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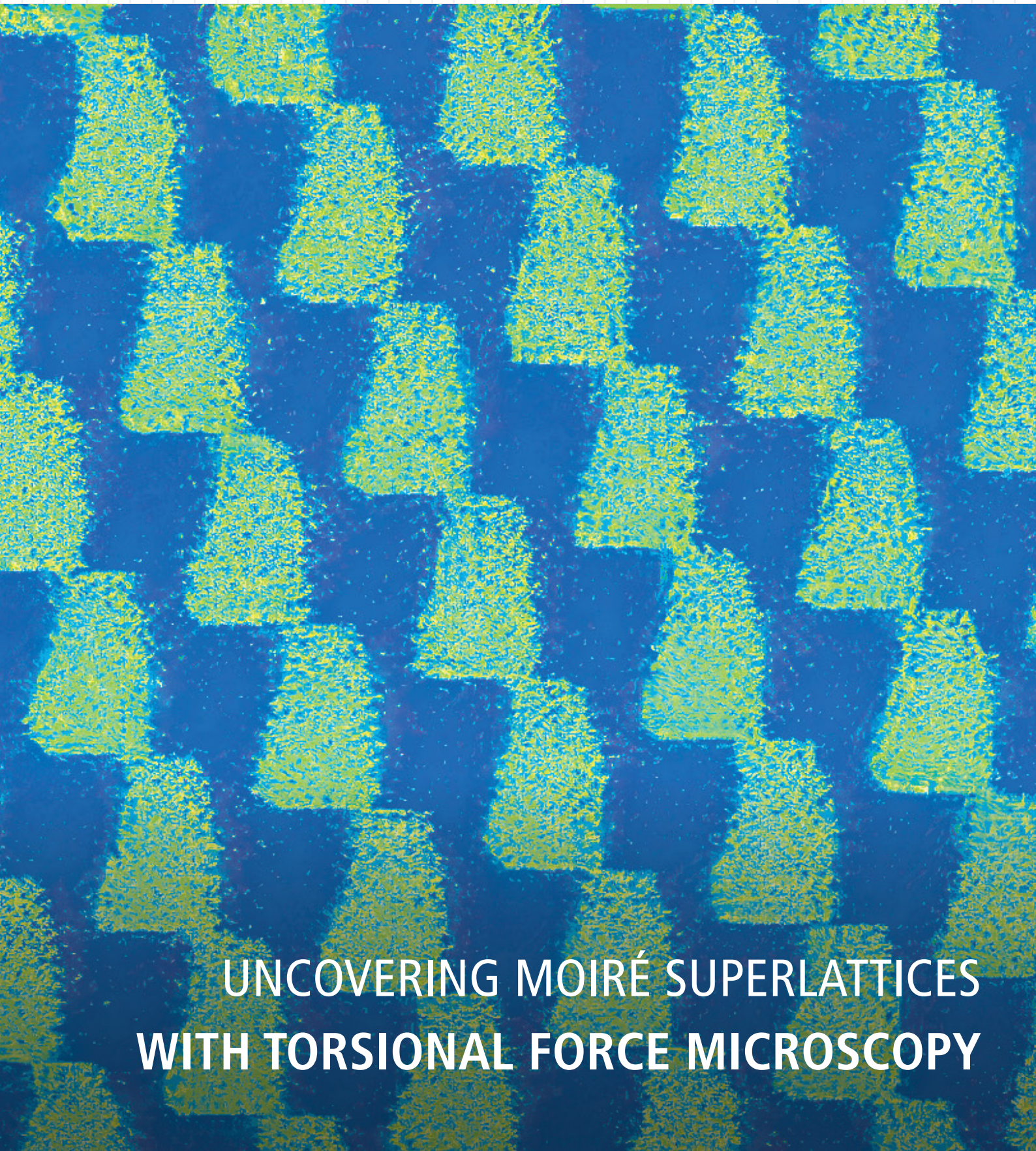
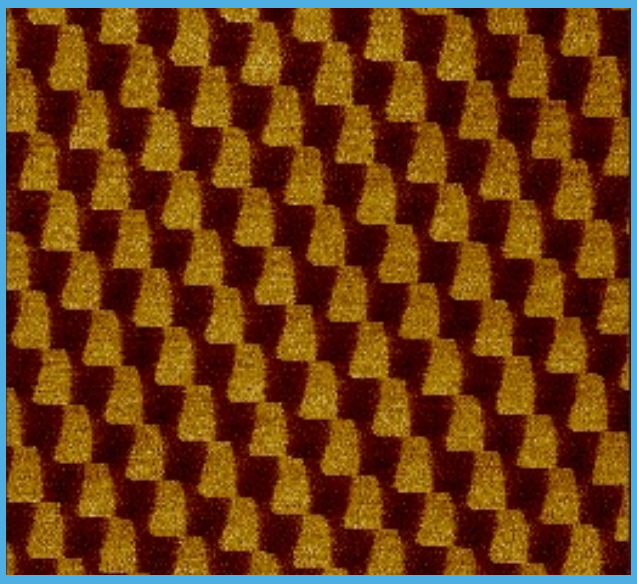


UNCOVERING MOIRÉ SUPERLATTICES WITH TORSIONAL FORCE MICROSCOPY

UNCOVERING MOIRÉ SUPERLATTICES WITH TFM



This high-resolution image captures the moiré superlattice pattern in twisted bilayer graphene, acquired using Torsional Force Microscopy (TFM) under ambient conditions. Subtle variations in the interlayer twist angle give rise to these periodic nanoscale patterns, which critically influence the material's electronic properties. By detecting lateral frictional variations with exceptional sensitivity, TFM provides a powerful, non-invasive method for visualizing both surface and subsurface structures in van der Waals heterostructures, directly supporting the development of next-generation quantum materials.

About this Issue:
Exploring New Frontiers in Nano Science

In this special edition, we present groundbreaking research and insights from the recent NanoScientific Symposium. This issue features advances in nanoscale metrology, including adaptive scalpel scanning probe microscopy for precise 3D volumetric sensing, FEM/KPFM hybrid methods for true surface charge quantification, and new AFM imaging revealing moiré superlattices with exceptional clarity. These articles showcase pioneering researchers pushing nanoscience boundaries to shape our quantum, electronic, and bio-integrated future. We invite you to explore these highlights of ingenuity, collaboration, and global impact from the NanoScientific community.

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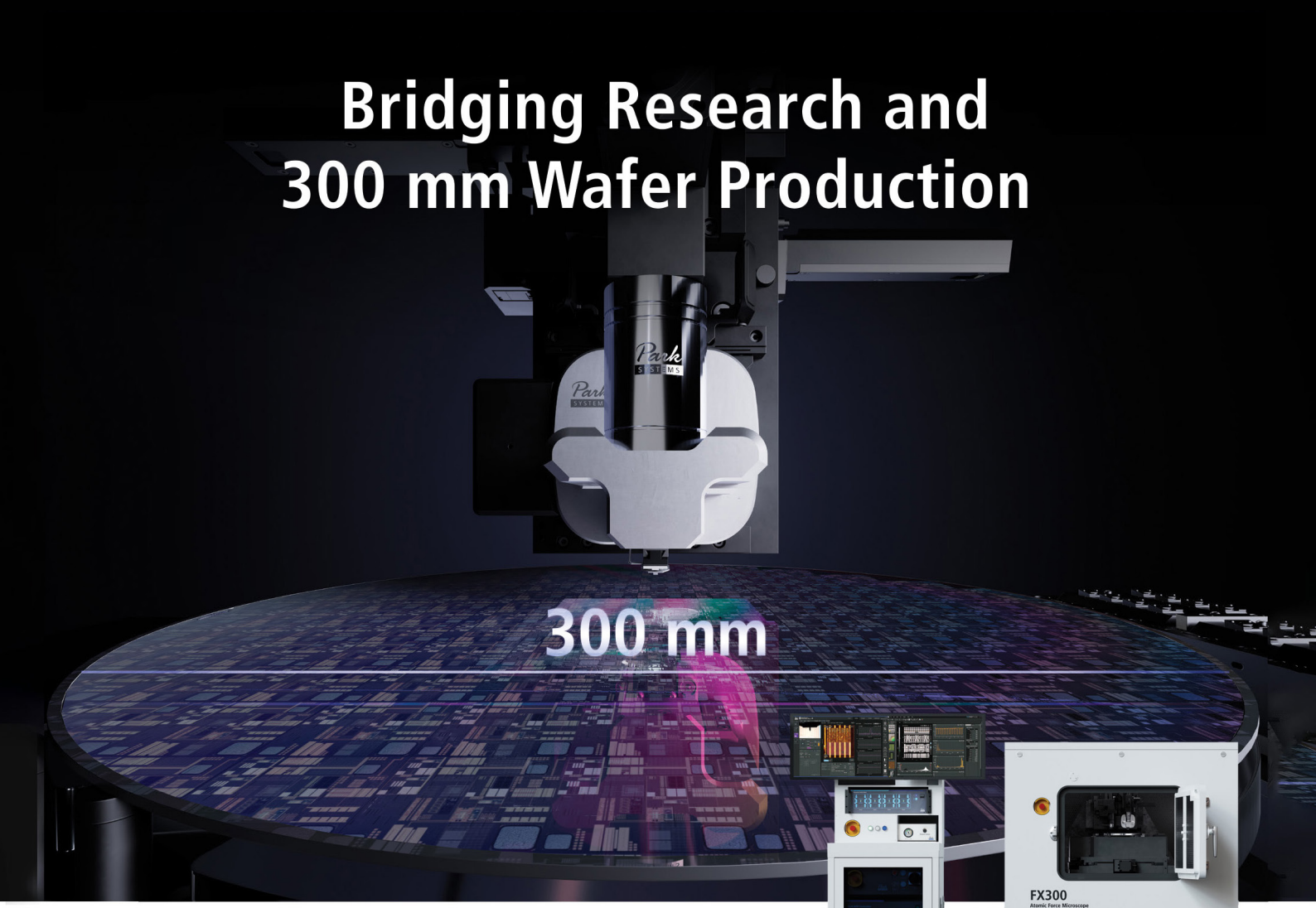
Bridging Research and 300 mm Wafer Production



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BEYOND POTENTIAL: A NEW FRONTIER IN SURFACE CHARGE QUANTIFICATION WITH FEM/KPFM-HYBRID APPROACH

Felix Pertl, Soft & Electrified Materials Lab, Institute of Science and Technology Austria (ISTA), Klosterneuburg, Austria
Adapted from Presentation, Edited by NanoScientific

From Balloons to Breakthroughs in Charge Mapping

Rubbing a balloon on your hair and watching it stick to a wall might seem like a simple childhood experiment, but for PhD student Felix Pertl from ISTA, it highlights a major unsolved problem in surface science. This effect, known as **contact electrification (CE)**, is the transfer of charge when two neutral materials touch. Despite being widely observed, its mechanisms remain unclear [1-2]. Key questions—such as which charge carriers are involved, what drives the transfer, and whether the resulting charge distribution is homogeneous or heterogeneous—are still unanswered. Possible explanations include transfer of electrons or ions via induced dipoles, adsorbed water layers, flexoelectric effects, or combinations thereof, with implications spanning electronics, lightning formation, and biology.

To study CE at the nanoscale, researchers use **Kelvin Probe Force Microscopy (KPFM)**. Pertl employs KPFM to examine whether surface charge is homogeneously or heterogeneously distributed (Figure 1) [3-4]. However, while KPFM measures voltage related to surface charge, converting this to true charge density remains a challenge.

At the NanoScientific Forum Europe 2024, Pertl presented a refined approach combining KPFM with **Finite Element Method (FEM) simulations** to overcome this issue, enabling rigorous extraction of true surface charge densities [5].

The Limits of Traditional KPFM

KPFM has long served as a stable technique in nanoscale characterization, capable of mapping the surface potential between a conductive atomic force microscope (AFM) tip and a sample. By applying an AC/DC voltage and measuring the resulting electrostatic force, KPFM determines variations in work function or surface potential at nanometer resolution.

However, Pertl emphasized a critical shortcoming of the conventional approach: “Everyone always shows the potential,” he said, “but I care more about the charge.” In other words, surface potential data—while useful—is only an indirect representation of the true quantity of interest: the charge distribution on the sample’s surface.

The most common workaround is the simplistic parallel-plate capacitor model, which assumes the AFM tip and the sample form a basic capacitor. The problem? This approach grossly underestimates charge density because it ignores the complex geometry and electrostatic interactions inherent in the real-world tip–sample system **(Fig. 2)**.

Toward a Physics-Accurate Charge Mapping

To address these limitations, Pertl proposes a more rigorous physical model based on energy equations and finite element simulations. His process begins with acknowledging the electrostatic energy of the system when a single point charge q is on the surface

$$W_{elec} = u_0 q^2 + u_1 qV + u_2 V^2$$

where the u_0, u_1, u_2 are geometric parameters and V is the potential between tip and back electrode. The first term, $u0q^2$, comes from the charge interaction with its images in the cantilever/insulator/ground capacitor. The second term comes from the charge’s interaction with the capacitor field. The last term is the energy of the capacitor itself. KPFM uses a voltage $V = V_{bg} - V_{DC} + V_{AC} \sin(\omega t)$, where V_{bg} is any background potential difference in the absence of charge added from contact (e.g. related to the contact potential differences or vertically separated bulk charges) and VAC/DC are AC/DC driving voltages. Taking the negative derivative of the electrostatic energy with respect to the tip deflection, z, gives the vertical force which can be separated into a DC component, a component at ω and a component at 2ω. Denoting z-derivatives as primed, the ω component is

$$F_\omega = -u'_1 q V_{AC} \sin(\omega t) - 2u'_2 (V_{bg} - V_{DC}) V_{AC} \sin(\omega t)$$

In AM-KPFM, a potential VDC is applied to nullifiy the force at frequency ω and solving for $V_{AM} = V_{DC}$, we have

$$V_{AM} = 1/2 \ (u'_1 q)/(u'_2) + V_{bg}$$

Thus, the presence of a point charge modifies the KPFM voltage of a neutral insulator surface by the addtion of the term $1/2\ u'_1\ q/u'_2$.

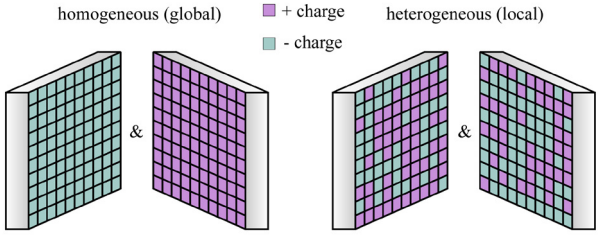

Fig. 1. *Is the charge transfer either homogeneous (global) or heterogeneous (local)?*

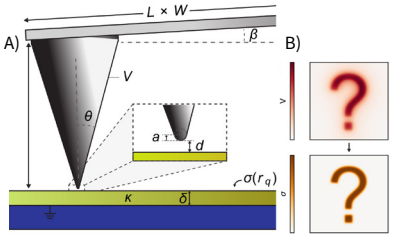

Fig. 2. A) *The system of an AFM probe above a grounded insulator with varying surface charge density σ is complex and depends on many parameters.* B) *In KPFM, one measures a potential map, V that is caused by the surface charge, but in a highly non-trivial way.*

Enter Green’s Functions and FEM Simulations

For realistic samples, which host continuous charge distributions rather than isolated point charges, u'_1 and u'_2 can be related to a Green’s function—a mathematical construct describing the response of a system to a unit input (in this case, a unit surface charge). The convolution between surface potential V and a continuous charge density σ can then be written as:

$$V(r_t) = \iint G(|r_t - r_q|)\sigma(r_q)\,dx_q\,dy_q$$

Obtaining an analytical expression of the Green’s function for this complex geometry is almost impossible. To circumvent this, Pertl uses finite element simulations to numerically construct the Green’s function for the exact setup used in experiments **(Fig. 3)**.

By systematically placing point charges beneath the tip and simulating the electrostatic response across the entire scan area, a full 3D map of the Green’s function is built. From here, the charge density can be extracted via deconvolution using Fourier transform techniques.

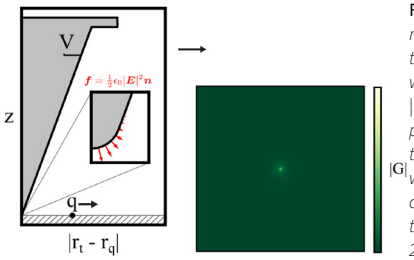

Fig. 3. *We perform axisymmetric COMSOl simulations to solve Poisson's equation with a 'point' charge q at |rt - rq| and voltage V at the probe/cantilever and 0 at the bottom plane. Then, we convert the numerically calculated 1D Green's function to a radially symmetric 2D map*

Synthetic Validation and Experimental Confirmation

Before applying the method to real data, Pertl validated it on synthetic samples with known charge distributions. A square patch with defined charge density was processed through the simulation pipeline, producing a potential map showing expected convolution effects, such as blurring and fringe fields. The inverse transformation accurately recovered the original charge density within a small margin of error **(Fig. 4)**, confirming the method's validity.

In the lab, Pertl tested this on a clean silicon oxide surface. By bringing the AFM tip into contact and slightly scratching it, he introduced localized surface charges. The resulting KPFM map showed a diffuse patch similar to the synthetic result **(Fig. 5)**.

Using FEM simulations based on precise setup parameters—tip geometry, material properties, and tip-sample distance—he generated the Green's function. Deconvolution of the potential data then yielded a charge density map with values matching the synthetic input (~40 nC/cm^2). This demonstrated quantitative accuracy and revealed fine spatial details invisible from potential data alone.

