Keysight Technologies
Electrical Measurement
Utilization of Keysight Nanoelectrix AFM Systems for Nanoscale Electrical Investigation

Application Note
Overview

Each Nanoelectrix system from Keysight Technologies, Inc. is precisely tailored to provide the advanced AFM-based electrical characterization methods best suited for specific applications and research interests. Leading scientists around the world have authored a multitude of published papers utilizing data acquired with Keysight’s innovative nanoelectrical measurement instrumentation and techniques.

Researchers in academic and industrial settings alike rely upon the powerful single-pass KFM, EFM, CS-AFM, magnetic force microscopy (MFM), piezo force microscopy (PFM) and SMM methods afforded by Keysight Nanoelectrix systems as they seek to continue expanding the frontiers of many diverse application areas.

The material presented here is a broad overview of this highly varied and rapidly growing body of work.

Technology advantages

- Single-pass kelvin force microscopy (KFM) and electrostatic force microscopy (EFM) for measuring surface potential and capacitance
- Current-sensing atomic force microscopy (CS-AFM) for measuring conductivity variation
- Scanning microwave microscopy (SMM) for calibrated capacitance and dopant density measurements

Data examples

- Single-pass KFM imaging of
  - Metals and semiconductors
  - Nanowires
  - Organic electronic materials
  - High spatial resolution on $F_{14}H_{20}$ on HOPG
- CS-AFM measurements
  - Ionic conductivity of nafion 212
  - Individual cnts in via arrays
  - Graphene/nBH hybrid material
- SMM investigations of
  - Dopant structures of semiconductor devices
  - Highly doped marker layers in gallium nitride (GaN) on sapphire
Single-Pass KFM/EFM Imaging

KFM has been widely used to map surface potential distribution at the nanoscale. There are two different approaches for KFM imaging, namely, lift mode and single pass. In lift mode, topography and surface potential images are acquired via two separate scans. The topography image is obtained first and the tip is then lifted a specified distance above the topography for a second scan that measures surface potential.

In single-pass KFM, topography and surface potential are acquired simultaneously using a multiple-frequency technique. The cantilever is excited simultaneously by two AC modulation signals at different frequencies. The first one is used for mechanical excitation to measure topography and the second one, usually at a much lower frequency, is for electric modulation to measure surface potential. Keysight’s single-pass KFM implementation utilizes a high-performance, triple lock-in AC mode controller. This unique method operates in the intermittent contact regime and the tip is always brought into the vicinity of the sample surface, thus significantly improving both detection sensitivity and spatial resolution. Additional details about the principles and instrumentation associated with single-pass KFM can be found elsewhere.

KFM contrast reveals important information about surface charging, molecular dipole orientation in organic thin films, band bending and dopant concentration in semiconductor materials, and much more. KFM can also be utilized to measure the work function of a conducting material (e.g., the change in work function with film thickness of few-layer graphene).

Figure 1 for example is the use of KFM for compositional imaging of metals and semiconductors of the test structures made by FIB deposition of Pt and SiO₂ lines on surfaces of Si wafer and graphite. The contrast of the lines differentiates them in the surface potential images. The quantitative data is seen from the cross-section profiles placed under the images. The potential of the Pt strips on both substrates are close to that of the conducting probe. The potential of SiO₂ strips, which are 20 nm in height, differs from that of Si substrate that has only 2–3 nm of naturally grown oxide film. Although the Pt and SiO₂ lines on graphite are partially “lost” amidst the numerous surface steps, the surface potential pattern shows only the material-related contrast.

