HD-KFM III Application Note

CSInstruments Nano-Observer II

1. Introduction

Since the invention of the Atomic Force Microscope (AFM) in 1986, various measurement modes have been developed to characterize interaction forces and surface properties beyond topography. One of the most intriguing applications of AFM is nano-electric characterization, which allows for the measurement of electric properties with nanometric lateral resolution. This capability is particularly valuable for the development of nanomaterials, including 2-D materials such as graphene, molybdenum disulfide, hBN, and black phosphorous. Understanding the electrical properties of these materials at the nanoscale is crucial for advancing applications in solar cells, energy storage, biosensing, and other fields.

Among the electrical modes of AFM, Kelvin Force Mode (KFM) stands out as a popular technique. KFM enables the measurement of the surface potential between the AFM tip and the sample surface, which can be related to material properties such as work function and bandgap.

$$V_{POTENTIAL} = \frac{\Delta\varphi}{e} = \frac{\varphi_{tip} - \varphi_{sample}}{e}$$

Figure 1: Equation relating surface potential to work function and bandgap:

2. HD-KFM: Principles of Operation

2.1 Multifrequency Excitation

HD-KFM (High-Definition Kelvin Force Microscopy) is characterized by its use of multifrequency excitation. Unlike single-pass setups, HD-KFM tunes the electric feedback to the second eigenmode frequency of the cantilever. This approach involves exciting the first flexural eigenmode of the cantilever mechanically for topography measurements, while the second flexural mode is excited electrically for surface potential measurements.

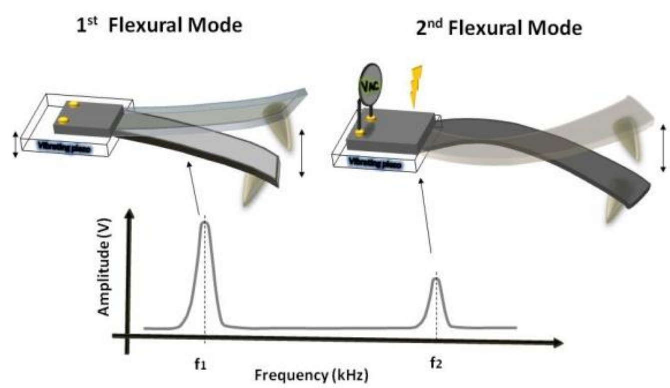


Figure 2: Illustration of multifrequency excitation concept in HD-KFM

The advantages of this method include:

- Signal amplification by the Q factor of the second eigenmode
- Ability to use smaller VAC values while maintaining an acceptable signal-to-noise ratio
- Increased stability due to the stiffer effective spring constant of the second eigenmode

2.2 Probing Close to the Surface

HD-KFM operates very close to the surface, with typical minimum tip-sample distances of 0.1-0.5 nm. This proximity allows for significantly higher sensitivity and lateral resolution compared to standard double-pass KFM techniques, which typically operate at distances of 10-100 nm.

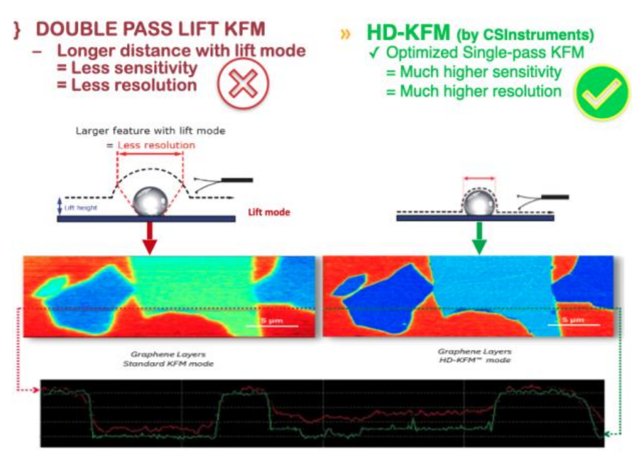


Figure 3: Comparison of standard KFM double pass and HD-KFM operation

2.3 Electric Field Cancelling (EFC)

The HD-KFM III introduces the Electric Field Cancelling (EFC) option, which allows for the compensation of electrostatic fields during measurements. This feature is particularly useful when combining HD-KFM with other measurement modes, such as Magnetic Force Microscopy (MFM).