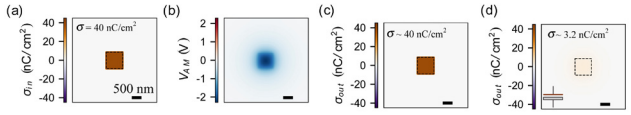

Fig. 4. **A)** *Synthetic input charge map with side lengths 1 µm and charge density 40 nC/cm^2.* **B)** *Forward convolution of surface charge with Green's function to generate voltage map.* **C)** *Deconvolution of surface charge with Green's function to exactly recover input.* **D)** *If instead we use the `capacitor method' to recover the charge, it is grossly underestimated; color bar same as* **C)** *to highlight this inaccuracy, with recovered charge so small it is barely visible. Additionally, sharp spatial features of original charge distribution are lost.*

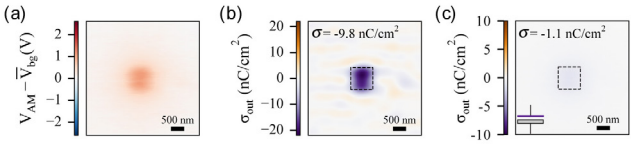

Fig. 5. **A)** *Background subtracted voltage map of surface after charging via scraping a square of side length 1 µm.* **B)** *Recovered surface charge density from* **A)**, *where deconvolution reveals average value -9.8 nC/cm² in the marked region.* **C)** *'Capacitor' recovered surface charge density from* **A)** *with average of -1.1 nC/cm² in the dashed region.*

Advantages Over the Capacitor Model

Pertl revisited the conventional capacitor model for comparison. When the same potential data was processed using the simplified model, the resulting charge density was underestimated by more than a factor of ten. This stark discrepancy underscores the importance of geometry-aware modeling, especially for high-resolution, quantitative analysis.

What makes Pertl's method transformative is its ability to move from qualitative imaging to quantitative metrology. It enables researchers to measure actual surface charge density at the nanoscale—turning KPFM from a relative mapping tool into an absolute characterization technique.

Implications and Future Applications

Pertl's approach paves the way for more precise studies of contact electrification, dielectric breakdown, charge trapping, and material aging. It holds particular promise for fields such as:

- **Flexible electronics**, where contact-induced charging can affect performance.
- **Energy storage**, where charge leakage or accumulation alters behavior over time.
- **Triboelectric devices**, where contact-induced charges are harnessed for energy harvesting.
- **Nanoionics and memristive devices**, which rely on ionic and charge transport mechanisms.

Moreover, his method could potentially be adapted for in-situ or operando experiments where the dynamics of charge movement and accumulation are tracked in real-time under electrical bias or mechanical stress.

Conclusion: A New Era for Surface Charge Microscopy

Felix Pertl's contribution is more than a technical enhancement—it represents a conceptual shift in how we analyze and understand surface charge phenomena. By integrating high-fidelity simulations with established KPFM techniques, he has opened new avenues for quantitative, spatially resolved charge mapping.

In doing so, Pertl offers a tool that could redefine how scientists study everything from friction and wear to semiconductor performance and sensor sensitivity. As the quest to understand contact electrification continues, his work stands as a crucial step toward unraveling the mysteries of surface charge behavior—one Green's function at a time.

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About Felix Pertl

The Waitukaitis group: Felix Pertl, 3rd from left

Felix Pertl is a PhD student in the Waitukaitis group, studying charge transfer after contact electrification using Kelvin Probe Force Microscopy. He began this work as an intern in 2021 after completing his master's in Technical Physics at the Technical University of Graz, where his thesis with Lam Research focused on defects on silicon wafers. He earned his bachelor's in physics there in 2019. Originally from Klagenfurt, Austria, Felix enjoys padel tennis, hiking, and skiing.

REDESIGNING MICROWAVE IMPEDANCE MICROSCOPY: TOWARD ENHANCED NANOSCALE DIELECTRIC IMAGING

Prof. Eric Y. Ma, Physics, Electrical Engineering & Computer Sciences, University of California, Berkeley
Adapted from Presentation, Edited by NanoScientific

In the realm of scanning probe microscopy, a quiet revolution is taking place—one that aims to democratize high-frequency electrodynamic imaging by radically simplifying the instrumentation without sacrificing sensitivity or spatial resolution. At the 2024 NanoScientific Symposium Americas, Prof. Eric Y. Ma of UC Berkeley introduced a groundbreaking redesign of Microwave Impedance Microscopy (MIM), a technique for mapping local dielectric and conductive properties at the nanoscale. His team's new architecture eliminates the need for a cancellation circuit and specialized probes—both long-standing technical bottlenecks. Instead, it harnesses self-referenced homodyne detection and commercially available monolithic silicon cantilever probes to achieve thermal-noise-limited sub-zeptofarad sensitivity and electrical spatial resolution limited only by the tip radius. This leap in design not only significantly reduces the cost and complexity barriers to MIM but also unlocks new measurement modalities, such as continuous-frequency local microwave spectroscopy, broadband photoconductivity nano-interferometry, and nonlinear MIM operation.

The Problem with Traditional MIM

Microwave Impedance Microscopy combines the spatial precision of atomic force microscopy with the electrical sensitivity of microwave reflectometry. It functions by delivering a high-frequency signal (typically in the GHz range) to a nanoscopic probe tip that interacts with the sample via near-field coupling. The reflected signal encodes changes in the tip-sample admittance—dependent on local dielectric constant, conductivity, and depletion/accumulation layers, among others—allowing high-resolution maps of local electronic behavior with minimal sample preparation even for buried samples (Fig. 1). With this unique capability, MIM has made significant contributions to wide-ranging scientific and technology fields (Fig. 2).

But conventional MIM systems face several technical hurdles:

1. **Specialized Probes:** Traditional MIM relies on highly-specialized, often-handmade, probes designed to minimize parasitic capacitance and signal loss at GHz frequencies. These probes are expensive, fragile, and suffer from relatively large and varying tip radii, severely limiting spatial resolution and robust, quantitative interpretation of MIM images.
2. **Cancellation Circuitry:** Another challenge lies in handling the strong reflected baseline signal. Since the signal of interest is a small fluctuation riding on top of a much larger baseline microwave reflection, a cancellation circuit is thought to be necessary to subtract the baseline via destructive interference and avoid amplifier saturation. This cancellation path, however, requires multiple stages of amplitude and phase controllers and is highly susceptible to environmental noise, temperature fluctuations, and vibration.
3. **Stability and Complexity:** These issues make MIM systems hard to operate stably for long periods to achieve higher sensitivity. Even in controlled lab environments, cancellation often degrades within an hour or two, capping integration time and limiting sensitivity. Moreover, the complexity of both the specialized probes and delicate cancellation circuits makes it challenging to deploy MIM in broader scientific or industrial settings.

A Simpler, More Robust Solution

Prof. Ma's group proposed a radical simplification: eliminate the cancellation circuit entirely and use standard, metal-coated monolithic silicon AFM cantilever probes (Fig. 3).

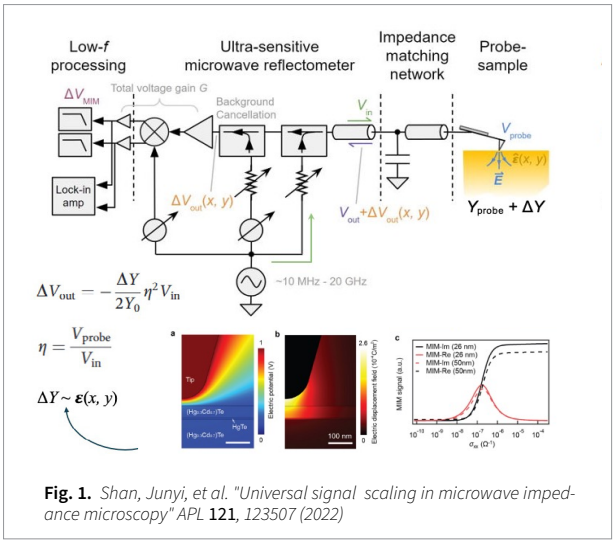


Fig. 1. *Shan, Junyi, et al. "Universal signal scaling in microwave impedance microscopy" APL* **121**, *123507 (2022)*

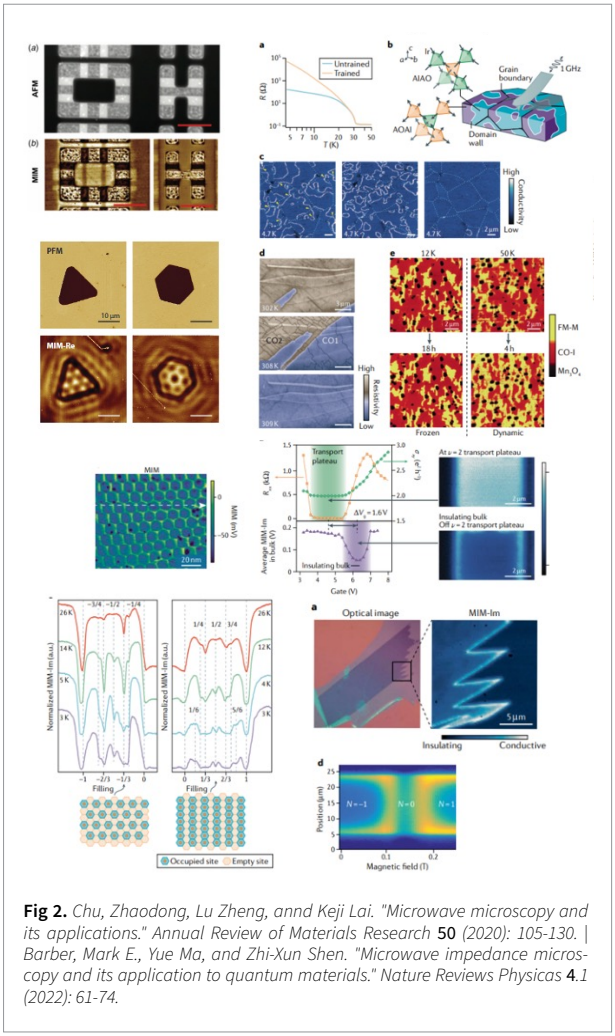


Fig 2. *Chu, Zhaodong, Lu Zheng, annd Keji Lai. "Microwave microscopy and its applications." Annual Review of Materials Research* **50** *(2020): 105-130. | Barber, Mark E., Yue Ma, and Zhi-Xun Shen. "Microwave impedance microscopy and its application to quantum materials." Nature Reviews Physicas* **4**.*1 (2022): 61-74.*

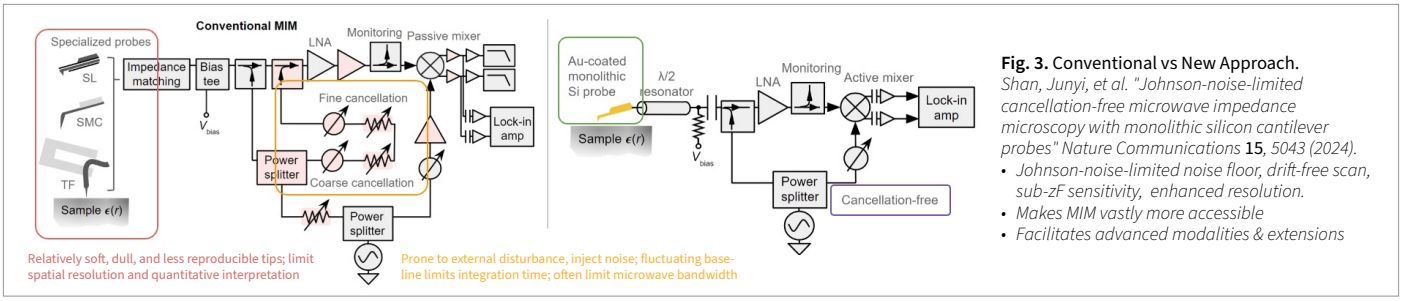


Fig. 3. Conventional vs New Approach.
Shan, Junyi, et al. "Johnson-noise-limited cancellation-free microwave impedance microscopy with monolithic silicon cantilever probes" Nature Communications **15**, *5043 (2024).*

- *Johnson-noise-limited noise floor, drift-free scan, sub-zF sensitivity, enhanced resolution.*
- *Makes MIM vastly more accessible*
- *Facilitates advanced modalities & extensions*

At first glance, this approach seemed counterintuitive. Standard AFM probes have higher self-capacitance and lack shielding, traits traditionally viewed as disqualifying for GHz signal delivery. Without cancellation, the large baseline reflection would saturate the amplification chain or preclude the necessary high signal gain. Yet, by careful impedance design and leveraging the inherent advantages of self-referenced detection and modern AFM probes, the team not only made it work—they achieved new state-of-the-art performance **(Fig. 4)**.

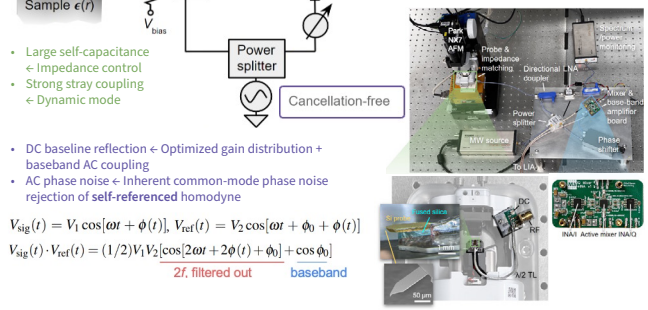


Fig. 4.
Why & How It Works.

Key Innovations

- **Impedance Engineering:** The probe structure is integrated into a 50-ohm transmission line by controlling the thickness and dielectric properties of the insulating spacer between the probe carrier chip and the ground plane. This minimizes stray capacitance and maximizes responsivity.
- **Dynamic Mode Signal Extraction:** Rather than measuring at DC or low frequencies, the MIM signal is extracted at the cantilever's mechanical resonance frequency (typically hundreds of kilohertz), where noise is reduced.
- **AC-Coupled Signal Path:** After the reflected signal is down-converted through a self-referenced homodyne mixer, it is AC-coupled, removing the DC baseline without any need for cancellation.
- **Common-Mode Noise Rejection:** The self-referenced homodyne detection scheme ensures that shared phase noise between the reference and signal paths cancels out, greatly reducing noise in the baseband output.
- **Tighter Integration:** All RF signal conditioning, demodulation, and baseband amplification electronics, including baluns, an active mixer and low-noise instrumentation amplifiers, are integrated onto a single printed circuit board. This minimizes wiring losses, thermal drift, and noise pickup.

Benchmarking Against Commercial Systems

To assess performance, the team performed side-by-side experiments using both their cancellation-free system with monolithic silicon probes and an expensive, cutting-edge commercial MIM platform. The test sample was an array of thin aluminum dots deposited on a silicon dioxide substrate—chosen for its strong capacitive contrast and sharp material boundaries **(Fig. 5)**.

Results:

- **Improved Spatial Resolution:** Thanks to the smaller (and more consistent) tip radius of commercial silicon AFM cantilevers, the new system resolved features more crisply, particularly small dust particles and edges, in both topography and MIM.
- **Higher MIM signal-to-noise-ratio (SNR):** The SNR in the MIM channels are comparable, if not better, despite a sharper tip and the absence of cancellation or shielding.

Breaking the Sensitivity Barrier: Thermal Noise-Limited Operation and Long Averaging Time

One of the most striking findings from Prof. Ma's group was that their system operated at the **Johnson-Nyquist thermal noise limit** for powers below approximately −10 dBm. This means the system's sensitivity is constrained only by fundamental physics, not by technical or environ-

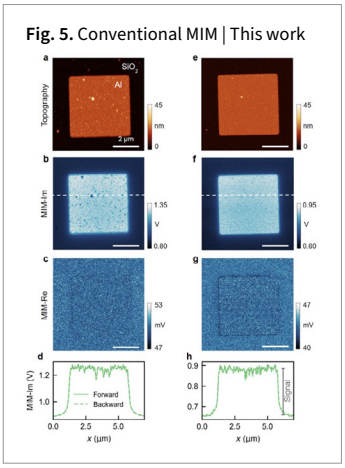


Fig. 5. Conventional MIM | This work

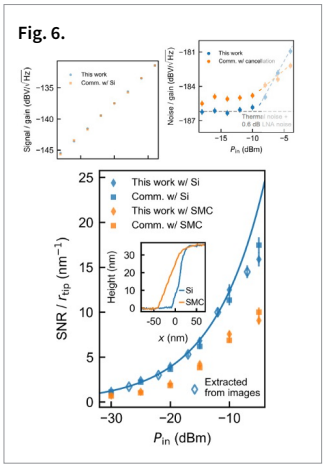


Fig. 6.

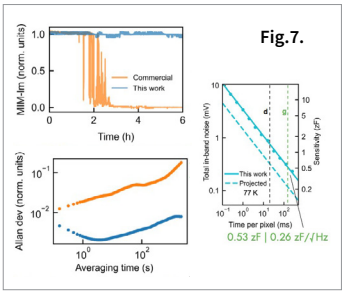
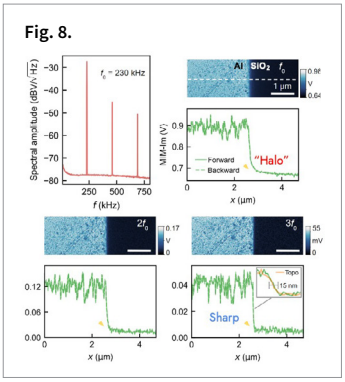


Fig.7.



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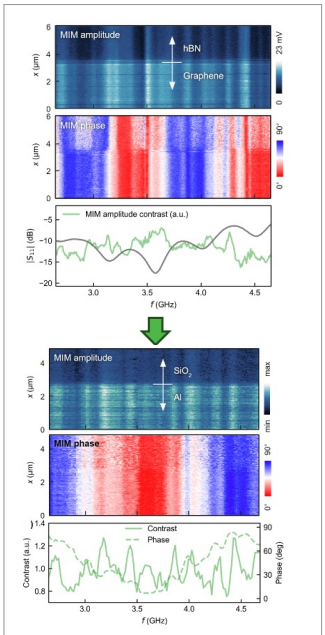


Fig. 9. Sig-Ref length matched

mental noise (Fig. 6).

