and other devices, is measured with force curves. Frictional behavior, which is critical in applications such as solid lubricant layers for nano- and micromechanical systems (NEMS and MEMS), is often characterized with LFM (see above). These techniques can image spatial variations in lateral forces or obtain friction loops of lateral force versus sliding distance. Calibrated measurements at different applied loads enable the coefficient of friction to be determined.



**Figure 10:** Investigating interfacial phenomena in graphene – elucidating the effects of water vapor layers could improve the accuracy of thickness measurements on 2D materials. These tapping mode images of AFM loss tangent tan  $\delta$  acquired at different relative humidity (RH) values indicate a water adlayer (arrows) between the few-layer graphene flake and a silicon dioxide (SiO<sub>2</sub>) film on silicon. The graph reveals that tan  $\delta$  is higher on graphene than SiO<sub>2</sub> at low RH but decreases for both materials with increasing RH, until it reaches similar values for both materials at ~70% RH. The results support a model of RH-dependent water adsorption developed from thickness measurements (not shown). Imaged with the MFP-3D AFM and the Humidity Sensing Cell. Adapted from Ref. 17.

#### Nanomechanical Measurements with Asylum AFMs

- Asylum Research offers a wide range of nanomechanical techniques, so that the most suitable one for a given application can be selected or results from different methods compared.
- The NanoRack Stretch Stage on MFP-3D AFMs enables measurements on samples under compressive or tensile strain.
- AM-FM Viscoelastic Mapping Mode is exclusive on all Asylum AFMs. It performs quantitative nanomechanical mapping faster than any other technique, when small cantilevers are used.
- Contact Resonance Viscoelastic Mapping Mode is integrated into software on all Asylum AFMs. It uses either Dual AC Resonance Tracking (DART) or the Band Excitation option.

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"Introduction and Innovations in High Speed Quantitative Nanomechanical Imaging"



**Figure 11:** Mapping subsurface variations in graphene – Understanding and controlling the interface between a 2D material and substrate is imperative for successful device engineering. In this work, the power of contact resonance AFM (CR-AFM) to sense subsurface structural and compositional variations was demonstrated. Epitaxial graphene on silicon was annealed in air to form deliberately modified subsurface regions via oxygen intercalation. (a) The topography image shows only graphene terrace steps, but (b) the CR-AFM frequency image contains numerous other features. (c) Topography and (d) CR-AFM frequency images corresponding to the boxes in (a) and (b). Using a combined density functional theory and continuum modeling approach (not shown), the nanomechanical response in regions 1, 2, and 3 could be attributed to three different interfacial atomic structures. Imaged on the MFP-3D in DART mode. Adapted from Ref. 18.

# **Explore Flatlands with Asylum AFMs**

The world of 2D materials has continued to experience explosive growth. Once limited to small flakes of graphene painstakingly prepared, it is now poised to encompass a plethora of materials, facile synthesis processes, and high-impact applications. The AFM is already an essential tool for characterizing 2D materials, but the enhanced power of today's models ensure it will maintain this role into the future. Improvements such as higher spatial resolution, faster imaging rates, and greater environmental control, as well as advanced capabilities for measuring electrical, functional, and mechanical properties, make AFMs more valuable than ever for 2D materials research. Learn how your work can benefit from AFM techniques by visiting www.oxford-instruments.com/AFM.

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## Oxford Instruments: Diverse Tools for 2D Materials

Oxford Instruments provides many capabilities beyond AFMs for 2D materials research:

- Systems for PE-CVD, MBE, and other processes enable growth and deposition of high-quality materials.
- Characterization solutions include optical spectroscopy and detection, electron spectroscopy and nanoprobing, electron beam diffraction (EDS and EBSD) and x-ray analysis.
- Cryostats, dilution refrigerators, and super-conducting magnet systems offer additional research possibilities.

To learn more, see "Graphene and 2D Materials" at www.oxford-instruments.com/graphene.

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