AFM Tools for Nanoscale Electrical Characterization

Abstract

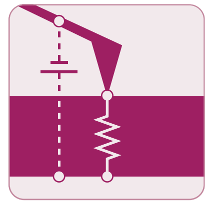
The importance of characterizing electrical functionality on nanometer length scales continues to grow as devices shrink and new nanomaterials emerge. Oxford Instruments Asylum Research offers a variety of AFM tools to evaluate local electrical properties, including current, surface charge and potential, dielectric breakdown, conductivity, and permittivity. Special features and unique capabilities for Cypher™ and MFP-3D™ family AFMs provide more opportunities for meaningful electrical measurements on a wide range of systems.

Introduction

Improving performance, power consumption, cost, and sustainability are key drivers in R&D on new electrical materials and devices. More and more, achieving these goals involves smaller and smaller length scales. Not only do the dimensions of silicon-based devices keep dwindling, but also next-generation processes with nanoscale components like nanotubes, graphene, and molecular building blocks are emerging. As a result, understanding physical processes that control electrical behavior increasingly requires measurements on smaller length scales.

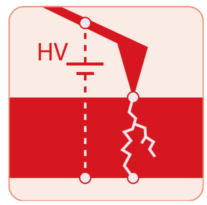
The inherent spatial resolution and high force sensitivity of the atomic force microscope (AFM) make it a powerful tool for nanoscale electrical characterization. Accordingly, a number of AFM techniques[1] have been developed for direct imaging of local electrical properties (Table 1). These techniques are valuable not just for probing failure modes in electric devices, but in many other applications to identify defects, assess uniformity, and otherwise assure quality.

Here we discuss several AFM electrical techniques. Modes like conductive AFM (CAFM), electrostatic force microscopy (EFM), and Kelvin probe force microscopy (KPFM) are well established and widely used. Fast Current Mapping Mode, nanoscale time-dependent dielectric breakdown (nanoTDDB), and scanning microwave impedance microscopy (sMIM) are newer modes with additional capabilities for electrical characterization available on Asylum Research AFMs.



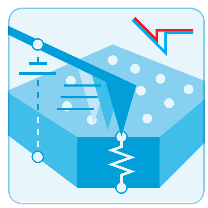
Conductive AFM

CAFM scans in contact mode while measuring any current flowing through the sample into the conductive tip. Additionally, this mode allows localized current vs. voltage (I-V) curves to be measured at specific user-defined points.



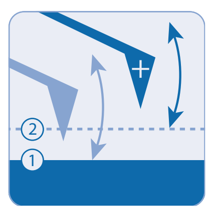
Nanoscale Time-Dependent Dielectric Breakdown

NanoTDDB is a point measurement mode that is used to study dielectric breakdown at the nanoscale. A high-voltage bias is applied to the sample until the breakdown event is detected.



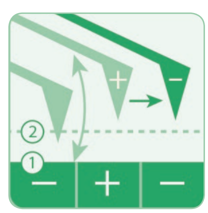
Fast Current Mapping Mode

Fast Current Mapping Mode measures current continuously during an array of force curves taken at high speed. Current maps of the sample can be generated with the benefit of minimal tip wear, since the tip is not scanning in contact with the surface.



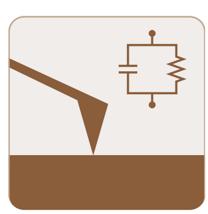
Electrostatic Force Microscopy

EFM is a two-pass imaging mode to qualitatively image the sample's longer-range electrostatic forces. Information on electric field gradients due to variations in trapped charge, potential, conductivity, or permittivity can be obtained.



Kelvin Probe Force Microscopy

KPFM can be performed in either single-pass or dual-pass mode to quantitatively image the sample's electrostatic potential. It is a surface-probing technique that provides information on surface potential and work function.



Scanning Microwave Impedance Microscopy

sMIM is a near-field technique that uses microwaves to measure variations in resistance and capacitance, as well as dC/dV and dR/dV gradients. It can be operated in contact, tapping, or fast force mapping modes.

Table 1. Nanoelectrical characterization tools available on Asylum Research AFMs. The icons graphically depict the measured interaction between the cantilever tip and sample.

