ATOMIC FORCE MICROSCOPY

The Definitive AFM Modes Handbook

A Comprehensive Guide to Expanding Your Materials Research Capabilities

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ScanAsyst
SECPM
TappingMode
TR-Mode
TR-TUNA

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Introduction

Since its development in the early 1980s, atomic force microscopy (AFM) has been used in groundbreaking research, leading to a constant stream of new applications. AFM has allowed researchers to map structures and properties on the micrometer and nanometer levels, which has led to its wide adoption in fields from semiconductors and piezoelectric materials to energy research and biology. As the technology has matured, AFM has given scientists the opportunity to characterize features at ever-smaller scales, including such firsts as mapping a live HeLa cell and achieving atomic resolution on a crystal lattice, among others.

The many advances in AFM technology over the past several decades have aimed to extend measurements to soft and fragile samples, broaden applications beyond mere topographical characterization, and improve measurement consistency and ease of use. These advancements have not been limited to specific experiments or applications but rather have been added to the common analytical toolboxes of researchers across a variety of applications. Central to all these new advances and approaches are AFM modes.

The Origins of AFM

The scanning tunneling microscope (STM) was developed by Gerd Binnig and Heinrich Rohrer in the early 1980s at IBM Research-Zurich. Like modern AFM, the STM scanned a probe tip across a surface and collected data based on the tip-surface interactions. Unlike AFM, STM was based on the concept of quantum tunneling—inducing electrons to move through the classically-impassable thin layer of vacuum separating the tip from the surface. For their work on STM, Binnig and Rohrer earned the 1986 Nobel Prize for Physics and laid the groundwork for the development of AFM. Binnig went on to develop the first AFM, along with his co-authors Quate and Gerber. The first commercially available atomic force microscope was introduced in 1989, and Bruker has been leading the expansion of AFM capabilities ever since with numerous patents and the development and expansion of dozens of innovative AFM modes.

Prior to the invention of AFM, surfaces were examined at the microscale using a combination of traditional and specialized optical microscopes and electron microscopes. Optical microscopes have resolution limits in the hundreds of nanometers, rely on the reflective properties of the surface to obtain quality data, and make it difficult to isolate surface-level data. In contrast, electron microscopes can achieve ultrahigh lateral resolution, but require atmospheric control, cannot directly measure sample height, and have certain sample restrictions. AFM, on the other hand, provides surface sensitivity at high resolution both laterally and vertically, can operate in a variety of conditions and across a wide variety of samples.

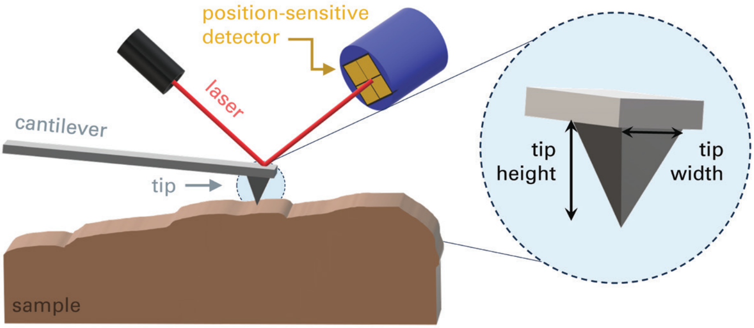
The basic working principle of an atomic force microscope (shown below) is:

1. A sharp tip at the free end of a cantilever is raster-scanned over a small area of sample.

2. As the tip passes over the surface, variations in height cause the cantilever to bend.

3. This bending, or change in deflection, is detected through movement of a laser or super luminescent diode (SLD) that is reflected off the cantilever into a position-sensitive photodetector (PSPD).

Throughout this process, piezo actuators operate within an electronic feedback loop to move the tip or sample closer or further away from each other to maintain the relative tip-sample distance and a constant setpoint.



What Is an AFM Mode?

In response to evolving research needs from an ever-widening number of application and material spaces, AFM methods and instrumentation have continually improved. Central to these improvements is the concept of AFM modes—adapting the probe itself or changing the way it interacts with the sample surface. An AFM mode can consist of one specific modification or method, or of many. All conditions that make up the test environment constitute a single mode.

Foundations: Topographical Modes

There are several AFM modes that evaluate sample topography and serve as the foundation for other AFM modes—contact mode, TappingMode, and PeakForce Tapping®. Broadly, all use the same concept of a cantilever and probe tip interacting with the surface, and all have a similar setup. However, the way the cantilever and probe tip are moved and the way the resulting signal is evaluated differs.

Evolution: Property Measurement Modes

In addition to topographic information, AFM can be used to collect and analyze material properties and responses at the nanoscale. The array of available property measurement modes makes AFM an incredibly powerful technique for materials analysis at the nanoscale. Property measurement modes often require a specialized probe, module, or analysis method. For example, nanoelectrical measurements require a conductive probe and a setup enabling applied voltage. Electrochemical response measurements require an electrochemistry cell hardware setup.

Complementary Technologies

Individual topography and property measurement modes can be enhanced with innovative capabilities, such as Bruker's DataCube and ScanAsyst® technologies. DataCube (Appendix A) is a hyperspectral imaging and data management method for rich, 3D datasets. ScanAsyst (Appendix B) conducts auto-optimization of imaging parameters, making data acquisition easy and consistent.

Constant Innovation

In response to emerging research and industry needs, the AFM modes list is ever-expanding. New hardware and software can enable innovative, cutting-edge experiments. For up-to-date information about available modes, visit www.bruker.com/afm-imaging.

How to Use This Handbook

This handbook illustrates the wide variety of operating modes available on Bruker AFMs, going well beyond the standard high-resolution topographic imaging capabilities of AFM. The modes are broken into seven separate categories: morphology, electromagnetic properties, thermal properties, mechanical properties, chemical properties, electrochemical properties, and manipulation. Each category begins with a dedicated page that groups the contained modes by subcategory. In this way, users should find it easier to identify relevant modes. Each individual mode page then describes what the mode is, how it works, and when to use it. These one-page summaries are accompanied by a number of datasets relating to that mode.

Terminology Clarification

Many AFM modes were developed by expanding upon the capabilities of other modes. This may complicate the language around modes discussions, since the foundational "mode" becomes only one element of the new mode. For example, PeakForce QNM® utilizes real-time force-distance curves collected during PeakForce Tapping operation to extract quantitative nanomechanical information. In this scenario, PeakForce QNM is the actual *mode* and PeakForce Tapping is a technology used as part of the PeakForce QNM mode. In this handbook, the primary mode being discussed will always be the title of the page. Any other mode names mentioned on the page are either component technologies or are secondary modes being compared to the primary mode.

AFM Probes

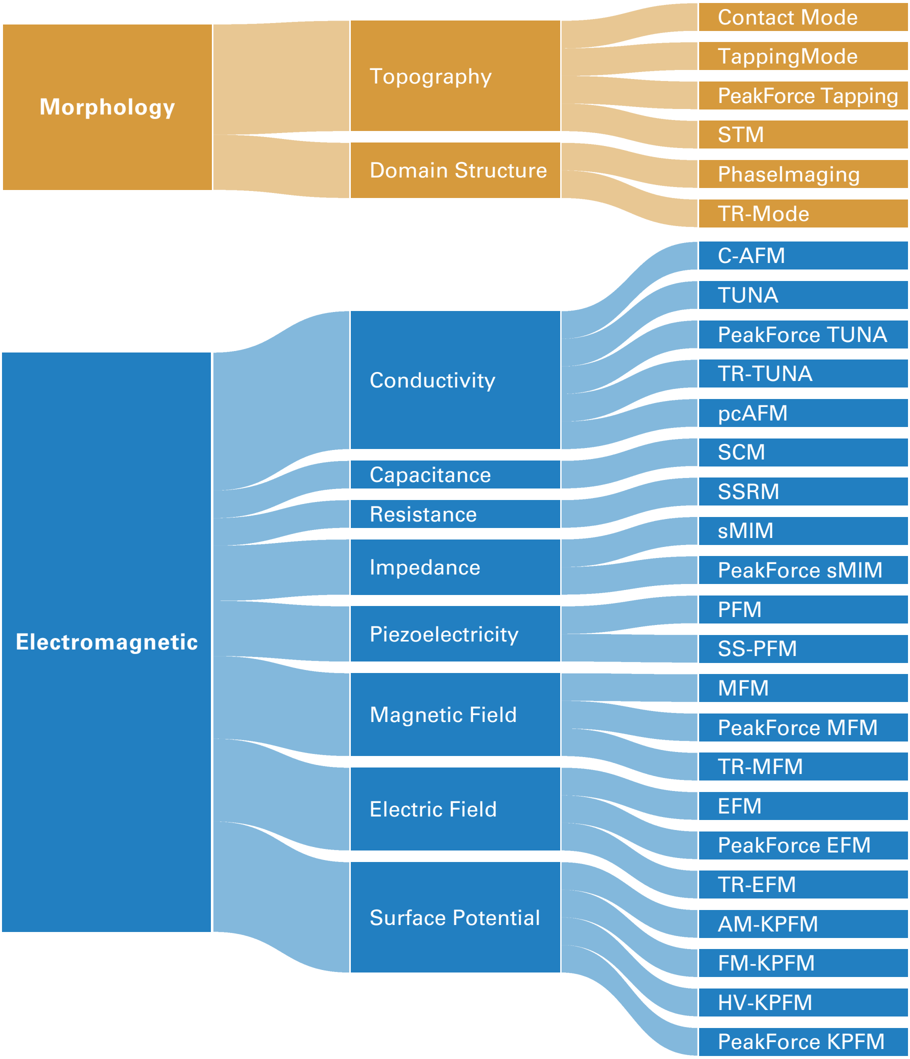
The mode pages in this handbook also list recommended Bruker probes for that mode. Note that probes are listed by their model name and, for brevity, some similar probe names are combined using parentheses. For example, the listing "SCANASYST-AIR(-HPI)" refers to both the "SCANASYST-AIR" probe and the "SCANASYST-AIR-HPI" probe, which are both recommended.

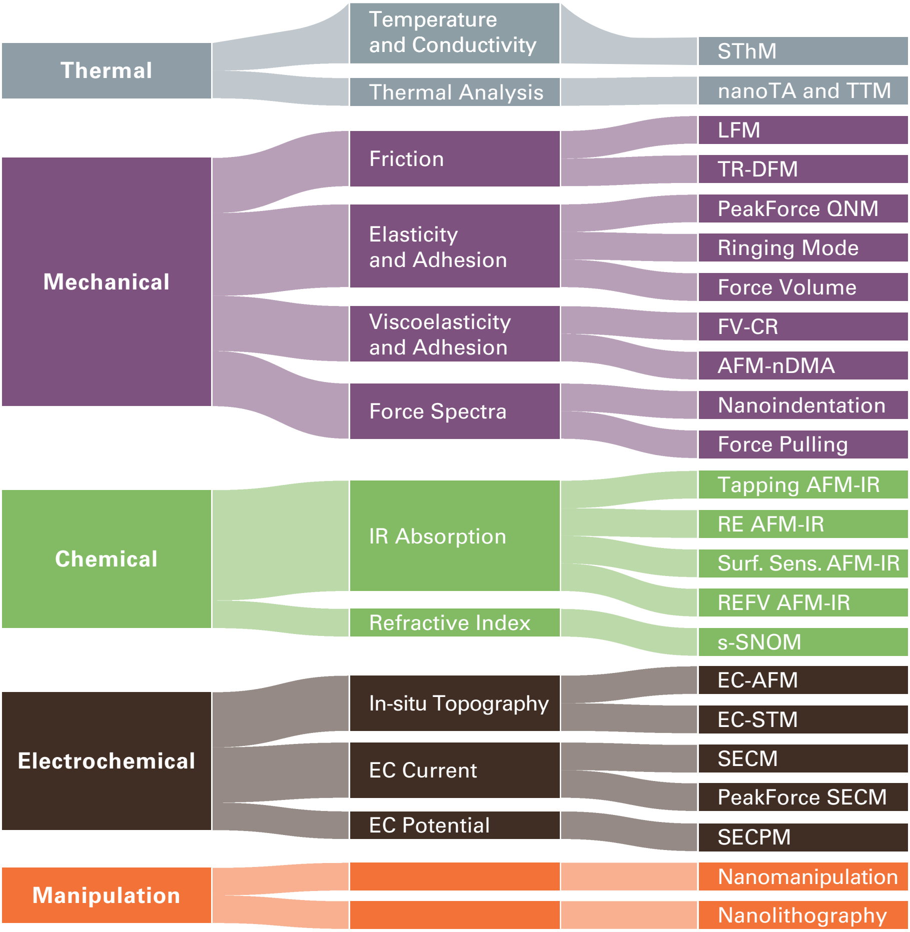
Though not exhaustive, these recommendations list popular probes for each category, and should give users a good place to start when beginning research. You can visit www.brukerafmprobes.com or review our probes selection guide for more detailed information on our probe offerings.

Find Your Mode

This handbook organizes and summarizes the most frequently used AFM operating modes. Each page gives a brief overview of a single mode, with exemplary data. Once you identify which modes you are interested in from these summaries, we encourage you to explore our online open-access AFM resource library to learn more.

Unsure which mode will work for your specific research? Contact Bruker experts through our website to ask about individual samples and applications.



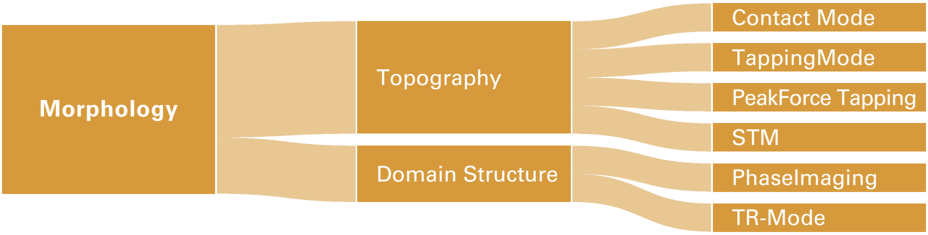


Customize Your Mode

We offer over 50 AFM modes to evaluate an extensive range of structures, properties, and responses, but sometimes custom experiments require custom modes. Bruker AFMs provide an open hardware and software platform where front panel BNCs afford easy access to most input/output signals, and an optional module expands access to all signal and control lines. The NanoScript™ software option provides an expansive set of SPM control functions for custom measurements, lithography, and manipulation. This flexibility has allowed users to adapt established operating modes for their specific needs or develop their own innovative operating modes, including Fast Fourier Transform SSRM, Time-to-breakdown C-AFM, Scanning Gate Microscopy, PeakForce AFM-IR, PeakForce Contact Resonance AFM, and Reverse Tip Scanning.

Morphology Modes

Morphology modes are used to measure the nanoscale 3D structure of a surface. This category of modes is split into those that evaluate topography and those that evaluate domain structure.

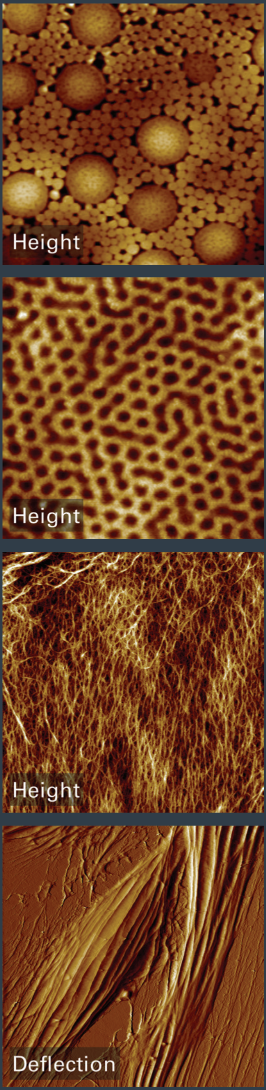


Contact Mode

In contact mode, the AFM tip is dragged across a surface while maintaining tip-sample contact. The sample topography induces a vertical deflection of the cantilever, which is tracked to reconstruct a topographic image. As the "original AFM mode," contact mode delivers the fundamental and straightforward imaging that is the basis for other advanced techniques. Contact mode uses a feedback loop to maintain constant deflection by moving the Z-scanner up or down to keep the preset load force constant. The motion of the Z-scanner (or sensor) is recorded to generate the topography signal.

Contact mode is used for topographical studies of surfaces and the study of friction in materials science, biological applications, and basic research. However, its application is limited for soft samples because dragging the sharp probe across the sample can cause damage. It is therefore more suitable for harder surfaces, such as hard polymers, ceramics, metals, and semiconductors.

Contact mode is considered a "fast" imaging mode, which can be applied for high-resolution imaging (down to atomic lattice resolution), as well as for large-area imaging.



(from top) Latex spheres, block copolymer, polypropylene, and endothelial cells. Scan sizes (X): 3 µm, 4 µm, 4 µm, 65 µm.

Environments: air, liquids, glovebox

Recommended probes: DNP-A, DNP-C, MLCT-F, DNP-D, RESPA-20, RESPA-10

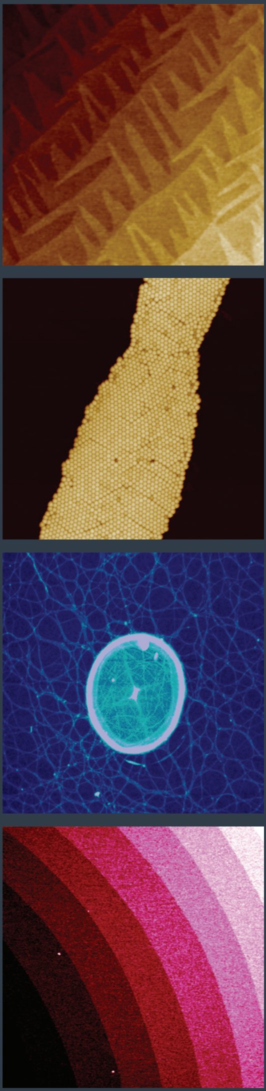
Related modes: LFM, AFM-nDMA, PFM, C-AFM, and others

TappingMode

TappingMode is the most popular AFM imaging mode in both air and liquids, and is also the backbone for many specialized modes, such as electric force microscopy (EFM) and magnetic force microscopy (MFM). The development of TappingMode enabled researchers to image samples too fragile to withstand the lateral forces of contact mode and use scan speeds much higher than could be obtained in contact mode.