The implications are profound. It suggests that the system has reached the theoretical maximum sensitivity for room temperature operation—and that any further improvement in noise performance must come from cooling the combined tip-sample system and the microwave pre-amplifiers.

Moreover, the cancellation-free architecture eliminates drift-induced saturation completely and enables significantly longer data acquisition times, resulting in higher SNR and improved sensitivity. In a 6.5-hour real-world scan of a graphene monolayer encapsulated in hexagonal boron nitride (hBN), the system detected minute variations in subsurface conductivity with an absolute capacitive sensitivity of **0.53 zF** (1 zF = 10-21 farad), corresponding to a normalized sensitivity of **0.26 zF/√Hz**—a new record for capacitance-based scanning probe technique, to the best of the group's knowledge (Fig. 7).

Enhanced Spatial Resolution via Harmonic Demodulation

Beyond improving sensitivity, the much higher baseband bandwidth (~1 MHz) of the new architecture also supports higher spatial resolution through **harmonic demodulation**. By extracting MIM signals not only at the cantilever's fundamental mechanical resonance but also its 2nd and 3rd harmonics, the team revealed increasingly localized electrical interactions.

This enabled them to resolve sharper transitions in conductivity with spatial resolution comparable to the topographic limit imposed by the tip radius, while suppressing artifacts such as halo effects seen in traditional MIM images.

New Modalities: Expanding What MIM Can Measure

1. Continuous-Frequency MIM Spectroscopy

Conventional MIM spectroscopy typically requires changing hardware and re-tuning multiple amplitude and phase controllers in the cancellation line to switch frequencies. This makes multi-frequency measurements cumbersome and inconsistent.

With the new architecture, Prof. Ma's group demonstrated continuous-frequency spectroscopy using a broadband resistive matching network coupled with matching signal and reference path lengths. In early data, they measured spatially resolved amplitude and phase variations between 2.7 and 4.6 GHz at a metal-insulator boundary—paving the way for more detailed modeling of frequency-dependent permittivity in nanostructures (Fig. 9).

2. Broadband Photoconductivity with Fourier Transform MIM

In a novel adaptation, the group used the MIM tip as a localized photoconductivity detector in a Fourier-transform interferometer. By focusing modulated light onto the tip-sample junction and capturing the resulting interferogram in the MIM signal, they extracted spectrally resolved photoresponse data with nanoscale spatial resolution and picometer-scale spectral resolution (Fig. 10).

3. Nonlinear MIM via Harmonic Detection

Finally, the team developed a formalism to interpret MIM signals at 2nd and 3rd harmonics of the excitation frequency, using concepts from nonlinear optics. By solving nonlinear admittance equations, they linked these harmonics to higher-order dielectric and conductive responses of materials— offering a powerful means of probing the rich information contained in local electrical nonlinearities, particularly relevant to semiconductors, strongly-correlated materials, and superconductors.

Implications and Outlook

The redesigned MIM architecture represents a transformative shift in non-invasive nanoscale electronic imaging. It dramatically reduces cost and complexity barriers while improving performance in both sensitivity and spatial resolution—making MIM accessible to broader research and industrial communities.

Future directions include integration with techniques such as STM, NSOM, and MFM, as well as operation at cryogenic temperatures to achieve yoctofarad (10^{-24} F) sensitivity. Additional goals involve applying MIM in biological and aqueous environments, developing highly quantitative methods for extracting local conductivity or doping concentrations, and enabling broader commercial deployment.

As Prof. Ma concluded, "By simplifying the system and letting the physics shine through, we've opened the door to broader adoption, deeper and more quantitative insights, and entirely new kinds of measurements that were previously out of reach."

Fig. 10.

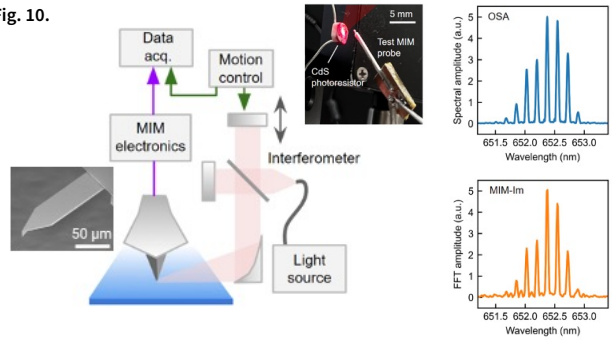
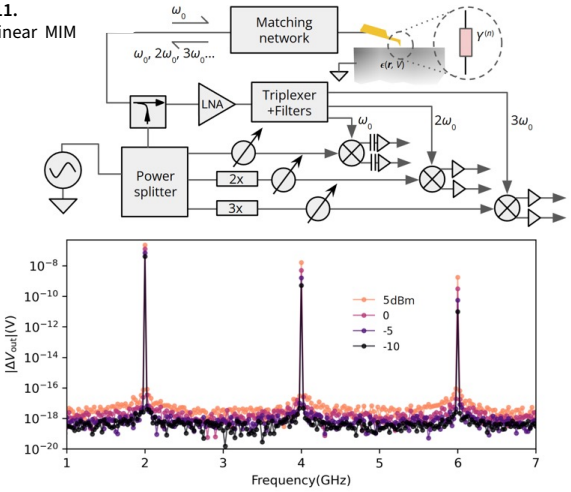


Fig. 11.
Nonlinear MIM



About Prof. Eric Ma

Dr. Ma is an Assistant Professor in Physics and EECS and holds the Georgia Lee Chair in Physics. He is also a Faculty Scientist at the Lawrence Berkeley National Laboratory. His research focuses on probing and controlling wave-matter interactions in condensed matter systems at deeply sub-wavelength scales using advanced instrumentation and AI/ML techniques. As an educator, Eric is passionate about using modern software and hardware tools to enhance undergraduate physics research and education, especially in laboratory courses.

For more information visit https://sites.google.com/berkeley.edu/ma-lab

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INSIDE THE ATOMICALLY THIN FRONTIER: HOW SPM IS ADVANCING 2D TMD CHARACTERIZATION AND DEVICE INTEGRATION

Dr. Albert Minj, imec R&D Nano Electronics and Digital Technologies, Belgium
Adapted from Presentation, Edited by NanoScientific

At the 2024 NanoScientific Symposium Americas, Dr. Albert Minj of imec presented a masterclass in precision metrology at the nanoscale. His talk offered a rare and revealing glimpse into the challenges and opportunities at the intersection of advanced scanning probe microscopy (SPM) and two-dimensional (2D) semiconductors—particularly transition metal dichalcogenides (TMDs), such as molybdenum disulfide (MoS_2) and tungsten disulfide (WS_2).

Imec, headquartered in Belgium, is a world-leading R&D hub known for its work in logic, memory, photonics, and energy. As part of imec's metrology department, Minj operates at the center of this ecosystem, helping drive breakthroughs in characterization tools critical for the semiconductor industry's transition to atomically engineered devices.

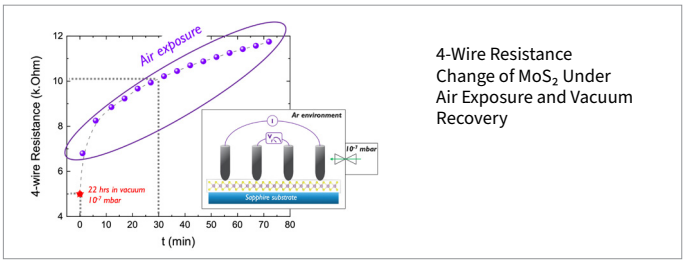
Monolayers, Mobility, and the Materials Challenge

The semiconductor industry is reaching physical and practical limits with traditional silicon-based scaling. Enter 2D materials. These atomically thin layers—just a few atoms thick—offer tantalizing potential for continued device miniaturization with improved electrostatics, scalability, and performance.

To make these materials viable for logic applications, however, imec is targeting the growth of high-quality, single-crystal monolayers across full wafer scales—up to 200 mm and beyond. Both n-type and p-type conduction must be achieved with high mobility. But the success of these efforts depends not just on growth processes, but also on the precision of the tools used to measure them.

Environment Matters: A Case Study in Vacuum vs. Air

A compelling early example Minj shared was the behavior of monolayer MoS_2 exposed to ambient conditions. Using four-point probe measurements on a sample grown on sapphire, imec researchers observed a dramatic increase in resistance—doubling within just 30 minutes of air exposure.



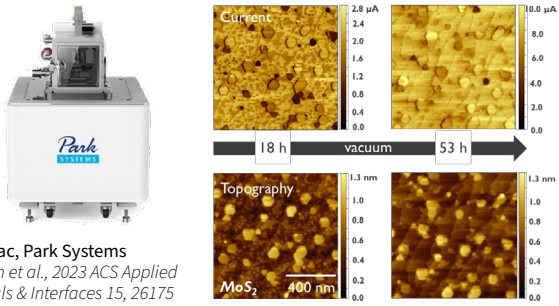
4-Wire Resistance Change of MoS_2 Under Air Exposure and Vacuum Recovery

This degradation is caused by interaction with environmental elements such as oxygen, water vapor, and airborne contaminants, which interfere with the material's electrical properties. Significantly, this effect was reversible. After prolonged exposure to high vacuum, the initial conductivity was largely restored.

Such results highlight the inadequacy of ambient characterization for truly understanding 2D materials. They also motivated the team to turn to high-vacuum conductive atomic force microscopy (C-AFM) using Park Systems instrumentation to delve deeper.

Conductive AFM: Revealing the Surface Transformation

C-AFM current maps of monolayer MoS_2 showed dramatic changes over time during prolonged scanning under vacuum. Initially, the maps revealed distinct inhomogeneities—patches of low conductivity and small features that suggested structural discontinuities or contamination. However, after more than 50 hours of continuous scanning, the current maps homogenized, and many of the patches vanished.



NX-Hivac, Park Systems
J Serron et al., 2023 ACS Applied Materials & Interfaces 15, 26175

The conclusion? The AFM tip, in vacuum, had physically displaced or removed intercalated water molecules and contaminants trapped between the 2D layer and its substrate. This finding was corroborated by STM (scanning tunneling microscopy) data, which visually captured intercalated water pockets and showed their disappearance over time.

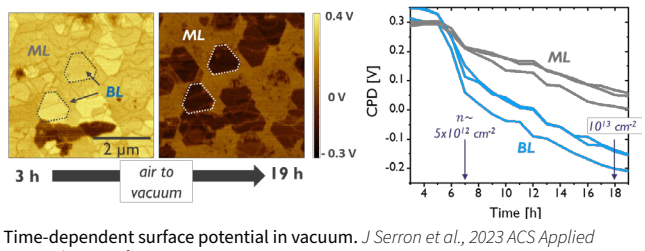
This result opens the door to a potentially transformative approach: correlating intercalation signatures with underlying defectivity—a path toward rapid screening of TMD quality.

Kelvin Probe Force Microscopy: Layer-Dependent Potential

To further analyze these transformations, Minj's team turned to Kelvin Probe Force Microscopy (KPFM), which maps the surface potential with nanometer precision. In 2D materials, the contact potential difference (CPD) measured by KPFM is not simply a function of the material's work function. Rather, it is heavily influenced by interface charges, environmental factors, and the number of layers.

Initial CPD maps showed that bilayer regions exhibited different potentials compared to the monolayer background. However, after prolonged vacuum exposure, this contrast reversed—an indication that charge distributions had changed. Applying theoretical models, the team estimated changes in carrier concentration, observing an increase from approximately 5×10^{12} to 1×10^{13} cm^{-2}.

Yet, even in deep accumulation conditions, some areas—especially grain boundaries and bilayer island edges—showed persistent difficulty in screening charges.



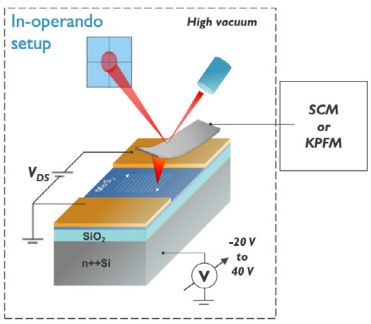
Time-dependent surface potential in vacuum. *J Serron et al., 2023 ACS Applied Materials & Interfaces 15, 26175*

From Passive Mapping to Active Devices: In Operando SPM

Beyond passive characterization, Minj emphasized the power of in-operando SPM—probing a device while it is functioning. Using a standard FET structure with a monolayer MoS_2 channel, nickel source–drain contacts, and a back-gated silicon substrate, the team used KPFM to monitor CPD profiles along the channel under varying gate voltages.

The maps showed band bending and potential shifts consistent with carrier injection and depletion. This extended to the contact edge—where the metal-semiconductor junction often limits device performance—allowing the team to observe how the interface responded to gate bias in real time.

By comparing the results to simulations, the team found that agreement broke down in some ranges—likely due to interface traps. Further, CPD delta analysis helped quantify local band bending behaviors.

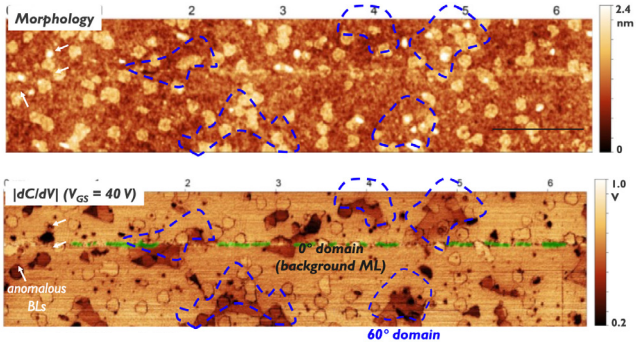


Grain Boundaries and Orientation Effects

One of the most illuminating applications of in-operando KPFM was the visualization of grain boundaries. Minj's team classified segments based on their alignment to current flow. Those perpendicular to current flow direction created carrier accumulation zones and acted as potential barriers, while parallel segments showed little impact.

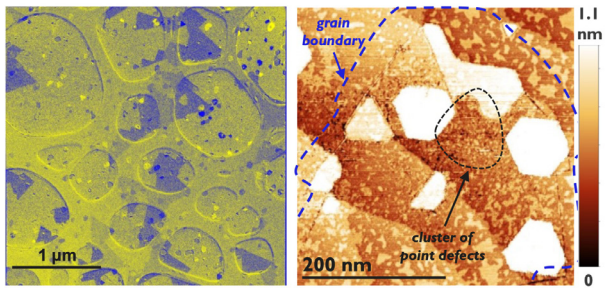
Scanning capacitance microscopy (SCM) further complemented these insights. Differential capacitance measurements showed that **misoriented grains and certain bilayer domains harbored higher electrically active defect densities**, as reflected in suppressed capacitance signals.

Statistical distribution of misoriented grains.
A Minj et al., 2024 ACS nano 18, 10653



Corroboration with TEM and STM

TEM analysis revealed that 60°-rotated domains—clearly mapped through polarity contrast—harbored point defect clusters, as confirmed by STM imaging. These findings validated SCM and KPFM measurements, showing that electrical behavior tracked closely with crystallographic and chemical non-uniformities.



Looking Ahead: Metrology Perspectives

Minj concluded by outlining key strategic directions for the field:

- **Standardization of characterization methods** is imperative to eliminate variability across labs and instruments. The 2D materials community urgently needs consensus on environmental control, measurement parameters, and interpretation models.
- **Integration of metrology tools with probe stations** will enable high-throughput, in-operando device measurements—transforming how labs screen new materials and optimize fabrication.
- **Fast, quantitative defect analysis** using tools like STM, c-AFM, friction mapping, and others should become routine to identify local disruptions at the atomic scale.

Conclusion

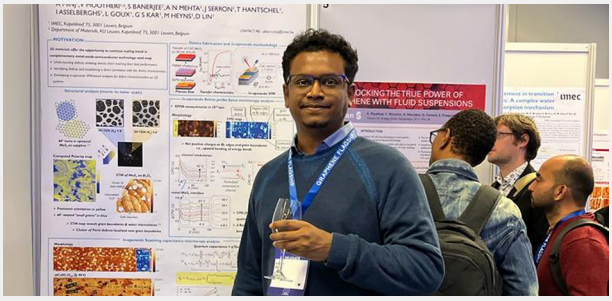
Dr. Albert Minj's work exemplifies the evolving role of scanning probe microscopy—not merely as an imaging technique, but as a quantitative platform that drives materials science forward. From water intercalation and defect mapping to in-operando device diagnostics, scanning probe microscopy is rapidly becoming the keystone technology for developing next-generation 2D semiconductors.

As the semiconductor industry marches into the atomic era, the future of logic devices may well hinge on what SPM reveals—one defect, one grain boundary, one layer at a time.

About Dr. Albert Minj

Dr. Minj has worked with Scanning Probe Microscopy (SPM) since 2009. He earned his PhD from the University of Bologna and completed postdoctoral research at CNRS in France, where he studied crystallographic defects in wurtzite crystals using Transmission Electron Microscopy and developed surface photovoltage KPFM for defect analysis in ultra-wide bandgap materials. In 2018, he joined imec in Belgium—the birthplace of Scanning Spreading Resistance Microscopy (SSRM)—where he is currently a researcher.
From 2020 to 2022, he led a Marie Curie Fellowship advancing SPM techniques for doping and defect analysis in GaN power devices and 2D materials. His ongoing research focuses on 2D systems, carbon nanotubes, and new AFM modes. He works within imec's 12-member SPM team, led by Dr. Thomas Hantschel, which supports both scientific innovation and metrology across imec's research programs.