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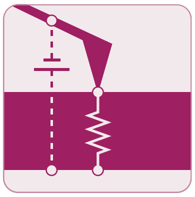


Conductive AFM (CAFM)

CAFM[2,3] measures current to determine local variations in conductivity. Its nanoscale spatial resolution complements macroscale methods such as four-point probes. CAFM measurements are performed simultaneously with topographic imaging. Thus it is especially useful for relating local electrical properties to local structure and morphology, for instance to improve efficiency in organic photovoltaics. Applications include a wide variety of systems, from ferroelectrics and piezoelectrics to semiconductor and data storage devices.[1] Because it directly measures conductivity, CAFM is sensitive to carrier concentration and therefore can be used for semiconductor dopant profiling. It is also used to inspect dielectric films and oxide layers for defects and uniformity.

In CAFM, a conducting AFM tip is placed in contact with the sample, and a bias voltage is applied between the sample and the tip. (Conducting tips can be made by adding metal coatings or by fabricating probes from materials such as solid metal, doped diamond, or doped silicon.) Any current flowing through the sample into the tip is recorded while the tip scans, creating a conductivity or current map. Figure 1 shows CAFM applied to a multiferroic film, and Figure 2 shows its use for evaluating a semiconducting polymer film.

Current measurements with AFM make stringent demands on the instrument. Measurements can range from picoamperes to microamperes depending on the sample, and need high sensitivity and low noise over this entire range—six orders of magnitude—of current. To meet these needs, Asylum's ORCA™ module for CAFM incorporates an amplifier with low noise levels (see sidebar, page 3). However, single-gain amplifiers typically only operate over about four orders of magnitude. Logarithmic amplifiers like those used for scanning spreading resistance AFM can cover a wider current range, but their measurement sensitivity and resolution are limited at lower currents. These obstacles can be overcome by using two separate amplifiers, such as in the Dual Gain ORCA module (see sidebar).

When performed with an illumination source, AFM current measurements are called photoconductive AFM. This technique is useful in optoelectronic and photovoltaic systems to evaluate nanoscale photoresponse such as dark current vs. photocurrent, or to map charge generation and transport. However, artifacts in the photoconductive AFM signal can arise from photocurrents induced by the AFM laser. Eclipse™ mode on Asylum AFMs prevents these artifacts by deactivating the AFM laser during the CAFM scan (see sidebar).

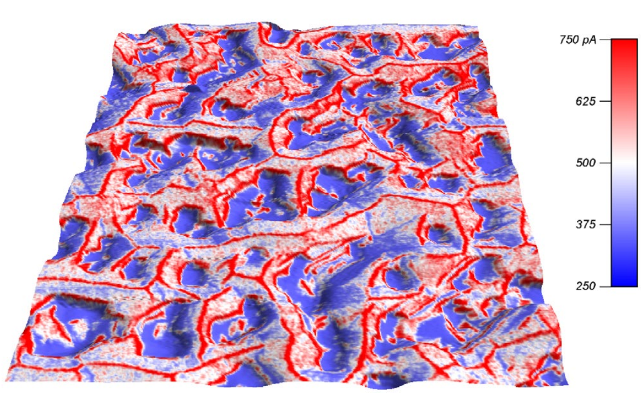


Figure 1: CAFM investigation of a (001) $Bi(Fe_{0.5}Mn_{0.5})O_3$ (BFMO) film on a (001) $SrTiO_3$ with 0.5% Nb substrate. BFMO's multiferroic and spin glass properties make it attractive for many applications. The image shows the tip-sample current overlaid on topography and reveals that the boundaries between crystalline grains (red) typically have much higher conductivity than the crystallite interiors (blue). The red conductivity "lines" occur in all directions on the sample surface, indicating that the contrast is not a topography artifact. Scan size 1 µm; acquired on the MFP-3D AFM.