TappingMode is a Bruker-patented technique that maps topography by lightly tapping the surface. The AFM cantilever oscillates at resonance with a typical amplitude of a few nanometers to a few tens of nanometers. The cantilever's oscillation amplitude changes with sample surface topography, and the topography image is obtained by monitoring these changes and closing the Z feedback loop to minimize them. When conducting phase imaging, the phase signal is also extracted (see PhaseImaging page).

TappingMode is used for topographical studies of surfaces in materials and life sciences. TappingMode can be applied in both air and liquid environments, and is applicable to virtually any sample type without causing damage to tip or sample. This has led to its use in thousands of publications. When used with high-bandwidth cantilevers, TappingMode can also be a fast imaging method.



Height images of (from top) epitaxial Si, 100 nm polystyrene spheres, diatom in marine gel, and atomic step terraces. Scan sizes (X): 1 µm, 6 µm, 5 µm, 5 µm.

Environments: air, liquids, glovebox

Recommended probes: TESPA-V2, RTESPA-300, OTESPA-R4, SNL-C, DNP-C

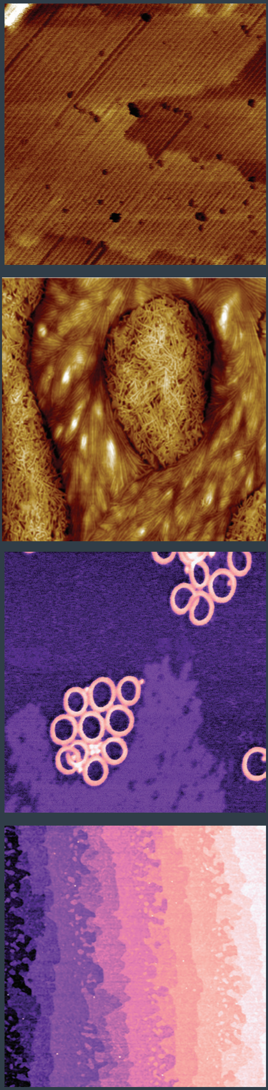
Related modes: PhaseImaging, EFM, MFM, and others

PeakForce Tapping

In PeakForce Tapping®, the probe periodically taps the sample, and the tip-sample interaction force is controlled very precisely in real time. This mode provides the lowest available imaging forces and simultaneously enables nanoscale mechanical and electrical property mapping. Exclusive to Bruker, PeakForce Tapping has become the most-published AFM technology since TappingMode.

PeakForce Tapping uses a cantilever oscillated at an off-resonance frequency. A feedback loop on cantilever deflection keeps the peak force down to 10–30 pN (cantilever dependent) at actuation rates up to 8 kHz in air and liquid. Because the tip is only in contact for a small portion of the oscillation cycle, lateral forces on the tip are minimized, reducing tip wear and providing non-destructive imaging of delicate and loosely bound samples. PeakForce Tapping technology uniquely enables and enhances other correlative and quantitative mapping techniques, delivering new possibilities in an ever-expanding set of topographical, mechanical, biological, electrical, and chemical applications at the nanoscale.

This versatile mode is ideal for highest-resolution imaging and property mapping in both materials research and biological samples due to its unprecedented low imaging forces and ease of use. It can be used to visualize topography and property variations in the widest variety of sample types, from the softest biological samples to very hard materials, and resolving even the smallest structures, such as atomic defects and double-helix DNA.



Height images of (from top) Cu phtalocyanine dye, sPP/PEO blend, polymer nanorings, and graphene multilayer. Scan sizes (X): 200 nm, 3 µm, 300 nm, 2 µm.

Environments: air, liquids, glovebox

Recommended probes: SCANASYST-AIR(-HPI), PEAKFORCE-HIRS-F-A, PEAKFORCE-HIRS-F-B, SCANASYST-FLUID(+), PFQNM-LC-V2

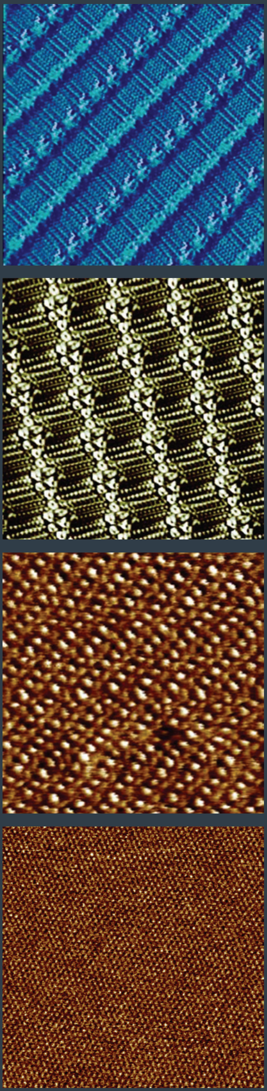
Related modes: PeakForce QNM, AFM-nDMA, PeakForce TUNA, PeakForce KPFM, PeakForce MFM, and others

Scanning Tunneling Microscopy (STM)

STM is the precursor technique to modern atomic force microscopy. It maps surface topography of surfaces with atomic resolution by monitoring tunneling current between tip and sample. STM has no tip-sample forces and has high lateral resolution, though it does require a conductive or semiconductive surface/substrate. In addition to extreme high spatial resolution imaging, STM can also be applied for local I-V and I-s spectroscopy.

In STM, a sharp conductive tip is scanned close to the sample surface while applying a bias voltage between tip and sample. At very close proximity, electrons tunnel between tip and sample; this tunneling current is highly sensitive to tip-sample distance. Surface height changes are detected through current changes, and a setpoint of either current or tip-sample distance is maintained through precise control of tip position.

STM can be operated in air and, in some cases, in fluids. It is a general atomic resolution imaging method for conductive surfaces and can be used to study electronic properties in condensed-matter physics, atomic-scale processes in catalysis, electrical properties of individual molecules or molecular assemblies, and behavior of individual qubits in quantum computing.



Height images of (from top) dinoadecanoylbenzene, monolayer at the phenyloctane/HOPG interface, selenol-based aromatic SAM, and HOPG atoms. Scan sizes (X): 15 nm, 20 nm, 10 nm, 12 nm.

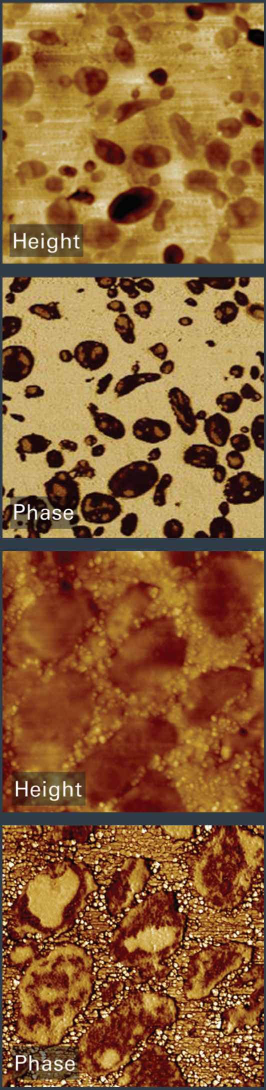
Environments: air, liquids, glovebox
Recommended probes: TT10, PT10
Related modes: EC-STM, SECPM

PhaseImaging

PhaseImaging, also known as *tapping with phase*, is a secondary imaging mode derived from TappingMode that detects sample variations, such as composition, adhesion, friction, and viscoelasticity, as well as other properties, including electric and magnetic. This mode's operation is identical to TappingMode, with the only difference being what data is collected and analyzed.

PhaseImaging maps the phase lag between the periodic signal from a piezo actuator that is driving the cantilever and the (near) resonance-frequency oscillations of the cantilever itself. Changes in phase lag often indicate changes in the properties of the sample surface, meaning that images of topography and material properties can be collected simultaneously.

This mode is a useful tool for simultaneous collection of topography and material properties. It is often used in applications where the samples contain inhomogeneities, such as contaminant identification or mapping components of composite materials. PhaseImaging is a key element in several other AFM modes, including magnetic force microscopy (MFM) and electric force microscopy (EFM).



(from top, in pairs) Impact-modified copolymer and rubber. Scan sizes (X): 8 µm, 4 µm.

Environments: air, liquids, glovebox

Recommended probes: RTESPA-150, RTESPA-300, RTESPA-525, RFESPA-75, OTESPA-R4, SNL-C, DNP-C

Related modes: TappingMode, EFM, MFM

Torsional Resonance Mode (TR-Mode)

Torsional resonance is an alternative to TappingMode for high-resolution topographic imaging on a wide range of samples. TR-Mode has been applied for high-resolution imaging on a variety of samples, including soft biological samples and polymers. The topographic images are complemented by torsional resonance phase images, which provide a highly sensitive contrast based upon anisotropic material properties.

In TR-Mode, the torsional resonance amplitude (or phase) is used to control the feedback loop and maintain the tip-surface relative position through lateral interaction. The nature of tip-surface interaction of TR-Mode facilitates phase measurements to resolve the in-plane anisotropy of materials, as well as measurements of dynamic friction at the nanometer scale.

TR-Mode can also be of interest for samples that require highly sensitive phase detection. In general, the Q-factor of the torsional resonance is considerably higher than the Q-factor for the flexural mode, resulting in higher sensitivity. A torsional resonance modulation can also be applied in a wide variety of operating modes. These typically have the same goal to resolve in-plane (often anisotropic) sample properties (e.g., frictional, magnetic field, electric field, and conductivity).

(from top, in pairs) HOPG with water at the step edges and PDES on Si substrate showing anisotropic property data in the TR phase channel. Scan sizes (X): 3 µm, 12 µm.

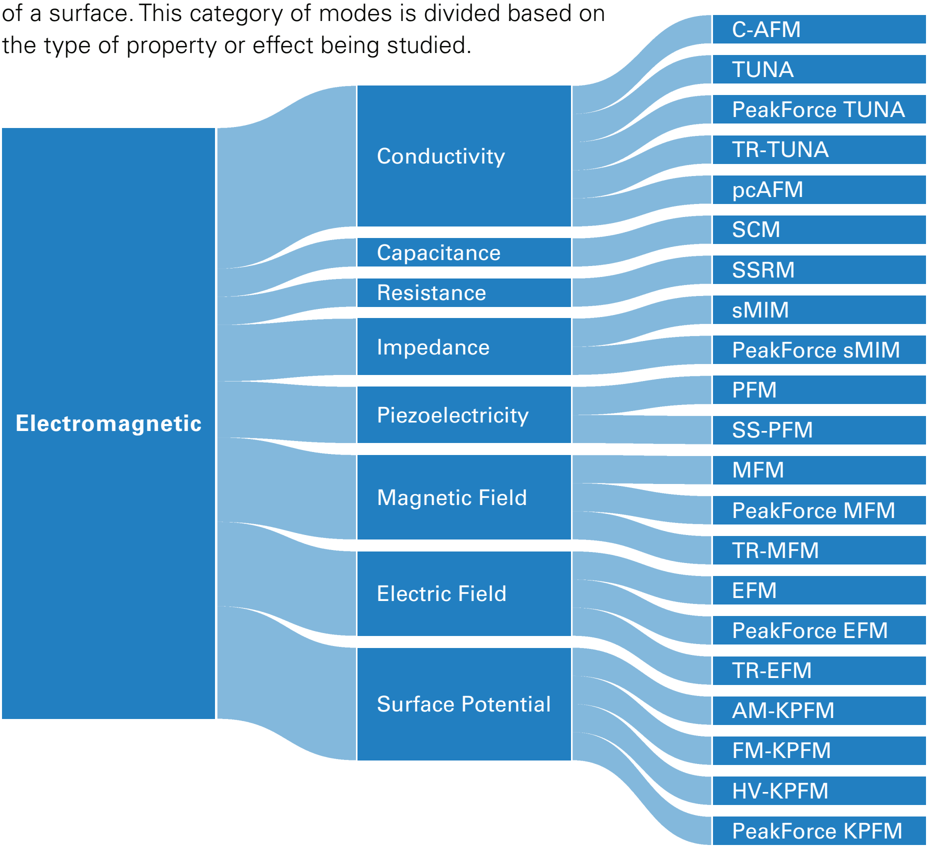
Environments: air, liquids, glovebox

Recommended probes: FESPA-V2, LTESPA-V2, MESP-V2, SCM-PIT-V2

Related modes: TR-DFM, TR-MFM, TR-TUNA

Electromagnetic Modes

Electromagnetic modes are used to characterize the nanoscale electrical and magnetic properties or responses of a surface. This category of modes is divided based on the type of property or effect being studied.

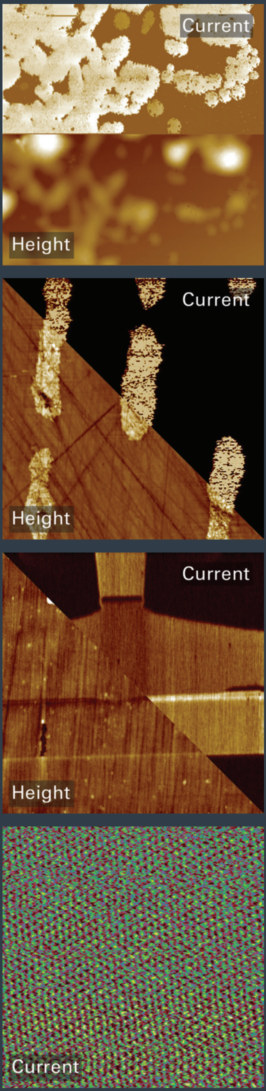


Conductive AFM (C-AFM)

C-AFM simultaneously collects data on electrical conductivity and sample height. It uses a conductive tip in contact with the sample and an applied voltage to map electric current and topography. This nanoelectrical characterization mode can be used to map conductivity, investigate triboelectric behavior, and characterize electrical defects.

During C-AFM scanning, a DC voltage is applied between the conductive tip and sample surface. By measuring the current flowing between tip and surface, conductivity variations across the sample can be mapped. Alternatively, the conductive tip can act as a single-point electrode to obtain local I-V curves. C-AFM requires a conductive probe, constant sample contact, and a (semi) conductive sample.

C-AFM is used to map variations in electrical conductivity across medium- and low-conducting or semiconducting materials, with current range 2 pA to 1 µA. Unlike STM, the current measured by C-AFM is not a tunneling current. Samples for C-AFM must be able to accommodate contact mode scanning.



(from top) Conductive polymer, cross-sectioned capacitor, magnetic read/write element, and HOPG atomic lattice. Scan sizes (X): 20 µm, 20 µm, 12 µm, 10 nm.

Environments: air, liquids, glovebox
Recommended probes: MESP-V2, SCM-PIT-V2, MESP-RC
Related modes: TUNA, pcAFM, PeakForce TUNA

Tunneling AFM (TUNA)

TUNA uses a conducting AFM tip to scan the sample surface while a DC voltage is applied between tip and sample. TUNA provides local electrical conductivity maps along with surface topography. It offers higher current sensitivity than C-AFM, enabling characterization of ultralow currents (<1 pA) through highly resistive samples, and detailed conductivity mapping over low-conductivity materials.

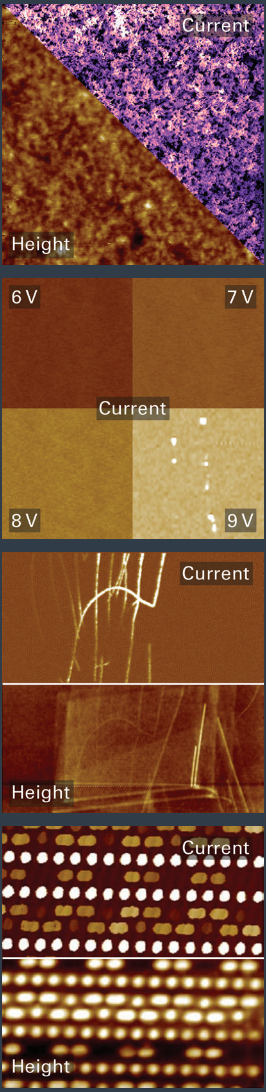
TUNA works similarly to C-AFM but with a different design of the current amplifier, which allows for higher current sensitivity and six gain levels that can be adjusted. During operation, a conductive tip scans the sample surface while a DC voltage is applied, and the resulting current is measured.

TUNA is valuable for ultralow current measurements on low-conductivity samples. It can be used to map electrical defects in semiconductors, data storage devices, conductive polymers, or organics. Its high current sensitivity makes it ideal for conductivity mapping over dielectric thin films and other low-conductivity materials where the current 'tunnels' through the dielectric, as well as for generating I-V curves.

Environments: air, liquids, glovebox

Recommended probes: MESP-V2, SCM-PIT-V2, MESP-RC, SCM-PIC-V2, DDESP-FM

Related modes: C-AFM, pcAFM, PeakForce TUNA



(from top) Conductive polymer, SiO$_2$ showing breakdown at higher voltages, conductive nanowire network, IC contact-level failure analysis. Scan sizes (X): 10 µm, 1 µm, 4 µm, 3 µm.

PeakForce Tunneling AFM (PeakForce TUNA)

PeakForce TUNA was specifically designed for local electrical conductivity mapping on soft and fragile samples. This technique offers high spatial resolution, high current sensitivity, and the ability to directly correlate quantitative nanomechanical and nanoelectrical property imaging. It is non-destructive and avoids probe-tip contamination caused by lateral forces, since it is based on PeakForce Tapping operation.

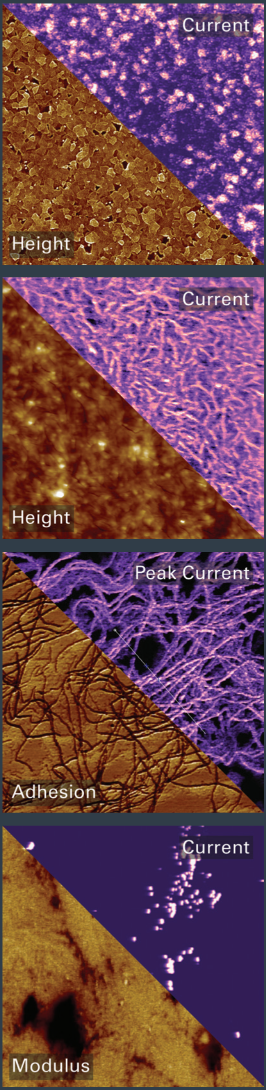
PeakForce TUNA can be used either to map current, topography, and mechanical properties, or to collect I-V curves. When imaging, the electrically conductive probe is scanned over the surface as a feedback loop keeps peak force on the tip constant. A DC tip-sample bias can be applied, allowing the measurement of current passing through the sample during the short contact time. For I-V curves, tip location is fixed and sample bias is ramped.