Figure 1. Topography and surface potential images of FIB-deposited lines of Pt and SiO₂ on Si substrate. Scan size 40 μm. The contrast covers the height and potential changes in the 0–60 nm and 0–1V ranges. The cross-sections profiles taken along the directions pointed with the white dotted lines are placed underneath the images.
More sophisticated standards, which have surface layers with different doping type and level, are employed for verification using AFM based electric techniques capable of quantitative tracking these industrially important properties. KFM images of the IMEC standard (n-type Si staircaseT9 on the surface with the doped layers running perpendicular) are shown in Figure 2 together with the resistivity and doping profiles determined by different methods. The face with the layers having doping levels increasing from $1 \times 10^{14}$ to $1 \times 10^{16}$ cm$^{-1}$ does not show noticeable surface corrugations. The layers do manifest themselves in the surface potential image as the bands whose contrast increases with the increase of the doping level. The potential varies 250 mV on the locations covering 4 orders of magnitude of dopant density. This data set is in good agreement with the earlier experimental results obtained on Si wafers with different doping density. The difference of 0.240 eV between Fermi levels of the n-Si samples with $1.1 \times 10^{14}$ and $7 \times 10^{19}$ cm$^{-1}$ doping densities was estimated from the surface potential data. The well-pronounced steps of the potential profile (Figure 2) correlates well to the width of the differently doped layers. However, it is rather premature to claim that surface potential provides the direct quantitative measure of the doping density. In many cases the potential differences detected within the single image are relatively reliable. Less reproducible are absolute potential values recorded in different images. Surface contamination, varying oxide coverage and environmental effects are among the possible reasons of the poor reproducibility.

Figure 2. Topography and surface potential images of T9 standard sample with layers having different doping level. Scan size 25 μm × 30 μm. The contrast in the topography image covers the height corrugations in the 0–20 nm range. The resistivity (dark blue) and doping (magenta) profiles are placed underneath the topography image. The cross-section profile along the direction marked with a white dashed line in the surface potential image is shown below it.
KFM on Low-Dimensional Semiconductor Structures

Low-dimensional semiconductor and metal systems offer variety of unique structural, optical and electric properties. Some of these highly ordered materials can be prepared in mild conditions by self-assembly. The formation of CdTe nanowires is one of the examples. It has been shown that crystalline nanowires are built up of CdTe nanoparticles in water solution upon controlled removal of organic stabilizers. The strong dipole–dipole interactions were suggested as the driving force of self-organization of the nanoparticles into the pearl necklace aggregates that re-crystallized into the nanowires. The formation of the CdTe nanowires from suspension of the nanoparticles during its drying on mica substrate was monitored with KFM. The topography and surface potential images, which were recorded at the end of this process, are shown in Figure 3. These images show individual CdTe nanowires, which are 20–30 nm in width and few nm in height, as well as their arrays whose growth was likely nucleated from thicker linear structures. The surface potential image is dominated by bright contrast (approx. 0.5V) of the nanowires. Additionally to nanowires, there are also the small nanoparticles (20–30 nm in diameter), which are brighter than the nanowires in the topography images but darker in the surface potential image. These features can be assigned to the traces of the organic stabilizers of the CdTe nanoparticles.

The KFM results suggest that self-assembly of CdTe nanoparticles into crystalline nanowires is accompanied by structural transitions leading to the formation or realignment of molecular dipoles. Dipole moments in CdTe nanoparticles with cubic lattice are not expected unless they appear as structural defects that induce asymmetry of molecular structure. Therefore, the formation of the nanowires entails the radical changes of local symmetry of molecular groups.

Figure 3. Topography and surface potential of CdTe nanowires formed from a suspension of CdTe nanoparticles. Scan size 2 μm. The contrast covers the height and potential variations in the 0–22 nm and 0–1V ranges.
KFM on $\text{F}_{14}\text{H}_{20}$ on HOPG

On KFM studies of $\text{F}_{14}\text{H}_{20}$ and related compounds we examined their self-assemblies on different substrates and in different environments in pursuit of a better understanding of their structures and behavior. In Figure 4 we can see fine features in the sub-10 nm range, in the topography and surface potential images of the $\text{F}_{14}\text{H}_{20}$ adsorbate. This area is filled by the toroids and self-assembled ribbons. The surface potential of the ribbons is only slightly different from that of the toroids. Nevertheless, there are few locations with very pronounced contrast: the voids between the toroids and the slits between the ribbons. At these locations the probe “feels” the substrate better than elsewhere.

The high-resolution surface potential images of the area outlined with a red dotted square is given in Figure 4(c). The insert shows the profile across one of the slits in the location pointed with a white arrow. The width of the 0.1 V peak seen in this profile is around 2 nm that can be used as a measure of spatial resolution of KFM AM-FM operation in the intermittent contact mode. This result suggests that the sensing apex area of the AFM probe, which is much smaller than the tip diameter (~20 nm), dominates in the electrostatic measurements. The achieved high-resolution is in line with the expectation of where the force gradient detection combined with a small tip-sample distance was advocated as a way to enhance resolution.