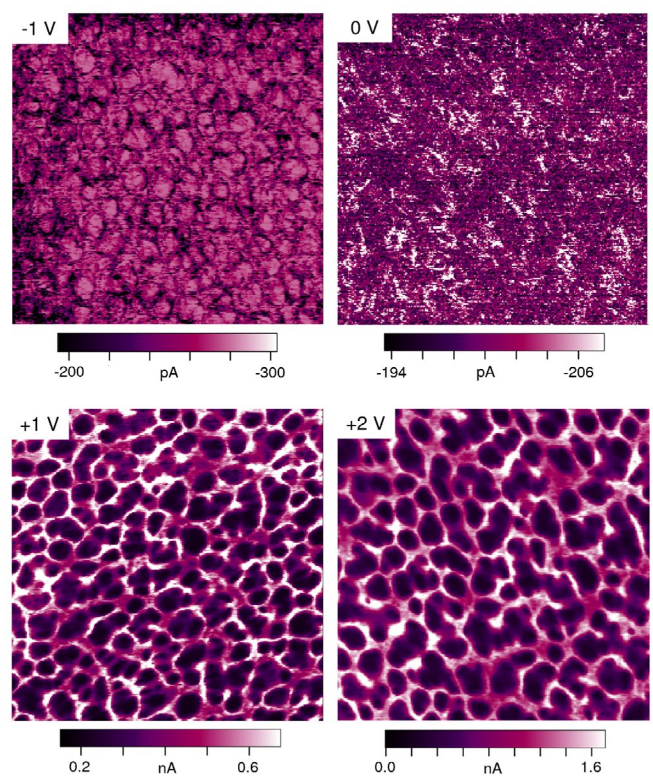


Figure 2: Imaging conduction networks in a semiconducting polymer film with CAFM. Learning how a film's morphology and conduction network depend on the casting solvent is important for optimizing performance in photovoltaic applications. This film was made by blending a low-bandgap, diketopyrrolopyrrole-benzothiadiazole-based copolymer (PDPP-BT) and phenyl-C71-butyric acid methyl ester (PC71BM) with chloroform as the casting solvent. CAFM images at different DC bias voltages show bright regions of positive current in polymer-rich phases and dark regions of PC71BM aggregates. The positive current increases with increasing positive bias, indicating increased hole transport through the blend. Scan size 3 µm; acquired on the MFP-3D AFM under dark conditions. Adapted from Ref. 4.

Asylum Research AFM Features for Current Measurement

The ORCA module enables CAFM on Cypher and MFP-3D AFMs. It consists of a cantilever holder with an exclusive low-noise transimpedance amplifier for sensitive measurements over a wide current range (~1 pA to 20 nA). The Dual Gain ORCA option uses two separate amplifiers for an even wider current range, from ~1 pA to 10 µA.

MFP-3D-BIO AFM For experiments that require a light source, the MFP-3D-BIO AFM simplifies measurements and eliminates system integration hassles. It sets the industry standard for seamless integration of AFM and optical microscopy while maintaining high measurement quality.

On all Asylum AFMs, Eclipse mode improves photocurrent measurements with a dual-pass scanning approach. In the first pass, topography is measured in contact mode. In the second pass, the laser is turned off while CAFM measurements are performed at the same height.

The Probe Station option for MFP-3D family AFMs allows easy electrical probing, biasing, and other measurements during AFM scanning. A variety of electrical connections can be made and combined with various imaging modes.

Localized current-voltage (I-V) curves can also be acquired with CAFM. The tip is placed in contact at a user-defined position, and the bias voltage is ramped while the current is measured. As shown in Figure 3, 2D arrays of I-V curves can be acquired at precise sample positions. These arrays can be analyzed to obtain maps of properties such as charge carrier mobility, or simply averaged for statistical information and failure analysis. Varying the tip-sample DC bias voltage or applied contact load also yields insight on properties such as charge generation and injection, or contact resistance.

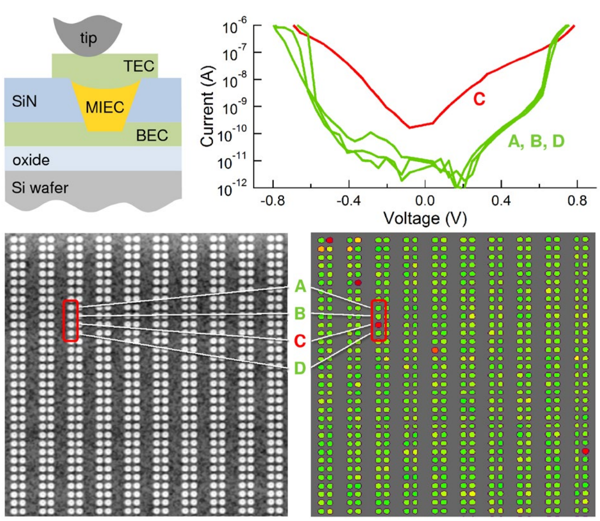
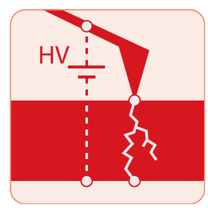


