

Scanning Microwave Microscopy for Nanoscale Electrical Characterization

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Introduction

Recently, a highly sensitive imaging mode for, complex, calibrated electrical and spatial measurements was made available to atomic force microscope (AFM) users [1, 2]. Scanning microwave microscopy (SMM), an award-winning AFM mode of operation developed by Agilent Technologies, combines the comprehensive electrical measurement capabilities of a microwave vector network analyzer (VNA) with the nanoscale spatial resolution of an AFM.

This imaging mode outperforms traditional AFM-based scanning capacitance microscopy techniques, offering greater application versatility, the ability to acquire calibrated quantitative results, and higher sensitivity and dynamic range. A new, second-generation SMM nose cone brings a simplified user experience to these advanced measurements. Probe exchange is now more streamlined, reducing the complexity of experimental setup and reducing the time to results. The new SMM nose cone also brings these capabilities to all members of the Agilent 5000 series AFM family, specifically the 5420, 5500, and 5600LS AFM systems.

SMM mode is useful for a diverse set of applications. Its ability to provide calibrated, highly sensitive electrical and spatial measurements is particularly useful in semiconductor test and characterization. In addition to enabling complex impedance (resistance and reactance) measurements, SMM mode can be used to acquire calibrated, traceable capacitance and dopant density measurements.

The SMM mode works on all common semiconductor types: Si, Ge, III-V (for example, GaAs, InAs, GaN) and II-VI (for example, CdTe, ZnSe). Unlike other scanning-probe capacitive techniques, it does not require an oxide layer, simplifying sample preparation. However, the properties measured have wide applicability outside the semiconductor field in such areas as materials science and life science research. The 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 insight into fundamental sample characteristics. For example, SMM's high capacitance sensitivity (~ 1.2 aF) is ideal for the study of ion channels. Graphene studies represent another area in which the use of SMM mode is valuable to researchers, yielding detailed electrical properties in conjunction with high-resolution surface morphology measurements. This article gives a description of the SMM technique and shows results from two important applications.

Principles of Operation

Scanning Probe Microscopy (SPM). SPM uses a physical, small radius probe to delicately trace the surface topography and resolve features under 1 nm. A SPM system will employ a silicon cantilever with a sharp-tipped probe to interact with

the sample and to measure the interaction with the sample at each point in the scan area (or field of view). The most common embodiment of a SPM is the AFM, where the control signals that are monitored are the forces between the probe and the surface.

Being a physical “contact” technique, the employment of conductive or magnetic coated probes, and special control feedback mechanisms, allow the method to be often extended to various related measurements including surface potential, capacitance, electrostatic force measurement, and magnetic domain mapping.

SMM couples a solid PtIr probe in contact mode imaging with a VNA to measure complex impedance and capacitances of a variety of samples.

Reflected microwave signals. In SMM mode, the system's VNA sends an incident microwave signal through a diplexer to the sub-7 nm conductive tip of a solid platinum-iridium cantilever. The signal is reflected from the tip and measured by the VNA. The magnitude and phase of the ratio between the incident and reflected signals are calculated, and a model is then applied in order to calculate the electrical properties of the sample. The AFM scans the sample and moves the tip to specific locations to perform point probing.

Measuring impedance. The VNA provides three different methods for measuring complex impedance, depending on the frequencies and magnitudes involved. The impedance of a device under test is measured by comparing the reflected return from the incident signal on the device and extracting the device impedance. This “reflectance” method works best at microwave frequencies and at impedances that are near the characteristics of transmission lines (50Ω or 75Ω). To ensure the best performance, the device under test is placed in parallel with a load to match the 50Ω ideal load and hence to optimize the responsiveness of the VNA's measurements.

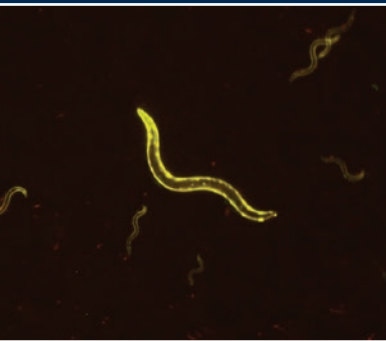
The SMM mode couples the high-resolution complex impedance measurement capability of the VNA with the metallic AFM probe, providing high spatial resolution for reflectance measurements of impedance. This simple yet effective probe for the device under test can detect very small changes in impedance—that of the metallic AFM probe in contact with the semiconductor sample. A wide range of excitation frequencies (2 GHz to 20 GHz) ensures that SMM users can select the optimal frequency to maximize the signal-to-noise and to attain the best sensitivity.

