Application Note
AN.R.092017

RESISCOPE II

ADVANCED CONDUCTIVE NANO CHARACTERIZATION WITH RESISCOPE II AND SOFT RESISCOPE II ATOMIC FORCE MICROSCOPY

1. ABSTRACT

In this Application Note we will describe the most advanced module for conductive measurements with Atomic Force Microscopy named ResiScope II. This module allows to perform current/ resistance measurements over 10 orders of magnitude (from 100 fA to 1 mA). By connecting ResiScope to a conductive tip, surfaces with both highly conductive and insulating domains can be mapped overcoming undesired side-effects like probe-induced local oxidation, bimetallic effect or melting of the conductive coating by Joule effect. Additionally it will be described the Soft ResiScope mode, that allows to carry out electrical characterization in intermittent contact (point by point) at regular scanning speeds (few lines per second). This module prevents the tip from wear due to continuous scanning in contact and is especially suitable for soft samples.

2. RESISCOPE AFM

Since the invention of Atomic Force Microscope (AFM) in 1986 by Quate, Binnig and Gerber, many alternative modes have been developed to map (with nanometric lateral nanometric) different physical properties or interactions that can be correlated to the surface topography.
ResiScope mode is a different approach to perform conductive measurements that was first proposed in 1996 by Boyer et al, (first conductive experiments were carried out by O'Shea et al in 1993).

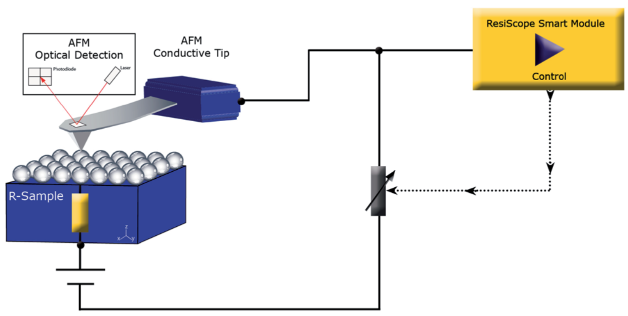

Figure 1 : Schematics illustrating the ResiScope concept.

In Figure 1 it is shown a schematics of the ResiScope concept: total current flowing through the tip and sample is limiting by an internal device driven by the controller.
Most of the conductive techniques simply use passive electronics (amplifiers with predefined gains) which not gives flexibility on large dynamic range and sensitivity.
The ResiScope electronics use a Processor (DSP) to select in realtime the best configuration and the appropriate gain that better matches the sample resistance (current).

The ResiScope module provides 10 orders of magnitude of measurement range (10 orders of magnitude, from 100 fA to 1 mA for current, 1 kΩto 1TΩ for resistance) thus allowing to measure both highly conductive and insulating domains simultaneously. On the other hand actual current flowing through the tip and sample is limited which minimizes associated side effects like Joule self-heating (which may induce melting of the metallic coating), local oxidation induced by the probe or bimetallic effect. (see ref. 5 for more details about this phenomena).

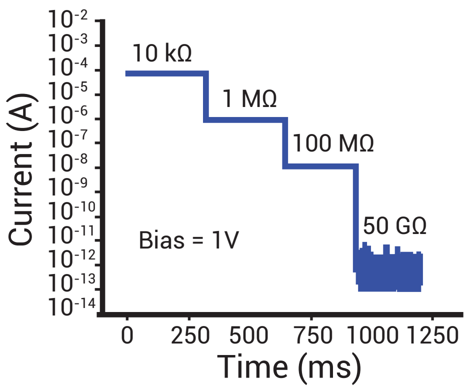

Figure 2 : Measurement of different resistance values with the ResiScope (from 10 kohm to 50 Gohm).

In Figure 2 is shown a graph illustrating the high dynamic range and the fast velocity of the ResiScope. A set of different resistors with known values was used to simulate a sample.

The resistor values were 10 kΩ, 1 MΩ, 100 MΩ and 50 GΩ respectively. The graph depicts the measured current values while keeping the bias at 1 V. The resistors were switched every 250 ms approximately, the fast switching can be appreciated as the measured current does not show transients in between two resistors.

3. CONDUCTIVE VS RESISCOPE

In Figure 3 it is shown a comparison between a standard conductive AFM measurement and a ResiScope AFM measurement on a sample of graphene oxide domains over gold substrate. This sample provides a good test for conductive measurements with the ResiScope module as it has well-defined conductive and non-conductive domains. In Figure 3a and 3b it is shown the topography and current images respectively of a standard conductive AFM mode (amplifier gain = 1 nA/V) over the oxide graphene/Au sample. The current image shows typical features of an uncontrolled conductive measurement: on the top part of the image, stable current image is obtained with well defined values for the gold (saturated to maximum value 10 nA-red color) and graphene oxide (<1nA values-blue color). After few lines, current signal decrease, increases again and eventually conductivity is lost. This behavior is an example of the influence of the several phenomena described in ref 5.