Figure 3: Yield testing of mixed-ionic-electronic-conduction (MIEC)-based access devices with CAFM. MIEC materials are attractive for high-density, non-volatile memory applications. (Top left) "Short loop" devices were fabricated with copper-containing MIEC surrounded by silicon nitride (SiN) between top and bottom electrodes (TEC and BEC, respectively). (Bottom left) Topography image of a short loop device array; scan size 20 µm. (Top right) I-V curves for devices A, B, C, and D circled in the topography image. Note the logarithmic current scale that spans six orders of magnitude. Device C (red curve) is leaky, with relatively high "off" state current. (Bottom right) Yield map obtained by programming the AFM to automatically acquire and analyze I-V curves on every device in the array. Each pixel represents a different device and is color coded to its off state current. With this map, leaky devices such as device C are quickly detected. Acquired with the Dual Gain ORCA module on the Cypher AFM. Adapted from Ref. 5.



Nanoscale Time-Dependent Dielectric Breakdown

Understanding dielectric breakdown is critical for reliable manufacturing in the semiconductor industry and beyond. Catastrophic dielectric breakdown occurs when a conducting path forms in an electrical insulator such as a transistor gate oxide. Integrating new materials such as low-k dielectrics into a device introduces different mechanical, thermal, electrical and adhesive properties. These altered properties potentially create new breakdown mechanisms that must be characterized and understood. Although mechanical probe stations are typically used for wafer-level breakdown testing, they cannot interrogate dielectric properties on the single-device level. In contrast, the spatial resolution of the AFM tip enables local measurements of dielectric breakdown.[1,6]

NanoTDDB is an exclusive mode on Asylum Research for dielectric breakdown measurements on much smaller length scales (~20 nm) than possible with conventional probe stations. As in CAFM, in nanoTDDB the tip-sample current is measured with the tip in contact. However, in nanoTDDB the tip remains at the same position and the measurement continues until a breakdown event is detected. The voltage can be held constant or ramped (up to ±150 V or ±220 V depending on model). Measurement locations can be precisely selected from high-resolution images, or a grid of points can be mapped and analyzed.

The wide range of Asylum's ORCA amplifiers (see sidebar) enables both very small pre-breakdown currents and much larger post-breakdown currents to be measured sensitively and accurately.

Fast Current Mapping Mode

In CAFM, lateral tip-sample forces are created while scanning in contact mode. Depending on the applied force needed for good electrical contact, these forces may damage the sample or the tip. For instance, lateral forces can irreversibly deform soft polymers or rearrange loosely-bound nanotubes, degrading image quality. Damage to the special conductive probes may alter the tip-sample contact area or interrupt the electrical path. In either case, wear affects the measured current and hence complicates image interpretation.

Asylum Research's Fast Current Mapping Mode addresses these issues. Like CAFM, it maps current and conductivity distributions. However, current in Fast Current Mapping Mode is measured while acquiring a high-speed array of force curves instead of scanning in contact. It uses the same principles as Asylum's Fast Force Mapping Mode, moving the cantilever vertically in a continuous sinusoidal motion while scanning laterally. Saving complete curves of current versus time enables a wealth of analysis options, both during the experiment and at any later date. Pixel rates up to 300 Hz on MFP-3D Infinity AFMs and even faster on Cypher family AFMs mean that 256×256 pixels can take less than 10 min to acquire.

Fast Current Mapping Mode enables current imaging with gentler forces, making it ideal for delicate samples and high spatial resolution. For example, the integrity of the carbon nanotube film in Figure 4 could easily be compromised by contact mode scanning. The same low-noise, high-bandwidth ORCA and Dual Gain ORCA amplifiers used for CAFM ensure high measurement sensitivity over a wide current range. A further benefit is that force curves are acquired simultaneously with current in Fast Current Mapping Mode.

Force curves are obtained from the cantilever deflection versus Z sensor position during a load-unload cycle and can be used to measure elastic modulus and adhesion. With concurrent topography, current, and mechanical data, Fast Current Mapping Mode yields deeper insight into relations between local structure, properties and functional behavior.