System components. SMM has been engineered to leverage the accuracy and flexibility of a VNA for impedance measurements, particularly for quantitative capacitance measurements on a dielectric sample. Furthermore, it can be tailored to measure differential capacitance (dC/dV) by applying a low-frequency (RF) modulation signal to the

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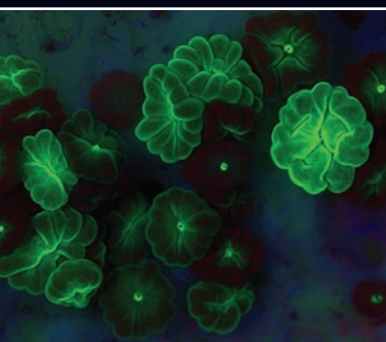
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microwave measuring signal [1, 2]. A standard SMM system, which comprises an AFM and a VNA, can be configured to include a dopant profile measurement module (DPMM) and a lock-in amplifier for dC/dV measurements. The components of a typical SMM system configuration are schematically illustrated in Figure 1. It can be used to perform capacitance and dC/dV imaging simultaneously.

System operation. 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 an RF signal is also applied to the tip from an external source (for example, a function generator).

Because of 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; it is used to measure the capacitance of the sample.

Results

Carrier concentrations. For the case of semiconductor devices, the mobile charge carriers in the doped region can either accumulate or deplete in the vicinity of a contact electrode depending on the DC bias applied [3, 4]. 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 V_{ac} will be introduced, as illustrated in Figure 2. V_{dc} is usually chosen at that point where the C-V curve has the largest slope, which is around the flatband voltage. Note that “flatband voltage” refers to a term used in metal oxide semiconductor (MOS) devices: a voltage at which there is no electrical charge in the semiconductor and no voltage drop across it; in a band diagram, the energy bands of the semiconductor are horizontal (flat). In this way, maximum sensitivity is achieved. Under these conditions, a higher dC/dV value corresponds to low carrier concentrations, whereas 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

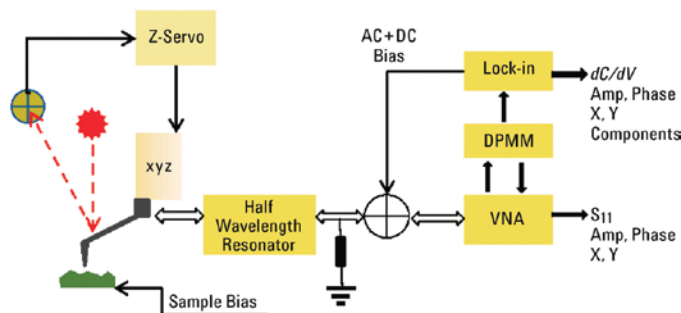


Figure 1: Block diagram of the SMM configuration for capacitance and dopant measurements.

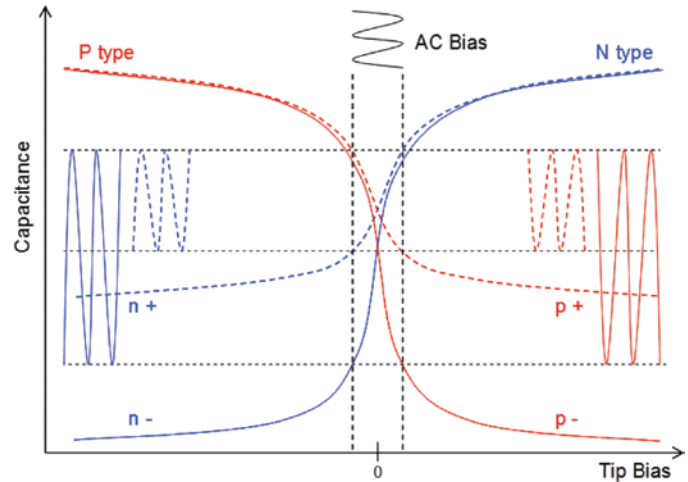


Figure 2: Capacitance vs. voltage plot for n-doped (blue) and p-doped (red) semiconductors. For the same ac modulation signal applied, the change in capacitance is larger for lower doping density (solid line) and smaller for higher doping density (dashed line). For the p- and n-doped samples shown, these signals are identical in amplitude, but opposite in phase.

carrier concentrations. Therefore, the modulation index of the reflected microwave signal (that is, 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.