Minj at Graphene week, Gothenberg, Sweden 2023.



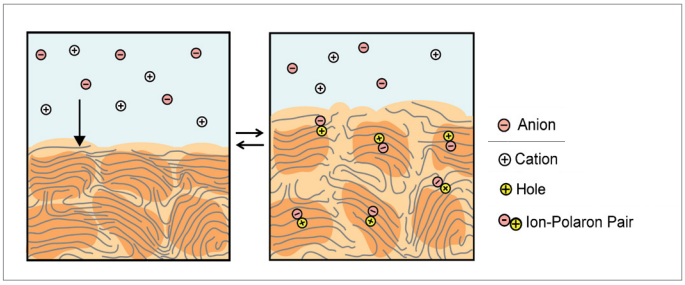
MAPPING IONS AND POLARONS WITH PHOTOINDUCED FORCE MICROSCOPY: UNLOCKING THE INNER WORKINGS OF ORGANIC MIXED CONDUCTORS

Prof. Connor Bischak, Department of Chemistry, University of Utah
Adapted from Presentation, Edited by NanoScientific

Organic mixed ionic-electronic conductors (OMIECs) are ushering in a new paradigm in the field of soft electronics. Distinguished by their ability to transport both electronic and ionic charges, OMIECs offer an unprecedented platform for interfacing electronic devices with dynamic environments such as biological systems. In a recent lecture delivered at the 2024 NanoScientific Symposium Americas, Prof. Connor Bischak presented his group's latest advances in visualizing charge dynamics in these complex systems using **Photoinduced Force Microscopy (PiFM)**.

Mixed Conduction and Its Challenges

Unlike traditional inorganic semiconductors like silicon, which are purely electronic conductors, OMIECs combine the conductive properties of conjugated polymers with side chains that facilitate ionic transport. This dual transport mechanism enables **volumetric electrochemical doping**, in which ions from an adjacent electrolyte penetrate the bulk of the polymer to compensate for injected electronic carriers. This is in stark contrast to the surface-confined charge accumulation in conventional materials, which is limited by electric double layer formation.



Prof. Bischak introduced the core concepts behind these materials, focusing on conjugated polymers functionalized with ethylene glycol-type side chains. These side chains allow the ingress of ions—typically from aqueous electrolytes—into the polymer backbone under applied bias, forming polaron-ion pairs. These pairs represent the basic charge storage and transport units in OMIEC-based devices such as organic electrochemical transistors (OECTs) and bioelectronic interfaces.

However, a major challenge in the development and optimization of these materials is the lack of spatially resolved techniques that can directly visualize where ions and polarons accumulate during operation. Without such information, the design of more efficient devices remains largely empirical.

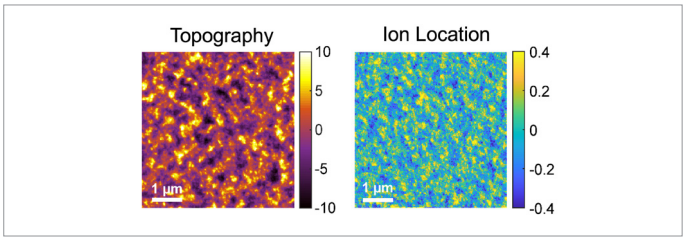
PiFM: A Window into Nanoscale Charge Distribution

To address this gap, Prof. Bischak's group with graduate student Seth Jackson has employed Photoinduced Force Microscopy (PiFM)—a hybrid scanning probe technique that combines the spatial resolution of atomic force microscopy (AFM) with the chemical sensitivity of infrared spectroscopy. PiFM detects the photoinduced forces between the AFM tip and the sample, enabling the mapping of specific vibrational modes associated with distinct chemical species such as ions in a polymer matrix.

Through PiFM, the team achieved nanoscale chemical imaging of ion and polaron distributions in polymer thin films under electrochemical bias. This method allowed them to observe how different regions of the film respond heterogeneously to doping, revealing dynamic rearrangements during charge injection and extraction.

For instance, they identified domains within the polymer where ions preferentially accumulated, forming more heavily doped (polaron-rich) regions, while other areas remained relatively undoped. These spatial heterogeneities are likely rooted in the semicrystalline morphology of OMIECs, where crystalline domains offer different levels of ionic accessibility compared to amorphous regions.

Such insights are crucial, as they help explain the observed electrical behavior of devices and guide the synthesis of polymers with more uniform or controllable doping profiles. The ability to directly image the formation and distribution of polaron-ion pairs at the nanoscale represents a significant advance in the field.



Beyond Microscopy: Integrated Materials Discovery

While PiFM has become a central tool in the Bischak Lab's arsenal, the group also adopts a broader strategy for materials development. Their research is supported by robotic synthesis platforms inspired by arcade-style claw machines, capable of performing **automated deposition and characterization** of polymer films. This setup enables rapid screening of candidate materials under reproducible conditions, with integrated photoluminescence and absorption spectroscopy for immediate feedback.

These automation tools allow the lab to systematically explore the effects of processing variables—such as solvent choice, coating speed, and electrolyte composition—on film morphology and electrochemical response. The data generated can be coupled with PiFM insights to construct comprehensive structure–function relationships.

In addition, Prof. Bischak's team investigates **phase transitions in 2D hybrid perovskites** and other soft materials, using time-resolved optical spectroscopy to track melting transitions and dynamic structural rearrangements. This work complements their study of OMIECs by expanding their understanding of ion–lattice interactions and charge transport in mixed conduction systems.

Toward Better Bioelectronic Devices

One of the key application targets for OMIECs is in bioelectronics, where materials must interface effectively with the ionic environments of cells and tissues. The volumetric capacitance enabled by OMIECs allows

them to transduce weak biological signals with high fidelity and low impedance, making them ideal for neural interfaces, biosensors, and drug delivery systems.

By uncovering the spatial dynamics of electrochemical doping, Prof. Bischak's work provides foundational knowledge to optimize these interfaces. More homogeneous ion and polaron distributions could lead to more stable device operation, faster switching times, and improved biocompatibility.

Conclusion and Future Directions

Prof. Connor Bischak's work represents a fusion of advanced characterization, synthetic chemistry, and device engineering, all aimed at solving one of the central challenges in soft electronics: understanding and controlling mixed conduction at the nanoscale. By leveraging the unique capabilities of Photoinduced Force Microscopy (PiFM), his group has demonstrated the ability to map the location of ions and polarons within conjugated polymer thin films with high spatial precision.

Their findings indicate that ions tend to preferentially localize within crystalline regions of the polymer—a critical insight that may influence how these materials are processed and engineered for device performance. The group has also proposed a promising strategy to improve n-type OMIECs by blending polymers with complementary electronic and ionic transport properties, potentially overcoming historical challenges in n-type material design.

Looking ahead, the Bischak Lab is exploring how to extend PiFM into solid–liquid interfaces, which would enable real-time probing of electrochemical processes in environments that more closely mimic operating devices or biological conditions.

As the field moves toward increasingly complex and integrated devices, the ability to "see" where charges go—and how they interact with the surrounding medium—will be essential. PiFM is proving to be not just a tool of analysis, but a guidepost for the rational design of the next generation of responsive, bio-integrated, and high-performance soft electronics.

What Is PiFM?

Photoinduced Force Microscopy (PiFM) is a hybrid analytical technique that combines elements of Atomic Force Microscopy (AFM) and infrared (IR) spectroscopy to achieve label-free, non-destructive chemical imaging at the nanoscale.

While AFM excels at imaging topography by detecting minute forces between a sharp tip and the sample surface, it traditionally lacks chemical specificity. IR spectroscopy, on the other hand, identifies molecular vibrations specific to chemical bonds but suffers from diffraction-limited spatial resolution (typically >1 µm). PiFM cleverly integrates the two.

How Does PiFM Work?

At the heart of PiFM is the detection of photo-induced forces between an AFM tip and the sample surface under infrared illumination:

1. **Excitation:** A tunable IR laser illuminates the sample. If the frequency of the IR light matches a vibrational mode of a chemical group in the sample, it absorbs the light, leading to localized photothermal expansion or a change in dipole moment.
2. **Force Generation:** These light-induced phenomena generate a localized attractive force between the tip and sample, which oscillates at characteristic frequencies.
3. **Force Detection:** The AFM cantilever senses these oscillating forces. By demodulating the response at specific harmonics of the cantilever's resonant frequency, PiFM distinguishes the photo-induced force from background noise.
4. **Spectroscopy and Imaging:** By scanning across the surface and tuning the laser, PiFM maps out both the topography and chemical composition of the sample with nanometer resolution—often better than 10 nm.

Why Is PiFM Unique?

PiFM stands out because it offers:

- Simultaneous topographic and chemical imaging
- Sub-10 nm spatial resolution in chemical maps
- Non-contact, non-destructive analysis
- Label-free identification of functional groups and material phases
- Sensitivity to local dielectric, optical, and vibrational properties

This makes it ideal for investigating heterogeneous, soft, or nanostructured materials—including polymers, biological tissues, 2D materials, and hybrid devices.

Applications of PiFM

PiFM has been successfully applied in diverse scientific and technological domains:

- Polymer and composite materials: Mapping domain compositions, phase separation, and chain orientation
- 2D materials and semiconductors: Probing heterostructures, grain boundaries, and local doping
- Energy materials: Investigating degradation and ion migration in batteries and fuel cells
- Biomaterials and tissues: Identifying proteins, lipids, and other macromolecules in situ
- Organic electronics: Studying morphology and doping dynamics in organic solar cells and transistors

Watch how PiFM works on YouTube:
https://www.youtube.com/watch?v=by3tNOCWw1w

About Prof. Connor Bischak

Dr. Bischak is an Assistant Professor of Chemistry at the University of Utah (appointed 2021). He earned a B.S. in Chemistry from Haverford College (2010), completed a Fulbright Fellowship at Ruhr University Bochum (2010-11), and received his PhD in Chemistry from UC Berkeley in 2017 under Prof. Naomi Ginsberg. He then served as a Washington Research Foundation and Cottrell Postdoctoral Fellow with Prof. David Ginger at the University of Washington (2018-21), focusing on organic mixed ionic–electronic conductors.

Bischak's lab develops in situ nanoscale imaging and spectroscopic platforms to unravel how structural phase transitions, ion transport, and electronic processes couple in soft hybrid semiconductors—such as layered metal-halide perovskites and conjugated polymers—aiming to inform next-generation energy, lighting, and bioelectronic technologies. His honors include an ACS PHYS Young Investigator Award, a Cottrell Postdoctoral Fellowship, and appointment to the Early Career Board of ACS Applied Electronic Materials. He is committed to training a diverse cohort of scientists through automation-enabled, interdisciplinary materials research.

For more information, check out Prof. Bischak's website at www.bischaklab.com.

Prof. Bischak (first from left standing) and his team

INFORMATIVE VIBRATIONS: QUANTITATIVE IMAGING OF ELECTROSWELLING IN ORGANIC MIXED IONIC–ELECTRONIC CONDUCTORS

Prof. Tobias Cramer, Dept. of Physiscs, University of Bologna, Italy
Adapted from Presentation, Edited by NanoScientific

In the rapidly evolving field of bioelectronics, one of the grand challenges lies in bridging the material divide between biology and electronics. Our current technological landscape is dominated by rigid, inorganic semiconductors, while biological systems operate through soft, ionic mechanisms. Prof. Tobias Cramer of the University of Bologna addresses this critical disjunction through a fascinating study of organic mixed ionic electronic conductors (OMIECs) and a pioneering AFM-based technique that enables the **quantitative imaging of electroswelling**—a key electromechanical response in these materials.

This innovative work, presented at the 2024 NanoScientific Forum Europe (NSFE), provides deep insights into the electromechanical dynamics of OMIECs, particularly focusing on materials such as PEDOT:PSS and polypyrrole doped with dodecylbenzenesulfonate (PPy:DBS). These insights carry significant implications for designing bioelectronic interfaces, actuators, and organic electrochemical transistors (OECTs).

The Biological-Inorganic Divide

The human brain processes information through ions and neurotransmitters in a soft, aqueous environment. In contrast, modern electronics rely on electrons moving through solid-state semiconductors. Attempts to interface these disparate systems—such as using metal electrodes to connect neurons with circuits—often result in functional limitations and long-term biocompatibility issues. The search for materials that can mimic biological ionic transport while maintaining electronic conductivity is at the heart of OMIEC research.

OMIECs, such as PEDOT:PSS, offer a unique duality. They consist of a π-conjugated polymer network that allows electronic charge transport, embedded in a polyanionic matrix that supports ionic conduction. This dual functionality enables these polymers to interact intimately with biological systems while still interfacing with traditional electronic devices.

The Phenomenon of Electroswelling

When OMIECs undergo electrochemical doping (oxidation or reduction), ions and water are inserted or removed from the polymer matrix, leading to a volumetric change—an effect known as **electroswelling**. This phenomenon has been exploited in applications ranging from soft actuators to neural probes. However, electroswelling can also be problematic, leading to mechanical strain and delamination in thin films used in transistors and sensors.
Understanding the dynamics of electroswelling—its speed, magnitude, and underlying mechanisms—is essential for engineering OMIEC-based devices with greater reliability and performance. Traditional measurement techniques, however, fail to resolve these processes at the nanoscale with sufficient spatial and temporal resolution.

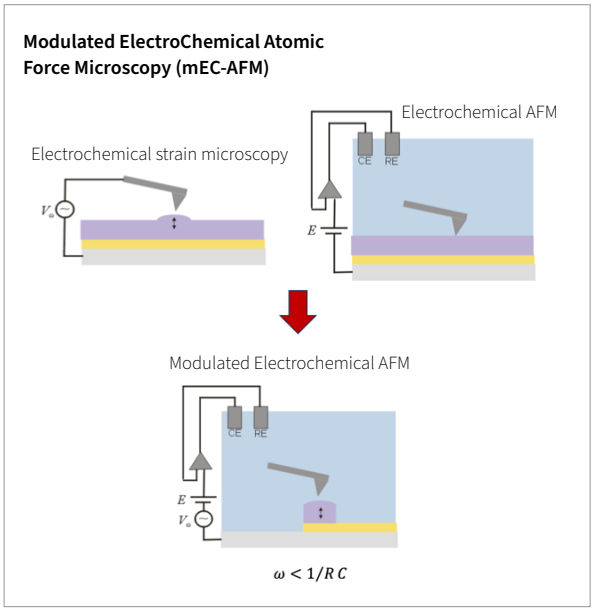
A New AFM Paradigm: mEC-AFM

To tackle these challenges, Prof. Cramer's team developed a novel **modulated ElectroChemical Atomic Force Microscopy (mEC-AFM)** approach. By integrating the principles of electrochemical AFM with those of electrochemical strain microscopy, the method introduces an AC voltage modulation to microelectrodes coated with OMIEC films. This enables simultaneous acquisition of:

- Ionic current responses
- Height modulation (swelling amplitude)
- Phase shifts of mechanical deformation

These nanoscale measurements are achieved using **PinPoint mode** AFM, ideal for soft polymer films where conventional contact mode could damage the sample.

One of the key advantages of mEC-AFM is its ability to perform **multidimensional spectroscopy**—tracking the electromechanical response over a wide frequency range and film thicknesses. The results can be visualized spatially (as images) and spectroscopically (as frequency-dependent plots), offering a uniquely holistic view of OMIEC behavior.

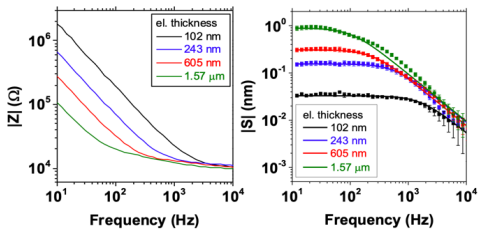


Key Findings in PEDOT:PSS

Using PEDOT:PSS as a model system, the group demonstrated that the swelling response scales linearly with the injected ionic charge—regardless of film thickness, frequency, or position. The **electroswelling coefficient** they extracted was approximately **0.08 nm per nanoCoulomb of charge**, correlating directly with the hydration volume of sodium ions.

This linearity and consistency across experimental conditions pointed to a simple yet powerful model: the swelling is governed by the volume of hydrated cations entering the polymer matrix. Additional experiments with polyanions confirmed that swelling is indeed due to cation ingress and not anion egress.

Interestingly, the hydration radius measured inside the film was about **50% smaller than in free water**, attributed to coordination of the ions with the polyanionic PSS matrix. This reduced hydration reflects the constrained environment within the polymer and provides a window into the microscopic interactions driving electromechanical behavior.



Modulated ElectroChemical Atomic Force Microscopy (mEC-AFM).

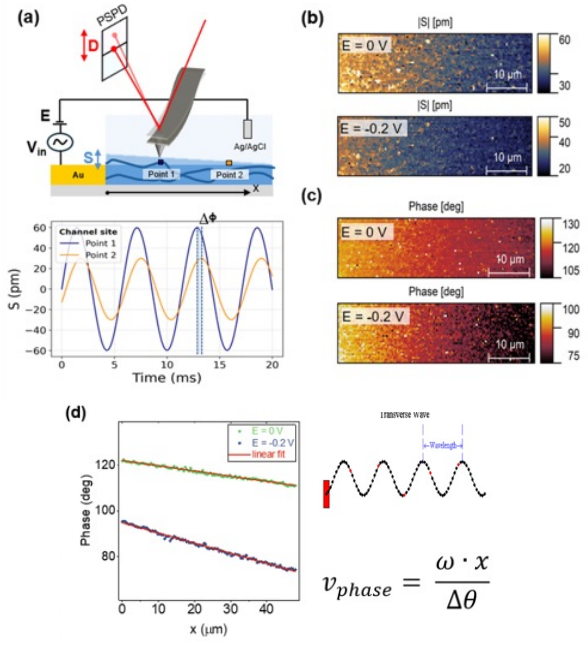
Complex Dynamics in Polypyrrole (PPy:DBS)

While PEDOT:PSS presents relatively stable and predictable electroswelling, other OMIECs like **PPy:DBS** exhibit more dramatic and complex responses. Thicker PPy:DBS films (up to 3 µm) showed electroswelling coefficients nearly **10 times higher** than PEDOT:PSS, with a strong dependence on both thickness and electrochemical potential.