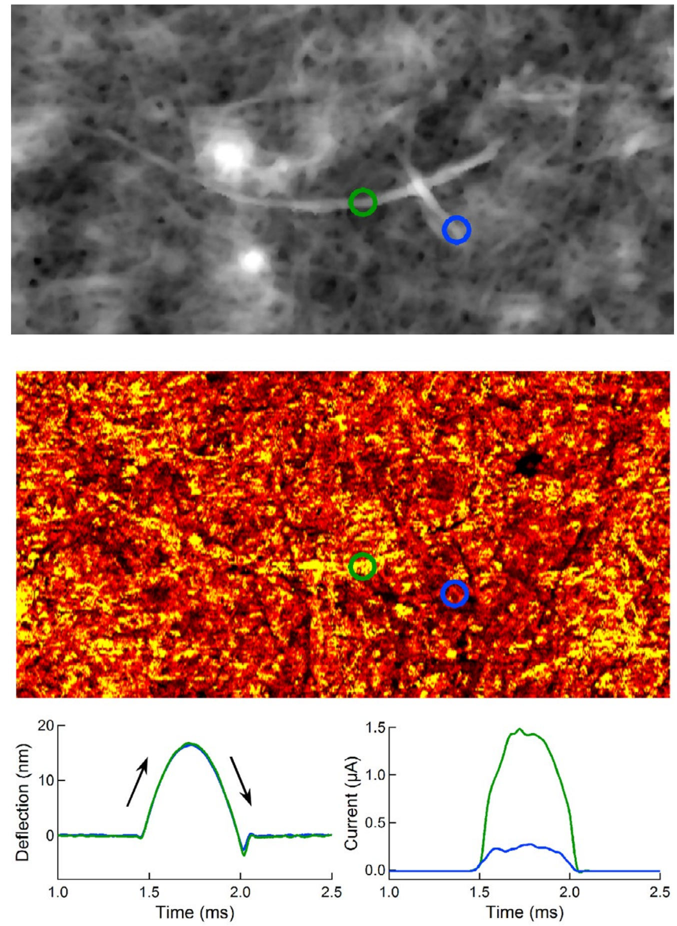
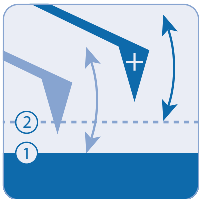


Figure 4: Fast Current Mapping of single-walled carbon nanotubes (SWCNTs). Photoactive layers of SWCNTs could enhance inorganic solar cell devices by simplifying fabrication and lowering costs. The images show topography (top) and peak current (bottom) of a SWCNT film on silicon acquired simultaneously in Fast Current Mapping Mode. Individual nanotubes can be resolved that display a range of conductivity from low (black) to high (yellow). In previous work on similar films (see Ref. 7), ~9% of the SWCNTs were metallic. The graphs show the cantilever deflection (left) and the current (right) versus time at the positions marked by the color-coded circles. The up and down arrows indicate the extend and retract segments, respectively, of the force curve. Scan width 2 µm, topography scale 60 nm, current scale 1.5 µA. Acquired on the MFP-3D Infinity AFM.



Electrostatic Force Microscopy

EFM[8,9] is an electrical AFM mode that is useful for visualizing electric field variations. It is especially effective for detecting near-surface, isolated conductors in insulating or semiconducting materials, for instance carbon nanotubes in a polymer matrix, or carbon inclusions in rubber tires. This makes it an attractive alternative to more invasive electron microscopy methods in some applications. As shown in Figure 5, EFM is also valuable for characterizing the performance of electronic devices.

A standard mode on all Asylum Research AFMs, EFM is based on the fact that an oscillating cantilever is sensitive to long-range electrostatic force gradients. Variations in tip-sample capacitive coupling created by surface charges or potentials are sensed by a dual-pass measurement approach. The first pass scans the conductive probe in standard tapping mode to measure topography. In a second "nap" pass, the topography data allows the tip to retrace the scan line at a constant height above the surface. (The term "nap pass" derives from the aviation expression "nap-of-the-earth flight," in which a plane makes a contour-hugging flight at low altitude.) The height is user-controlled and typically a few tens of nanometers. Separating the tip and sample enhances sensitivity to long-range electrostatic forces, while maintaining constant height minimizes effects from other interactions such as van der Waals forces.