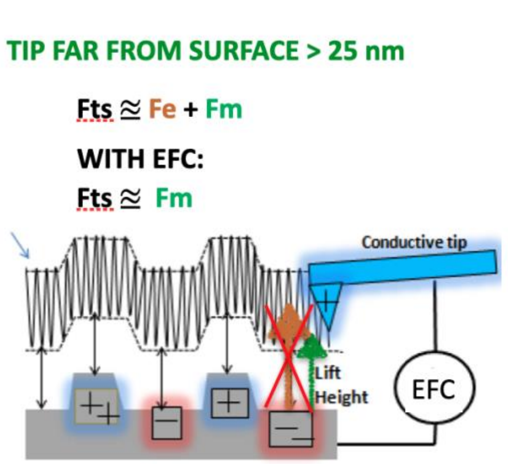


Figure 4: Illustration of EFC concept

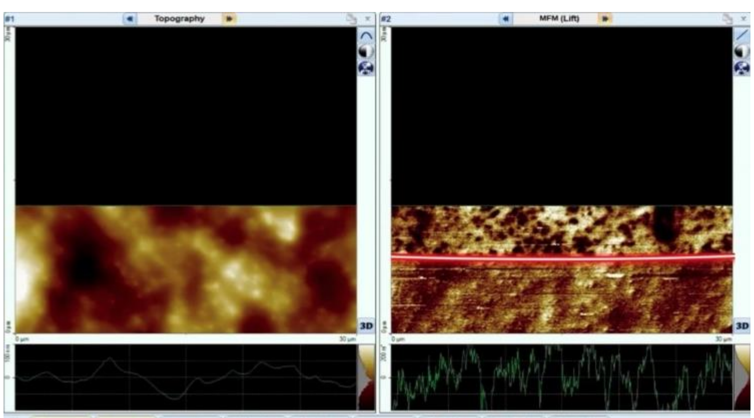


Figure 5: Example of MFM measurement with and without EFC

2.4 Capacitance Gradient (dC/dz) Measurements

HD-KFM III can simultaneously measure the capacitance gradient (dC/dz), providing information about the local dielectric permittivity of the sample. This is achieved by tuning the internal lock-in amplifier to the double frequency component of the electrostatic force.

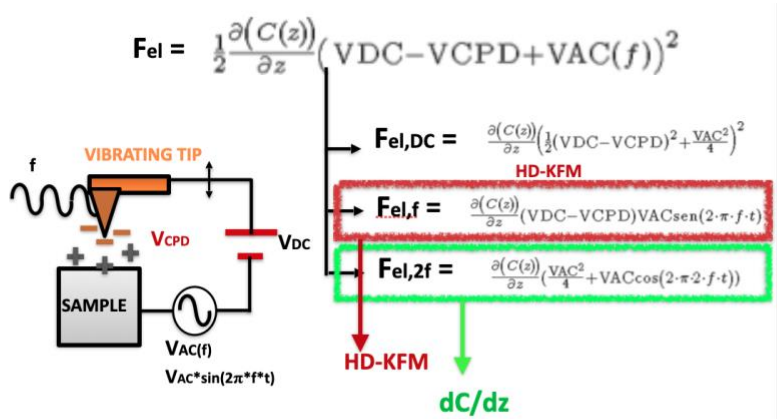
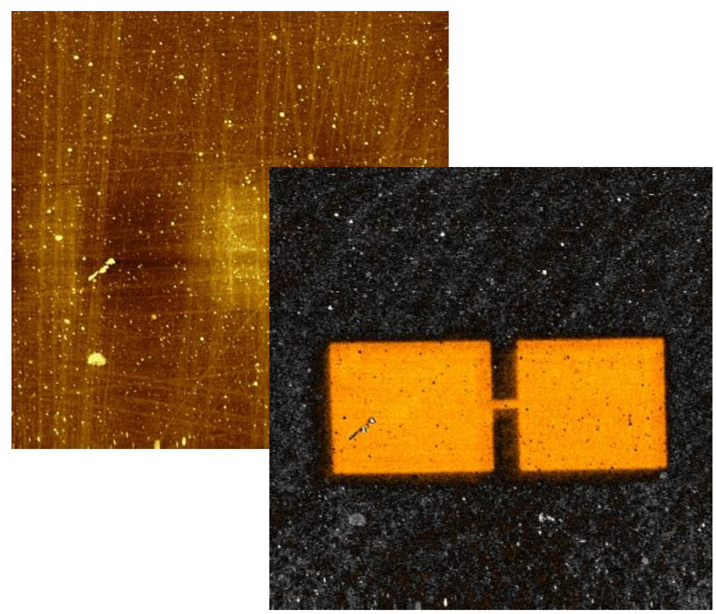


Figure 6: Theoretical background of HD-KFM and dC/dZ operation



Metallic structure embedded on epoxy left topography and right dC/dz results.