To unravel these effects, Prof. Cramer's team employed advanced impedance modeling using the **Transfer Line Method (TLM)**. This technique models the film as a 1D transmission line of resistors and capacitors, allowing the reconstruction of **subsurface swelling and charge profiles** from spectroscopic data. The analysis revealed that in reduced films, most swelling occurs deep within the bulk, whereas in oxidized states, swelling is confined to near the surface.

These findings suggest that **swelling is not merely a surface phenomenon** but a result of dynamic microstructural rearrangements within the film—shifts that are essential for optimizing actuation performance.

Determination of phase velocity inn OMIEC channels with mEC-AFM.
F. Bonnafe et al. in preparation



Mapping Signal Propagation

In another extension of their work, the team applied mEC-AFM to study **signal propagation** along OMIEC channels. In these experiments a modulated AC signal is injected from one side into the OMIEC channel to generate a combined excitation of potential and swelling. The resulting electrochemical strain waves propagate along the channel and their local amplitude and phase shift is measured with the AFM experiment.

From these findings, the team could directly determine the **phase velocity**, that is the speed at which combined ionic/electronic signals propagate along thin OMIEC channels. Interestingly, it was found that the speed is **slower than signal propagation along biological axons**, underscoring the limitations of synthetic mixed conductors. This velocity was found to depend primarily on **electronic mobility** and **ionic capacitance** of the material. Notably, highly ionic hydrogels, while biocompatible, tended to slow down signal transmission due to excessive ion loading.

Implications and Outlook

The implications of Prof. Cramer's research are both foundational and practical. His mEC-AFM technique opens a new window into the electromechanical behavior of soft, multifunctional materials, offering unprecedented resolution and interpretability. The work also advances our understanding of how ions and electrons coexist and interact in confined, hydrated environments—insights critical to improving bioelectronic devices, sensors, and soft actuators.

Moreover, the research redefines how we view "material performance" in biointerfaces—not just in terms of conductivity or swelling, but in the **spatiotemporal orchestration of ionic and electronic processes**. As artificial tissues, neural prosthetics, and wearable electronics become increasingly viable, tools like mEC-AFM will be indispensable in guiding their design and ensuring their compatibility with the body's delicate electrochemical symphony.

Prof. Cramer and his team at the University of Bologna have thus illuminated a crucial frontier—where physics meets physiology, and where the future of bioelectronics takes shape not only through molecules but through movement.

About Prof. Tobias Cramer

Dr. Cramer is an Associate Professor in the Department of Physics at the University of Bologna, Italy. He and his research team study semiconductors and charge transfer processes using advanced microscopic techniques. Their work aims to translate fundamental scientific insights into the development of novel sensors and transducer devices, with a strong emphasis on applications in bioelectronics. Originally from northern Germany, Tobias values the cultural richness and historical legacy of Bologna, drawing inspiration from the city's vibrant academic environment and the University of Bologna's long-standing tradition of excellence.

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For more information, contact *tobias.cramer@unibo.it* or look at *https://site.unibo.it/semiconductor-physics/en*

Prof. Cramer (2nd from right, bottom) in Bologna Semiconductor Physics Group

ENABLING NANOELECTRONICS WITH ADVANCED SPM TECHNIQUES

Dr. Bikas C. Das, Associate Professor, School of Physics, IISER Thiruvananthapuram, Kerala, India
Adapted from Presentation, Edited by NanoScientific

As electronics and optoelectronics evolve in a data-driven world, the need for smaller, faster, and more efficient devices grows. At the 2024 NanoScientific Symposium SE Asia in Bangkok, Professor Bikas C. Das of IISER Thiruvananthapuram showcased how advanced scanning probe microscopy (SPM) techniques are addressing this challenge. His talk highlighted how tools like Conductive AFM, Kelvin Probe Force Microscopy, and Electrostatic Force Microscopy are uncovering nanoscale mechanisms essential to next-generation devices—from memristors to photodetectors and engineered 2D semiconductors.

Motivation: Smaller, Smarter, More Efficient

Modern electronic systems–from data centers to edge devices–demand components that are increasingly miniaturized, energy-efficient, and capable of advanced functionalities like in-memory computing or neuromorphic learning. This technology convergence calls for new materials, architectures, and diagnostics.

Prof. Das emphasized that merely shrinking devices is insufficient; understanding their internal physics at the nanoscale is equally vital. “We are not only interested in storing information but also in mimicking the brain’s learning pathways through resistive switching,” he noted. For such breakthroughs, SPM tools provide unparalleled insight into the electro-mechanical behavior of novel materials.

What is Resistive Switching?

At the heart of Prof. Das’s memristor research is the phenomenon of **resistive switching (RS)** - the ability of a material or device to reversibly switch between different resistance states when subjected to an external voltage.

Unlike traditional resistors that maintain a constant resistance, RS devices exhibit nonlinear and bistable I-V characteristics. At a critical voltage threshold, they "switch" from a high-resistance (OFF) state to a low-resistance (ON) state, and vice versa. This behavior is non-volatile, meaning the device retains its state even when the power is turned off.

Resistive switching is the foundational mechanism behind emerging **Resistive Random-Access Memory (ReRAM)**, offering advantages such as:

- **High switching speed** (nanoseconds range)
- **Low energy consumption**
- **Scalability to nanometer dimensions**
- **Potential for in-memory and neuromorphic computing**

RS can be **filamentary** (localized conductive paths formed by ion migration or defect rearrangement) or **interface-driven**, depending on the material system and device configuration.

The wide range of materials capable of RS - including oxides, organic films, chalcogenides, and 2D materials like MXenes - opens up vast possibilities for customizing device performance.

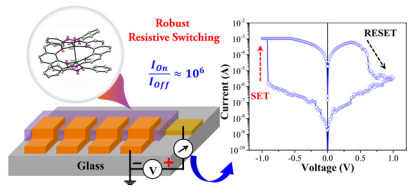


Fig. 1. A typical pinched hysteresis I-V characteristic of bistable resistive switching device with the device schematic.

Part I: C-AFM for Memristor Innovation

One of the most intriguing parts of Prof. Das’s research focuses on RS in memristors—devices capable of toggling between multiple stable resistance states. These devices hold promise for data storage, logic operations, and neuromorphic computing.

The MXene Advantage

At the core of this work lies **MXene**, a family of two-dimensional materials derived from etching MAX-phase compounds. With high conductivity, mechanical strength, and tunable surface chemistry, MXenes are attractive candidates for memristive devices.

To fabricate these, Prof. Das’s group utilized a biphasic technique—depositing ultrathin films at the interface of two immiscible liquids, allowing better control over film uniformity and quality. Characterization using Raman spectroscopy and TEM confirmed the formation of high-quality 2D MXene sheets with clear D- and G-band features and a lattice spacing of 0.24 nm.

Visualizing Resistive Switching

Using C-AFM, Prof. Das’s group probed local I-V characteristics of these MXene-based memristors. The results revealed:

- A SET process at +0.8 V, representing a switch to a high-conducting state.
- A RESET process upon reverse bias, bringing the device back to low conduction.
- Temporal response showing switching speeds of 30–45 nanoseconds, consuming just ~130–170 picojoules.

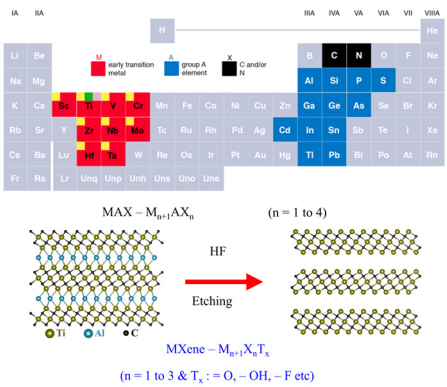


Fig. 2. MXene is the delaminated MAX phase after selective etching of aluminium atoms.

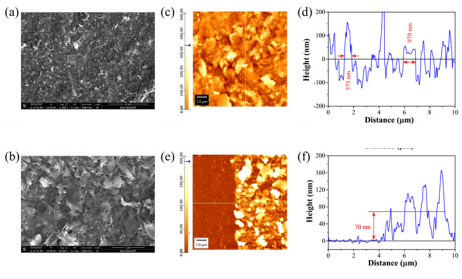


Fig. 3. Successful thin-film deposition of titanium carbide ($Ti_3C_2T_x$) MXene nanoflakes visualized using scanning electron microscope (SEM) and atomic force microscope (AFM).

Most significantly, **C-AFM current mapping before and after voltage pulses provided direct visual evidence** of filamentary conduction—a long-hypothesized yet rarely captured mechanism in RS. Post-SET images displayed a sharp increase in current confined to specific nanoscale regions, supporting the formation of conductive filaments.

"This was the first time we directly visualized the conductive path formation and annihilation during RS in MXene thin film memristor device," said Prof. Das, underscoring the unique capability of C-AFM in nanoscale device diagnostics.

Part II: KPFM for Self-Powered Photodetectors

In another segment of his talk, Prof. Das turned to **Kelvin Probe Force Microscopy (KPFM)**—an SPM variant sensitive to work function differences and contact potential variations.

Designing the Device

He described experiments using **$CsPbBr_3$ perovskite crystals** integrated into planar photodetector devices with symmetric Au electrodes. Surprisingly, even with identical electrode materials, the devices exhibited photovoltaic behavior—generating current under light without any external power.

The answer, he proposed, lies in **asymmetric contact areas** between the perovskite and each electrode. Crystals grown directly on one electrode surface had greater overlap, leading to distinct local work functions.

KPFM Validation

KPFM scans confirmed this hypothesis: the **contact potential difference (CPD)** was significantly higher at the larger contact area than at the smaller one, creating a built-in electric field sufficient to drive charge separation. This internal asymmetry allows the device to function in **self-powered mode**, an essential feature for low-power, next-gen sensors.

These findings, backed by meticulous electrical characterizations and modeling using Schottky barrier theory, were recently accepted for publication in *Physical Review Applied*.

Part III: EFM and the Role of Defects in 2D Semiconductors

Defects, especially grain boundaries (GBs), are inevitable in 2D material growth. Their impact on device performance—particularly in optoelectronics—is not fully understood. To address this, Prof. Das's team used **Electrostatic Force Microscopy (EFM)** to probe **monolayer MoS_2 flakes** with controlled geometries and defects introduced during chemical vapor deposition (CVD) growth.

Optical and Electronic Signatures of GBs

- Raman and photoluminescence studies showed red shifts and line broadening at GBs, indicative of **strain, defect states, and reduced band gaps**.
- Devices fabricated with and without GBs showed stark performance contrasts. Devices without GBs exhibited ON/OFF ratios as high as 10^5 and superlinear photocurrent response—suggestive of **photothermal enhancement**.
- Devices with GBs showed lower photocurrent and sluggish response times.

EFM Insights

EFM mapping under different sample bias conditions (+1 V, 0 V, –1 V) demonstrated charge puddling at GBs. These regions acted as **potential barriers** for both electrons and holes, suppressing carrier transport and increasing recombination. By correlating line profiles of electrostatic force variations with

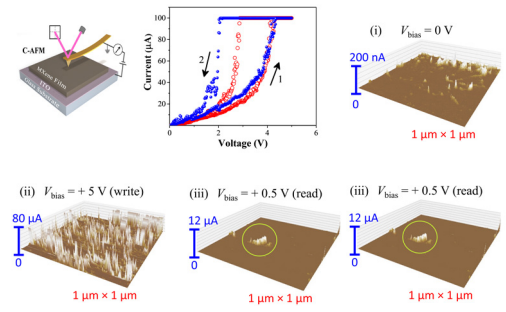


Fig. 4. Conductive AFM I-V characteristics and current mapping images at different applied bias voltages reveal resistive switching behavior in the MXene film, indicating the formation and rupture of filamentary conductive paths.

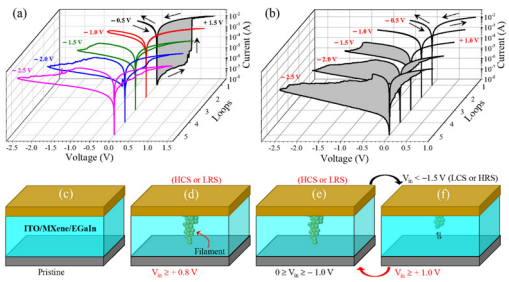


Fig. 5. Current-voltage (I-V) characteristics in loops and demonstration of the electroforming or filamentary conduction mechanism to display the bistable nonvolatile resistive switching behavior.

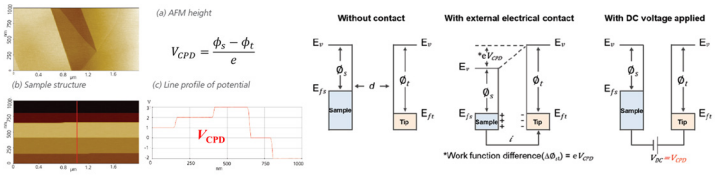


Fig. 6. Schematic illustration of the KPFM technique showing the measurement of the contact potential difference (CPD) across the sample surface.

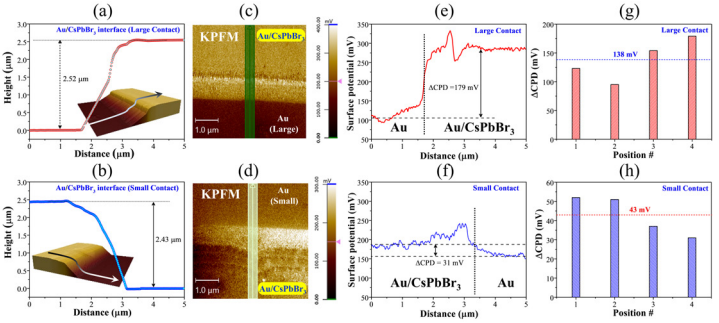


Fig. 7. AFM height profiles and KPFM surface-potential mapping images of Au/$CsPbBr^3$ interfaces.

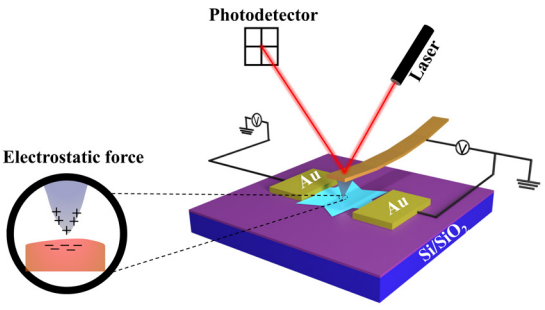


Fig. 8. The schematic of EFM technique to measure the electrostatic frictional force between the sample surface and a biased tip.

device geometry, the group was able to visualize how GBs alter internal band structures and inhibit performance.

"This nanoscale visualization helps us understand how to engineer 2D material synthesis for better device outcomes," said Prof. Das.

A New Microscope, A New Horizon

A key enabler of this work has been the installation of a Park Systems NX7 AFM in Prof. Das's lab. Since November 2023, this instrument has powered new modes of exploration—from ultrathin film thickness measurements to defect profiling and electrostatic analysis—underscoring the growing importance of **advanced SPM platforms** in electronic materials research.

Conclusion: SPM at the Crossroads of Nanotechnology and Functionality

Prof. Bikas C. Das's lecture at the 2024 NanoScientific Symposium SE Asia highlights how **advanced scanning probe microscopy is no longer a niche characterization tool—it is a strategic enabler of device innovation**.

- **C-AFM** not only maps current but confirms conduction mechanisms at nanoscales.
- **KPFM** deciphers complex interface physics critical to self-powered optoelectronics.
- **EFM** reveals how defects modulate local fields and impact carrier dynamics.

Together, these tools provide an integrated framework for understanding, designing, and optimizing future electronics. As we march toward neuromorphic computing, energy-efficient photodetectors, and high-performance 2D semiconductors, the insights delivered by SPM will remain indispensable.

And thanks to the vision of researchers like Prof. Das, the roadmap to this nanoscale frontier is becoming increasingly clear.

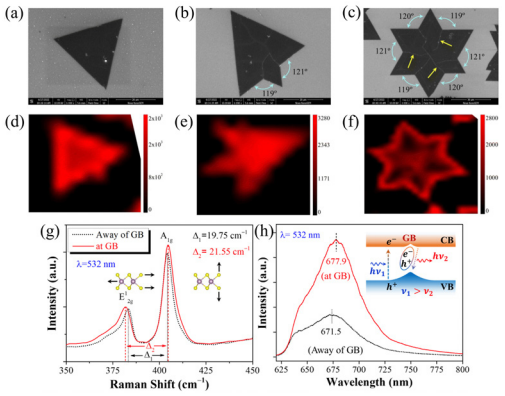


Fig. 9. Shape evaluation of CVD grown 2D MoS_2 with grain boundaries (GBs) to higher vertices. **(A-C)** SEM images. **(D-F)** Photoluminescence (PL) mapping images. **(G and H)** Raman scattering and PL spectra near and away of the GB, respectively.