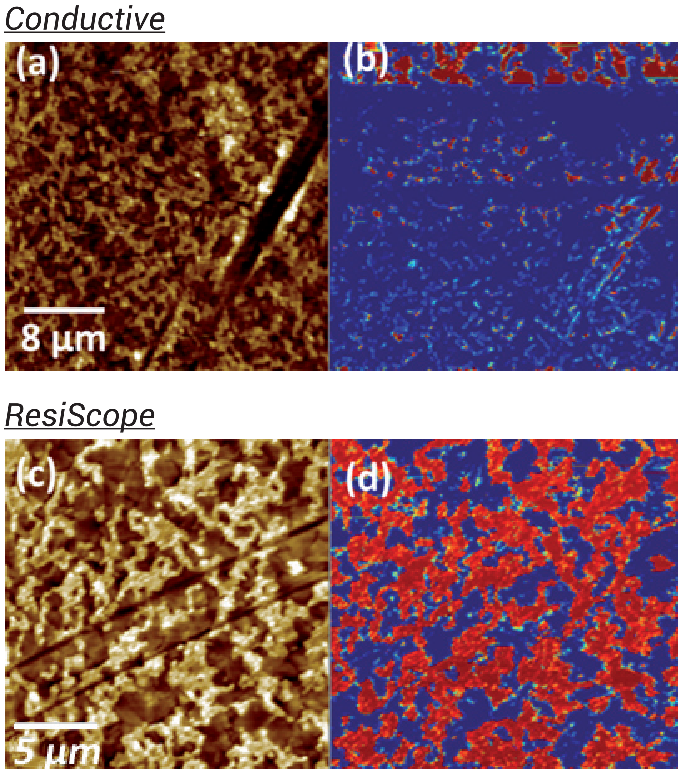

Figure 3 : Comparison of conductive and ResiScope mode on a gold sample covered with domains of graphene oxide. (a) and (b) topography and current in C-AFM. (c) and (d) topography and current in ResiScope mode.

In Figure 3c and 3d are plotted similar images when operating in ResiScope mode with a similar cantilever and same applied force. The comparison with the results shown in Figs. 3a and 3b reveals the benefits of performing conductive measurements with ResiScope mode: the image is completely stable from top to bottom parts of the scanned area. Additionally the high dynamic range allows to measure the electric signal in the gold areas without saturation (blue areas) and the graphene oxide covered areas (read areas)...

4. RESISCOPE AFM CHARACTERIZATION ON SEMICONDUCTOR DEVICES

ResiScope is the suitable mode for conductive characterization of electrical devices with high lateral resolution. In this section we will show several examples of it.

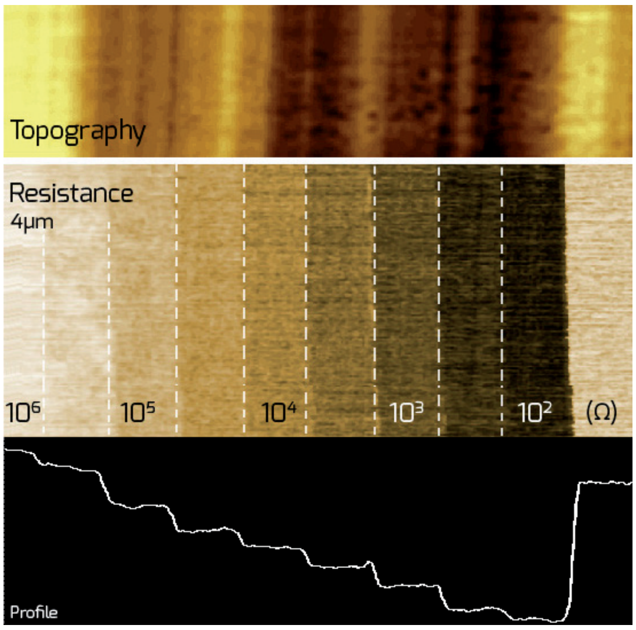

Figure 4 : ResiScope characterization of a SiC doped sample with an staircase dopping profile.

In Figure 4 it is shown an example of a ResiScope characterization on a SiC sample with a staircase-type n-doping concentration profile made by ionic implantation. The doping concentration is increased from left to right. The image size is 4 µm.

Figure 4 (top) shows the topographic image while Figures 4 (middle and bottom) show the resistance image and a resistance cross-section line respectively. Topography show some vertical lines due to the cleaving and polishing process of the sample. Resistance image show clearly the different doping levels of approximately 400 nm width showing clearly defined borders.