3. Applications and Examples

3.1 Semiconductor Characterization

HD-KFM is particularly useful for characterizing semiconductor devices, allowing for both electrical response measurement with nanometric lateral resolution and detection of manufacturing defects. For example, HD-KFM can clearly distinguish between n and p regions in SRAM memory devices.

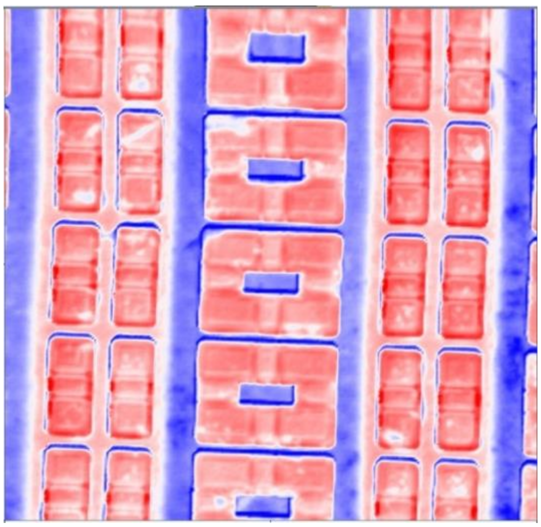


Figure 7: HD-KFM measurement of SRAM memory showing n and p domains (50x50 micron scan size)

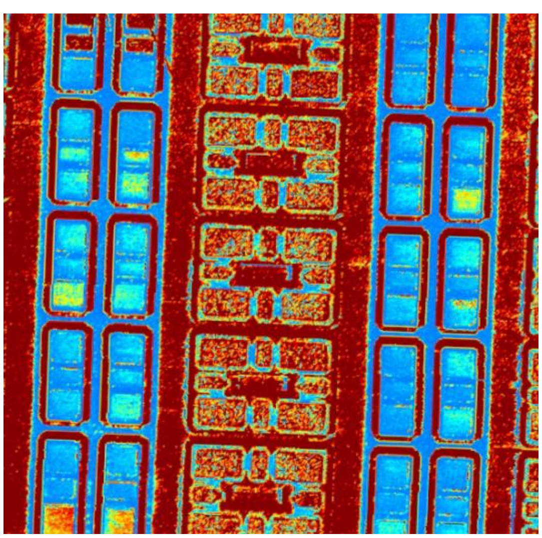


Figure 8: ResiScope measurement of the same SRAM memory area (50x50 micron scan size)

3.2 Solar Cell Analysis

In the field of solar cell research, HD-KFM provides valuable insights into the behavior of new materials and designs. For instance, in perovskite solar cells, surface potential measurements help understand recombination efficiency in active areas. HD-KFM can reveal the positions of active layers and detect defects that may arise from sample preparation processes.