As a PeakForce Tapping–based mode, PeakForce TUNA is ideal for probing organic photovoltaics, conductive nanotubes, and nanoparticles. It is particularly useful for applications that require high-resolution current mapping and correlated nanomechanical and nanoelectrical property imaging. This mode provides better repeatability and consistency, making it preferred over C-AFM or TUNA, unless lateral force is specifically required.

Environments: air, liquids, glovebox

Recommended probes: PFTUNA, SCM-PIT-V2, SCM-PIC-V2, DDESP-FM

Related modes: TUNA, C-AFM, pcAFM



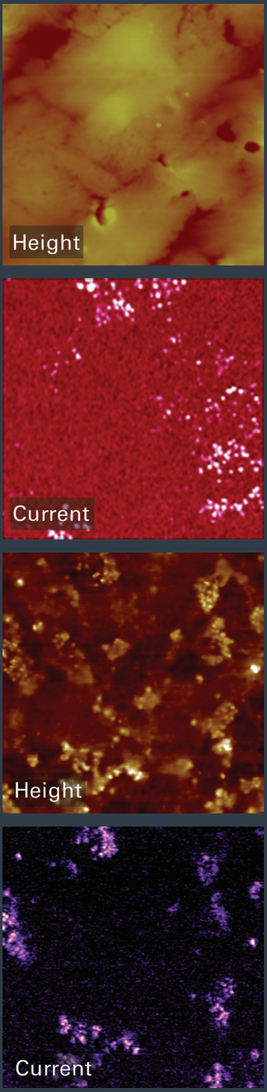
(from top) ITO, polymer fibrils, P3HT organic conductive nanowires, and TPV (PP/rubber blend, with conductive carbon black particles) Scan sizes (X): 10 µm, 5 µm, 3 µm, 5 µm.

Torsional Resonance TUNA (TR-TUNA)

In TR-TUNA, the sample topography and conductivity are acquired simultaneously, similar to TUNA and PeakForce TUNA modes. As with PeakForce TUNA, TR-TUNA can overcome some of the limitations of the contact mode–based techniques, which are often not suitable for soft and fragile materials (e.g., some organic materials or nanowires), as the relatively high lateral forces can cause artifacts (e.g., plowing or piling-up of material) or tip wear.

In TR-TUNA, topographic imaging is performed in TR-Mode (with amplitude feedback), and current is collected simultaneously while applying a DC voltage to the sample. The torsional resonance feedback keeps the tip-sample distance constant at a very small value. This is sufficiently small to enable measurements of sample conductivity. The TR-Mode scan also provides correlated mechanical property (dynamic friction) information.

Typical samples where TR-TUNA can be applied include nanowires, nanoparticles, organic device materials, conductive polymers, and composite materials. Materials where there is an interest to correlate dynamic friction and conductivity are particularly well served by TR-TUNA. If other mechanical properties (e.g., adhesion, modulus, and deformation) are of interest, it is recommended to use PeakForce TUNA.



(from top, in pairs) Rubber modified PP with carbon nanoparticles and carbon sticky tape with conductive carbon nanoparticles. Scan sizes (X): 4 µm, 5 µm.

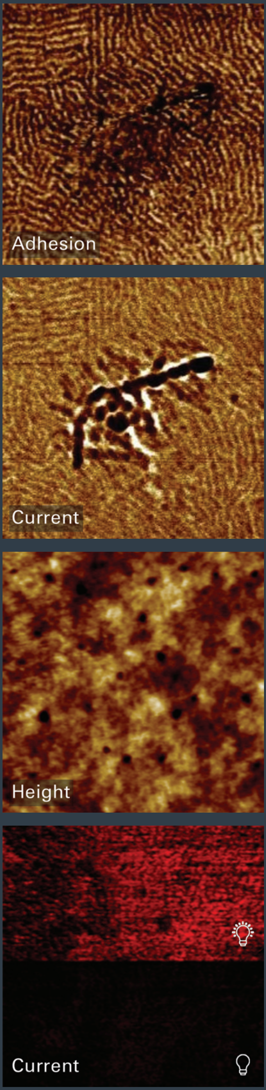
Environments: air, liquids, glovebox
Recommended probes: MESP-V2, SCM-PIT-V2
Related modes: C-AFM, TUNA, PeakForce TUNA, pcAFM

Photoconductive AFM (pcAFM)

The pcAFM mode is used to study nanoscale electrical properties of materials that exhibit change in electrical conductivity when exposed to light. When the material surface is illuminated by a light source, charges are generated in the photoactive layer. A conductive probe is used to collect the charges, and as the probe raster scans on the surface, the local photo current is mapped with nanometer resolution.

In essence, pcAFM is very similar to C-AFM, but is specifically applied for photoactive material systems with the addition of a varying illumination onto the sample surface, creating photo-induced electrical effects. This approach is applicable to C-AFM, TUNA, PeakForce TUNA, TR-TUNA, and DataCube TUNA. A similar approach can also be applied to other electrical modes (EFM, KPFM, PeakForce KPFM, PFM, etc.) to study the effect of specific illuminations.

Typically, pcAFM is used for the characterization of photovoltaic (PV) samples, including organic PV, semiconductor PV and perovskite-based PV. Photoelectrical effects studied can include photoconductivity, as well as photoinduced changes in surface potential, charge density, or piezoelectric properties.



(from top, in pairs) Illuminated composite CNT/P3HT sample and P3HT/PCBM sample illuminated and dark without bias. Scan sizes (X): 700 nm, 1 µm. (courtesy Dr. Philippe Leclère, University of Mons, Belgium)

Environments: air, glovebox

Recommended probes: MESP-V2, SCM-PIT-V2, MESP-RC, SCM-PIC-V2, DDESP-FM

Related modes: C-AFM, TUNA, PeakForce TUNA

Scanning Capacitance Microscopy (SCM)

SCM is used to analyze charge carrier concentration and carrier type in semiconductor materials and devices. Variations in capacitance between the AFM tip and the sample are measured using an capacitance sensor with high sensitivity operating at ~1 GHz. These capacitance variations directly relate to the variations in carrier concentration.

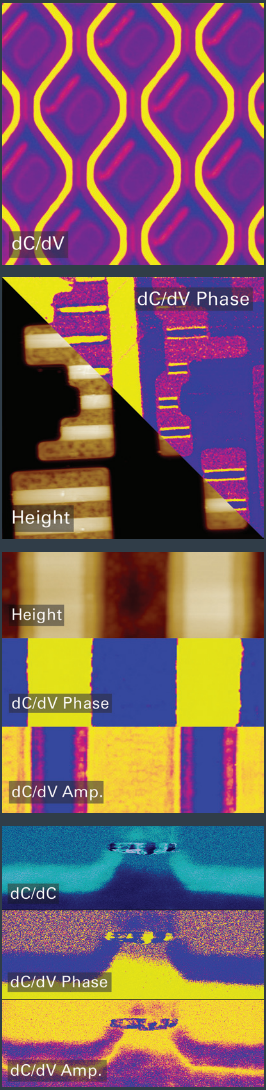
In SCM, a conducting tip is scanned in contact mode on the sample surface. An electrical bias (AC+DC) is applied between the tip and the sample, and a capacitance sensor, which is essentially an LC oscillator, measures dynamic capacitance changes induced by the applied AC voltage. The capacitance changes are detected by a lock-in to create dCdV amplitude and dC/dV phase channels relating to the carrier concentration and carrier type (n- or p-type).

Along with SSRM and sMIM, SCM is one of three commonly applied modes for carrier profiling on semiconductor devices. It can be applied on both silicon and compound semiconductor devices. Sample preparation might be required to provide access to the area of interest on the device under study. Cross-sectioning techniques, such as precision cleaving, polishing, or optimized FIB sectioning, are commonly applied. SCM is utilized for both failure analysis and process development.

Environments: air, glovebox

Recommended probes: SCM-PIT-V2, SCM-PTSI, MESP-V2, MESP-RC

Related modes: SSRM, sMIM



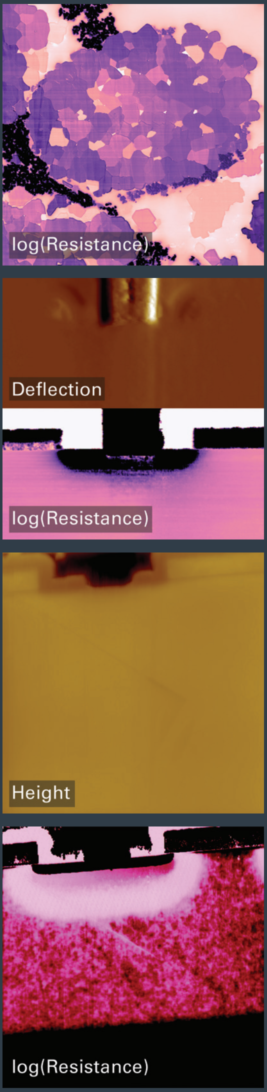
(from top) SCM carrier profile images on Si CCD sensor, Si SRAM memory, two adjacent transistors (top surface), and a cross-sectioned PMOS transistor. Scan sizes (X): 10 µm, 22 µm, 1 µm, 600 nm.

Scanning Spreading Resistance Microscopy (SSRM)

SSRM simultaneously collects data on electrical resistivity and sample height, and is of particular interest for samples where the resistivity (or conductivity) varies over many orders of magnitude. Semiconductor samples and devices fall into this category, and SSRM provides access to their concentration, which can vary from 1015 to 1021 atoms/cm^3.

Like C-AFM, SSRM uses a conductive tip in contact with the sample and the current induced by an applied voltage is measured. To expand the dynamic range in SSRM, a logarithmic current amplifier is used. This makes SSRM extremely useful for samples where linear amplifiers either saturate or do not have the required sensitivity. When applied on semiconductor structures, a reference sample with known carrier concentration is often used to quantify SSRM data into carrier-concentrion maps. Single-nanometer spatial resolutions have been reported.

Along with SCM and sMIM, SSRM is one of three commonly applied methods for carrier profiling on semiconductor devices. Of these three modes, SSRM has the highest spatial resolution. It can be applied on both silicon and compound semiconductor devices. Sample cross-sectioning techniques, such as precision cleaving, polishing, or optimized FIB sectioning, might be required to provide access to the area of interest. SSRM can also be applied to nanomaterials with strong variations in electrical conductivity, such as battery anodes and cathodes.



(from top) Cross-sectioned battery cathode, and carrier concentration maps in cross-sectioned Si transistor and Si DMOSFET device. Scan sizes (X): 15 µm, 6 µm, 12 µm.

Environments: air, glovebox

Recommended probes: DDESP, DDESP-FM, AD-40-AS, AD-2.8-AS

Related modes: SCM, sMIM

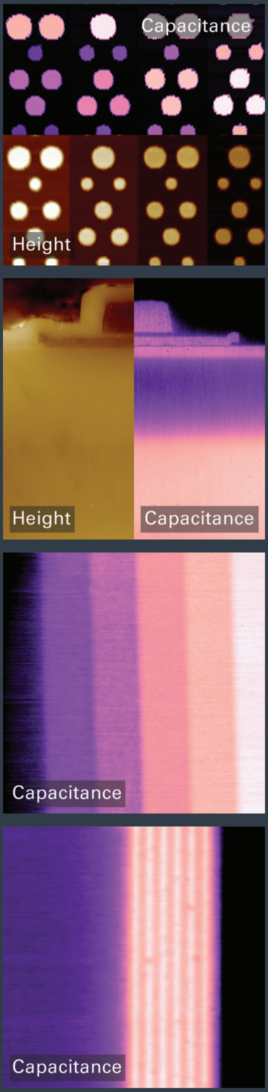
Scanning Microwave Impedance Microscopy (sMIM)

The sMIM mode is a near-field AFM technique that uses an RF generator (operating at ~3 GHz) and a specialized, shielded AFM probe to measure local changes in capacitance and conductance. The measured capacitance directly relates to charge carrier concentration or variations in the dielectric constant.

Operating in contact mode, sMIM-C and sMIM-R channels represent static variations in sample capacitance and resistivity. Similar to SCM, an AC voltage can be applied between tip and sample to get dC/dV. The dC/dV amplitude gives the magnitude of charge-carrier concentration and the dC/dV phase gives the type of the carriers (p- or n-type). Point spectroscopy can be performed by ramping the DC voltage to obtain C-V and R-V spectra. As sMIM uses reflecting microwave signals, no electrical back contact is required, which simplifies sample preparation.

The sMIM mode is ideal for probing capacitance or resistivity variations in nanomaterials, including 1D and 2D materials, semiconductors, dielectrics and ferroelectrics, photovoltaic materials, and polymers. The method is well suited for highly sensitive carrier profiling in semiconductors, as well as to map out variations in dielectric constant or thickness. On certain samples, sMIM can also reveal sub-surface features.

Environments: air, glovebox
Recommended probes: SMIM-150
Related modes: PeakForce sMIM, SCM, SSRM



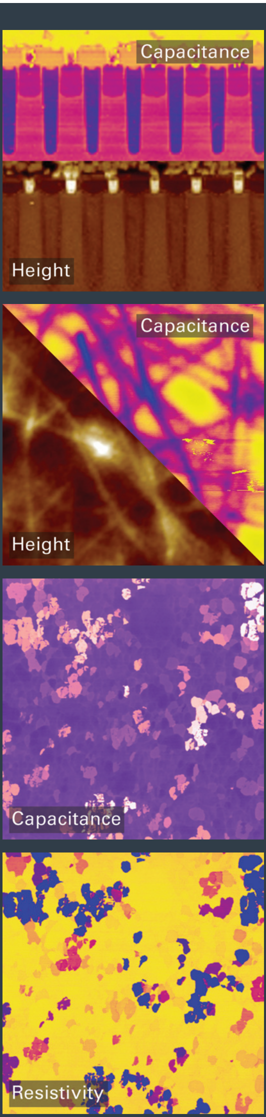
(from top) Array of small capacitors, and carrier profiling on cross-section of PIN diode, Si sample with staircase dopant profile, and sample with alternating doped Si/SiO_2 layers. Scan sizes (X): 46 µm, 25 µm, 13 µm, 2 µm.

PeakForce Scanning Microwave Impedance Microscopy (PeakForce sMIM)

PeakForce sMIM was specifically designed for local electrical impedance mapping on soft and fragile samples. Similar to standard sMIM, both capacitive and resistive information is acquired. However, PeakForce sMIM offers the ability to directly correlate quantitative nanomechanical and nanoelectrical property imaging. Since it is based on PeakForce Tapping operation, it is non-destructive and avoids probe tip contamination caused by lateral forces.

PeakForce sMIM can be used either to map capacitance, resistivity, topography, and mechanical properties, or to perform electrical spectroscopy (e.g., C-V spectroscopy). When imaging, the electrically conductive probe is scanned over the surface as a feedback loop keeps peak force on the tip constant. The impedance measurements can be performed with or without an applied tip-sample voltage. For spectroscopy, tip location is fixed and sample bias is ramped.

As a PeakForce Tapping–based mode, PeakForce sMIM is ideal for soft and fragile samples, such as organic photovoltaics, conductive nanotubes, and nanoparticles. It is particularly useful for applications that require correlated nanomechanical and nanoelectrical property imaging. On certain samples, this mode provides better repeatability and consistency than standard sMIM, making PeakForce sMIM preferred unless lateral force is specifically required.



(from top) Cross-sectioned IGBT devices, silver nanowires embedded in PET polymer, and iron oxide nanoparticles. Scan sizes (X): 12 µm, 2 µm, 5 µm.

Environments: air, glovebox
Recommended probes: SMIM-150
Related modes: sMIM, SCM, SSRM

Piezoresponse Force Microscopy (PFM)

Ferroelectric materials switch polarization under an applied electric field and maintain their state when the field is removed. To understand these materials, it is important to characterize their behavior at the nanoscale. Piezoresponse force microscopy (PFM) is ideal for this, since it combines imaging of the piezoelectric response with spectroscopy and domain manipulation, all localized to the nanoscale by an AFM probe tip.

In PFM, a sinusoidal drive voltage is applied between a conductive AFM probe and the sample substrate. The inverse piezoelectric effect then causes the sample to expand and contract sinusoidally, which is detected by an AFM probe in contact with the sample. PFM phase data can help to determine the electrostrictive coefficient sign. Since the AFM can measure deflection normal to the sample surface or parallel to the surface, it is possible to simultaneously measure two orthogonal components of the piezoelectric displacement vector.

PFM can be applied to map the piezoelectric and ferroelectric domains on a variety of samples, with both a positive (such as PZT) or negative (such as PVDF) electrostrictive coefficient. Both the in-plane and out-of-plane domain information can be collected. If the material is ferroelectric, a large enough applied voltage can cause the domain polarization to flip. Spectroscopy modes, such as SS-PFM and DataCube PFM, enable the study of these domain polarization dynamics.

Environments: air, liquids, glovebox
Recommended probes: MESP-RC, MESP-V2, SCM-PIT-V2, DDESP-FM
Related modes: SS-PFM

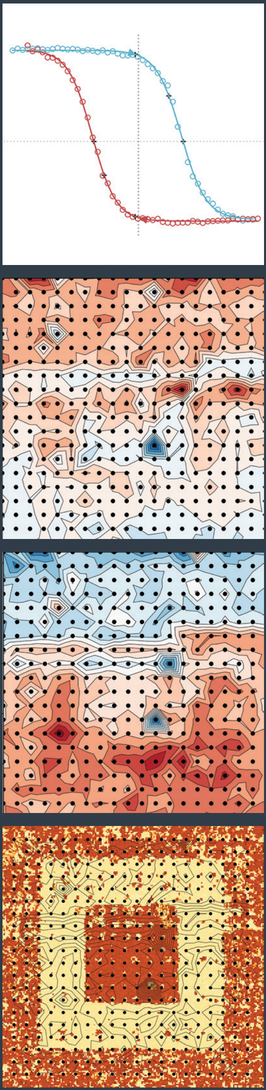
(from top) Thin films of BFO (after domain manipulation), PMN-PT, and LTO. (bottom) DataCube PFM Phase switching displayed vs. applied voltage on a BFO film. Scan sizes (X): 5 µm, 10 µm, 1 µm, 1 µm.

Switching Spectroscopy PFM (SS-PFM)

SS-PFM improves the accuracy and sensitivity of PFM spectroscopy by separating measurements of the PFM response during read voltages from those during write voltages. It provides accurate nanoscale characterization of the properties of ferroelectric materials and enables hyperspectral maps of ferroelectric key parameters, revealing information about XY heterogeneity and its correlation with other properties.