Figure 4. (a–b) The topography and surface potential images of the $\text{F}_{14}\text{H}_{20}$ adsorbate on graphite. (c) The surface potential image of an area, which is marked with a white dotted box in (b). The insert in this image presents the cross-sectional profile taken at the location marked with a white arrow in the direction perpendicular to the bright strip.
KFM on Organic Electronic Materials

Current developments of organic materials for electronics strongly depend on understanding structure property relationships in molecular films applied in field effect transistors, light emitting diodes, and organic solar cells. Getting such knowledge becomes non-trivial for objects consisting of a single sheet of molecules where electron transport has a two-dimensional character and the molecular scale imperfections need to be examined. AFM and, particularly, electric modes can be very helpful in the characterization of such structures and related devices. This has been proved in numerous studies of semiconducting molecular layers of pentacene. Single-pass KFM can further improve the characterization of these materials. KFM images of a pentacene adsorbate, which was evaporated on Si substrate in vacuum, are shown in Figures 5. This is a single layer film with several 2nd layer dendritic structures. The 1st layer is composed of tightly packed vertically standing molecules and it has the domain substructure. The domains boundaries are best seen as bright features in the surface potential image. This contrast is observed only when the stimulating AC voltage is above 3 V that indicates on the voltage-induced dipoles at the domains’ edges. The surface potential of the dendritic structures, whose height also indicated on the vertical orientation of the molecules, is more positive (~80 mV) than that of the 1st layer, and it does not depend on a magnitude of the stimulated voltage. Actually, the smaller potential change of 50 mV was observed between the 1st and 2nd pentacene layers on Si using the Lift mode. The nature of the surface potential contrast of pentacene layers is not yet established. The possible contribution of the interfacial dipoles is considered as a factor leading to the observed surface potential.

Figure 5. Topography and surface potential images of a pentacene adsorbate on Si. Scan size 8 μm. The contrast covers the height and surface potential variations in the 0–10 nm and 0–1 V ranges. AC stimulated voltage was 4 V.
CS AFM Imaging of Nafion 212

CS-AFM, also known as conducting AFM, is particularly useful for studying the transport process of protons in proton exchange membranes. The CS-AFM data presented in Figures 6 was performed using a CS-AFM nose cone with a 1 nA/V preamplifier that measures the current flowing through the AFM. The conductive tips are Pt/Ti-coated Si probes with a nominal spring constant of 0.15 N/m and a bulk resistivity of 0.01–0.05 Ω*cm. Before each measurement is taken, the Nafion sample and tip are allowed to settle in the Nanoelectrix system’s environmental chamber for 2–3 hours to let the humidity level stabilize.

The surface potential image reveals cluster- and fiber-like structures on the surface that have a lower conductivity compared to the rest of the surface. The lower conductivity of these fiber-like structures suggests that they correspond to the hydrophobic polymer region that forms the backbone of the Nafion membrane. This conclusion is also consistent with the surface potential measurement.

However, unlike the potential measurement that measures the ionic site on the surface, the CS-AFM measurement detects a conductive ionic current only when the tip is in contact with an ion transport channel that runs through the membrane (i.e., CS-AFM measures only the “active channel” in the membrane). Because the ion conductivity measured with CS-AFM depends on the contact area between the tip and the surface, it is important to maintain constant force during imaging.

Figure 6. CS AFM of Nafion 212. Image reveals cluster and fiber like structures on the surface that have a lower conductivity compared to the rest of the surface. The lower conductivity of this fiber like structures corresponds to the hydrophobic polymer region that forms the backbone of the Nafion membrane.
CS AFM imaging of individual CNTs in Via Arrays

Figure 7 shows a CS AFM mapping of a via array. The vias are localized close to each other. Within a single via the I-V characteristics of three individual MWCNTs were obtained. The inset shows the I = V characteristic of the CNTs within one via does not change.

However, a large variation of the CNT resistances between different vias in the 10 \( \mu \text{m} \times 10 \mu \text{m} \) region were observed in the scan. Comparing the I-V characteristics of the via with the highest resistance (427 M) and the one with the lowest resistance (1.4 M), a difference of two orders of magnitude were found.