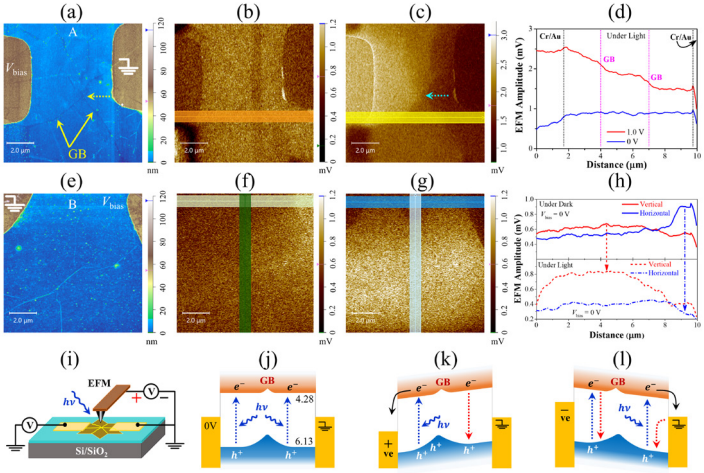


Fig. 10. The EFM analysis of devices of with and without GBs accompanied by potential energy band diagrams corresponding to various testing conditions.

About Prof. Bikas C. Das

Dr. Bikas C. Das, Associate Professor at IISER Thiruvananthapuram's School of Physics, leads a research group at the intersection of materials engineering and device physics. His team develops energy-efficient electronic devices—such as memristors, memtransistors, and autonomous photodetectors—using advanced materials like 2D TMDs, halide perovskites, organic semiconductors, quantum dots, MXenes, and functional oxides. A central focus is neuromorphic hardware that mimics brain-like computing with fast response and ultra-low energy use, addressing the limits of CMOS-based systems. His work advances intelligent, adaptive, and sustainable electronics through innovative device design and material integration.

For more information, visit https://bikas.iisertvm.ac.in

Prof. Bikas Das (center front) group photo

Prof. Bikas Das

Prof. Bikas Das (2nd from right) and his students

REVEALING TWISTED REALITIES: MAPPING MOIRÉ SUPERLATTICES IN HBN WITH AFM PHASE IMAGING

Dr. Stefano Chiodini, Italian Institute of Technology (IIT - Milan), Italy
Adapted from Presentation, Edited by NanoScientific

In the cutting-edge world of twistronics, where simple twists lead to revolutionary shifts in material properties, few phenomena are as visually and scientifically compelling as moiré superlattices. These striking nanoscale interference patterns, born from the rotational misalignment of two-dimensional (2D) materials, have captivated physicists and material scientists alike. At the 2024 NanoScientific Forum Europe, Dr. Stefano Chiodini of the Instituto Italiano di Tecnologia presented an elegant, experimentally robust method for visualizing and characterizing these moiré patterns in twisted hexagonal boron nitride (t-hBN)—using atomic force microscopy (AFM) in an inventive phase imaging modality.

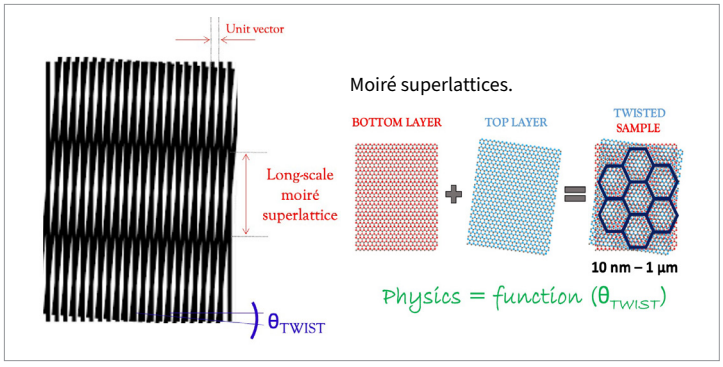
This groundbreaking work, funded by the ERC CoG "METAmorphoses" and achieved as collaborative effort between the groups of Dr. Antonio Ambrosio at IIT Milano and Prof. Andrea Ferrari at University of Cambridge, not only deepens our understanding of interlayer van der Waals (vdW) interactions in layered materials, but also uncovers a surprising new aspect of moiré physics: in-plane polarization patterns that extend the complexity of known electromechanical responses. His findings offer new frontiers for nano-engineered materials, where atomic misalignments may one day be tailored for advanced photonic, electronic, and quantum devices.

Twistronics: When Geometry Transforms Physics

Twistronics refers to the manipulation of electronic properties through the deliberate twisting of atomically thin materials like graphene or hBN. A small twist angle—often less than 1°—between two stacked layers of such materials creates a long-range periodic pattern, or moiré superlattice. This geometrical trick, deceptively simple, unlocks entirely new physical behaviors by altering interlayer couplings and symmetries.

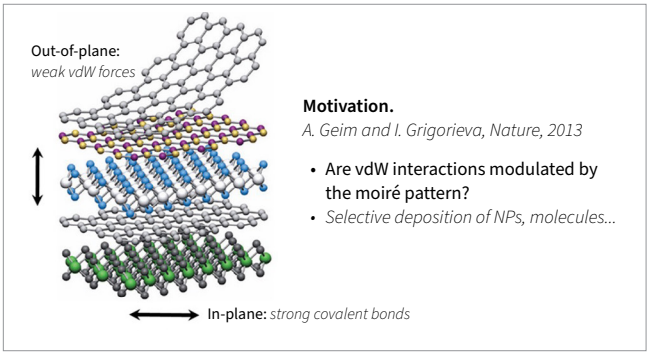
As Dr. Chiodini explains, "The entire physics of your twisted sample becomes a function of the twist angle. With no chemical modifications, just geometric control, you can induce entirely new material properties."

This was famously demonstrated in 2018, when twisted bilayer graphene at the "magic angle" of ~1.1° was found to exhibit superconductivity. But the concept is not limited to graphene. In layered insulators like hBN, moiré patterns have been shown to modulate optical, electrical, and electromechanical properties—manifesting as triangular domains in near-field, Kelvin probe, and piezoresponse force microscopy.



AFM Phase Imaging: A Van der Waals Interaction Microscope

While previous work explored moiré-induced modulations in surface potential and piezoelectricity, Chiodini's team posed a novel question: **Are van der Waals interactions themselves modulated by the moiré pattern?**



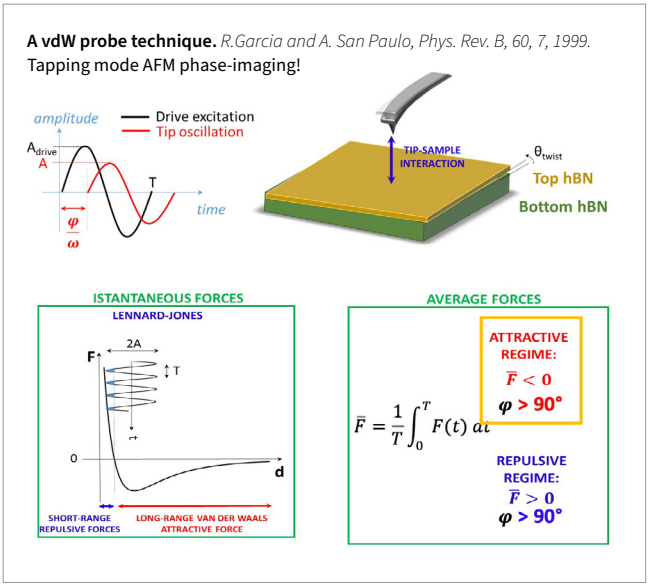
To probe this, they turned to an unexpected ally: AFM phase imaging in tapping mode. This well-established technique, often used to infer material stiffness or energy dissipation, can also act as a sensitive detector for vdW forces when configured in the attractive regime.

"In tapping mode," Chiodini notes, "the phase lag between the cantilever's oscillation and the drive signal reveals the nature of tip-sample interactions. If the average interaction is attractive, dominated by van der Waals forces, the phase exceeds 90°."

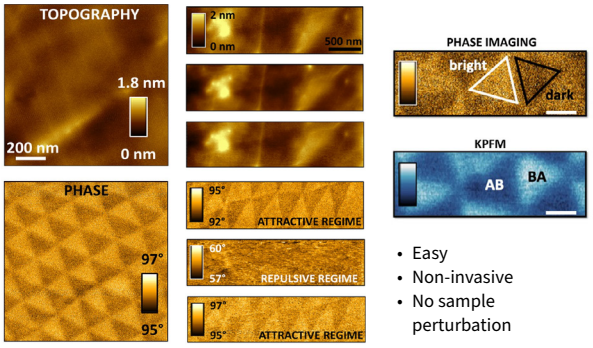
By tuning the AFM to operate in this vdW-sensitive regime, Chiodini and his collaborators could map out the local interaction energy landscape without applying voltage or making contact—ideal for delicate 2D materials. Remarkably, they discovered a spatial modulation of the phase signal that mirrored the moiré lattice itself.

Images of twisted hBN revealed familiar triangular domains—absent in topographic scans but vividly apparent in phase contrast, confirming a direct correlation between moiré geometry and vdW interaction strength.

"This approach is non-invasive, requires no conductive tip or substrate, and avoids sample perturbation," Chiodini emphasizes. "It's a powerful, underutilized way to image moiré physics."



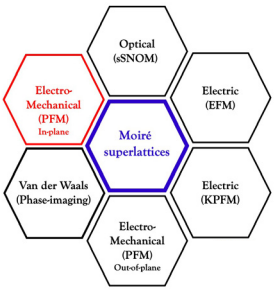
Tapping mode phase imaging results.
S. Chiodini et al., ACS Nano 16, 5, 2022.

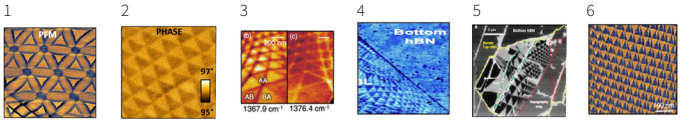


- Easy
- Non-invasive
- No sample perturbation

Moiré superlattices properties by SPM.

1. S. Chiodini, arXiv:2406.02195, **2024**
2. S. Chiodini, *ACS Nano* 2022
3. S. Moore, *Nat. Commun.*, 2021
4. C. Woods, *Nat. Commun.*, 2021
5. V. Stern, *Science*, 2021
6. K. Yasuda, *Science*, 2021

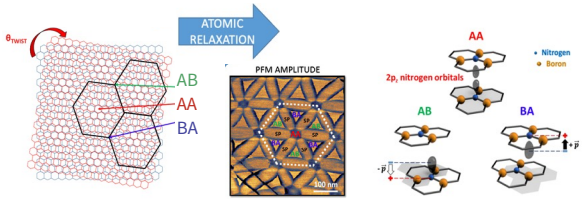




Electromechanical Depth: From Out-of-Plane to In-Plane Polarization

In the second part of his presentation, Chiodini ventured into less-charted territory: in-plane electromechanical responses of moiré-patterned hBN. While prior work had demonstrated out-of-plane polarizations in AB and BA stacking domains—arising from slight shifts in atomic registry—Chiodini hypothesized the existence of in-plane dipoles, forming a more intricate 3D polarization lattice.

Motivation. *Interfacial array of* AB/BA out-of-plane *polarization.*



To probe this, his team used vertical piezoresponse force microscopy (V-PFM), exploiting a subtle phenomenon known as the "buckling effect." While V-PFM typically detects vertical motion due to out-of-plane polarization, it can also register cantilever buckling when an in-plane polarization vector aligns with the cantilever axis.

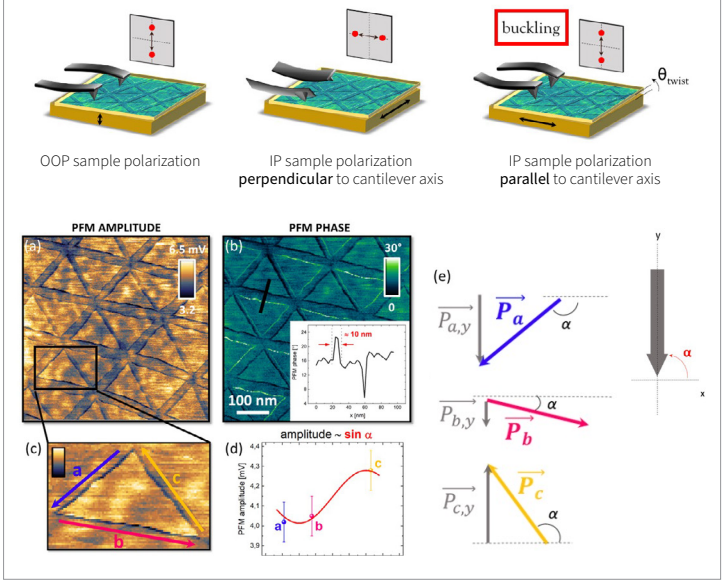
"In this buckling geometry, the cantilever bends vertically due to in-plane forces, and the photodiode detects a vertical signal just as if there were out-of-plane motion," he explains. "But it's really the in-plane polarization being picked up."

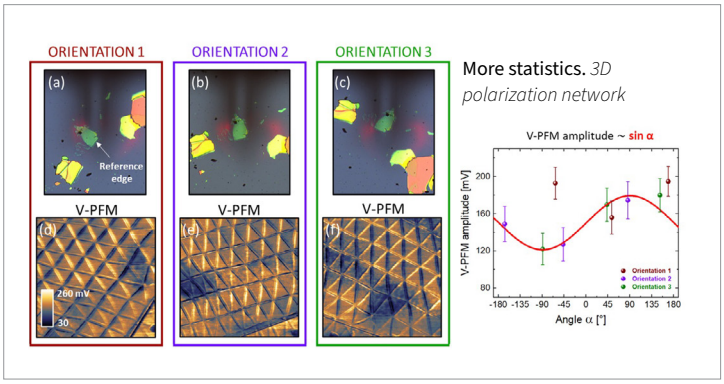
By analyzing amplitude signals along different edges of the triangular moiré domains—each corresponding to a different orientation—Chiodini's team observed a sinusoidal dependence of amplitude on the angle between polarization vector and cantilever axis. When the edge aligned with the cantilever axis (90° projection), the buckling signal peaked. This angular modulation is a direct fingerprint of in-plane dipole activity.

To strengthen their case, the researchers reoriented the same hBN flake in multiple directions relative to the cantilever and repeated the measurements. Each time, they mapped nine distinct domain edges with consistent sinusoidal trends, providing compelling statistical evidence of in-plane polarization.

This work marks the first demonstration of in-plane polarization in t-hBN moiré superlattices, enriching the previously one-dimensional picture of electromechanical behavior. "We are looking at a true 3D polarization network," Chiodini concluded. "This opens new questions about the internal field distributions and their coupling to external stimuli."

In-plane polarizations in t-hBN. *S. Chiodini et al., arXiv:2406.02195,* **2024**





More statistics. *3D polarization network*

Implications and Future Directions

The implications of Chiodini's dual discoveries—the modulation of vdW forces and the existence of in-plane polarization—are far-reaching. First, they suggest that even fundamental interatomic interactions are sensitive to geometric phase effects in layered materials. This could influence how nanoparticles, molecules, or even atoms selectively adsorb on such surfaces, enabling directed self-assembly on a moiré template.

Second, the observation of multidirectional polarization fields invites a rethinking of piezoelectric and ferroelectric applications in 2D materials. Could twist-controlled dipole architectures be used in nanoscale transducers, energy harvesters, or logic elements? What role might they play in light-matter interactions or topological excitations?

Moreover, Chiodini's results serve as a call to revisit older AFM techniques under a new theoretical lens. Phase imaging—often sidelined in favor of more quantitative methods—has shown its value as a highly sensitive, artifact-free probe for subtle nanoscale phenomena.

Final Reflections

Dr. Chiodini's meticulous and creative approach demonstrates the continued relevance of fundamental scanning probe microscopy in frontier physics. His ability to extract profound insights from familiar tools speaks to the evolving interplay between instrumentation and imagination in nanoscience.

At a time when quantum materials and flat-band phenomena are reshaping electronics, understanding the hidden landscapes of interlayer interactions and internal fields becomes crucial. Twisting hBN is no longer just a geometrical curiosity—it is a platform for engineering the invisible forces that shape the future of materials.

As twistronics matures from serendipity to design, Chiodini's work lights the path with clarity, rigor, and just the right angle.

About Dr. Stefano Chiodini

Dr. Chiodini is a researcher in the Vectorial Nano-Imaging Group at the Italian Institute of Technology, where since July 2020 he has been using scanning probe microscopy to investigate the optical, electronic, and mechanical properties of twisted 2D materials, small molecules, and polymers. From 2017 to 2019, he was a postdoctoral fellow in the PLATON group at the University of Zaragoza, contributing to the MAGIC CELL GENE project with AFM-based studies of cell membranes and self-assembled molecular electronics. Prior to that, he worked as a postdoc at ICMM-CSIC in Madrid starting in 2015, focusing on atomic-scale studies of metal-organic frameworks and advancing techniques in atomic-resolution and multifrequency AFM. He earned his PhD in Physics in 2015 from the National Research Council (ISMN-CNR) in Bologna, where his research under Prof. Fabio Biscarini centered on the growth of organic semiconductors using real-time AFM in UHV. He received his bachelor's and master's degrees cum laude from the University of Bologna in 2010.

For more information on the work, funding and research groups involved, please visit:
https://pubs.acs.org/doi/10.1021/acsnano.1c11107
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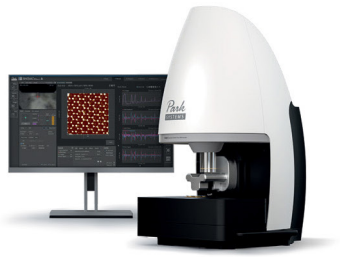
One Nanostep for Microscopy
One Giant Leap for Science



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UNVEILING THE TRUE SHAPE OF CONDUCTIVE FILAMENTS: ADAPTIVE SCALPEL SPM REDEFINES 3D VOLUMETRIC SENSING

Poster Winner at NanoScientific Symposium Americas 2024
Md Ashiqur Rahman Laskar and Team, Arizona State University
Adapted from Presentation, Edited by NanoScientific

In the nanoscopic realm of advanced materials and electronic devices, precision is everything. The need to understand, control, and characterize structures at the nanoscale has driven the evolution of microscopy into a high-art of engineering and physics. At the 2024 NanoScientific Symposium, Researcher Md Ashiqur Rahman Laskar and his interdisciplinary team from Arizona State University, INRiM (Italy), and NenoVision (Czech Republic) presented an award-winning breakthrough in this domain—**Adaptive Scalpel Scanning Probe Microscopy (SPM)**, a transformative approach to three-dimensional tomographic analysis.