SS-PFM improves signal-to-noise ratio by applying an AC voltage near the cantilever's contact resonance frequency and measuring response, providing accurate measurements of amplitude (*A*) and quality factor (*Q*) of the resonance to enable quantification of the response (*A*/*Q*). It achieves more accurate measurements of *A*, *Q*, and phase by using frequency sweeps and lock-in amplifiers to measure response, enabling longer integration times for ultimate low-signal performance.

SS-PFM has the important ability to characterize low-response ferroelectrics without domain flipping. It is used frequently in piezoelectric materials R&D to characterize the properties of ferroelectrics and quantify key parameters in the ferroelectric hysteresis loop. This is an important capability for comparing material performance across instruments, labs, and probes. Used alone or in conjunction with other modes, SS-PFM provides insight into the relationship between material microstructure and polarization switching.



(from top) PZT film ferroelectric hysteresis loop, coercive voltages V_0^- and V_0^+ extracted from loops, and V_0^+ map overlaid on PFM phase map. Scan size (X): 6 µm.

Environments: air, glovebox

Recommended probes: MESP-V2, SCM-PIT-V2, SCM-PtSi, DDESP-FM

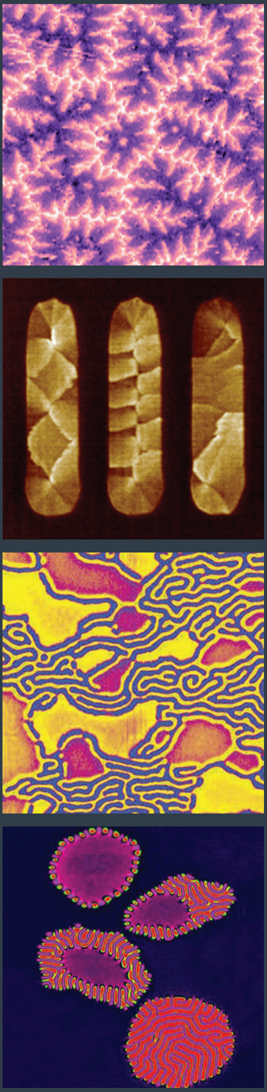
Related modes: PFM

Magnetic Force Microscopy (MFM)

MFM is a high-sensitivity, high-spatial-resolution method for mapping magnetic field distribution on surfaces. MFM has a wide range of applications in various fields, including materials science, magnetism, data storage, and even life sciences. Magnetic domain information is directly correlated to the sample's topography, and can also be acquired while applying external magnetic fields to the sample.

In MFM, a magnetically coated probe tip interacts with the magnetic field gradients emanating from the sample, inducing a force on the tip. Detection is performed by phase, amplitude, or frequency measurement of the resonating cantilever. To ensure that the measured force is primarily due to magnetic interactions, MFM is typically performed in a two-pass approach called *LiftMode*.

MFM is ideal to acquire high-resolution maps of magnetic field distribution. MFM can be applied to virtually any type of sample, since TappingMode is used to acquire the topography map. To obtain the desired signal sensitivity without modifying the sample magnetization, MFM probes are available with different magnetic moment and/or coercivity.



(from top, all MFM phase) Nickel alloy, Permalloy, Co-Pd multilayers showing clear domain structures, and magnetic heterostructures (Courtesy: AG Hellwig, TU Chemnitz). Scan sizes (X): 13 μm, 30 μm, 9 μm.

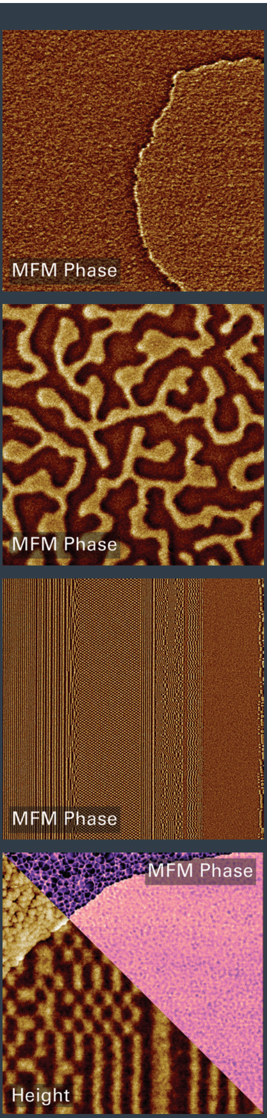
Environments: air, glovebox
Recommended probes: MESP-V2, MESP-RC
Related modes: EFM, PeakForce MFM, TR-MFM

PeakForce Magnetic Force Microscopy (PeakForce MFM)

In PeakForce MFM, the capabilities of MFM and PeakForce QNM are combined. PeakForce MFM provides simultaneous acquisition of topography, magnetic field distribution, and mechanical properties, such as adhesion, modulus, and deformation.

Similar to standard MFM, PeakForce MFM uses a two-pass approach. In a first pass (main scan) topography is acquired, while in the second pass (LiftMode scan) cantilever oscillation amplitude and phase are acquired, representing magnetic field variations in the sample. In PeakForce MFM, the topography scan is done in PeakForce Tapping, providing simultaneous extraction of mechanical properties (i.e., modulus, adhesion, dissipation, indentation). This approach also allows the use of cantilevers with lower spring constant, resulting in lower force regimes and enhanced magnetic field detection sensitivity (scaling with Q/k).

Imaging of magnetic domains with PeakForce MFM can be of interest in material science, magnetics, data storage, and even some life sciences applications. PeakForce MFM can be preferred over conventional MFM when there is an interest in correlating mechanical properties to magnetic properties, or when a higher sensitivity or higher spatial resolution is required.



(from top) 5x PtCoPt multilayer and 25x PtCoPt multilayer (courtesy K. Bouzehouane, Université Paris-Saclay, Thales), 20 TB HDD, 20 TB HDD with ferrofluid drop. Scan sizes (X): 3 µm, 2 µm, 10 µm, 1 µm.

Environments: air, glovebox
Recommended probes: PFMFM-LM, MESP-V2
Related modes: MFM, EFM, PeakForce EFM, TR-MFM

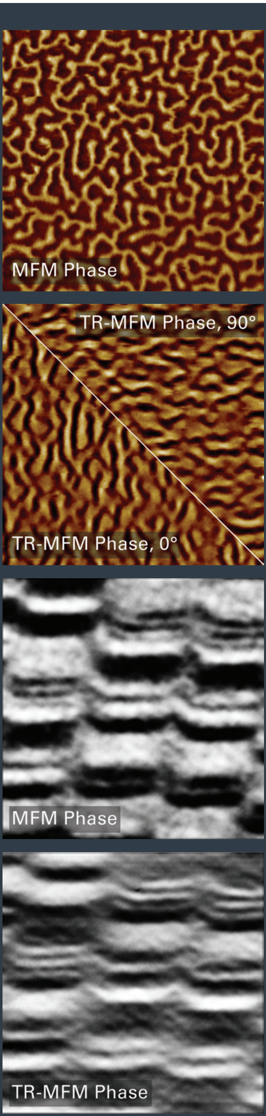
Torsional Resonance MFM (TR-MFM)

TR-MFM maps magnetic field distribution with a sensitivity to the lateral fields (in contrast to the vertical fields in conventional MFM). Magnetic domain information is directly correlated to sample topography, and can also be acquired while applying external magnetic fields to the sample.

In conventional MFM, the cantilever is oscillated vertically, whereas in TR-MFM this oscillation is replaced by an (essentially) horizontal one, creating a torsional mode. The tip selectively detects the field that has a force gradient parallel to the tip. These in-plane force gradient components can be measured with the same resolution and signal-to-noise ratio as flexural modes at the same location, providing magnetic information complementary to conventional MFM.

Studies evaluating the use of TR-MFM for imaging magnetic structures have shown its ability to obtain magnetic contrast free from topographic influence, with similar lateral resolution as standard MFM. When combined with standard MFM, TR-MFM is especially useful for studying the 3D nature of magnetic field distributions, as it provides access to the lateral field information.

Environments: air, glovebox
Recommended probes: MESP-V2, SCM-PIT-V2
Related modes: MFM, PeakForce MFM



(from top) Magnetic domains on perpendicular magnetization multilayer [Pt/Co/Pt]x25 (courtesy: K. Bouzehouane, Université Paris-Saclay, Thales), and on magnetic tape. Scan sizes (X): 3.6 µm, 3.8 µm, 3 µm.

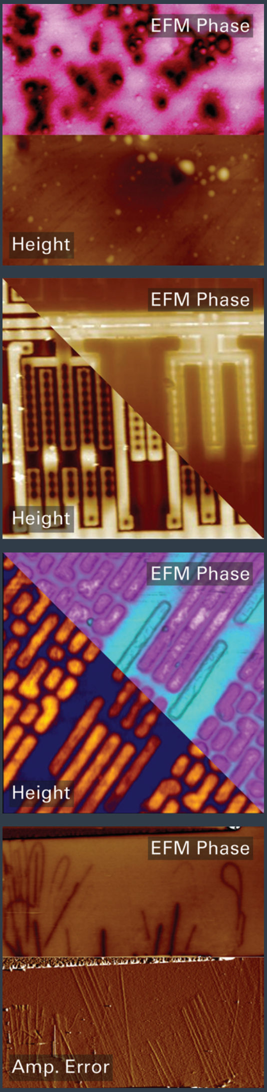
Electric Force Microscopy (EFM)

EFM is a high-sensitivity, high-spatial-resolution method for mapping electric field distribution on surfaces. EFM has a wide range of applications in various fields, including materials science, 2D materials, and semiconductor materials and devices. Electric field information is directly correlated to the sample's topography, and directly relates to the charges present at or near the sample surface.

In EFM, a metal-coated probe tip interacts with the electric field gradients emanating from the sample, inducing a force on the tip. Detection is performed by phase, amplitude, or frequency measurement of the resonating cantilever. To ensure that the measured force is primarily due to electric interactions, EFM is typically performed in a two-pass approach (LiftMode).

EFM is ideal to acquire high-resolution maps of electric field distribution. EFM can be applied to virtually any type of sample, since TappingMode is used to acquire the topography map. EFM is particularly effective for mapping the nanoscale distribution of electrical charges at or near the sample surface, as well as the electric field emanating from an active, operating electric device.

Environments: air, glovebox
Recommended probes: MESP-V2, SCM-PIT-V2, MESP-RC
Related modes: MFM, PeakForce EFM, KPFM



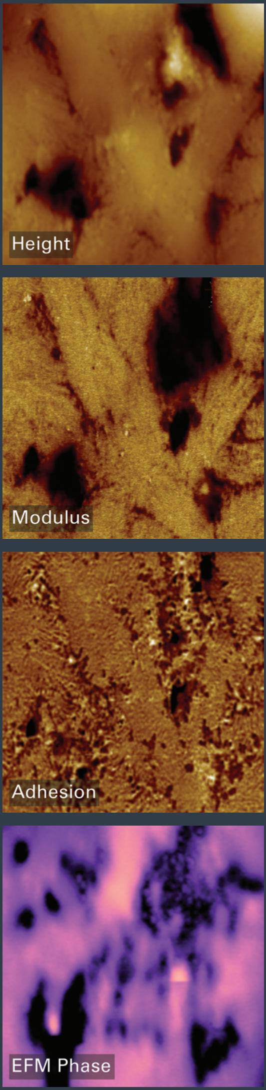
(from top) Carbon nanoparticles embedded in polymer, faulty (leaky) IC device, SRAM memory device, and carbon nanotube network. Scan sizes (X): 5 µm, 80 µm, 1.4 µm, 10 µm.

PeakForce Electric Force Microscopy (PeakForce EFM)

In PeakForce EFM, the capabilities of EFM and PeakForce QNM are combined. PeakForce EFM provides simultaneous acquisition of topography, electric field distribution, and mechanical properties, such as adhesion, modulus, and deformation.

Similar to EFM, PeakForce EFM uses a two-pass approach. In a first pass (main scan) topography is acquired, while in the second pass (LiftMode scan), cantilever oscillation amplitude and phase are acquired, representing the electric field variations in the sample. However, in PeakForce EFM, the TappingMode topography scan is replaced with a PeakForce Tapping scan, providing simultaneous extraction of mechanical properties (i.e., modulus, adhesion, dissipation, indentation). This approach also allows the use of cantilevers with lower spring constant, which results in lower force regimes and enhanced electric field detection sensitivity (scaling with Q/k).

Imaging of electric fields and charges with PeakForce EFM can be of interest for semiconductors, 2D materials, photovoltaics, perovskites, and general nanoelectrical materials. PeakForce EFM can be preferred over conventional EFM when there is an interest in correlating mechanical properties to electric properties, or when a higher sensitivity or higher spatial resolution is required.



(from top) Thermoplastic vulcanizate with low modulus EPDM, harder polypropylene, and (charged) carbon black nanoparticles. Scan sizes (X): 3 μm, 3 μm, 3 μm.

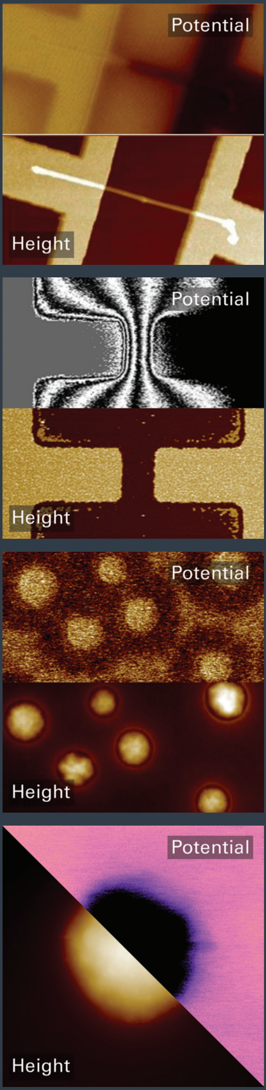
Environments: air, glovebox
Recommended probes: PFMFM-LM, MESP-V2, SCM-PIT-V2
Related modes: EFM, PeakForce MFM, PeakForce KPFM

Amplitude Modulation Kelvin Probe Force Microscopy (AM-KPFM)

KPFM, also referred to as surface potential microscopy, is a method to map out the surface (electrostatic) potential, contact potential difference (CPD) or work function on samples. Along with C-AFM, KPFM is one of most widely used modes for nanoscale electrical studies. AM-KPFM was the first of several KPFM methods implemented on the Bruker AFMs, and is based upon electric force detection.

AM-KPFM is a dual-pass technique. In a first pass, topography is collected in TappingMode, while during the second pass (LiftMode), an AC bias is applied between the tip and sample at the resonance frequency of the AFM cantilever. The modulated electric force is measured using the oscillation of the cantilever, which is used as input to a Kelvin feedback loop where DC bias is adjusted until oscillation amplitude drops to zero. In this way, the applied voltage equals the CPD value of interest.

AM-KPFM has found broad applications in corrosion studies of alloys, evaluation of photovoltaic effects on solar cells, and surface analysis. This mode can also be applied to map the potential distribution in active samples, such as operating semiconductor devices. AM-KPFM is an easy-to-use KPFM method, but for enhanced accuracy or resolution, consider one of the other KPFM implementations: FM-KPFM, PeakForce KPFM, or HV-KPFM.



(from top) InP nanowire between two differently biased contacts, between differently biased (isolated) contacts, PS-LDPE, and toner particle. Scan sizes (X): 2 m, 25 m, 5 m, 10 m.

Environments: air, glovebox
Recommended probes: MESP-V2, SCM-PIT-V2, MESP-RC
Related modes: FM-KPFM, PeakForce KPFM, HV-KPFM, EFM, PeakForce EFM

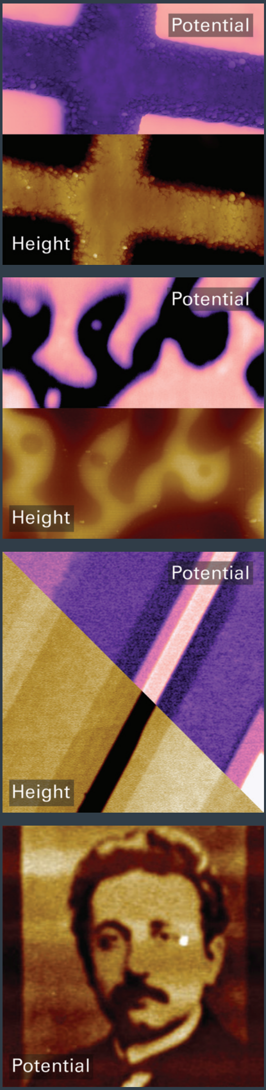
Frequency Modulation Kelvin Probe Force Microscopy (FM-KPFM)

KPFM, also referred to as surface potential microscopy, is a method to map out the surface (electrostatic) potential, contact potential difference (CPD) or work function. Today, along with C-AFM, KPFM is one of most widely used AFM methods for nanoscale electrical studies. FM-KPFM is based upon electric force gradient detection and provides improved accuracy and spatial resolution over AM-KPFM.

Unlike AM-KPFM and PeakForce KPFM, FM-KPFM does not use a dual-pass technique. During the TappingMode scan, a low-frequency AC bias is applied between the tip and sample, modulating the resonant frequency and creating sidebands. The modulated electric force gradient is measured using two cascaded lock-in amplifiers and is used as input to a Kelvin feedback loop, adjusting a DC bias until the oscillation amplitude drops to zero. In this way, the applied voltage equals the CPD value of interest.

FM-KPFM has found broad applications in corrosion studies of alloys, evaluation of photovoltaic effects on solar cells, mapping of trapped charges in dielectrics, and surface analysis. This mode can also be applied to map the potential distribution in active samples, such as operating semiconductor devices. FM-KPFM provides higher accuracy, repeatability and spatial resolution than AM-KPFM, and using a single pass also reduces image acquisition time.