Capacitance images. As stated earlier, SMM can be used to obtain capacitance and dC/dV images simultaneously. Capacitance (VNA amplitude) and dC/dV images of an individual capacitor (IC) sample are presented in Figure 3. The capacitance image, Figure 3A, clearly reveals differences in the varied shallow doping that creates devices within the active areas. Dramatic contrast differences in the dC/dV phase image, Figure 3B, 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 3B) is shown in Figure 3C. The measurement shows that the different bands in the IC are of 180 degrees different in phase, which suggests that these two regions of the sample are dominated by opposite charge carriers, in correspondence with the existing bands of n-type buried layer (NBL) and p-type buried layer (PBL) under the active components.

Figure 4 shows the topography (A), dC/dV amplitude (B), and dC/dV phase (C) images of a smaller area on the IC sample surface. The dC/dV amplitude reveals the difference in dopant level. The dC/dV phase reveals two types of information in this case: first, the opposite carriers between the bands; second, the small variations due to changes of each individual component. These variations become visible only when looking at the n- or p-type region with the contrast properly adjusted, as shown in the zoomed image of Figure 4D.

Thus, SMM mode can be used for capacitance and dopant profiling measurements on semiconductor devices. It is possible to identify certain structures below the surface level because of the penetration length and interaction volume of the microwave signal.

Graphene. Another key use of SMM is in the study of graphene. Graphene, a new member of the carbon family, has stimulated extensive interest in both academia and

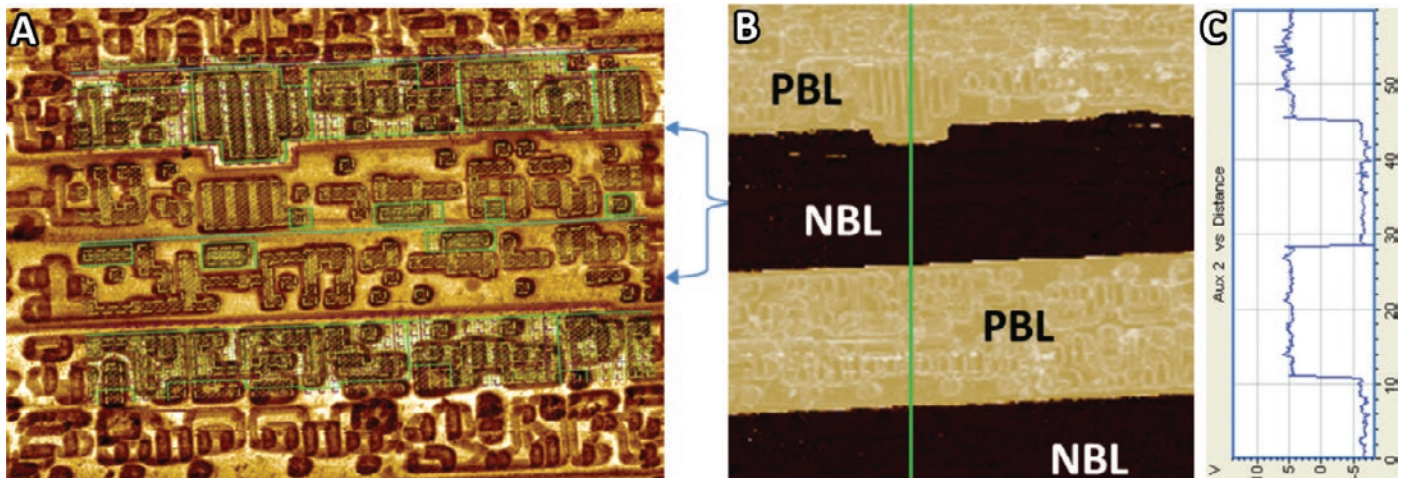


Figure 3: Integrated circuit plan view. (A) Capacitance image, (B) dC/dV phase image, and (C) phase line profile at green line in (B). This is a BiCMOS silicon IC sample that has been delayed to reveal the doped regions within the active devices. The IC process stack includes a lightly doped p-substrate with an epitaxial layer approximately 3.0 μm thick. Highly doped N+ (NBL) and P+ (PBL) buried layers are positioned at the interface. Shallow doping of various impurities forms the wells and contacts of the active devices.

