ORCA[™] – Conductive AFM Imaging Using the MFP-3D[™] AFM

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Conductive AFM is a powerful current sensing technique for electrical characterization of conductivity variations in resistive samples. It allows current measurements in the range of hundreds of femtoamps to ten microamps. Conductive AFM can simultaneously map the topography and current distribution of a sample. It is a measurement useful in a wide variety of material characterization applications including thin dielectric films, ferroelectric films, nanotubes, conductive polymers, and others.

How It Works

The ORCA module consists of a specially-designed cantilever holder that includes a transimpedance amplifier. The gain of the amplifier can be chosen by the user. Standard values range from 5 x 10^7 to 5 x 10^9 volts/amp. The cantilever holder is used with conductive AFM probes to make the measurement. The easiest technique for measuring the localized conductivity of a sample is to combine the current measurements with contact mode AFM imaging. All images in this application note were acquired using contact mode with a PtIr coated Electri-Lever (Olympus), with a nominal spring constant of 1-2 N/m and good wear characteristics. Note that coated cantilevers are vulnerable to imaging artifacts associated with irreversible changes in the tip shape or coating. This is an important consideration when interpreting ORCA measurements.

Current Measurements

Data in this application note was made using a gain of 5 x 10⁸ volts/amp on the initial stage (see ORCA-58 in Figure 1). On the MFP-3D, the ORCA output was digitized with one of the auxiliary 100 kHz ADCs and then digitally filtered at 1 kHz. The measured RMS noise for these settings was 0.5 pA, consistent with the Johnson noise performance predicted in Figure 1.

Figure 2 shows an example image made at a 1.5 volt bias. The sample is a 10 nm thick film of Europium doped ZnO. This is a relatively high resistivity sample, particularly challenging for conductive AFM measurements. The contact mode topographic image "A" shows a relatively uniform grainy structure. The current image "B", however, shows patches of high conductivity surrounded by very low conductivity regions (see Figure 3 for a higher resolution scan). The NPS[™] nanopositioning closed loop sensors on the MFP-3D make it possible to reproducibly position the cantilever at a point of interest as shown by the colored circles in Figure 2B. The tip was positioned in the center of the colored circles using the MFP-3D's "pick a point" force curve interface. The bias voltage was then swept from -5 to 5 volts and the response current measured. Figure 2C shows the resulting current-voltage (IV) curves. The conductivity curves are consistent with the contrast observed in Figure 2B. Specifically, the conductivity is highest at the position marked with the black circle, in between at the red, and lowest at the blue.



ORCA Sample Mount (left), and Cantilever Holder (right).



Figure 1: Gain selection chart. Johnson noise and the relevant current ranges for a transimpedance amplifier digitized at 16 bits. At a gain of nearly 10¹⁰ volts/amp, Johnson noise is equivalent to the best resolution of a 16-bit ADC. At smaller gains, the main limitation is the resolution of the ADC. At higher gains, Johnson noise dominates.



Figure 2: Topography (A) and current (B) image of a Europium-doped ZnO sample at a bias of 1.5 volts, 2 µm scan sample courtesy of the Krishnan Lab, Univ. of Washington. Corresponding IV curves (C) recorded at three specific positions indicated in B. The curves are consistent with the current contrast observed in 2B. Specifically, the conductance is highest at the black location, medium at the red and lowest at the blue.



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Current as a Function of Loading Force

Using the flexible IGOR Pro software interface and the ARC2[™] Controller, ORCA can also be used for a variety of transport measurements. Figure 4 shows the effects of loading force on the IV curves. For this experiment, the cantilever was positioned at an XY point on the sample. The cantilever deflection was maintained at a constant value with a feedback loop. At the same time, the tip bias was swept sinusoidally at an amplitude of 1 volt and a frequency of 10 Hz. The measured current was then plotted vs. the drive voltage (see Figure 4). Not surprisingly, as the loading force is increased, the conductance increases. There is also more noise in the signal at smaller loading forces.

Combined Force and Current Measurements

Figure 5 shows another measurement with the current flow as a function of the load at two fixed biases. There is an asymmetry in the responses at the different biases consistent with the IV curve measurements in Figures 2C and 4. There are also some additional features in these curves. First, the conductance seems to be highest at a point where there is a step in the contact portion of the force curve. There is also a difference between the extension and retraction curves. These features are consistent with the contact resistance of the sample varying as a function of load, although other explanations are certainly plausible.

Specifications

- The standard ORCA cantilever holder has a gain of 5 x 10⁸ volts/amp (~1 pA to 20 nA).
- The Dual Gain ORCA cantilever holder has gains of 1 x 10^{6} and 1 x 10^{9} volts/amp (~1 pA to 10 μ A).
- Other gain values are available.
- Includes cantilever holder, sample holder, and test resistor.

Conclusion

The ORCA conductive AFM option for the MFP-3D provides low-noise, flexible transport measurements at the nanoscale. The flexible software environment enables a variety of standard measurements to be made, as well as allowing the researcher to define their own experiments.



Figure 3: High resolution topography (I) and current (r) at a bias of 1.5 volts, 50 pA scale, 2 µm scan. Sample courtesy K. Krishnan Lab, University of Washington.









Figure 5: Current (above) and force (below) as the cantilever extends towards the ZnO sample surface and retracts away. The blue curves show the positively biased response while the red shows the negatively biased response.

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