Environments: air, glovebox
Recommended probes: PFQNE-AL, SCM-PIT-V2
Related modes: AM-KPFM, PeakForce KPFM, HV-KPFM, EFM, PeakForce EFM



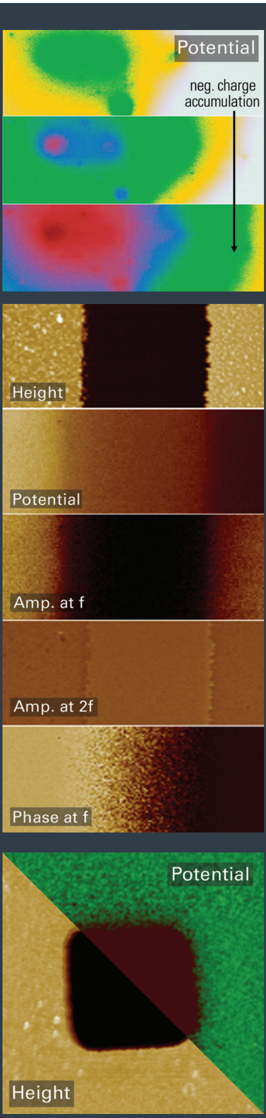
(from top) Al lines on Cu, PS-PMMA polymer, hBN, and SiO_2 with intentionally injected charges using nanolithography. Scan sizes (X): 25 µm, 8 µm, 6 µm, 16 µm.

High-Voltage Kelvin Probe Force Microscopy (HV-KPFM)

HV-KPFM belongs to the family of KPFM methods to map out surface (electrostatic) potentials. In HV-KPFM, electrostatic potential can be measured beyond ±10 V, which is often a limit in other KFPM implementations. HV-KPFM can be applied to a variety of samples.

KPFM-HV is a dual-pass technique. In a first pass, topography is collected in a PeakForce Tapping scan that also provides nanomechanical information. On the second pass (LiftMode), an AC bias is applied between the tip and sample. The vertical deflection signal is simultaneously fed to two synchronized lock-in amplifiers (drive frequency and second harmonic). A calculation is carried out to obtain electrostatic potential difference. HV-KFPM allows measurement of voltages in the range of ±200 V.

HV-KPFM is of particular interest for samples where the surface potential values vary over a large range. A typical example is the measurement of trapped (immobilized) charges in insulators. As HV-KPFM applies a so-called open-loop detection, it can also be of interest in experiments where avoidance of a closed-loop Kelvin loop is desired, for example to overcome the limited response time. KPFM experiments that require a high sensitivity generally use one of the other methods, such as PeakForce KPFM.



(from top) PDMS, Al, and Au films on Si showing the different measurables that compose the potential property channel, and on Si_3N_4 film on Au in liquid. Scan sizes (X): 30 µm, 10 µm, 6 µm.

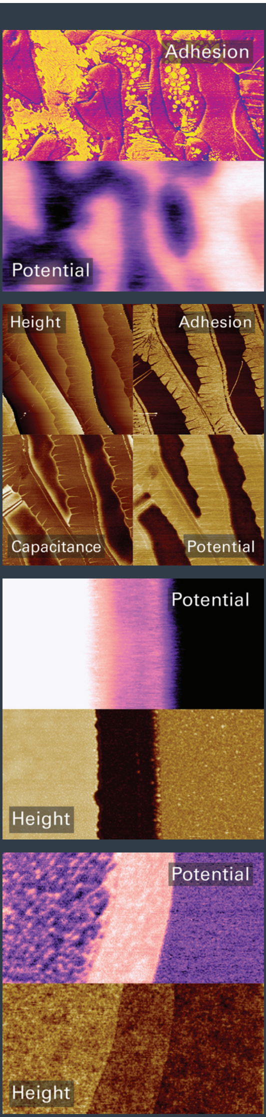
Environments: air, glovebox
Recommended probes: SCM-PIT-V2
Related modes: AM-KPFM, FM-KPFM, PeakForce KPFM, EFM, PeakForce EFM

PeakForce Kelvin Probe Force Microscopy (PeakForce KPFM)

In PeakForce KPFM, the capabilities of KPFM and PeakForce QNM are combined. PeakForce KPFM provides simultaneous acquisition of topography, surface potential (or CPD) and mechanical properties, such as adhesion, modulus and deformation. PeakForce KPFM can be operated with both amplitude modulation (AM) or frequency modulation (FM) detection, and can provide higher sensitivity than the TappingMode–based AM-KPFM and FM-KPFM.

PeakForce KPFM is a dual-pass technique. In a first pass, topography is collected using PeakForce Tapping, providing simultaneous extraction of mechanical properties (modulus, adhesion, dissipation, indentation). During the second pass (LiftMode), the surface potential is detected using a Kelvin feedback loop with amplitude modulation (AM) or frequency modulation (FM) detection.

PeakForce KPFM can be applied to a broad range of applications, including corrosion of alloys, photovoltaic effects on solar cells, trapped charges in dielectrics, potential distribution in active devices, and surface analysis. PeakForce KPFM is of particular interest for composite samples, to directly correlate electrical properties to mechanical properties. Compared with other KPFM implementations, PeakForce KPFM provides higher sensitivity, as it can be operated with softer cantilevers that have higher sensitivity.

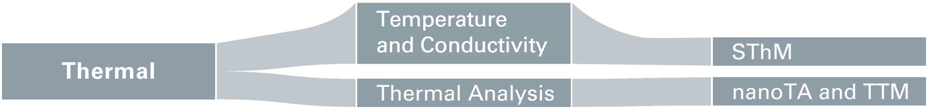


(from top) Graphene on SiC, SnB solder, Al and Au on Si, and hBN. Scan sizes (X): 20 µm, 4 µm, 12 µm, 5 µm.

Environments: air, glovebox
Recommended probes: PFQNE-AL, SCM-PIT-V2
Related modes: AM-KPFM, FM-KPFM, HV-KPFM, EFM, PeakForce EFM

Thermal Modes

Thermal modes are used to characterize the nanoscale thermal properties or responses of materials. These modes require local temperature control and/or local thermal sensing.

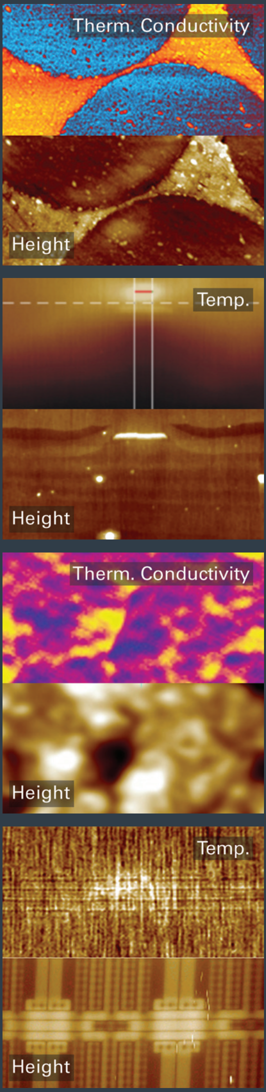


Scanning Thermal Microscopy (SThM)

In SThM, a thermal probe is used to measure variations in sample temperature or thermal conductivity on the nanoscale. SThM can be applied to both active samples, such as operating semiconductor devices, or passive samples. Thermal information is directly correlated to the sample's topography.

In SThM, a thermal probe with a resistive element near the tip apex is scanned across the sample surface in contact mode or PeakForce Tapping. This resistor acts as a thermistor and is part of a Wheatstone bridge setup. SThM probes can be used to detect temperature variations with high sensitivity, perform heated-tip experiments, and detect variations in thermal conductivity of the sample. A voltage applied to the probe slightly heats the probe, which enables detection of local variations in thermal conductivity through monitoring of probe resistance.

SThM is used to acquire high-resolution maps of temperature distribution or thermal conductivity variations. This mode can be applied to a wide variety of samples. SThM can be operated on both passive samples and active samples, such as an operating semiconductor or data storage device. The capability to heat the tip can also be applied to perform heated-tip AFM experiments, such as heat-assisted nanolithography.



(from top) Fibers embedded in epoxy, active edge-emitting laser structure, 20 nm nanoparticles, and active IC device with failure. Scan sizes (X): 8 µm, 10 µm, 450 nm, 90 µm.

Environments: air, glovebox

Recommended probes: VITA-xx-GLA-1 (where xx = AFM model)

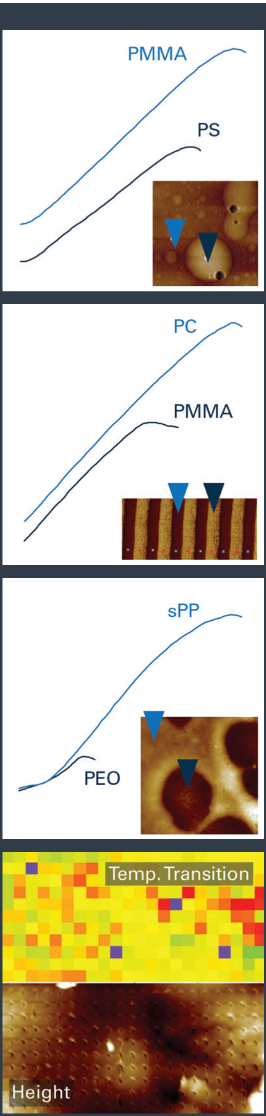
Related modes: nanoTA, TTM, Nanolithography

Nanothermal Analysis (nanoTA) and Temperature Transition Microscopy (TTM)

The nanoTA mode is an analytical technique used to determine the melting or glass transition temperature of a material based on the penetration of a heated AFM cantilever into the material. As a point spectroscopy method, nanoTA is performed in positions selected by the user in a previously acquired AFM map. When performed in an array of positions, the method is referred to as TTM.

In nanoTA, a thermal probe is used to image the topography of a sample in contact mode. At user-selected positions, a small region of the sample around the point of contact is heated by increasing the voltage applied to the thermal probe. During the thermal ramp, probe deflection shows an initial increase due to thermal expansion of the sample. When its thermal transition temperature is reached, the sample softens or melts, leading to probe penetration into the sample and a sudden decrease in probe deflection.

Both nanoTA and TTM provide a nanoscale equivalent to standard bulk thermal techniques, such as DSC and TMA, to which these modes show a high degree of correlation. While nanoTA and TTM can be applied to a variety of samples, the most common are polymer blends and composites.



(from top) nanoTA spectra on PS-PMMA, polycarbonate-PMMA, PEO-sPP blend, and TTM map on pharmaceutical copolymer. Scan sizes (X): 3.5 µm, 10 µm, 10 µm, 10 µm.

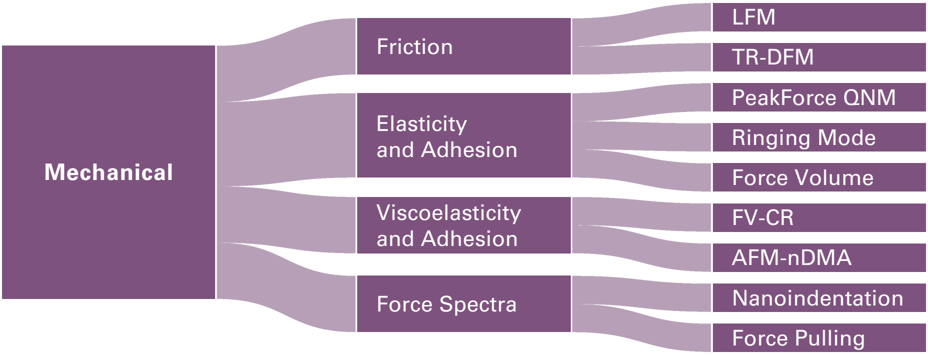
Environments: air, glovebox

Recommended probes: VITA-xx-NANOTA-200, VITA-xx-NANOTA-300 (where xx = AFM model)

Related modes: SThM

Mechanical Modes

Mechanical modes are used to characterize the nanoscale (elastic, plastic and viscoelastic) mechanical properties and responses of materials. Quantitative mechanical measurements are attainable in some cases.

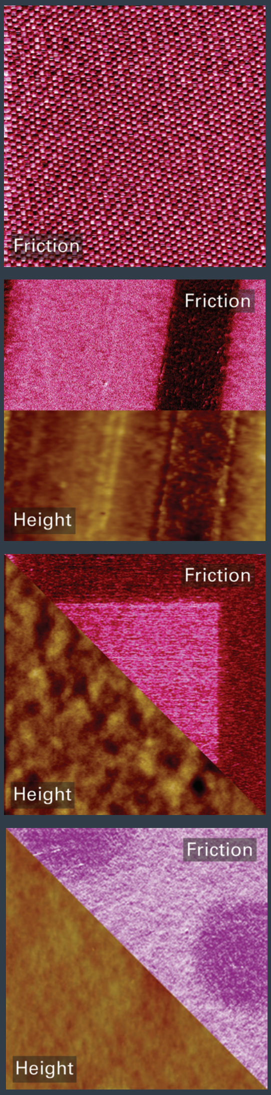


Lateral Force Microscopy (LFM)

LFM, also referred to as *friction force microscopy*, provides access to the frictional properties of the sample. The lateral bending of the cantilever is used to access frictional characteristics.

LFM is performed in contact mode. In addition to measuring the vertical bending of the cantilever, the lateral bending is also recorded. To decouple vertical and lateral forces, the scan direction is typically set to 90 degrees, and both the trace and retrace data are collected. The difference is used to represent the surface friction characteristics of a sample. The origin of lateral forces can arise from changes in the frictional coefficient of a region on the sample surface or from onsets of changes in height.

LFM can be applied to all samples that can be imaged in contact mode, and operates in both air and liquid. Its application can be challenging on soft samples where dragging the sharp probe across the sample can cause damage. It is therefore more suitable for harder surfaces, such as hard polymers, ceramics, metals, and semiconductors. LFM is a fast mode that can be applied for high-resolution imaging (down to atomic lattice resolution) as well as large-area imaging.



(from top) HOPG atoms, MLPE polymer, patterned brush polymer, and locally functionalized Si. Scan sizes (X): 10 nm, 10 µm, 8 µm, 8 µm.

Environments: air, liquids, glovebox
Recommended probes: DNP-A, DNP-C, MLCT-F, RESPA-20
Related modes: Contact Mode, TR-DFM

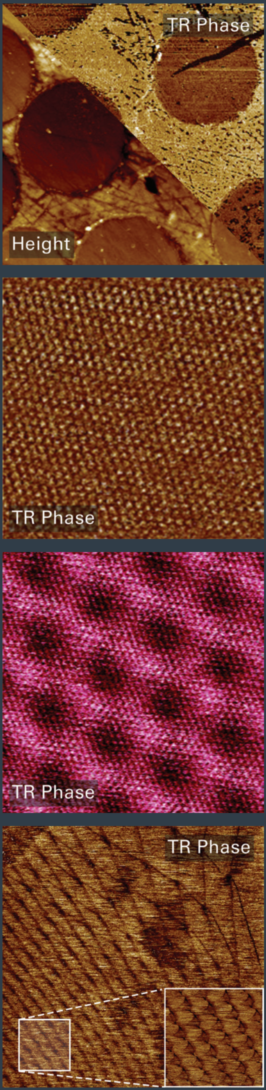
Torsional Resonance Dynamic Friction Microscopy (TR-DFM)

TR-DFM is a unique mode providing highly sensitive frictional property maps while acquiring topography images in contact mode. When using small torsional resonance oscillation, ultrahigh spatial resolution can be obtained while maintaining high sensitivity. This mode is sometimes also referred to as d*ynamic friction microscopy* or *dynamic lateral force microscopy*.

In TR-DFM mode, a torsional modulation is applied during contact mode operation. The torsional amplitude and phase are monitored and are highly sensitive to tribological properties, such as friction. When operated at or near the contact torsional resonance (TR) frequency, the TR friction force images show much higher sensitivity than conventional contact mode–based friction measurements. The TR friction force images are also less affected by surface topography and are almost independent of the scanning direction of the tip, in contrast with conventional contact mode friction force measurements.

TR-DFM is applied for friction measurements that require the highest sensitivity levels. This mode has also proven to be extremely useful for high-resolution imaging of 2D materials and the moiré patterns they can create when stacked. Resolution down to the atomic scale can be obtained.

Environments: air, liquids, glovebox
Recommended probes: FESPA-V2, AD-2.8-AS, AD-2.8-SS
Related modes: TR-Mode, LFM



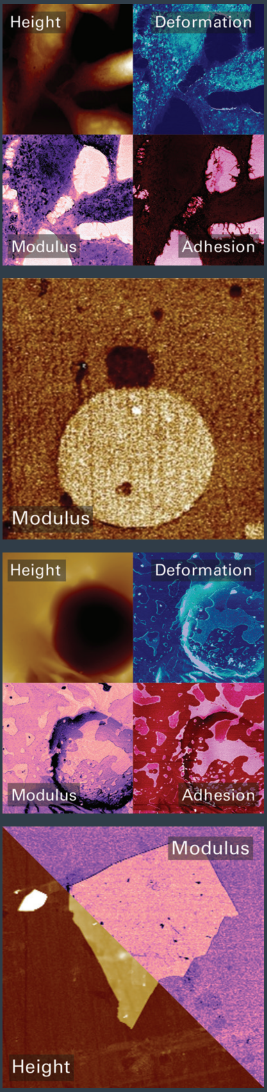
(from top) Cross-sectioned carbon fibers in epoxy, HOPG atoms, moiré pattern with underlying graphene lattice (courtesy M. Pendharkar et al., Stanford University), and moiré pattern of stacked 2D materials. Scan sizes (X): 20 µm, 10 nm, 10 nm, 1 µm (inset 200 nm).

PeakForce Quantitative Nanomechanics (PeakForce QNM)

PeakForce QNM can non-destructively map important mechanical properties, such as Young's modulus, adhesion, energy dissipation, and deformation, while providing detailed high-resolution surface topography.

PeakForce QNM utilizes real-time force-distance curves collected during PeakForce Tapping operation to extract quantitative nanomechanical information by fitting to specific models. Ramp frequencies range from 100–8000 Hz. Interaction forces are meticulously controlled using maximum (peak) force control, ensuring non-destructive measurements. Pre-calibrated probes can further enhance the reliability and accuracy of nanomechanical mapping results.

PeakForce QNM provides quantitative data to complement topographic images and is essential for detailed nanomechanical characterization in diverse scientific applications. Its basis in PeakForce Tapping and ability to be used on a range of soft to hard materials (~1 kPa up to 100 GPa) makes PeakForce QNM exceptionally versatile and applicable across a wide range of fields, including materials science, biomaterials, polymers, and advanced materials. For life sciences, it facilitates high-speed, quantitative mapping of live cells and the study of biological interactions. In polymers and composites, it aids in characterizing mechanical properties and mapping molecular interactions.



(from top) Tri-polymer PS-PE-PP blend, cells, PS-PMMA-PVC polymer blend, and perovskite nanosheet. Scan sizes (X): 6 µm, 50 µm, 5 µm, 20 µm.