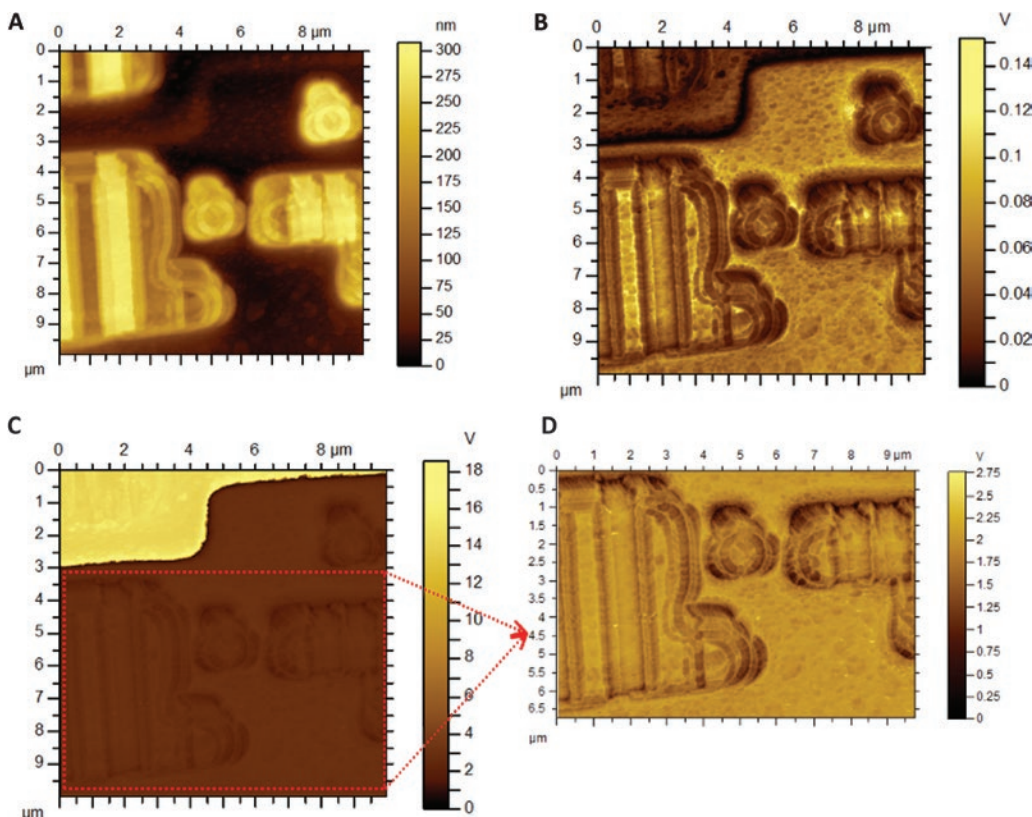


Figure 4: Higher magnification images of the same integrated circuit as Figure 3. (A) Topography image, (B) dC/dV amplitude image, and (C) dC/dV phase image. (D) is a zoomed region of (C) showing the small variations of phase corresponding to individual structures.

industry owing to its unique electrical, mechanical, and optical properties. With a hexagonal monolayer of sp^2 -bonded carbon atoms, graphene is a basic building block for all graphitic materials.

One future direction of graphene-related research is to seek alternative layered inorganic materials as analogues of graphene—materials exhibiting some needed attributes that

graphene lacks. Hexagonal boron nitride (h-BN), for example, attracts much attention because it is a III-V compound with a wide band gap (more than 5 eV). The monolayer h-BN is called “white graphene.”

Figure 5A is a high-resolution topographic image of a few-layer h-BN specimen synthesized via chemical vapor deposition. It exhibits a structural heterogeneity. Some regions corresponding to high-quality h-BN films are observed. Figure 5B is the close-up of one such location, indicated as a red square in Figure 5A. As can be seen, its surface morphology is smooth and uniform, and the quality is very close to those films produced using a mechanical exfoliation method. In addition, several protrusion islands corresponding to regions with one or more additional layers of BN are resolved.

The capacitance images (VNA amplitude and VNA phase) reveal that the double-layer films are fairly uniform across the region (Figure 6). Even though the existence of wrinkles caused by the transfer process or grain boundaries of the underlying substrate are resolved in the topographic images, they are not shown in the capacitance images. This indicates that the capacitance images are dominated by the film thickness and the dielectrics of the film. It is interesting,

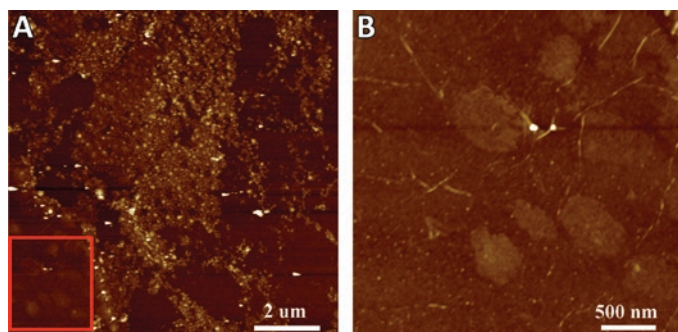


Figure 5: SMM of few-layer h-BN ultrathin films. (A) One example of a topographic image revealing rich surface structures of few-layer h-BN ultrathin films. (B) A closer look at the high-quality h-BN location indicated by the red square in (A). Samples for imaging generously provided by Columbia University (New York), Rice University (Texas), and University of Malaya (Malaysia).