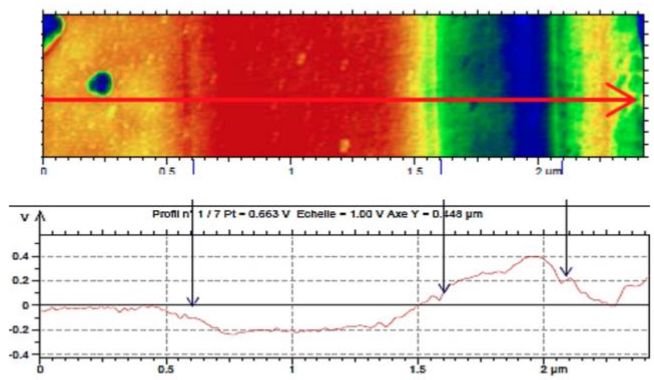


Figure 9: Sketch of the cross-section of a perovskite solar cell

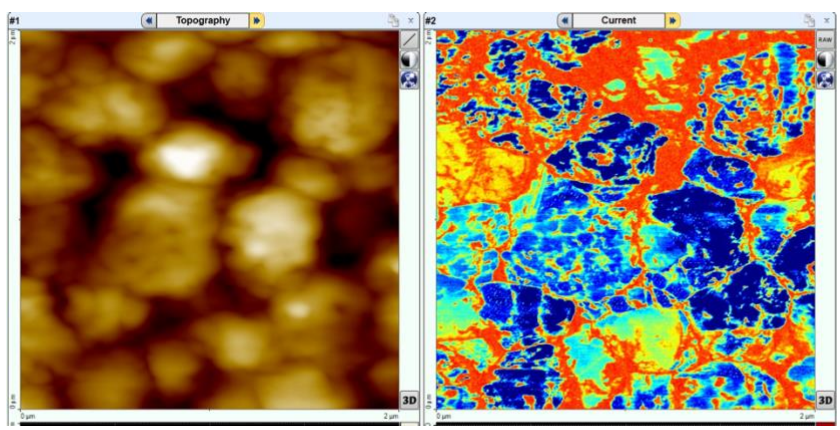


Figure 10: Topography and surface potential of a polished perovskite solar cell (2x2 micron scan size)

3.3 Oxide Materials

HD-KFM is effective in characterizing both polarized and conductive oxide surfaces. It can detect areas of different polarization in materials like ZnO and reveal crystal domains with varying surface potential in conductive oxides such as SrVO3.

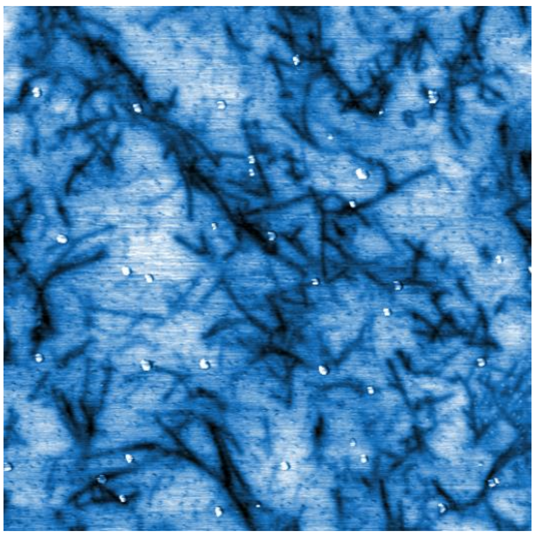


Figure 11: Surface potential image of alternatively biased areas on ZnO thin layer (40x40 micron scan size)

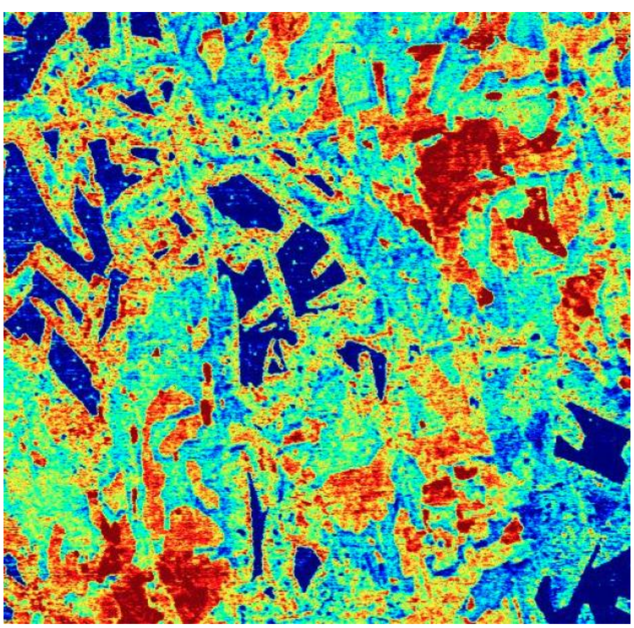


Figure 12: HD-KFM SP image of SrVO3 perovskite

3.4 Buried Structures

The long-range nature of electrostatic interactions allows HD-KFM to detect features below the surface. This capability is particularly useful for characterizing nanocomposites, where reinforcement phases like carbon nanotubes may be embedded within a matrix material. HD-KFM can reveal the distribution of these subsurface structures, which may not be visible in topography scans.