Environments: air, liquids, glovebox

Recommended probes: SCANASYST-AIR(-HPI), SCANASYST-AIR-HPI-30, RTESPA-150-30, RTESPA-300-30, RTESPA-525-30, SCANASYST-FLUID(+), PFQNM-LC-V2

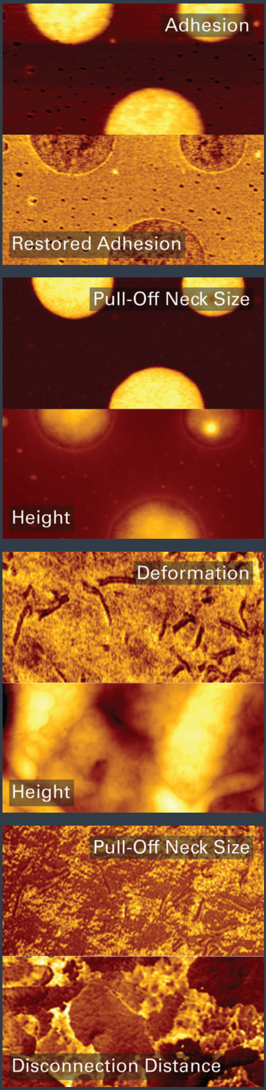
Related modes: TR-Mode, LFM

Ringing Mode

Ringing mode is a powerful extension of PeakForce QNM that simultaneously records up to eight quantitative compositional imaging channels in a single scan, providing previously inaccessible information on surface adhesion properties: restored adhesion, adhesion height, zero-force height, disconnection height, pull-off neck size, disconnection distance, disconnection energy loss, and dynamic creep phase shift.

In ringing mode, the ringing signal resulting from surface detachment (or "pull-off") of the AFM probe in PeakForce Tapping is anlayzed in detail for every force-distance cycle. This signal provides new insights into the often-complex unbinding process of surface molecules. As a result, ringing mode extends the available data in PeakForce QNM to provide up to fourteen different data types.

Ringing mode is of particular interest for characterizing neck formation in soft matter, evaluating tensile deformation of surface layers at the nanoscale. Pull-off neck size provides insights into processes, such as polymer necking or membrane tubule formation in cells. Ringing mode can also be applied to study the extension length of surface molecules stretched from the surface by the AFM probe, to measure the energy dissipation during tip-sample interactions, and to collect enriched adhesion information.



(from top, in pairs) PS/LDPE polymer and human melanoma cell. Scan sizes (X): 3 µm, 2 µm.

Environments: air, liquids, glovebox

Recommended probes: SCANASYST-AIR(-HPI), SCANASYST-FLUID(+)

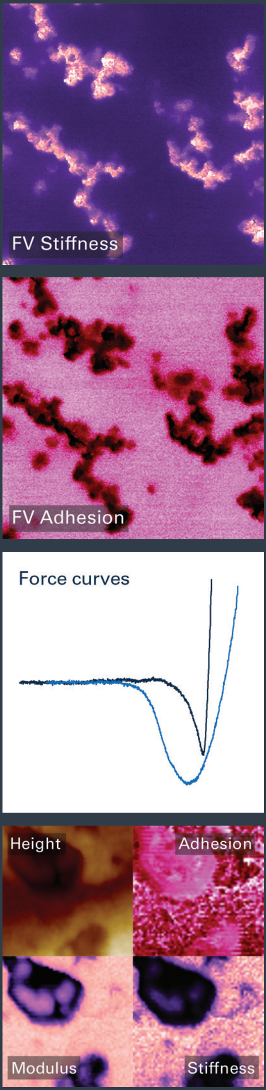
Related modes: PeakForce QNM

Force Volume (FV)

FV provides maps of sample mechanical properties by collecting a volume (data cube) of deflection data over the scan area. The deflection data can be analyzed to provide maps of sample properties, such as modulus, stiffness, and adhesion. FV data cubes can also be used to investigate more specialized behaviors, such as deviations from the standard contact mechanics models and specific binding in liquid.

FV collects a force curve at each XY pixel in an image. The system starts with the tip above the surface and ramps the Z position to bring tip and sample into contact (approach). The Z position and force are recorded and, once a user-defined force is reached, the system retracts the tip (retract). The system then moves on to the next pixel and repeats the process. Combining all of the curves provides two data cubes, one for the approach and one for the retract. These data cubes are then analyzed to provide maps of sample properties.

FV is useful in measuring the elastic modulus and adhesion of samples in both air and liquid over a range of temperatures. It works with a wide selection of probes, allowing the cantilever and tip to be optimized for sample stiffness and adhesion. Since the tip is not in contact with the sample during XY motion, lateral forces are minimized. Minimal lateral forces maximizes tip lifetime and allows operation on soft, delicate samples, such as fibers and nanoparticles, as well as biological cells and tissues.



(from top) Carbon sticky stub images and force curves, and polypropylene impact copolymer images. Scan sizes (X): 2 µm, 1.5 µm.

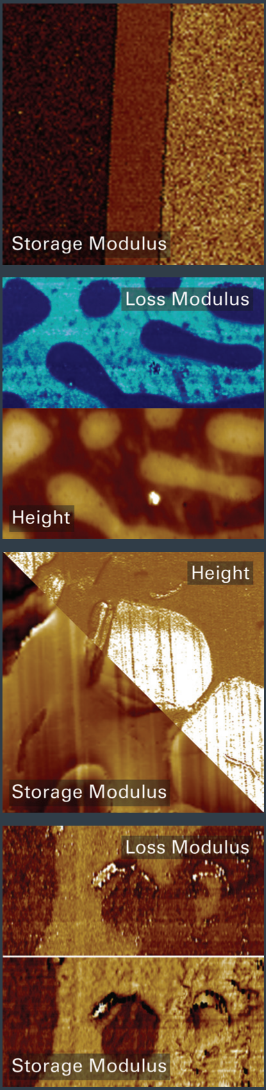
Environments: air, liquids, glovebox
Recommended probes: DNP-A, DNP-B, DNP-C
Related modes: FV-CR, PeakForce QNM, AFM-nDMA

Force Volume Contact Resonance (FV-CR)

FV-CR uses a combination of force mapping through force volume (FV) imaging and contact resonance (CR) frequency sweeps to access quantitative viscoelastic nanomechanical properties. FV-CR offers fast, accessible, and robust nanomechanical measurements. It also inherits FV benefits, i.e., simultaneous acquisition of adhesion and avoidance of lateral forces.

In FV-CR, the system stops to touch the sample surface at each pixel, sweeping the frequency of mechanical excitation. The resonance frequency and shape of the CR peak shifts in response to changes in tip-sample properties, such as stiffness and dissipation. With integrated models and calibration, this results in quantitative determination of viscoelastic properties (e.g., storage modulus, loss modulus, and loss tangent) as well as topography and adhesion data.

FV-CR is applied for the viscoelastic characterization of a variety of samples, from relatively stiff polymers, polymer composites, and metals to harder materials, such as ceramics. FV-CR works better than standard FV on stiff samples, because the CR enhances signal-to-noise ratio. Additionally, FV-CR is preferred over contact mode–based CR methods because there are no lateral forces, maximizing tip lifetime and allowing measurement on more delicate samples. Simultaneous acquisition of adhesion provides additional information and improves the accuracy of estimating contact radius, storage modulus, and loss modulus.



(from top) 50 nm films of aluminum and chromium on silicon, PS-PMMA, microtomed fibers embedded in epoxy, and PE-PP-PS blend. Scan sizes (X): 12 µm, 5 µm, 40 µm, 4 µm.

Environments: air, glovebox

Recommended probes: DDESP-FM-V2, DDRFESPA-40, DDLTESPA-V2

Related modes: FV, AFM-nDMA, PeakForce QNM, PFM

AFM Nanoscale Dynamic Mechanical Analysis (AFM-nDMA)

AFM-nDMA provides quantitative viscoelastic property measurements of nanoscale domains in heterogeneous polymeric materials. AFM-nDMA directly addresses frequency and temperature dependence of viscoelastic properties in the rheologically relevant range (0.1–20 kHz). In contrast with traditional AFM modes, AFM-nDMA compares well with established mechanical characterization techniques while enabling studies of microstructure and bulk properties.

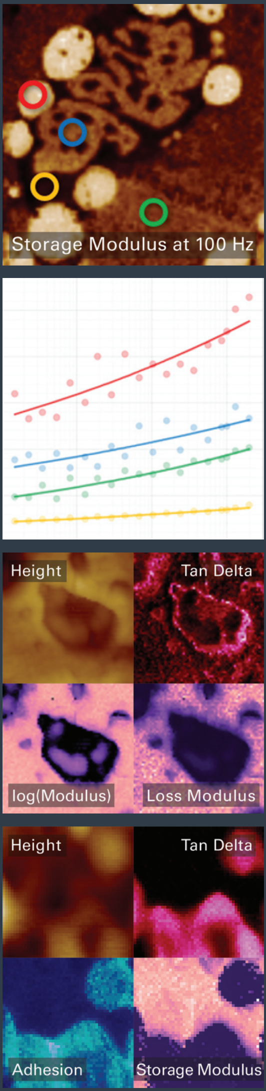
AFM-nDMA includes two main modes of operation. First, viscoelastic moduli images can be collected through single-frequency data collection in force volume mode. Second, frequency sweep measurements are performed in selected positions on the sample. This advanced ramp spectroscopy uses a ramp scripting routine with a series of segments to control preload, relaxation, modulation, and calculation of contact radius. Analysis then leads to the creation of mastercurves, as well as quantitative data for loss tangent, loss modulus, and storage modulus.

AFM-nDMA is the most advanced AFM method for the quantitative characterization of viscoelastic properties at the nanoscale. It is preferred over other AFM-based nanomechanical methods for characterizing a wide variety of polymer and nanomaterial samples.

Environments: air, glovebox

Recommended probes: RTESPA-150-125, RTESPA-300-125, RTESPA-150-30, RTESPA-300-30

Related modes: FV



(from top) Quaternary polymer blend with correlated rheological frequency spectra, PEE, and PC-ABS polymers. Scan sizes (X): 8 µm, 1.5 µm, 1 µm.

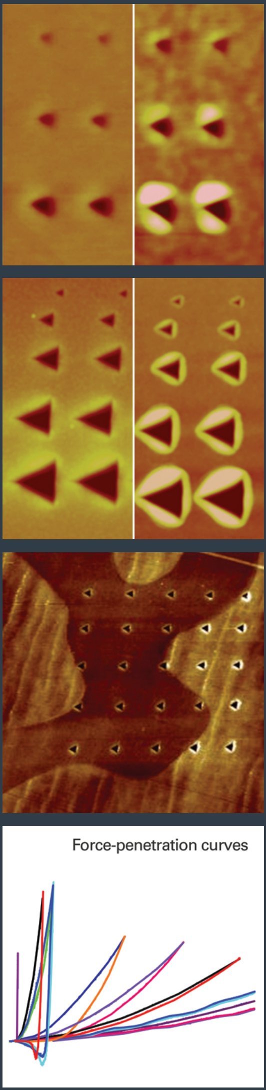
Nanoindentation

Nanoindentation is a point spectroscopy technique where force-distance (or force-separation) spectra are collected to extract the nanoscale mechanical properties of hardness and modulus. The sample surface is typically imaged both prior to indentation for site selection and after indentation for viewing indentation marks. AFM-based nanoindentation has a lot of similarities to instrumented nanoindentation, but differs in the use of a cantilever-based indenter.

In nanoindentation, a pre-calibrated, high-spring-constant cantilever with diamond tip is first used to image the sample surface in TappingMode. This image is used to position the tip in specific locations where force-distance (or force-separation) spectra are collected. The resulting indentation curves are fitted to specific models to extract quantitative nanomechanical properties. Lastly, the same sample surface area is imaged again to obtain an accurate 3D image of the indents, which is used to analyze indentation depth, pile-up, and other details. The same setup can also be applied for wear and scratch tests.

Nanoindentation can be applied on a wide variety of samples in material science and is complementary to mechanical property imaging modes, such as PeakForce QNM, AFM-nDMA, and FV-CR. As the cantilevers typically have a high spring constant, the method is of particular use for materials with high hardness values.

Environments: air, glovebox
Recommended probes: DNISP, DNISP-HS
Related modes: Force Pulling



(from top) Height maps of two DLC and polymer samples indented at various loads, and on TMPC-PS at 275°C. (bottom) Testing on samples with different hardness. Scan sizes (X): 300 nm, 1.5 µm, 3 µm.

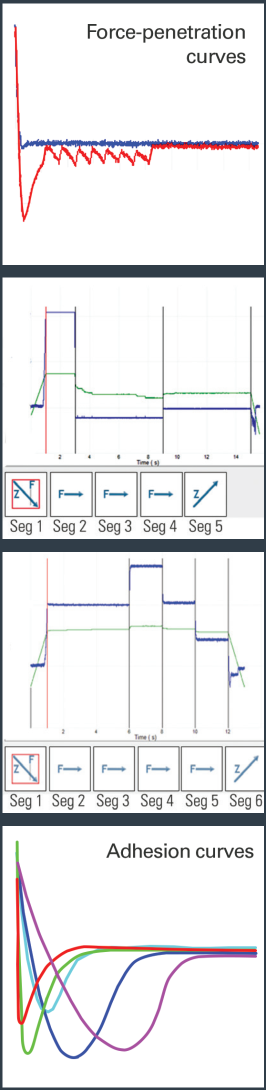
Force Pulling

Force pulling is a point spectroscopy technique where force spectra are collected to extract quantitative nanomechanical property information related to adhesion, molecular unfolding, and single molecule (un)binding events.

In force pulling, a pre-calibrated AFM probe is ramped in the Z direction, bringing it into and out of contact with a sample surface. The retract portion of the resulting force-distance curve is analyzed to obtain quantitative measurements of the forces acting on the probe as it is pulled away from the surface. These forces can provide insights into structural and mechanical properties, such as molecular unfolding and adhesion. AFM probes can also be functionalized with small ligands to study (un)binding forces with specific molecules on a sample surface. In general, the spring constant of the AFM probe used should be closely matched to the type of interaction forces that are being measured.

Force pulling can be applied to a wide range of materials. It is complementary to other mechanical property AFM modes, such as FV and PeakForce QNM that conduct full material property mapping over the entire surface area. Force pulling is often preferred over these other modes in cases where isolated, individual force measurements, or a small array of force curves, are sufficient for studying specific location(s) on a sample surface.

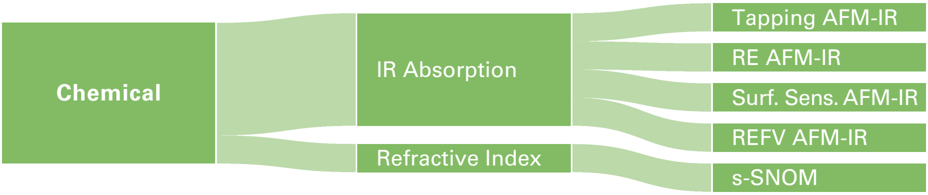
Environments: air, liquids, glovebox
Recommended probes: DNP-B, DNP-C, DDESP-FM-V2
Related modes: Nanoindentation



(from top) Titin force-separation curves, titin force-time script, live fibroblast cell force-time script, and rubber polymer blend adhesion curves.

Chemical Modes

Nanoscale infrared spectroscopy offers a range of chemical analysis capabilities. Bruker's proprietary photothermal AFM-IR technique detects thermal expansion caused by the absorption of infrared radiation and provides nanoscale chemical mapping and spectroscopy directly correlated to FTIR spectroscopy, allowing chemical identification. AFM-IR also works with hyperspectral implementation, where a full IR spectrum is collected at each pixel. Detailed information about the working principles and applications of AFM-IR can be found in A Comprehensive Guide to Photothermal AFM-IR Spectroscopy. A number of AFM-IR modes have been developed to characterize materials more comprehensively.

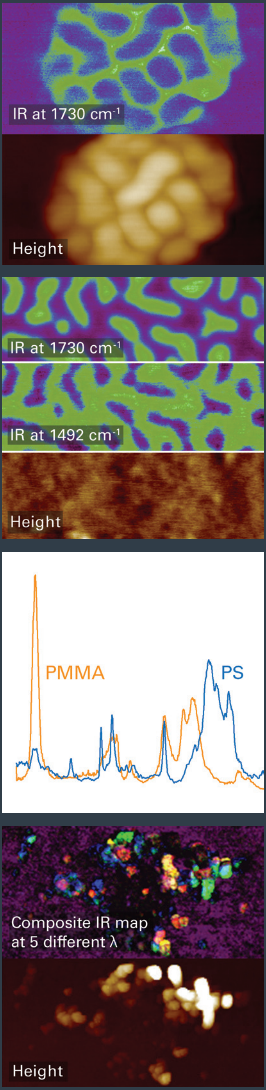


Tapping AFM-IR

Tapping AFM-IR provides simultaneous topography and chemical images with excellent spatial resolution. It measures IR spectroscopy with high sensitivity and extends the applications of nanoscale IR spectroscopy to a broader range of samples.

In Tapping AFM-IR, the probe is driven by a piezo actuator with periodic oscillations near one of the cantilever resonance frequencies. Depending on parameters, the tip-sample interaction can be in a net-repulsive or attractive regime. The Tapping AFM-IR signal is detected at another resonance frequency, and laser pulse rate is tuned to match the sum or difference of the two cantilever resonance frequencies. A phase-locked loop is used to automatically adjust laser pulse rate according to resonance frequencies of the cantilever on the sample, compensating for resonance shifts from the mechanical differences during IR measurement.

Tapping AFM-IR is particularly useful for studying rough, soft, or adhesive samples that are difficult for contact mode–based AFM-IR modes. Its high spatial resolution makes it possible to obtain meaningful infrared absorption images on samples with fine structures, such as block copolymers and some semiconductor samples with small domains. Its high sensitivity also enables measurements on thin samples, such as self-assembled monolayers on non-metal substrates.



(from top) Block copolymer core shell, PMMA/PS block copolymer with accompanying IR spectra, and nanocontaminants on a wafer. Scan sizes (X): 400 nm, 500 nm, 2 µm.