During the nap pass, the tapping piezo is still driven so that the cantilever continues to oscillate at the tapping frequency. A DC bias between the tip and sample is also applied. Electrostatic forces between the tip and sample shift the resonance frequency of the probe. The resulting changes in the cantilever amplitude and phase at the oscillation frequency are recorded in the EFM amplitude and phase channels.

EFM is an effective way to obtain qualitative contrast with minimal setup in many applications. However, quantitative interpretation is more challenging. The measured signals have a complex dependence on numerous experimental parameters such as tip and sample geometry, charge distribution, surface chemistry, and even atmospheric conditions.

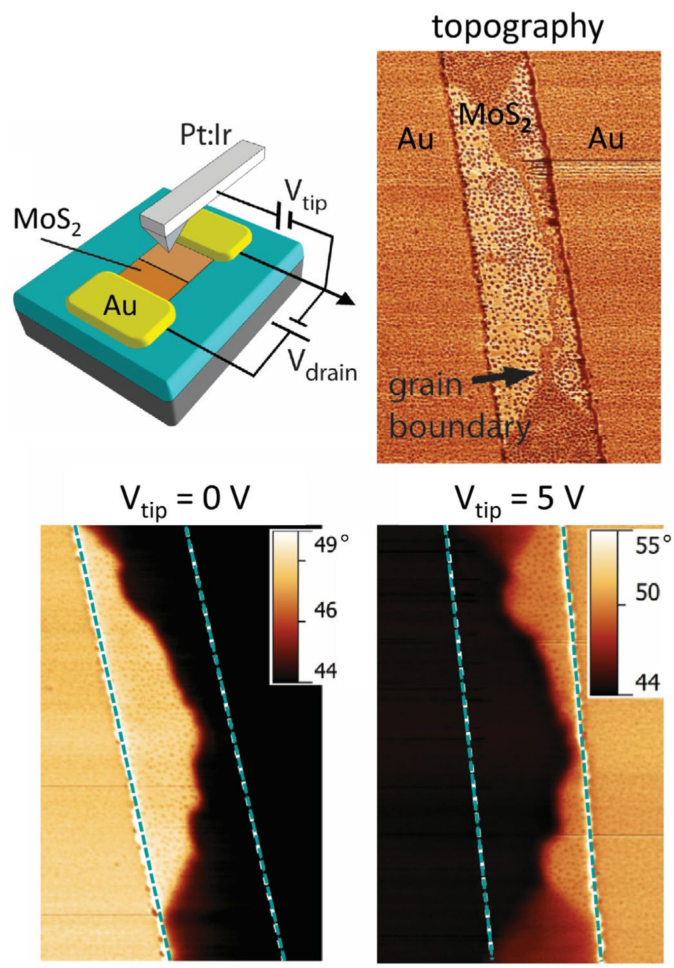
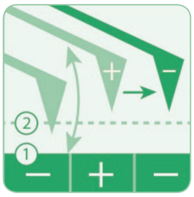


Figure 5: Elucidating switching mechanisms in a 2D memristor with EFM. Memristors are nonvolatile memory devices for enhanced performance in high-speed computing. The schematic (top left) shows the first 2D memristor created from a MoS_2 monolayer between two gold (Au) electrodes. The straight black line in the MoS_2 region represents a grain boundary that bisects the memristor. In the topography image of an actual device (top right), the bisecting grain boundary is curved. The dark, straight lines are the Au-MoS_2 junctions spaced 2 µm apart. EFM phase images (bottom) were acquired at different tip bias voltages V_{tip} with the same device conditions (V_{drain} = 5 V, V_{source} = V_{gate} = 0 V). The dotted lines have been added to help identify the Au-MoS_2 junctions. The abrupt EFM phase drops across the grain boundary reveal its resistive nature and support hypotheses on the critical role of sulfur vacancy migration in switching. Acquired on the Cypher ES AFM. Adapted from a figure originally published in Ref. 10.



Kelvin Probe Force Microscopy

KPFM[11,12] is standard on all Asylum Research AFMs and is suitable for any system where electric fields are present. Although EFM can probe a small volume of a sample, KPFM is purely a surface probing technique. Nonetheless, KPFM provides valuable information on a wide range of materials including biased electrodes and operating devices, oxide layers, and semiconductors. In metallic alloys, it can be used for corrosion studies and to monitor catalytic activity. In polymers, it can visualize molecular conformation and dipole orientation, and the distribution of phase-separated domains.