It can be seen that the resistance decreases from 1 MΩ to 100 Ω as the mobility of the carriers is decreased with the increasing doping concentration. It can also be noticed that there is no cross-talk between the lines produced with the polishing (topography) and the electric behavior (resistance).

Figure 5 illustrates another example of the ResiScope capabilities on a more challenging device: a PMOS transistor. CMOS transistors are the most widely used among all of the Integrated Circuit (IC) technologies. A PMOS transistor consists on a source and drain p+ regions on an n-type bulk. When the bias is applied to the gate (in between the source and the drain) a current flows from source to drain.

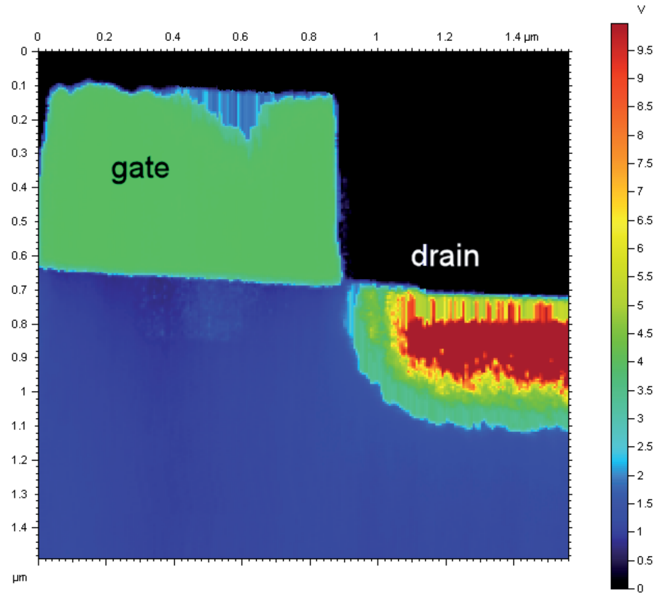

Figure 5 : ResiScope characterization of a PMOS transistor.

The ResiScope image shows part of the structure of the PMOS transistor (gate and part of the drain) and shows different values of resistance for gate and drain and the n-bulk (blue color in the image). It can also be seen some defect in the gate induced by the cleaving process. It is interestingly to notice that the drain shows to well defined areas, the outer one (green colored) delimits the borders of the depletion region while the red area clearly defines the p+ implanted regions.

5. HIGH RESOLUTION RESISCOPE AFM

Beside the high sensisitvity of the ResiScope, another important issue regarding the conductive measurements is the lateral resolution achieved in the electrical measurement. This is strongly dependent on the total radius of the tip and the conductive coating. However, as the measurement is made in continuous contact mode, the tip coating may be worn after several images due to both mechanical and electrical stress. CSInstruments provides a new family of doped diamond probes made of single crystal with an outstanding performance, durability and sharp tip-radius (up to 5 nm).

In Figure 6, it is shown an example of the characterization of the Back Surface Field of a solar cell. Creation of BSF prevents surface recombination of electrons and holes witch reduces the efficiency of the performance of the solar cell. Fig. 6 shows comparison of a resistance measurement of the BSF on a solar cell with a standard conductive diamond tip and a high resolution single crystal diamond probe (Concept Scientific Instruments, France). Fig. 6(left) shows the image obtained with the high-resolution boron-doped single crystal tip (tip-radius <10 nm), while Fig. 6 (right) shows the similar image with a standard boron-doped diamond probe (tip radius >50 nm). As it can be clearly noticed in the respective profiles, the 30 nm barrier for the carriers can be clearly resolved only with the high resolution tip.

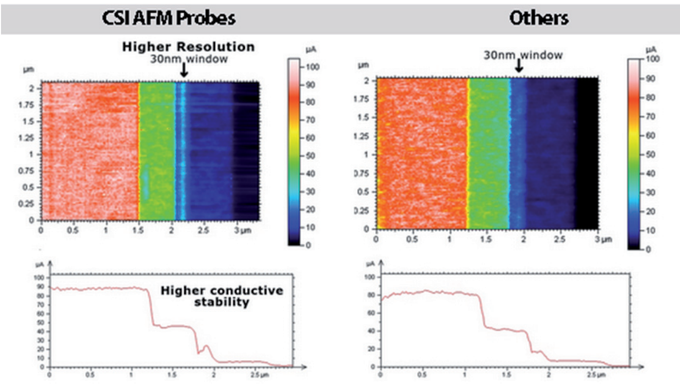

Figure 6 : ResiScope characterization of the Back Surface Field of a solar cell with a high resolution single crystal diamond tip (left) and an standard diamond tip (right).