Environments: air, glovebox

Recommended probes: PR-xx-TnIR-A, PR-xx-TnIR-D (where xx = AFM model)

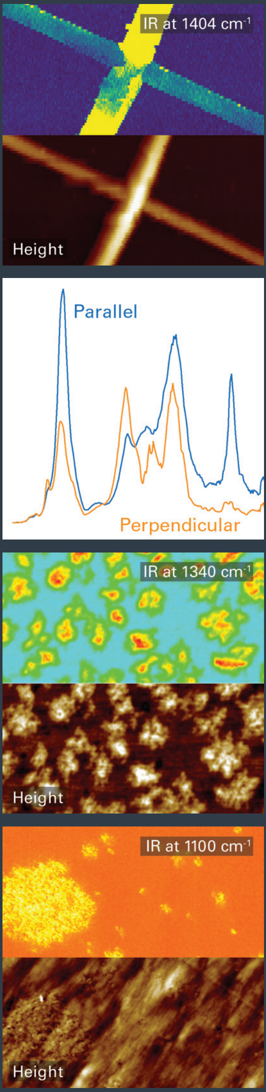
Related modes: RE AFM-IR, Surf. Sens. AFM-IR, REFV AFM-IR, s-SNOM

Resonance Enhanced AFM-IR (RE AFM-IR)

RE AFM-IR provides simultaneous topography and chemical images, and measures IR spectroscopy with high sensitivity. In this mode, the laser pulse rate matches a contact resonance frequency of the probe, improving sensitivity and data collection efficiency. RE AFM-IR is one of the most-published photothermal AFM-IR modes due to its excellent sensitivity and relatively long adoption history.

In RE AFM-IR, the pulse rate of a mid-IR laser is tuned to match a contact resonance frequency of a soft cantilever scanning the surface in contact mode. The photothermal IR response is detected at the same frequency. To compensate for the resonance shift from mechanical property differences, a phase-locked loop (PLL) synchronizes laser pulse rate and contact resonance frequency of the cantilever on the sample.

RE AFM-IR should be used in situations where spectral identification is the primary goal and ease of use is a key concern. It is particularly effective for chemical analysis on very thin samples, such as self-assembled monolayers on non-metal substrates. RE AFM-IR is very versatile and can be used for nanoscale chemical identification of thin films, bulk-faced polymers, microtomed sections, and biological samples.



(from top) PVDF nanofibers with accompanying polarized IR spectra, PP/SiO_2 nanocomposite, and PEG monolayer. Scan sizes (X): 12 µm, 2 µm, 10 µm.

Environments: air, glovebox

Recommended probes: PR-xx-CnIR-B (where xx = AFM model)

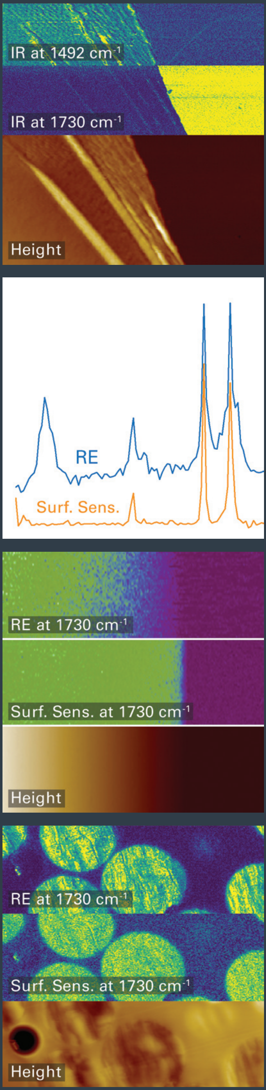
Related modes: Tapping AFM-IR, Surf. Sens. AFM-IR, REFV AFM-IR, s-SNOM

Surface Sensitive AFM-IR (Surf. Sens. AFM-IR)

Surf. Sens. AFM-IR provides simultaneous topography and chemical maps, exhibiting top-layer sensitivity by limiting signal contributions from sub-surface material. It is then possible to distinguish top surface data from that of layers and bulk material beyond the probing depth.

In Surf. Sens. AFM-IR, a soft AFM cantilever is engaged with the sample in contact mode. It achieves top-layer sensitivity through a heterodyne detection scheme, where the laser pulse repetition rate is tuned to match the sum or difference of two contact resonance frequencies of the cantilever. Typically, the cantilever is driven at a high resonance frequency and detected at a lower resonance frequency, with a relatively high laser pulse repetition rate.

Surf. Sens. AFM-IR is particularly useful for studying multilayer, thin coatings on bulk materials. It provides many of the same capabilities as RE AFM-IR but confines the measured signal to the top surface (<30 nm) of the material. This technique is valuable for applications in advanced materials, devices, and fundamental light/matter interactions, especially for distinguishing surface chemistry from bulk properties and reducing the impact of spectral saturation in thick materials.



(from top) Thin PS film on thick acrylate with accompanying IR spectra, PMMA wedge on Si, PMMA in epoxy. Scan sizes (X): 5 µm, 2.4 µm, 10 µm.

Environments: air, glovebox

Recommended probes: PR-xx-CnIR-B (where xx = AFM model)

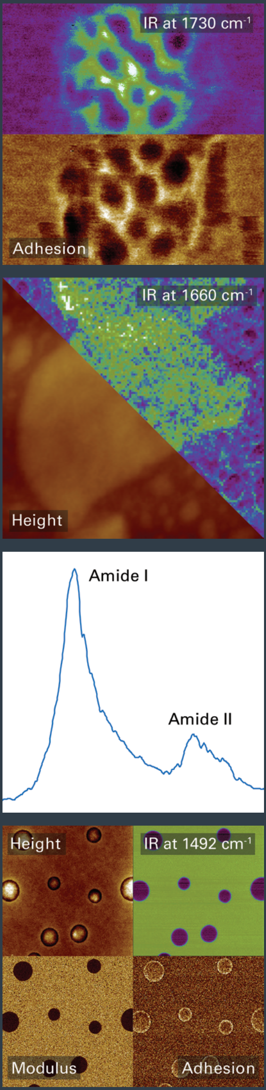
Related modes: Tapping AFM-IR, RE AFM-IR, REFV AFM-IR, s-SNOM

Resonance Enhanced Force Volume AFM-IR (REFV AFM-IR)

REFV AFM-IR is a force volume–related approach where RE AFM-IR imaging or spectroscopy is performed during the hold time. It offers the capability to perform simultaneous multimodal imaging to collect mechanical properties, such as elastic modulus and adhesion, together with chemical information.

REFV AFM-IR uses a force-volume method (linear approach/retract) with an adjustable hold (dwell) time. It leverages resonance-enhanced detection—a method in which the pulse rate of the laser source is set to a resonance frequency of the AFM cantilever—to boost signal. At each pixel, REFV AFM-IR provides chemical information from the photothermal AFM-IR spectrum collected during the dwell time, and mechanical information from the retract part of the force curve.

REFV AFM-IR is an accurate, accessible, and easy-to-use means to obtain quantitative nanoscale chemical and mechanical characterization. Since the probe is not dragged on the surface, there are no lateral forces, enabling investigations of previously inaccessible samples, such as fragile nanoparticles and sticky polymers. One distinct advantage of this mode is the capability to use frequency tracking to easily eliminate mechanically induced artifacts.



(from top) PS-b-PMMA core shell, thin purple membrane with accompanying IR spectrum, and PS-LDPE blend. Scan sizes (X): 240 nm, 2.5 µm, 500 nm.

Environments: air, glovebox

Recommended probes: PR-xx-CnIR-B, PR-xx-TnIR-A, PR-xx-TnIR-D, PR-xx-FnIR-A (where xx = AFM model)

Related modes: Tapping AFM-IR, RE AFM-IR, Surf. Sens. AFM-IR, s-SNOM

Scattering Scanning Near-Field Optical Microscopy (s-SNOM)

In s-SNOM, light backscattered by a nanometer-scale area directly under the probe tip is detected and analyzed. It is used to measure complex optical properties at the nanoscale. Data extracted from s-SNOM are analogous to those obtained from ellipsometry, giving full access to the refractive index and absorption coefficient.

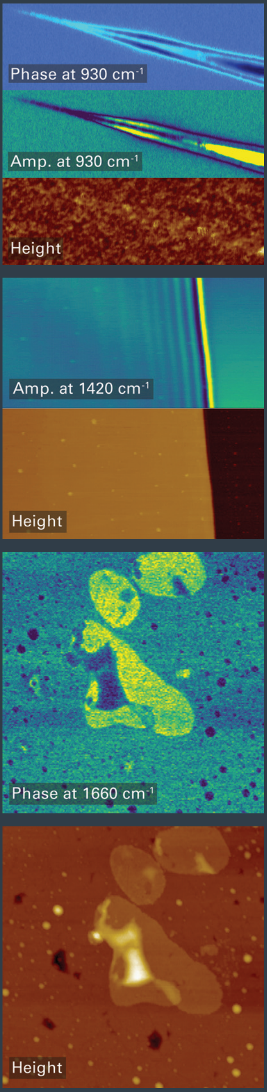
The AFM probe operates in TappingMode during s-SNOM, and the backscattered light is detected through an interferometer. The signal is demodulated at a higher harmonic of the probe tapping frequency to give the near-field component. Amplitude and phase of the near-field signal are used to derive the complex optical properties. Raster scanning across the sample while detecting the elastically scattered light results in an image of the optical response.

The s-SNOM technique provides <10 nm spatial resolution and can be used for high-resolution chemical mapping by tuning the laser wavelength to specific chemical resonances. It is useful for imaging nanoscale contrast in optical properties, with diverse applications in advanced materials, devices, and fundamental light/matter interactions. It is especially valuable for studying 2D materials and nanophotonics.

Environments: air, glovebox

Recommended probes: PR-EX-SNM-B, PR-EX-SNM-C

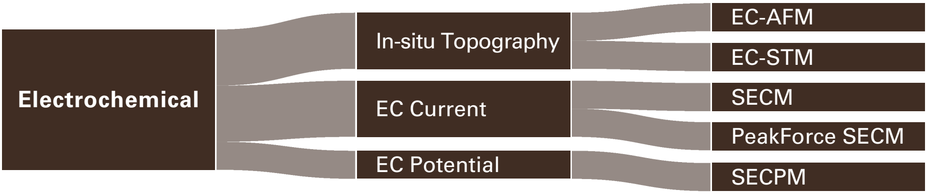
Related modes: Tapping AFM-IR, RE AFM-IR, Surf. Sens. AFM-IR, REFV AFM-IR



(from top) Graphene wedge, hexagonal boron nitride (hBN), and purple membrane (image pair). Scan sizes (X): 2 µm, 10 µm, 3 µm, 3 µm.

Electrochemical Modes

Electrochemical modes are used to observe in-situ changes in materials under electrochemical control or during electrochemical reactions. All electrochemical modes require an electrochemical cell.



Electrochemical AFM (EC-AFM)

EC-AFM is used to visualize nanoscale features and changes in an in-situ electrochemical (EC) environment. This mode is conducted within an EC cell and is used to track surface structure, properties, and reactivity during EC reactions. Typical EC experiments use a fixed working electrode potential/current, a potential sweep, or measurements at open circuit potential.

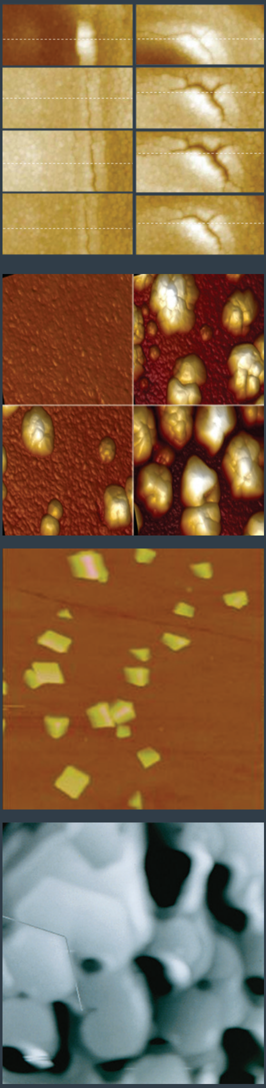
An EC fluid cell used for EC-AFM contains the sample as a working electrode, plus (typically) an additional quasi-reference and counter electrode. All electrodes are electrically connected to a potentiostat or galvanostat. By using this EC cell, in-situ studies are made possible under EC control and in volatile solvents. Note that the probe is unbiased for EC-AFM and acts as a passive nanoscale imager. Multiple AFM imaging modes are therefore possible (e.g., contact mode, TappingMode, PeakForce Tapping, etc.)

EC-AFM is frequently used in energy research for the study of charging/discharging of battery materials, deposition/galvanization processes, and structural/mechanical stability of solid electrolyte interfaces (SEI). More broadly, it can also be used for studies on corrosion and (bio)catalytic reactions/formation. It is important that the EC cell used fits the sample and experiment requirements, such as precise environmental control and ability to engage with poorly reflective samples, such as lithium cathode materials.

Environments: liquid (EC cell), glovebox

Recommended probes: SCANASYST-FLUID(+), DNP-C, DNP-D

Related modes: EC-STM, SECM, PeakForce SECM, SECPM



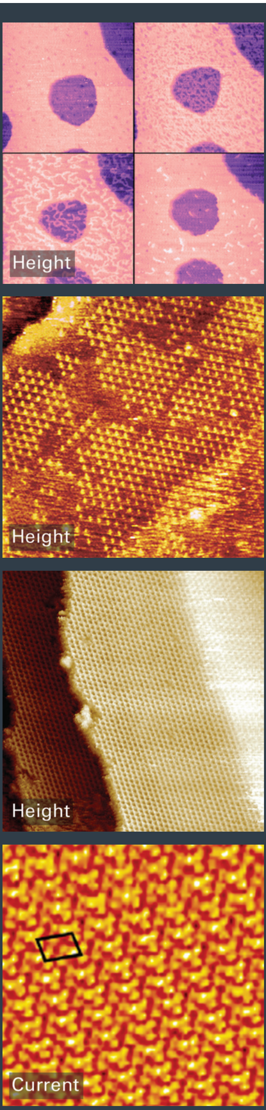
(from top, all in-situ height) SEI failures in Li-ion batteries through lithation/delithiation, Cu deposition on glassy carbon, deposited Fe nanoparticles, and overpotential deposition of Cd on Cu(111). Scan sizes (X): 5 µm, 1 µm, 2 µm, 650 nm.

Electrochemical Scanning Tunneling Microscopy (EC-STM)

EC-STM is STM in an electrochemical (EC) cell with a four-electrode setup (two working electrodes, a reference electrode, and a counter electrode). This enables in-situ topographic and electrical imaging of electrode surfaces during EC experiments, as well as current versus distance or current versus voltage spectrum collection to measure local conductivity, work function, or density of electronic states.

EC-STM uses the tunneling current under a bias between a (semi)conducting sample and a conducting tip for distance control and feedback. The tip, which is coated with an insulating layer to expose only the very end, can be scanned in constant current mode (with feedback control) or in constant height mode (feedback switched off). Current-distance and I-V spectroscopy is also possible. An EC fluid cell used for EC-STM contains the tip and sample as working electrodes plus (typically) an additional quasi-reference and counter electrode, connected to a bipotentiostat. By using this EC cell, in-situ studies are made possible under EC control and in volatile solvents.

EC-STM is particularly useful for characterization of metallic layers, molecular adsorbates, electrochemically active species, and catalytic sites, as well as high-resolution atomic- or molecular-scale imaging of electrochemical processes on a conductive sample. It can also be used to dynamically track deposition/stripping processes or conformational changes.



(from top) Cu underpotential deposition on Au (courtesy Dr. Joseph Campbell), K$^+$ cation formation on Pt(111), Cu monolayer underpotential deposition on Au, and SO$_4^{2-}$ adsorbed on Au. Scan sizes (X): 300 nm, 30 nm, 20 nm, 6 nm.

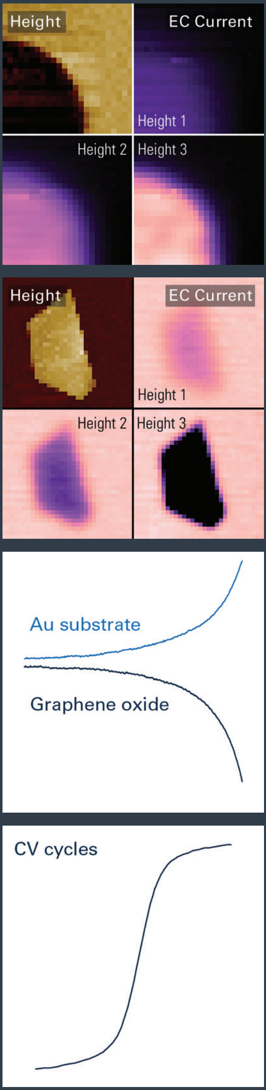
Environments: liquid (EC cell), glovebox
Recommended probes: TT-ECM10, PT-ECM10
Related modes: EC-AFM, SECM, PeakForce SECM, SECPM

Scanning Electrochemical Microscopy (SECM)

AFM-based SECM simultaneously maps topography and electrochemical (EC) current, providing images with <100 nm spatial resolution that show local EC activity. It uses an (ultra)microelectrode or a nanoelectrode scanned (in close proximity) across a surface in an electrolyte under EC control. The probe is highly specialized and has a nanoscopic electrode area at or around the tip apex.

In SECM, the probe tip is the working electrode in a low-noise 3- or 4-electrode EC cell. Applying potential between electrode and solution triggers an EC reaction of redox species at the probing electrode and generates a measurable current. This current is indicative of geometrical accessibility of the probe and, more importantly, the local concentration of the redox active species.

AFM-based SECM (rather than its macroscopic counterparts) is best suited for fundamental and functional investigations of nanoscale EC reactivity and diffusion characteristics. This includes such applications and samples as catalytic nanoparticles, fuel cell membranes, reactivity of battery and energy materials, early stages of corrosion, etc. Generally, the force-controlled PeakForce SECM is preferred over standard SECM due to preserved probe shape/quality, lack of sample damage, and possibility for complementary mechanical data.



(from top) Graphene oxide flake on Au, Si_3N_4 on Pt, current versus distance and cyclic voltammogram. Scan sizes (X): 2 µm, 2 µm.