![Figure 7. Investigation of an array of carbon nanotube vias (CNT via) Conductivity Mapping exhibiting more or less successful forming of back contacts. Inset: Resistance measurement of three individual CNT vias inside the green circle.](image)

CS AFM Studies of Graphene/nBN Hybrid Material

Graphene and hexagonal boron nitride (h-BN) are layered materials with similar crystal structures but dramatically different electronic properties. If two-dimensional domains of graphene and h-BN, with controlled shapes and sizes, can be stitched together seamlessly and engineered into extended hybrid atomic layers, it opens up possibilities for new two-dimensionally engineered materials. Figure 8 on the right is the AFM topographic image of an in-plane G/h-BN heterostructure consisting of randomly distributed domains. The domain boundaries between graphene and h-BN are almost indistinguishable.

The left image was the simultaneously acquired with the current image (right) and the two different materials are clearly identified. Those brighter regions (i.e., higher current) are corresponding to the conductive graphene domains.
SMM Imaging of Dopant Structures of Semiconductor Devices

SMM is an award-winning imaging mode developed by Keysight that combines the comprehensive electrical measurement capabilities of a microwave vector network analyzer (VNA) with the nanoscale spatial resolution of an atomic force microscope. This innovative AFM mode of operation offers unprecedented utility for a diverse set of applications. Its ability to provide high-sensitivity, complex electrical and spatial measurements is particularly useful for semiconductor test and characterization. In addition to enabling complex impedance (resistance and reactance) measurements, SMM mode can be used to acquire calibrated capacitance and dopant density measurements when studying sidewall diffusion.

A wide range of excitation frequencies (2 to 20 GHz) ensures that SMM users can select the optimal frequency to maximize the signal-to-noise ratio and achieve the best sensitivity. A special dopant profile measurement module (DPMM) extends the capabilities of SMM to provide calibrated, absolute measurement of dopant densities, critical for advanced device physics studies. A lock-in amplifier for differential capacitance (dC/dV) measurements can be added. SMM is capable of obtaining capacitance and dC/dV images simultaneously.

The microwave signal from the VNA is divided into two parts within the DPMM. The first part is amplified and used as the local oscillator (LO) signal for the dC/dV mixer. The second part is amplified and sent through the main arm of the coupler to the AFM probe tip, where a low-frequency (RF) signal is also applied to the tip from an external source (e.g., a function generator).

Due to changes in the capacitance of the sample induced by the RF signal, the microwave signal is reflected and modulated at a rate equal to the RF frequency. The reflected, modulated microwave signal is then divided into two parts as well. The first signal is amplified and directed to the DPMM internal mixer, where it is mixed with the LO signal and demodulated. This demodulated signal is further processed using a lock-in amplifier for dC/dV amplitude and phase signal. The second part of the reflected microwave signal is amplified and delivered to the VNA receiver, and is used to measure the capacitance of the sample.

Figure 8. CS-AFM studies of Graphene/h-BN Hybrid Material. Left is the AFM topographic image of an in-plane G/h-BN heterostructure consisting of randomly distributed domains. The domain boundaries between graphene and h-BN are almost indistinguishable. The right image is the simultaneously acquired current image and the two different materials are clearly identified. Those brighter regions (i.e., higher current) are corresponding to the conductive graphene domains.
In the case of semiconductor devices, the mobile charge carrier in the doped region can either accumulate or deplete in the vicinity of a contact electrode depending on the DC bias applied.\textsuperscript{8,9} By applying an AC voltage, $V_{ac}$, around a fixed working potential, $V_{dc}$, a change of capacitance $dC$ in response to the modulation voltage $Vac$ will be introduced.\textsuperscript{10} $V_{dc}$ is usually chosen at that point where the C-V curve has the largest slope, which is around the flatband voltage. In this way, maximum sensitivity is achieved. Under these conditions, a higher $dC/dV$ value corresponds to low carrier concentrations, while a lower $dC/dV$ value corresponds to higher carrier concentrations. It is also clear that for both p- and n-type Si, $dC/dV$ is the same in magnitude but of opposite sign (positive for p-type, negative for n-type) for identical carrier concentrations. Therefore, the modulation index of the reflected microwave signal (i.e., the magnitude and phase of the modulated signal measured by SMM) can be used to characterize the structure and type of dopant in semiconductor devices.