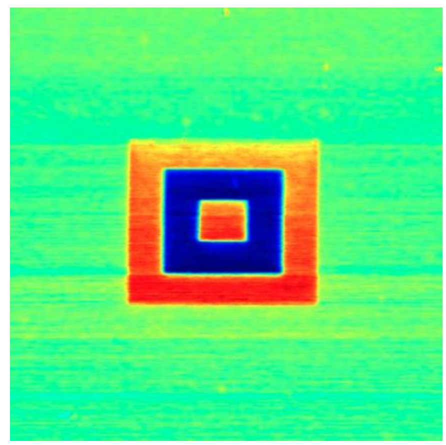


Figure 13: HD-KFM image on buried CNTs (10x10 micron scan size)

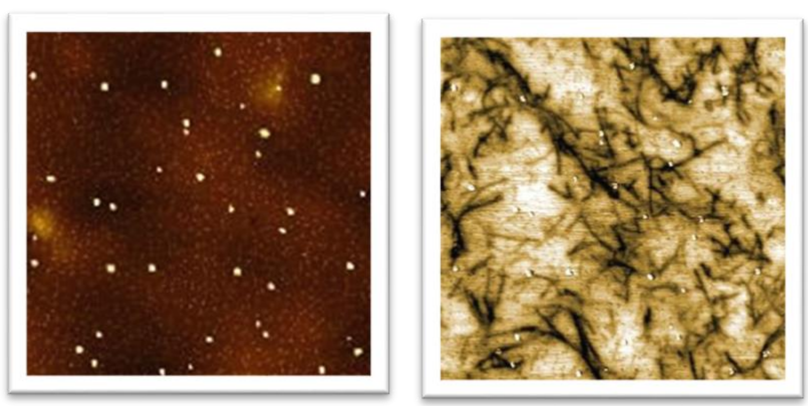


Figure 14: Topography and HD-KFM on Nanotubes embedded on a polymer

3.5 Organic Molecules

HD-KFM can characterize the electrical properties of organic molecules such as fluoroalkanes. It can reveal the dipole orientation of these molecules and provide insights into their structure and arrangement on surfaces.

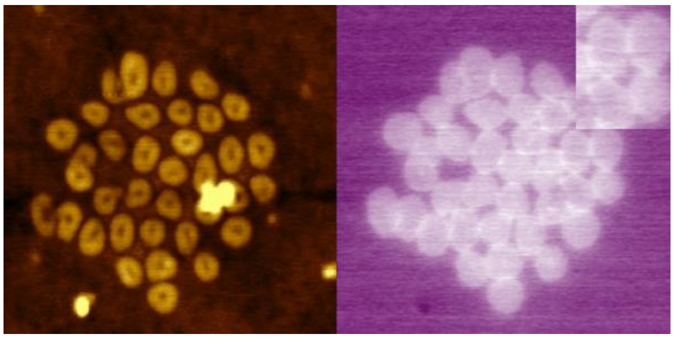


Figure 15: Topography and surface potential of fluoroalkenes molecules

3.6 Magnetic Materials

When combined with Magnetic Force Microscopy (MFM), HD-KFM III allows for simultaneous measurement of both electrical and magnetic properties. This is particularly useful for samples that exhibit both magnetic and electric domains.

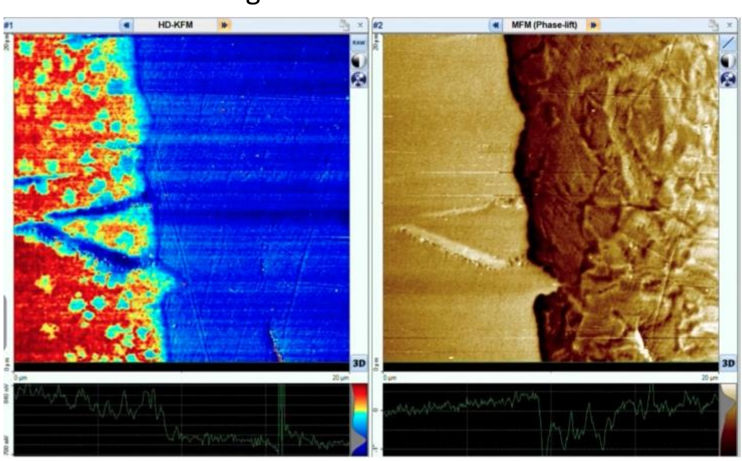


Figure 16: HD-KFM and MFM images of a chrome steel sample

3.7 Battery Research

HD-KFM III has emerged as a valuable tool in battery research, complementing other energy-related applications such as solar cell analysis. The technique's ability to measure surface potential with high resolution makes it particularly useful for studying battery materials and interfaces.