Another example of the high resolution that can be achieved by combining both the benefits of using ResiScope (high dynamic range and less electrical stress) and high resolution tips is shown in Figure 7. A resistance image of a complex sample alloy shows multiple small domains of different metallic and intermetallic compounds giving different values of resistivity. Even domains separated by grain boundaries of 5-10 nm can be resolved, which allows to characterize the distribution of phases in the alloy.

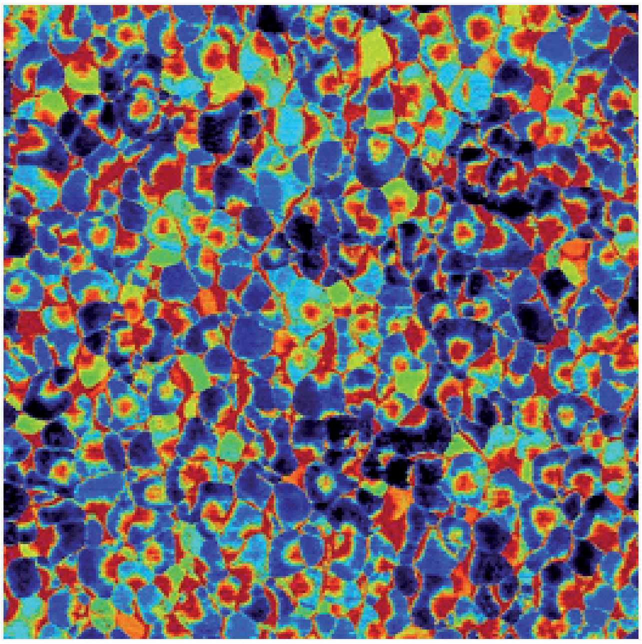

Figure 7 : ResiScope characterization of the domains of a complex alloy.

6. SOFT RESISCOPE MODE

The main drawback of ResiScope mode is the fact that the topographic image is obtained in contact mode, which implies continuous mechanical contact between tip and sample. This originates friction forces that may damage the sample (i.e., soft polymers, biomolecules) or the tip coating. This disadvantage can be overcome with the recently developed Soft ResiScope mode.

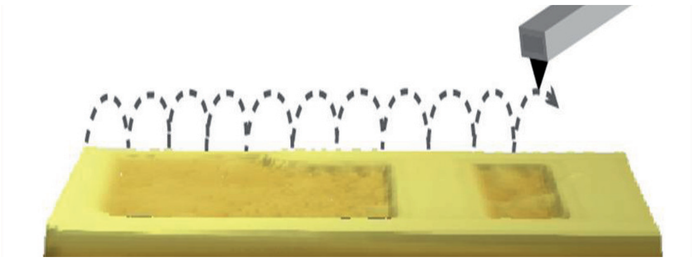

Figure 8. Schematics illustrating the Soft ResiScope mode.

Figure 8 shows the concept of Soft ResiScope mode. The tip separation is modulated from one point to the other of the surface so that mechanical contact is avoided. Then, on every point, the tip is approached to the sample at the desired force setpoint, and the bias is applied to collect the current. This measurement cycle can be made at regular scans velocities (1-3 Hz) without ringing and, more importantly, ensuring that the tip-sample contact (setpoint force) is constant during the electrical measurement. This last condition is vital to obtain accurate and reproducible measurements.

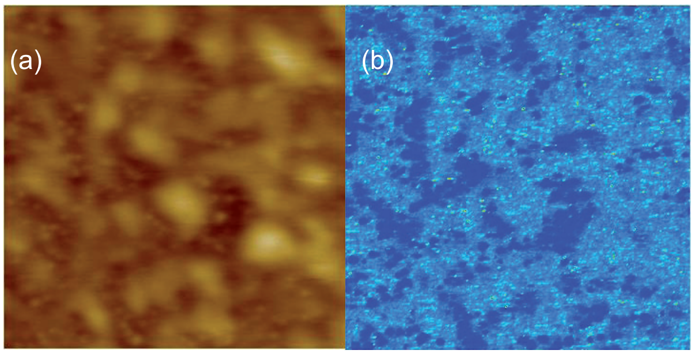

Figure 9. Soft ResiScope characterization of an organic solar cell. (a) topography, (b) resistance

Soft electrical materials are becoming more interesting as flexible electronics is based on organic compounds. Electrical characterization of conjugated polymer-based organic solar cells made from poly (3-hexylthiophene) - P3HT- is of interest for this application. Operating in standard conductive AFM would damage the organic polymer as discussed above. This is a clear example where the Soft ResiScope mode is the right choice for electrical characterization. Figures 9a and 9b show topography and resistance images respectively on a P3HT organic solar cell. Topography shows a surface with some waviness, due to the fabrication process. The resistance map shows a correlation of the deeper areas (less thickness) with a smaller value of resistance, which allows to better control the deposition process.

8. REFERENCES.

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