As stated earlier, SMM can be utilized to obtain capacitance and $dC/dV$ images simultaneously. Capacitance (VNA amplitude) and $dC/dV$ images of an IC sample are presented in Figure 9.\textsuperscript{10} The capacitance image, Figure 9(a), clearly reveals differences in the varied shallow doping that constructs devices within the active areas. Dramatic contrast difference in the $dC/dV$ phase image, Figure 9(b), confirms the presence of sub-surface highly doped layers. A vertical line profile across the phase image (as indicated by the green line in Figure 9(b) is shown in Figure 9(c). The measurement shows that the different bands in the IC are 180 degrees in phase mode, which suggest that those two different regions of the sample are dominated by opposite charge carriers, in correspondence to the existing bands of n-type buried layer (NBL) and p-type buried layer (PBL) under the active components. The circuitry map is also overlaid atop the VNA amplitude image to show the regions of the NBL and PBL. A small difference in capacitance can be seen between the two regions.

Figure 9. Capacitance (a), $dC/dV$ phase (b), phase line profile (c), and design layout of an IC sample. (BiCMOS silicon IC sample that has been delayered to reveal the doped regions within the active devices. The IC process stack includes a lightly doped p-substrate with an epitaxial layer approximately 3.0 $\mu$m thick. Highly doped N+ and P+ buried layers are positioned at the interface. Shallow doping of various impurities forms the wells and contacts of the active devices.)
SMM Imaging of Highly Doped Marker Layers in GaN on Sapphire

SMM was used here to investigate the origin of unintentionally doped regions in gallium nitride (a III-V compound semiconductor with a wide band gap) grown on a sapphire substrate. During the growth process, thin marker layers were introduced that are snapshots of the surface configuration. Figure 10 shows SMM topography, a capacitance map, a dopant density map of the film cross-section, and a line profile across the dopant density map.  

The sapphire substrate is located on the left edge of the dataset; the wafer surface would be further towards the right but it is not within the scan range shown. The topography shows a step from the substrate to the GaN layers, several steps within the GaN, and some undefined contaminants at the right edge. The capacitance map shows some contrast at the substrate-film interface and a regular pattern of bright and dark lines towards the wafer surface. In SMM dopant density maps, undoped as well as highly doped material yield lower signal or darker regions. The dark regions comprise the sapphire substrate, the highly doped marker layers, and the undoped GaN layers. The regions are indicated in Figure 10(d). The bright features are regions with a low (but not too low) density of charge carriers. This is the case in the regular stripes towards the wafer surface. Between the layers dubbed “undoped” a layer of highly doped material was grown. Both materials have little dC/dV signal and show dark. Due to diffusion of carriers from the doped into the undoped layers a low density of carriers is present at the edge of the undoped region and thus shows a high dC/dV signal. The straight regular stripes indicate that the film growth of these layers was regular and smooth.

In between the substrate and the smooth layers is another region of doped material. This region was unintentionally doped during the growth process. In this region, there are one to three dark bands meandering from left to right. The bands are highly doped marker layers. They mark the position of the growth surface at those times when dopant material was introduced. In the unintentionally doped region the doped layers show a strong fluctuation, indicating a rough surface during growth. Furthermore, SMM dopant density maps of larger regions (not shown here) appear to support the assumption that inclined surfaces are crucial for the growth of unintentionally doped material.

Beyond its ability to measure semiconductors, glasses, polymers, ceramics, and metals, SMM mode allows users to perform high-sensitivity investigations of ferroelectric, dielectric, and piezoelectric materials. In studies of organic films, membranes, and other biological samples, SMM can provide unique insight into fundamental characteristics. For example, SMM’s high sensitivity (1.2aF) is ideal for looking at ion channels.

Graphene studies represent another area in which the use of SMM mode is rapidly proving invaluable to researchers.

Figure 10. (a) – (d) SMM topography, capacitance map, dopant density map, and cross-section of dopant density map along the green line in (c).
References


AFM Instrumentation from Keysight

Keysight Technologies offers high-precision, modular AFM solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Keysight’s leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative and easy-to-use AFM technologies.

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