Sometimes called scanning Kelvin probe microscopy (SKPM) or simply "surface potential imaging", KPFM maps variations in the surface potential. It can be operated in either a single or dual-pass approach. Single-pass KFPM imaging is faster and provides additional information from the cantilever's response at higher-harmonic frequencies. Dual-pass KPFM is used to minimize crosstalk from topography effects. Like EFM, the first pass acquires topography in tapping mode, and the second, nap pass, rescans the conducting tip at constant relative height above the surface. Unlike EFM, however, the tapping piezo is not driven during the nap pass in KPFM. Instead, an AC bias voltage is applied between the tip and the sample at the tapping frequency. Differences in electric potential between the tip and the sample will cause the cantilever to oscillate at the tapping frequency. With this approach, the KPFM signal is the DC bias voltage that must be applied to null the potential and prevent the cantilever from oscillating.

This DC-nulling voltage is a direct, quantitative measure of the contact potential difference between the tip and the sample. As seen in Figure 6, the potential difference can yield convenient contrast in cases where topography does not. However, KPFM has many other uses. In particular, it can measure the work function in conductors and semiconductors because, in these materials, the potential difference typically arises from differences in work function. (The work function represents the energy necessary to move an electron from the Fermi level into vacuum.) Thus, KPFM measurements are sensitive to many surface phenomena. For instance, differences in work function can be used to distinguish the orientation of different metallic crystalline grains or to profile the effects of band bending and dopant density in semiconductors.

Quantifying the local work function from surface potential measurements involves a model to describe the electric interaction between the tip and the sample, and knowledge of the tip's work function. The work function of the tip can be assumed or determined from calibration measurements on a reference sample. Figure 7 shows an example of KPFM work function measurements on nanoparticle films.

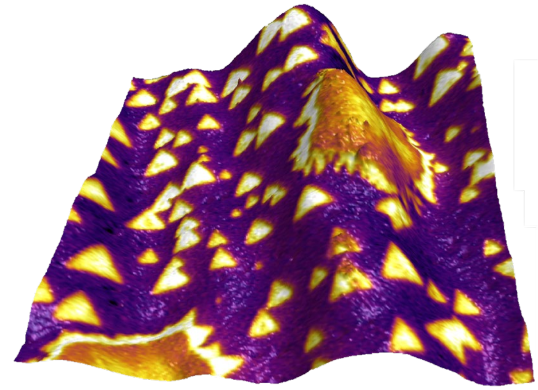


Figure 6: KPFM surface potential overlaid on topography for flakes of boron nitride (small triangles) and graphene (large irregular features) grown on a copper foil substrate. At a single atom thick, the flakes can be difficult or even impossible to resolve in topography images due to the roughness of the copper substrate. However, they are readily identified by variations in the tip-sample potential. The surface potential also distinguishes between the graphene and boron nitride, and reveals boron nitride regions growing epitaxially out from the graphene islands. Scan size 50 µm. Imaged on the MFP-3D AFM.

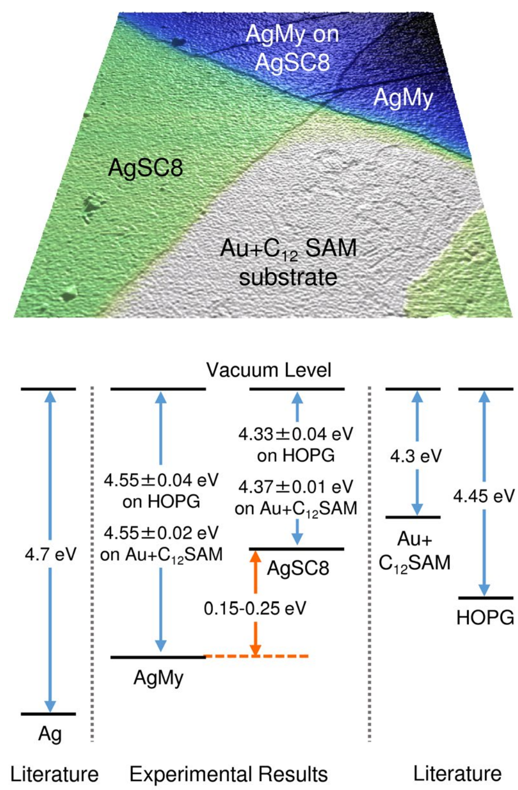
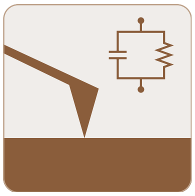