A significant advantage of HD-KFM III is its compatibility with other measurement techniques, particularly when combined with ResiScope. This combination allows for a comprehensive characterization of battery materials without changing the experimental setup.

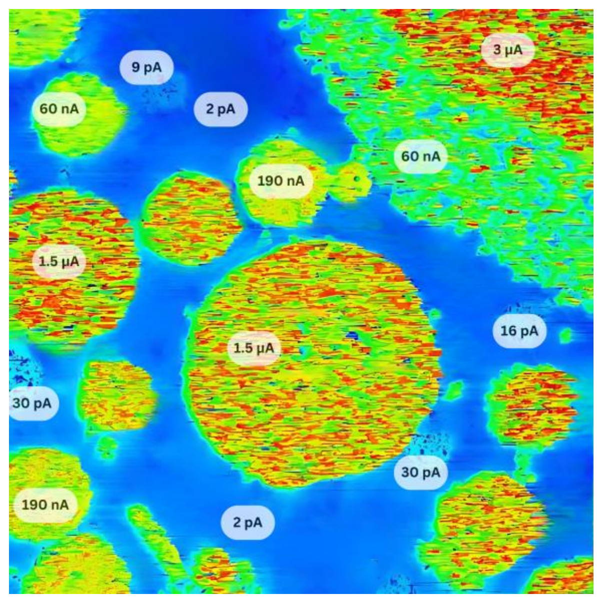


Figure 17: ResiScope current/resistance map of a polymer battery sample (50x50 micron scan size)

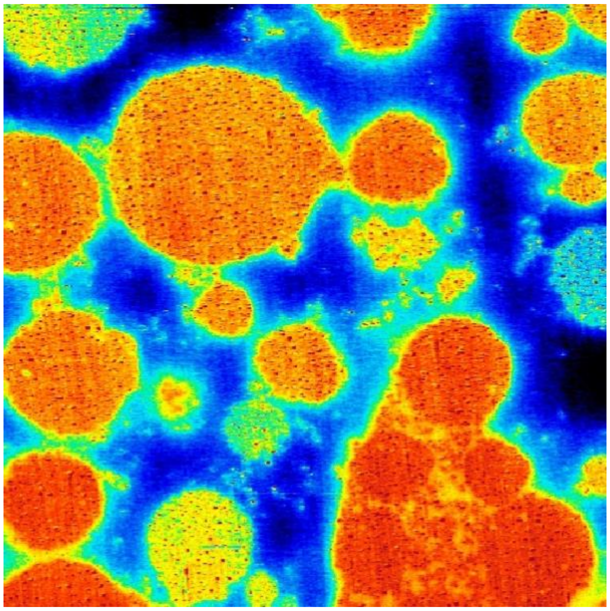


Figure 18: HD-KFM surface potential image of the same area on the polymer battery sample (50x50 micron scan size)

Figure 17 shows the current/resistance map obtained using ResiScope, while Figure 18 displays the surface potential image of a close area acquired with HD-KFM. The correlation between these two datasets provides invaluable insights into the electrical properties and charge distribution within the polymer battery material.

The seamless integration of these complementary techniques demonstrates the versatility and power of HD-KFM III in advanced battery research, paving the way for new insights into energy storage materials and devices.

4. Conclusion: Benefits of HD-KFM III

HD-KFM III represents a significant advancement in electrical characterization at the nanoscale. Its key benefits include:

1. Enhanced sensitivity and lateral resolution due to close-proximity probing
2. Multifrequency excitation, allowing for simultaneous topography and electrical measurements
3. Compatibility with other measurement modes, such as MFM and ResiScope
4. Ability to detect subsurface features and structures
5. Advanced features like Electric Field Cancelling (EFC) and capacitance gradient (dC/dz) measurements
6. Wide applicability across various materials and research fields, including semiconductors, solar cells, oxides, nanocomposites, and organic molecules

In conclusion, HD-KFM III provides researchers with a powerful tool for comprehensive nanoscale electrical characterization, offering insights that are crucial for the development of next-generation materials and devices.