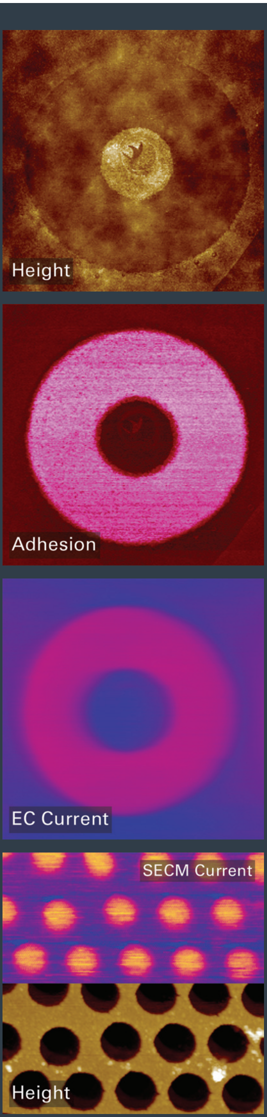
Environments: liquid (EC cell), glovebox
Recommended probes: PF-SECM
Related modes: EC-AFM, EC-STM, PeakForce SECM, SECPM

PeakForce Scanning Electrochemical Microscopy (PeakForce SECM)

PeakForce SECM is similar to SECM, but uses PeakForce Tapping as its basis for tip-sample interaction. Like standard SECM, PeakForce SECM is used to primarily track and visualize electrical and chemical processes in liquid. Unlike standard SECM, PeakForce SECM can also be used to simultaneously capture complementary mechanical data, correlating biological, chemical, and physical properties with morphological structures at the nanoscale.

PeakForce SECM utilizes a specialized nanoelectrode AFM probe that is scanned across a surface in an electrolyte under electrochemical (EC) control. Applying potential between tip and solution triggers an EC reaction of a redox active species at the probing tip and generates a diffusion limited current (pA to nA level), which is indicative of the local concentration of the redox active species and the geometrical accessibility of the probing electrode.

PeakForce SECM is best suited for fundamental and functional investigations of nanoscale EC reactivity and diffusion characteristics, e.g., for electrochemically active species, corrosion hotspots, and battery materials/processes. The PeakForce version of SECM is essential when complementary mechanical and electrical properties are of interest, when samples are sticky or fragile, and when highest resolution is required. With PeakForce SECM, tip shape and quality lifetimes are also extended, ensuring accuracy throughout longer experiments.



(from top) Three data channels from donut-shaped thiol self-assembled monolayer on Au, Si_3N_4 layer with openings on Pt. Scan sizes (X): 25 µm, 5 µm.

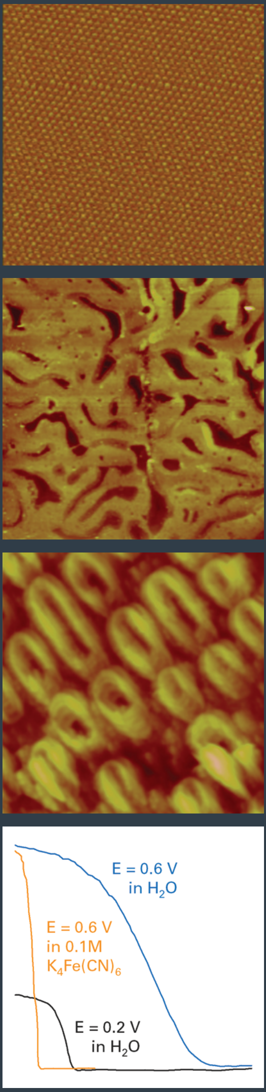
Environments: liquid (EC cell), glovebox
Recommended probes: PF-SECM
Related modes: EC-AFM, EC-STM, SECM, SECPM

Scanning Electrochemical Potential Microscopy (SECPM)

SECPM maps potential distribution and topography on a sample in an electrolyte and under electrochemical (EC) control. SECPM uses an STM tip, and feedback is based on the tip-sample potential difference in the EC double layer above the surface. SECM complements SECPM, since the former provides current mapping and the latter provides potential mapping.

SECPM utilizes a specially designed combined STM/SECPM head, which includes a current amplifier for current feedback in STM operation and a multiple-gain high-impedance potentiometer for potential feedback in SECPM operation. SECPM can be operated at constant distance for topography mapping (feedback loop maintaining a user-chosen potential setpoint) or constant height for potential mapping (feedback switched off).

SECPM enables potential-distance spectroscopy, potential imaging, and high-resolution topography imaging. When interleaved with STM, it can provide complementary, colocalized potential-based and current-based datasets. SECPM is best-suited for measurements in polar liquids, in higher-concentrated electrolytes, and with less-conductive samples. It is commonly used to evaluate EC double-layer thickness, an important factor for corrosion and deposition processes, as well as battery behavior.



Height images of (from top) HOPG in water, $Sn_{60}Pb_{40}$ in glycerol, and Cd-Ni coating. (bottom) Potential-distance profiles across electric double layers. Scan sizes (X): 10 nm, 5 µm, 10 µm.

Environments: liquid (EC cell), glovebox
Recommended probes: TT-ECM10, PT-ECM10
Related modes: EC-AFM, EC-STM, SECM, PeakForce SECM

Manipulation

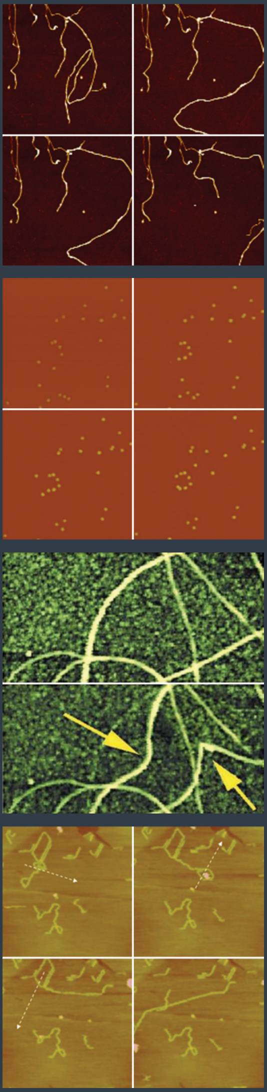
Manipulation modes are used to locally modify or pattern the sample surface. A typical workflow using these modes involves definition of a tip path then execution of manipulation, followed by imaging to verify the induced sample modifications.

Nanomanipulation

Through a set of nanomanipulation operating modes, AFM can also be applied for precise manipulation, modification, or fabrication of nanometer-scale structures. Most nanomanipulation methods make use of precise force control to alternate between imaging the sample and modifying it. A nanomanipulation experiment typically consists of a series of alternating imaging and modification steps using the same tip.

During nanomanipulation operation, the tip follows a user-defined path created by a series of user-selected positions, vector files, or scripts. While following the path, the operating mode (contact mode or TappingMode) and related tip-sample force interactions are precisely controlled to manipulate local sample features. Additional control of voltages can also be applied. Manipulation is typically followed by an imaging step (using the same probe) of the modified area.

Nanomanipulation is typically applied to move nanoparticles, nanowires, and other nanoscale objects that are loosely bound to the surface. A semiconductor industrial application for nanomanipulation is defect removal on EUV photomasks.



Height images during manipulation of (from top) carbon nanotubes, Au nanoparticles, carbon nanotubes, and polymer macromolecule on HOPG. Scan sizes (X): 2 µm, 2 µm, 900 nm, 500 nm.

Environments: air, liquid, glovebox
Recommended probes: FESPA-V2, TESPA-V2
Related modes: Nanolithography

Nanolithography

Through a set of nanolithography operating modes, AFM can also be applied for precise fabrication and interconnection of nanometer-scale structures. Various types of nanolithography are enabled by precise control of operating conditions for the probe, sample, and environment. A nanolithography experiment typically consists of a series of alternating imaging and modification steps using the same tip.

During nanolithography, the tip follows a user-defined path from vector files, scripts, or graphical inputs. Meanwhile, critical parameters, such as voltage, force, or temperature, are precisely controlled to locally modify the surface. For example, in anodic oxidation nanolithography, a voltage is applied to the AFM probe to create an oxide layer under the tip with high spatial resolution. Other lithography mechanisms include electrical charging, piezoelectric polarization control, thermal-induced transitions, force-induced modifications, and local electrochemical (EC) control.

Nanolithography can be applied on a wide variety of samples (e.g., nanowires, 2D material flakes, dielectrics, ferroelectrics) and is typically performed in a controlled environment. Environmental conditions, such as humidity or EC concentration, have an immediate impact on the lithographic operation. Nanolithography is often followed by another AFM mode providing property information of the modified sample area.

Environments: air, liquid, glovebox
Recommended probes: LTESPA-V2, MESP-RC
Related modes: Nanomanipulation

(from top) Anodic oxidation of dots on Si, vector line pattern on Si, charging of SiO_2 dielectric film, and piezoelectric poling of BFO ferroelectric film. Scan sizes (X): 3 µm, 5 µm, 16 µm, 5 µm,

Appendix A: DataCube

Hyperspectral AFM modes represent a big data approach with the potential to provide new information in a wide range of applications, especially on samples with high complexity. Extracting image slices, spatio-spectral slices, or spectra at every pixel opens the door to reveal sample properties that are either not accessible or are easily missed by conventional characterization techniques. Specific advantages include acquisition of full spectral information and images at a range of operating conditions, correlation between electrical and mechanical properties, longer tip lifetimes, the capability to measure soft and fragile samples, and mapping of new nanoelectrical properties

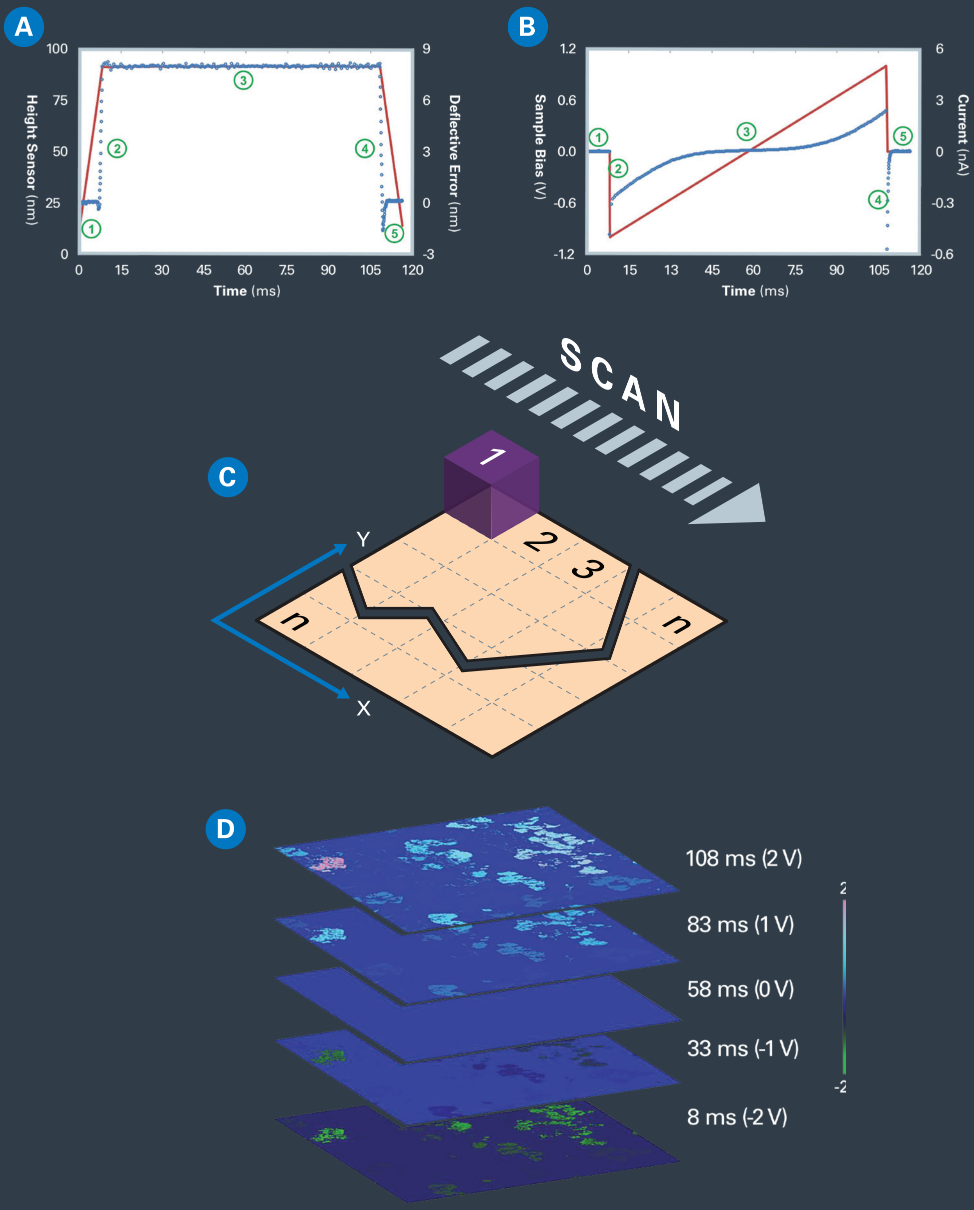
Bruker's proprietary DataCube hyperspectral modes use FASTForce Volume™ (FFV) to collect a force-distance spectrum at every pixel in a 2D array, including a hold or dwell segment at contact. A parameter ramp (e.g., AC or DC bias, frequency, temperature, etc.) is conducted during the hold segment at each pixel, generating a spectrum at each pixel. These spectra are analyzed and reconstructed into a multidimensional dataset. Image slices or points of interest can be extracted from that dataset as needed. The high-dimensional datasets enable the extraction of high-resolution maps of properties previously not acquirable using AFM, including Schottky barrier height, flat-band voltages, and piezoelectric switching voltages.

Data Collection

Bruker's DataCube modes are the result of seamless integration of existing high-performance property measurement modes with the FFV technique. In FFV, the tip is held in a fixed XY position, while a force distance cycle with dwell segment is executed in the Z direction. Force ramp rates up to 300 Hz are possible using Bruker's proprietary low-force trigger capability. Combined with sub-100 ms dwell times for the hold segment, good throughput is obtained, and regular imaging speeds can be maintained when acquiring dense arrays.

The DataCube method is illustrated in the following figure. Figure sections (A) and (B) show the height sensor plot and corresponding force curve, respectively. After acquisition of the spectra in one pixel, the tip is retracted from the surface before moving to the next pixel using the FFV raster scanning method, as shown in (C).

DataCube modes operating on FASTForce Volume approach: (A) height sensor (red) and deflection error (blue open circle) plots with segments showing extend (1–2), dwell (3), and retract (4–5) cycles; (B) pattern of sample bias (red) and corresponding TUNA current (blue); (C) illustration of scan pattern during acquisition of DataCube-mode data; and (D) TUNA current slices from DataCube TUNA results.



This avoids the shear force that is common in contact mode–based scanning. Section (D) of the figure shows five typical current slices extracted from a data cube acquired using this principle on γ-Fe_2O_3.

The 190 spectral data points from the dwell segment provide 190 of these current slice images. At the same time, the extend/retract segments of the deflection plot construct a complete force-distance curve and enable the extraction of quantitative nanomechanical images for modulus, adhesion, deformation, and stiffness.

Conditions can also be held constant during the dwell period, which can be short (milliseconds) or long (tens of seconds). In this way, signal can be averaged over a user-defined period, resulting in improved signal-to-noise ratios as compared to the equivalent contact or PeakForce mode. During the dwell period, the parameter of interest is measured as a function of time. This enables studies of time-dependent phenomena, e.g., mapping of time-dependent conductivity or photoconductivity after an external stimulus, observing time-dependent dielectric breakdown in dielectrics, and investigating charging/discharging effects after a voltage pulse.

Data Analysis

After acquiring the data, it needs to be analyzed. This is where the different DataCube modes truly excel, as they provide simultaneous capture of nanoscale electrical and mechanical characteristics in high-density data cubes. For materials scientists and engineers, this breaks long-standing efficiency and characterization barriers.

Bruker’s standard NanoScope® analysis offline software provides a suite of data tools for basic processing and visualization, such as the extraction of individual slices and spectra. Contour plots can also be extracted along a user-defined line.

Slices of the spectroscopic maps can be extracted for further processing, as one would do with regular AFM images. This allows surface topography and quantitative nanomechanical maps to be extracted. Any spectra can be selected, displayed, and exported through single or multiple point selection in the images. Dense collections of spectra can be visualized through statistical methods with the built-in density plots.

Bruker's DataCube modes include:

- DataCube TUNA
- DataCube SCM
- DataCube sMIM
- DataCube SSRM
- DataCube CR-PFM
- DataCube PFM
- DataCube EFM
- DataCube SThM
- DataCube SECM

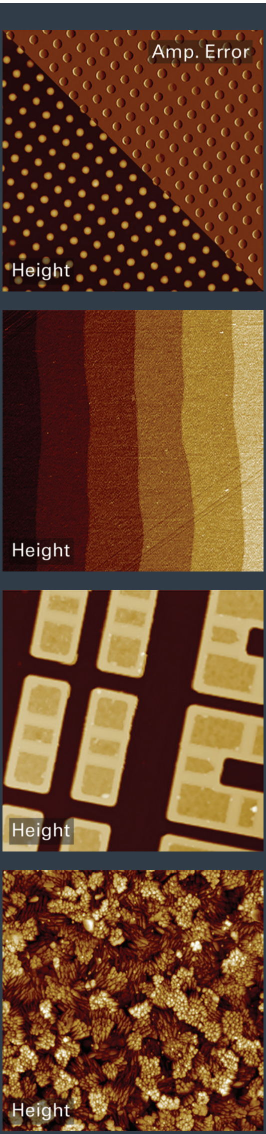
For more information and detailed DataCube examples,
download our e-book: The Latest Advances in AFM Nanoelectrical Modes.

Appendix B: ScanAsyst

ScanAsyst was the first automatic image optimization technology for AFM. Its intelligent algorithms continuously monitor image quality and make appropriate parameter adjustments. The original ScanAsyst is based on PeakForce Tapping, while ScanAsyst Plus (compatible with NanoScope 6 systems) can self-optimize for every AFM topography mode—PeakForce Tapping, TappingMode, and contact mode.

ScanAsyst has immense flexibility to accommodate advanced and novice AFM users. Advanced users may choose to manually control up to all relevant feedback parameters, while beginning users can choose to enable the system to fully self-optimize. ScanAsyst automatically provides consistent, expert-quality results independent of user experience.

An in-depth exploration of the original ScanAsyst can be found in Bruker Application Note AN133, *Introduction to Bruker's ScanAsyst and PeakForce Tapping AFM Technology*.



(from top, using ScanAsyst Plus) Nanopillars in TappingMode, sapphire in contact mode, SRAM in TappingMode, and ITO in PeakForce Tapping. Scan sizes (X): 5 µm, 5 µm, 20 µm, 2 µm.

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