Figure 7: Tuning work function for improved optoelectronic device efficiency. Tunability was achieved by capping silver (Ag) nanoparticles with self-assembled monolayers (SAMs) of organic molecules. AgMy particles were capped with a myristate SAM, while AgSC8 particles contained a 1-octanethiolate [$CH_3(CH_2)_7SH$] SAM. The image shows KPFM surface potential voltage overlaid on topography for a sample containing sheets of particles on gold (Au) with a 1-dodecanethiolate [$CH_3(CH_2)_{11}SH$ or C_{12}] SAM. The surface potential differs significantly for AgMy and AgSC8 sheets but is very similar for AgMy sheets on different substrates. (bottom) Work function values were calculated from KPFM data on both Au+C_{12}SAM and highly oriented pyrolitic graphite (HOPG) susbtrates. Values are consistent with behavior expected from differences in dipole moment. Scan size 10 µm; acquired on the Cypher AFM. Adapted from Ref. 13.



Scanning Microwave Impedance Microscopy (sMIM)

Understanding electrical response at microwave frequencies has proven valuable in both fundamental materials research and device development. In recent years, smaller device dimensions and new nanostructured materials have motivated the creation of AFM techniques[14] for microwave measurements. These techniques probe the near-field interactions between a microwave source and sample that occur on length scales much smaller than the radiation wavelength (submicrometers vs. centimeters).

sMIM is a near-field microwave technique[15] available on MFP-3D family and Cypher S AFMs. It is beneficial in a wide range of applications on both linear and nonlinear materials (conductors, semiconductors, and insulators). For example, sMIM can be used to evaluate ferroelectric domains and domain boundaries, analyze electric defects and failures, and visualize buried structures.

On dielectric films, the sMIM signal can be related to film thickness and dielectric constant; while on semiconductor materials, it enables high-sensitivity measurements of dopant type and concentration. As seen in Figure 8, sMIM can discern differences in conductivity for features that appear virtually identical in topography.

sMIM senses changes in sample resistance and capacitance using a microwave source coupled to a shielded probe. As the tip scans the sample, local variations in conductivity and permittivity affect the reflected microwaves. sMIM measures the complex reflection coefficient in quadrature, which allows changes in resistance to be distinguished from changes in capacitance. In contrast, techniques such as EFM, KPFM, and scanning capacitance microscopy (SCM) measure a combined response.

If desired, a modulated (AC) bias can be applied to sense gradients in resistance and capacitance. sMIM is typically performed in contact mode, but tapping mode operation is possible. sMIM can also be performed in a manner similar to Fast Current Mapping Mode for resistance and capacitance mapping with less risk of tip or sample damage.

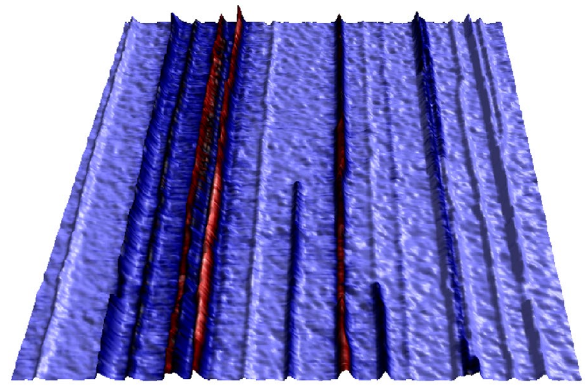


Figure 8: sMIM imaging of horizontally aligned single-walled carbon nanotubes (SWCNTs). Deeper knowledge of nanotube electrical properties will help realize their potential in applications including advanced microelectronics and thermal management. The image of sMIM relative resistivity signal overlaid on topography clearly distinguishes between metallic (red), semi-metallic (black), and semiconducting (blue) SWCNTs. Note that the semiconducting tubes are visible only in the topography channel. Scan size 7.5 µm; acquired on the MFP-3D AFM.

Summary

Asylum Research AFMs offer exceptional capabilities for nanoscale characterization of electric functionality. Different electrical modes give different insight, but they all reveal relations between structure, properties, processing, and performance in many systems. Contact Asylum Research to discover how your application can profit from electrical AFM techniques.

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