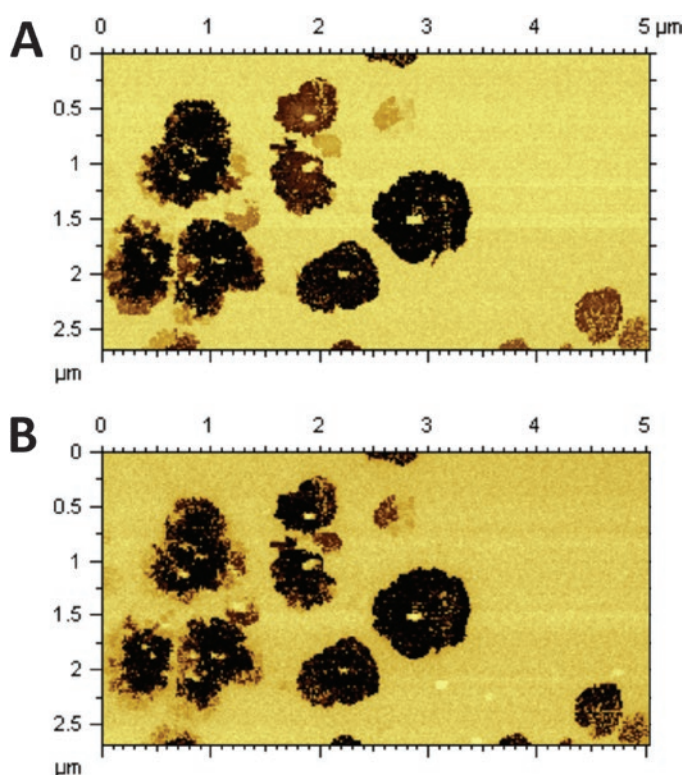


Figure 6: SMM of few-layer h-BN ultrathin films. (A) Acquired VNA amplitude image and (B) corresponding VNA phase image from SMM imaging of a different area of the same sample as Figure 5. Lighter regions are the dual-layer h-BN material, and the darker islands are tri-layer h-BN films. The thickness difference between 2-layer and 3-layer h-BN films is 1 nm. The contrast difference is caused by the sensitivity of SMM to measure the capacitance difference between the films. Samples for imaging generously provided by Columbia University (New York), Rice University (Texas), and University of Malaya (Malaysia).

however, to see that the contrast difference (likely a capacitance change) between the three-layer islands and the surrounding double-layer region are outstanding in Figures 6A and 6B. Because the difference in thickness between those layers is only about 1 nm, the overall film thickness is about 3 nm. These results demonstrate the sensitivity of the SMM technique.

Conclusion

SMM, an imaging mode available from Agilent Technologies, delivers electrical measurement capabilities of

a state-of-the-art microwave VNA, as well as the nanoscale spatial resolution of a high-precision AFM. The SMM mode is the only AFM-based electrical characterization method that provides dopant profiling calibration standards. There are two standards for dopant profiling calibration via SMM: one for n-type and one for p-type. This new quantitative information helps researchers better understand the response and behavior of nanoscale systems, particularly when device properties need to be assessed at their intended operation frequencies. The SMM mode can be applied in semiconductor test and characterization, biological science, materials science, graphene studies, and many more application areas.

References

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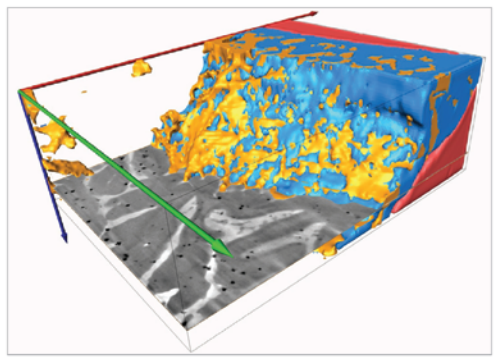
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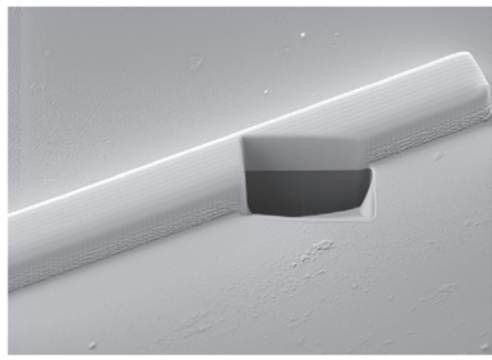
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