This work addresses a longstanding issue in conductive atomic force microscopy (C-AFM): the parasitic effects caused by tip-sample junction leakage and inhomogeneous electric fields, which distort lateral and vertical resolution in 3D reconstructions. Laskar's innovation offers a path to sub-1000 nm^3 tomographic precision, especially for characterizing resistive switching memories—ushering in a new era of volumetric sensing in nanoelectronics.

The Problem: Artifacts in Nanoscale Tomography

At the heart of modern nanoelectronics lies a growing class of devices that rely on resistive switching—such as valence change memory (VCM) systems based on transition metal oxides like NbO_x. These systems depend on the reversible formation and rupture of nanoscale conductive filaments (CFs), which define the ON and OFF states of a memory cell. Understanding these filaments, including their 3D morphology and evolution, is critical for optimizing device performance and longevity.

Yet a key barrier to true 3D insight has been **tip-induced artifacts** in conventional tomographic atomic force microscopy. These artifacts fall into two primary categories:

1. **In-plane leakage:** Current spreads laterally due to parasitic conduction pathways through the surrounding oxide matrix. As a result, the detected current footprint significantly overestimates the actual lateral dimensions of the conductive feature (i.e., the CF).

2. **Vertical overbiasing:** As material is removed layer by layer during the tomographic process, the oxide thickness reduces, causing an unintentional increase in electric field strength. This non-uniformity leads to variations in contrast and feature depth, skewing vertical resolution.

The result? A bloated and blurred picture of what should be nanosharp and sharply localized.

The Innovation: Adaptive Scalpel SPM

To combat these dual distortions, Laskar's team devised **Adaptive Scalpel SPM**, an approach that dynamically adjusts the tip-sample bias throughout the tomographic process. Instead of applying a constant voltage, which leads to uncontrolled increases in electric field as oxide layers thin, the adaptive method calibrates the bias to **maintain a steady internal electric field**—thus preserving contrast integrity as depth increases.

This innovation was tested on VCM cells with gold (Au) top electrodes and NbO_x-based switching layers. After removing the Au layer **(see Fig. 1)**, a sharp conductive probe was used to iteratively erode the NbO_x material while mapping current flow. Two acquisition modes were compared:

- **Constant bias mode:** Fixed voltage during the entire scalpel scan.
- **Adaptive bias mode:** Voltage tuned according to oxide thickness to stabilize the internal electric field.

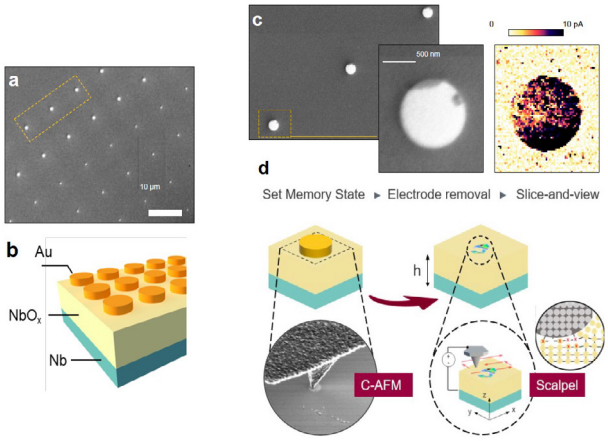

Fig. 1. VCM Device and Scalpel C-AFM: A) *Scanning electron microscopy image.* B) *Schematic of the Au dots forming the top electrodes of the VCM under study.* C) *Detailed view with SEM and conductive atomic force microscopy (C-AFM) of the AU top electrode of the VCM cell.* D) *Schematic of the tip-induced filament formation and top-electrode removal process.*

Uncovering the True Filament Geometry

The difference between these two approaches is vividly captured in 3D volumetric reconstructions of the conductive filament. Under constant bias **(Fig. 4a)**, the filament appears larger and less defined, a result of leakage spreading and overbiasing. In contrast, adaptive bias **(Fig. 4b)** yields a filament image that is smaller, sharper, and more confined—closely matching theoretical expectations and physical models.

This is reinforced in the cross-sectional comparisons of **x-y** and **x-z** slices, where the adaptive bias captures a **sharply bounded filament** located precisely at the core of the active device area. Such precision is crucial when designing or troubleshooting high-density memory arrays or emerging logic components.

Quantitative Insights: Resolution and Leakage Control

The experimental data quantifies the improvement:

- **Lateral resolution** improves due to reduced in-plane leakage, confirmed by varying the bias during scanning and observing a clear decrease in current footprint size.
- **Vertical sensitivity** is optimized through adaptive voltage control, leading to significant decrease in vertical leakage, charging artifacts

Introduction. *• Controlling nanoscale tip-induced material removal is crucial for achieving atomic-level precision in tomographic sensing with atomic force microscopy (AFM). • This work addresses in-plane and vertical tip-sample junction leakage as sources of parasitic contrast in tomographic AFM, hindering real-space 3D reconstructions. • An adaptive tip-sample biasing scheme is demonstrated for reduction in overestimation of the lateral dimensions and the mitigation of a class of artefacts induced by the high electric field inside the thin oxide when volumetrically reduced.*

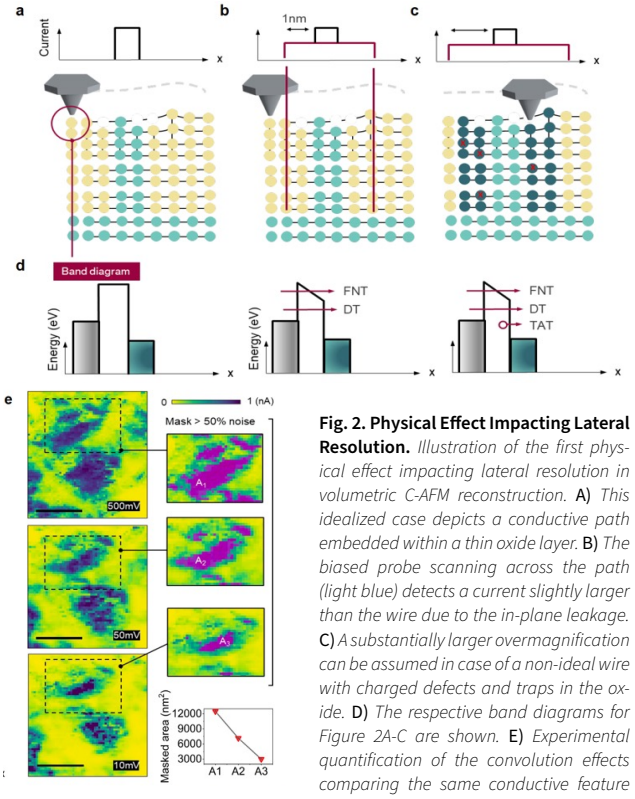

Fig. 2. Physical Effect Impacting Lateral Resolution. *Illustration of the first physical effect impacting lateral resolution in volumetric C-AFM reconstruction.* A) *This idealized case depicts a conductive path embedded within a thin oxide layer.* B) *The biased probe scanning across the path (light blue) detects a current slightly larger than the wire due to the in-plane leakage.* C) *A substantially larger overmagnification can be assumed in case of a non-ideal wire with charged defects and traps in the oxide.* D) *The respective band diagrams for Figure 2A-C are shown.* E) *Experimental quantification of the convolution effects comparing the same conductive feature probed with different bias (scale bar 200 nm). In the inset we quantify the area of the conductive spot with a clear reduction in size for the low bias stress conditions.*

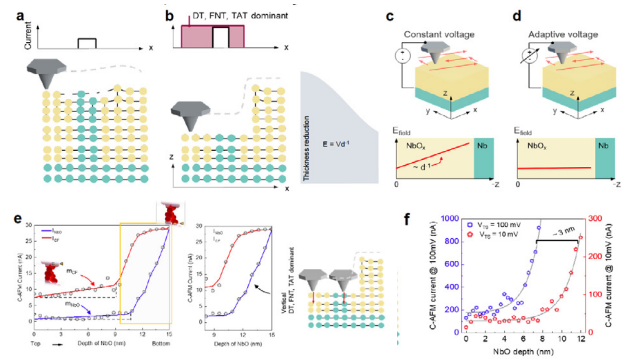

Fig. 3. Vertical Depth Sensitivity In Volumetric Reconstruction. *Illustration of the second physical effect impacting vertical depth sensitivity in volumetric C-AFM reconstruction.* A) *This idealized case depicts a conductive path (light blue) embedded within a thin oxide layer.* B) *The biased probe is used to remove material, thus eroding the oxide progressively.* C) *As the oxide thickness is reduced, the internal electric field will rise during the measurement.* D) *The concept of adaptive scalpel SPM is to maintain a constant electric field during the analysis by adjusting the tip-sample bias as the oxide is made thinner.* E) *Evolution of the tip-sample current as a function of depth for a CF (red) and pristine oxide (blue). In the inset, we show the schematic of the acquisition area.* F) *The evolution of the tip-sample current for a pristine oxide region compares the leakage in the case of constant bias (blue) and adaptive bias (red).*

and improved resolution.

Fig. 2 provides an instructive visualization of in-plane leakage effects, while Fig. 3 explains how thickness-dependent field increases can distort vertical profiling. The adaptive bias strategy not only restores fidelity but allows for reliable current-depth analysis (Fig. 3e-f), further substantiating its effectiveness.

Beyond Resistive Memories: Broader Impact

Although this study focuses on NbO_x-based VCM cells, the implications of Adaptive Scalpel SPM stretch far beyond memory technology:

- **Photovoltaics:** In organic and hybrid solar cells, 3D conductive networks and ion migration paths are critical to device stability and efficiency.
- **Ferroelectrics and Piezoelectrics:** Understanding charge domain structures and their evolution during operation demands similar volumetric precision.
- **2D and Layered Devices:** Heterostructures such as transition metal dichalcogenides (TMDCs) or perovskite-based architectures require reliable tomography to decode buried interface physics.

By enabling 3D reconstructions with sub-1000 nm^3 resolution, Adaptive Scalpel SPM positions itself as a universal tool for high-precision electrical nanocharacterization.

Practical Implementation: Tip-Device Interface

A detailed understanding of the tip-sample interaction was also key to the study. As the schematic in Fig. 7 shows, the team modeled the tip-Au-NbO_x stack as an equivalent resistive network. Once the Au top electrode is removed, the tip-sample junction becomes the primary resistance limiter. This naturally introduces a form of current compliance—a fortunate artifact that protects the device from damage during scanning.

Moreover, I-V curves (Fig. 6) demonstrate stable switching characteristics, and consistent current evolution under adaptive bias indicates improved reproducibility. Notably, Fig. 8 reveals that the filament area measured at the deepest slices of the NbO_x device was significantly smaller in adaptive mode, confirming better spatial confinement.

Collaboration and Outlook

This project is a testament to international scientific collaboration. Co-authors from ASU, INRiM, and NenoVision brought together expertise in materials science, metrology, AFM technology, and device engineering. The equipment and infrastructure support of ASU's Eyring Materials Center and funding from DOE and NSF (notably DMR 2111812) were critical to its success.

Looking ahead, Laskar and his colleagues envision integrating adaptive scalpel techniques into broader AFM platforms, potentially in combination with machine learning algorithms for automated reconstruction and classification of 3D features. The goal is clear: bring unprecedented clarity and confidence to nanoscopic tomography.

Conclusion

The work of Md Ashiqur Rahman Laskar and his team exemplifies the spirit of NanoScientific: combining creativity, technical mastery, and scientific rigor to break new ground in nanoscale exploration. Their Adaptive Scalpel SPM approach not only solves a real and pressing problem in AFM tomography—it redefines the resolution standards for the entire field.

As nanoscale systems become ever more complex, tools like Adaptive Scalpel SPM will be essential in decoding their structure-function relationships, helping engineers build the next generation of smart, efficient, and responsive devices.

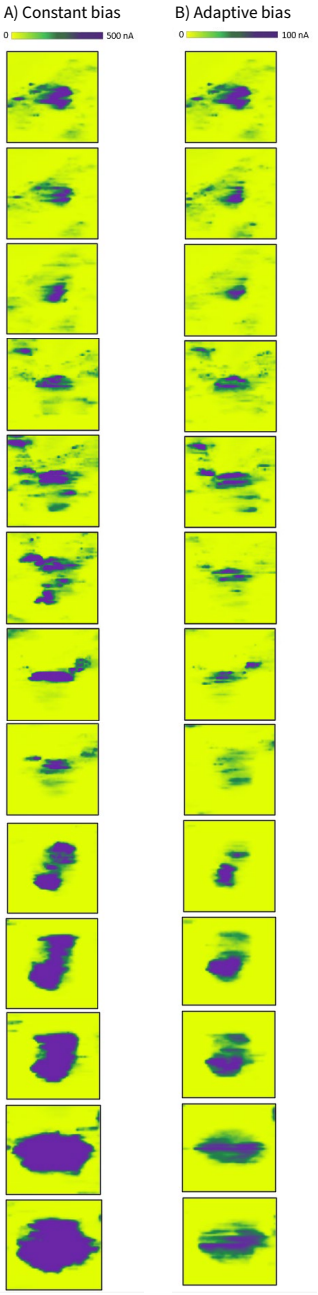

Fig. 5. 2D Slice Views. *Top view of selected 2D slices throughout the NbO_x device (scan size 100 x 100 nm) for* A) *constant voltage analysis, and* B) *for adaptive voltage. The CF appears in the middle of the active area after top electrode removal. The highly conductive features on the top and bottom corners are the exposed parts of the Nb electrode, which is completely exposed during the removal of NbO_x.*

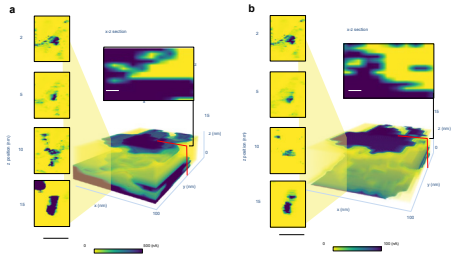

Fig. 4. 3D Observation Of The Conductive Filament. *3D observation of the conductive filament in NbO_x cell. The tomographic reconstruction is visualized by volume rendering comparing* A) *constant bias scalpel SPM with* B) *adaptive scalpel SPM. In the inset we compare the 2D observation of the CF section plane (x-z) and four section x-y planes for comparison of the resolution obtainable with the two modes of acquisitions (scalebar 5nm).*

Filament Characteristics

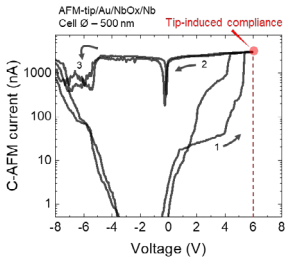

Fig. 6. *Electrical I-V readout from tip-induced resistive switching. (Note: here the current flowing in the filament is limited by the high series resistance of the tip-sample junction acting as a form of current compliance for the tip-sample device system).*

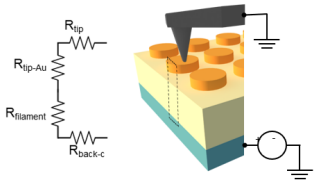

Fig. 7. *Schematic of the current collection scheme using the tip-Au-NbO_x-stack for switching. Note, the associated equivalent resistive network. Here, the emphasis should be placed on the high resistance of the tip-sample junction once the Au is removed.*

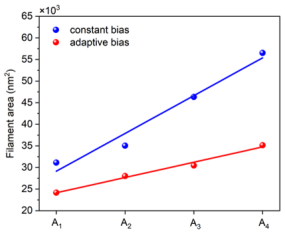

Fig. 8. *Filament area comparison under constant and adaptive bias for the last four slices at the very bottom of the NbOx layer.*

About Md Ashiqur Rahman Laskar

Md Ashiqur Rahman Laskar is a PhD candidate in Electrical Engineering at Arizona State University (ASU), specializing in semiconductor metrology and failure analysis. He is doing research with Dr. Umberto Celano at ASU. The ASU team aims to design experiments and develop advanced metrology techniques for the emerging devices and materials. The research goal is to understand and observe the fundamental processes that govern the operation of emerging devices, through nanoscale analytical imaging and correlative metrology. This generates fundamental understanding to engineer improved materials for nanoelectronics.

The ASU team behind the research: PhD students A. Laskar, S. Chakrabarti; Prof. U. Celano, the principal investigator of Nanoelectronics Metrology & Failure Analysis Lab at ASU.

For more information, https://labs.engineering.asu.edu/celano/

From L-R: Md Ashiqur Laskar, Srijan Chakrabarti, Dr. Umberto Celano

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ENABLING MOIRÉ AND ATOMIC LATTICE IMAGING IN 2D MATERIALS WITH TORSIONAL FORCE MICROSCOPY

Dr. John Kim and Edward Park, Park Systems Corp., S. Korea

Introduction

Torsional Force Microscopy (TFM) is a specialized atomic force microscopy (AFM) mode for detecting dynamic lateral forces at the nanoscale. It is particularly effective in visualizing moiré superlattices and atomic lattices in van der Waals (vdW) heterostructures such as bilayer graphene and graphene/hexagonal boron nitride (hBN) stacks, where even slight interlayer twist angle changes can dramatically modify electronic behavior[1],[2]. Unlike conventional AFM techniques, TFM does not require electrical connection to the sample or probe, making it ideal for electrically insulating or floating structures. Its operation under ambient conditions and ability to resolve surface and shallow subsurface features enable researchers to investigate two-dimensional (2D) materials on soft polymer substrates, even during intermediate fabrication stages.

Moiré superlattices arise from stacking two layers of graphene or other 2D materials at small twist angles. Although their unique physical properties have been widely explored, direct imaging under ambient conditions remains limited[3]. Tiny twist angle variations — even hundredths of a degree — can drastically alter electronic characteristics[2]. Existing imaging approaches often require complex instrumentation or extensive post-processing, and practical challenges such as inconsistent twist angles and poor spatial uniformity hinder reproducible fabrication and characterization[4]. Thus, there is strong demand for a fast, accessible, and reliable imaging technique that works under ambient conditions, requires no electrical connections or surface modifications, and is compatible with partially assembled vdW stacks on polymer substrates.

Park Systems has implemented TFM to meet these needs. This application note introduces TFM as a rapid, practical feedback solution and presents representative data acquired from 2D material samples using the Park FX40 AFM system, along with details of its hardware configuration optimized for TFM measurements.

Various SPM applications for imaging moiré superlattices

Several scanning probe microscopy techniques have been explored to visualize moiré patterns and atomic lattices in 2D materials. Conductive AFM (C-AFM) allows atomic resolution imaging but requires conductive samples and substrates[5]. Amplitude-modulation AFM (AM-AFM), including tapping mode, often struggles to clearly resolve fine moiré contrasts, whereas contact mode typically provides better contrast6. Scanning microwave impedance microscopy (sMIM) offers non-contact measurements but lacks atomic resolution[7]. Among these, Lateral Force Microscopy (LFM) and Friction Force Microscopy (FFM) have been effective for visualizing both moiré and atomic-scale structures in materials such as hBN and graphite[8,9]. However, their ability to detect subsurface structures remains unproven. TFM addresses this gap by utilizing torsional resonance modes, enabling high-sensitivity mapping of frictional contrast and thus offering clearer visualization of moiré structures across varying depths.

Torsional resonances sensitive to dynamic friction at the AFM tip-sample interface were employed to achieve high-resolution maps comparable to previous techniques. This proved highly effective for clearly resolving moiré contrast or even atomic lattices.

Fundamentals of TFM

TFM operation involves two simultaneous control schemes. First, closed-loop feedback tracks the sample topography, maintaining a constant normal loading force. Concurrently, an open-loop excitation at the cantilever's torsional resonance is applied to detect variations in lateral friction. This dual-mode configuration ensures high frictional contrast by combining sensitive torsional excitation with stable tip-sample contact control. To induce pure torsional motion without introducing vertical or flexural artifacts, a pair of bimorph actuators is driven 180° out of phase. This symmetric configuration ensures that vertical displacements are canceled out while generating a clean rotational torque at the cantilever base, thereby selectively exciting the torsional eigenmode with high efficiency. A single bimorph or asymmetrical drive would excite non-torsional modes and reduce signal clarity due to mechanical cross-talk and mode mixing. The lateral deflection resulting from this controlled torsional excitation is then measured to map nanoscale frictional variations. These signals, when analyzed at torsional resonances, provide high-resolution information about surface and subsurface structures without the need for electrical biasing.

TFM builds upon a two-mode control scheme that combines vertical feedback and lateral torsional excitation to detect nanoscale frictional variations with high spatial resolution. As illustrated in **Fig. 1**, the system simultaneously maintains topographic tracking through closed-loop control while driving the cantilever's torsional resonance via open-loop excitation. To ensure stable imaging conditions throughout the scan, the vertical Z-feedback maintains a constant normal force by adapting to the surface contours of the sample, following the principle used in conventional contact-mode AFM.

Torsional excitation is achieved by applying 180° out-of-phase signals to a pair of piezo actuators integrated into the cantilever holder, generating a rotational drive around the cantilever's long axis. The lateral deflection is detected by the photodetector's side quadrant channel (the PSPD), and its amplitude near the torsional resonance frequency indicates local variations in friction.

By scanning across the surface while maintaining this torsional excitation, TFM effectively maps nanoscale variations in friction linked to structural features such as moiré patterns or atomic lattices. Since the technique does not rely on electrical biasing or contact-based charge transfer, it avoids issues like electrostatic interference or sample charging. This makes TFM particularly well-suited for characterizing electrically insulating or floating samples, as well as fragile 2D materials or partially fabricated heterostructures.

By combining vertical force stability with lateral resonance sensitivity, TFM provides a powerful and non-invasive method for revealing fine mechanical contrasts in complex nanoscale systems, supporting advanced surface and interface characterization.

High-resolution TFM Imaging of Moiré Patterns and Atomic Lattices

To demonstrate the capabilities of TFM, high-resolution imaging was performed under ambient conditions on a variety of van der Waals (vdW) heterostructures, including a cleaved (001) surface of muscovite mica, single-layer graphene on hexagonal boron nitride (Gr/hBN) and twisted bilayer graphene (tBG). The results demonstrate TFM's ability to resolve both moiré patterns and atomic lattices with high spatial precision across multiple scales.

TFM was also applied to a cleaved (001) mica surface to examine atomic-scale lattice contrast. As shown in **Fig. 2**, TFM clearly resolved periodic atomic-scale features, whereas LFM measurements at the same

location resulted in noisy images with less distinctive structure and frequent spike-like artifacts in specific directions. The superior sensitivity of TFM to lateral friction enabled clean visualization of atomic lattice contrast even at the small scan size of 5 × 5 nm^2. The TFM measurements matched the reported atomic lattice periodicity of 0.52 nm, confirming measurement accuracy[10].

Fig. 3 shows the TFM results obtained on a single graphene layer on hBN (Gr/hBN). The optical image and wide-area topography (40 × 40 µm^2) confirm the multilayered structure. In the high-resolution small scans (100 × 100 nm^2), TFM clearly resolved moiré contrast linked to the twist angle.

TFM was applied to a twisted bilayer graphene sample to visualize the resulting moiré patterns. As shown in **Fig. 4**, TFM clearly resolved the periodic features, whereas LFM measurements at the same location produced noisy images with poorly defined structures. The superior lateral friction sensitivity of TFM enabled clean and distinct visualization of the moiré pattern contrast.

Conclusion

Torsional Force Microscopy (TFM), as implemented on the Park AFM systems, provides a powerful and reliable method for high-resolution imaging of moiré superlattices and atomic lattices in 2D materials under ambient conditions. By detecting lateral frictional variations without the need for electrical contacts or sample conductivity, TFM enables direct and minimally invasive visualization of both surface and shallow subsurface structural features. This capability is particularly valuable for characterizing twisted heterostructures, where subtle differences in twist angle and interlayer coupling critically influence material properties. The high spatial sensitivity, ability to operate in ambient conditions, and compatibility with soft or insulating substrates make TFM a practical and versatile tool for both research and process monitoring.

As interest in 2D quantum and electronic materials continues to grow, TFM is expected to serve as a valuable technique for providing structural insight and supporting the development of next-generation devices.

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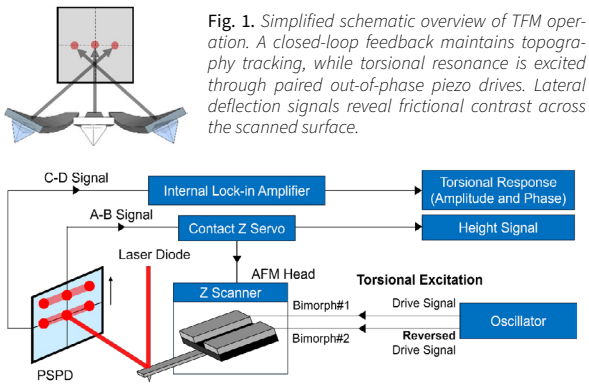


Fig. 1. *Simplified schematic overview of TFM operation. A closed-loop feedback maintains topography tracking, while torsional resonance is excited through paired out-of-phase piezo drives. Lateral deflection signals reveal frictional contrast across the scanned surface.*

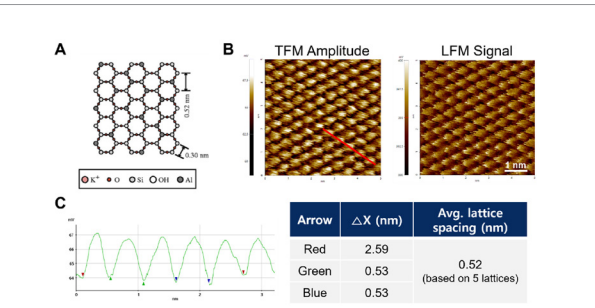


Fig. 2. *High-resolution images of mica surface measured by LFM and TFM.* **A)** *The crystal structure schematic of a cleaved mica surface with its lattice spacing distance.* **B)** *Comparative images of mica surface (101) acquired with TFM and LFM modes.* **C)** *Line profile of TFM image. The corresponding LFM and TFM measurement conditions were as follows: cantilever PPP-FMR, 512 × 512 pixel, scan rate of 8 Hz, and scan size 5 × 5 nm^2.*

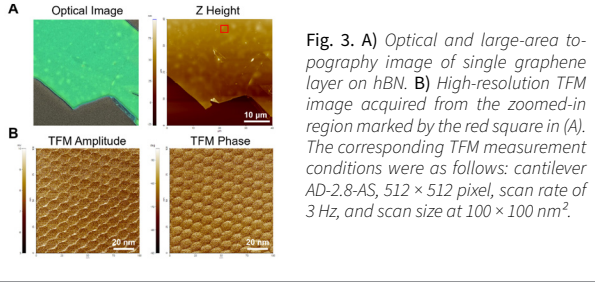


Fig. 3. **A)** *Optical and large-area topography image of single graphene layer on hBN.* **B)** *High-resolution TFM image acquired from the zoomed-in region marked by the red square in (A). The corresponding TFM measurement conditions were as follows: cantilever AD-2.8-AS, 512 × 512 pixel, scan rate of 3 Hz, and scan size at 100 × 100 nm^2.*

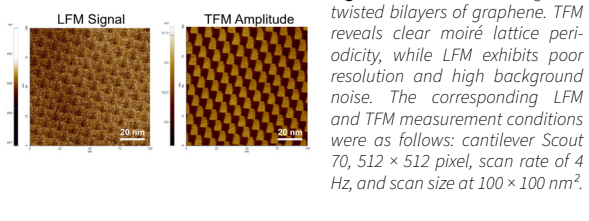


Fig. 4. *TFM and LFM images of twisted bilayers of graphene. TFM reveals clear moiré lattice periodicity, while LFM exhibits poor resolution and high background noise. The corresponding LFM and TFM measurement conditions were as follows: cantilever Scout 70, 512 × 512 pixel, scan rate of 4 Hz, and scan size at 100 × 100 nm^2.*

PARK SYSTEMS IN FOCUS

Lyncée Tec Acquisition Brings Holographic Microscopy to Park's Portfolio

In a strategic move to broaden its metrology portfolio, Park Systems Corp. announced the acquisition of Swiss-based Lyncée Tec SA, a pioneer in Digital Holographic Microscopy (DHM®). This acquisition follows Park's 2022 integration of Accurion GmbH, positioning the company as a provider of complementary nanometrology technologies across AFM, Imaging Spectroscopic Ellipsometry, and now holographic microscopy.

Founded in 2003 by researchers at EPFL in Lausanne, Lyncée Tec developed DHM® to acquire 3D surface data across an entire field of view at high speed—over 100 times faster than conventional interferometry. The technology serves a wide range of industries including semiconductors, life sciences, MEMS, and materials manufacturing.

"This acquisition is a pivotal step toward becoming an equipment company with a comprehensive suite of semiconductor metrology solutions," said Dr. Sang-il Park, CEO of Park Systems. Integration plans include building a fully automated DHM system that complements Park's AFM platforms, particularly for advanced packaging applications.

Founders of Lyncée Tec expressed confidence that joining Park Systems will enhance DHM's visibility and broaden its impact in global markets, leveraging Park's commercial infrastructure and international reach.

New Tools Bridge Research and Industry Needs

At SEMICON Korea 2025, Park Systems unveiled major additions to its FX Large Sample AFM series: the Park FX300, FX200 IR, and FX300 IR. Designed to address the semiconductor industry's transition to 300 mm wafers, the FX300 offers high-precision measurement without the complexity of a full inline system. It's suited for both industrial quality control and advanced research environments.

A key innovation is the integration of Photo-induced Force Microscopy (PiFM) in the FX200 IR and FX300 IR models, enabling nanoscale infrared (IR) spectroscopy with high spatial resolution. This allows chemical identification of nanoscale features without damaging wafer surfaces—opening applications in semiconductor analysis, polymers, and biomaterials.

The systems also include automation features like StepScan™, AI-based laser alignment, automatic tip exchange, and cleanroom-compatible options with Fan Filter Units (FFUs). These capabilities aim to streamline workflows while maintaining high resolution and reproducibility.

"The FX Large Sample AFM series represents the convergence of industrial robustness and research-grade performance," said Dr. Sang-Joon Cho, EVP at Park Systems. "With the new nano-IR systems, we're redefining how large-sample AFM can serve both research and production needs."

Interactive Platform Brings AFM Workflows Online

Park Systems has introduced SmartSimulator™, a virtual demonstration platform that enables remote users to explore AFM operation workflows. Announced during a global online showcase of the FX Large Sample AFM series, SmartSimulator is part of Park's broader strategy to support digital access and early-stage customer engagement.

Users can simulate key steps such as probe handling, scan setup, laser alignment, and data analysis through a browser-based interface. The tool includes 2D and 3D data visualization modes and replicates the experience of operating Park AFM tools in real lab settings—without needing the physical instrument.

By providing interactive access to AFM technology, SmartSimulator enables prospective users to evaluate capabilities remotely. It also supports workflow development for SmartAnalysis™ post-scan processing.

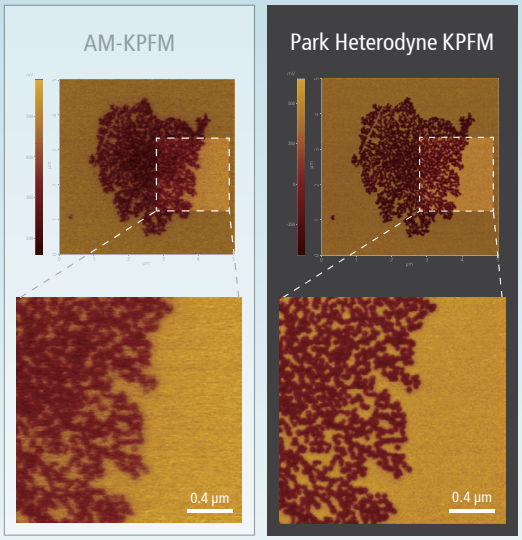
The virtual platform was demonstrated alongside live sessions in multiple languages during the showcase, where researchers discussed semiconductor and materials applications across Asia, Europe, and the Americas.

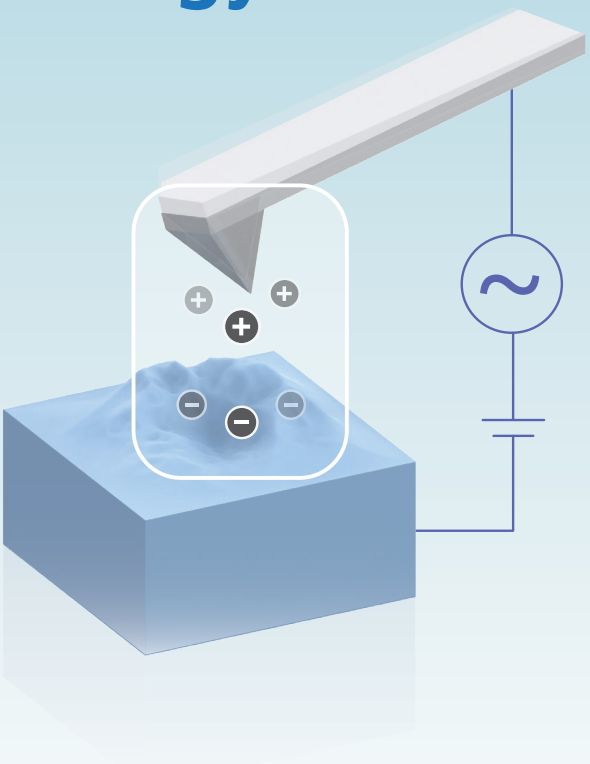
Company representatives indicated that SmartSimulator will be a cornerstone in Park Systems's expanding digital toolkit.

For more information, visit www.parksystems.com

Park Heterodyne KPFM

A New Standard in AFM Imaging Technology





The Heterodyne Effect:
How Does it Work?

Kelvin Probe Force Microscopy (KPFM) is a widely used AFM technique for quantitative measurement of surface potential and work function. Park Heterodyne KPFM advances this established method with a novel approach — leveraging second harmonic signal detection, which offers a higher Q factor and significantly reduces topographic crosstalk when measuring contact potential difference (CPD). Optimized for applications such as 2D materials, semiconductors, and nanostructured surfaces, Park Heterodyne KPFM delivers superior clarity and accuracy in nanoscale electrical characterization.

Key Features

- Superior Sensitivity & Resolution
- Localized Detection
- Minimized Frequency Artifacts
- Ideal for Diverse Applications

System Compatibility

- Included in Park FX Series
- Available via Paid Hardware Upgrade on Park NX Series

*Excluding Park NX15 & NX7

Park Systems Corp.

To learn more about **Park Heterodyne KPFM**, please visit www.parksystems.com/heterodynekpfm or e-mail inquiry@parksystems.com

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