Quantitative Electrical Measurements with Atomic Force Microscopy

Jennifer E. Greene
Nanoscience Instruments, 9831 S. 51st Street, C119, Phoenix, AZ 85044
jgreene@nanoscience.com

https://doi.org/10.1017/S1551929515000991 Published online by Cambridge University Press

Introduction

Since the introduction of atomic force microscopy (AFM) from the landmark publication by Binnig, Quate, and Gerber [1], the field of study has exploded well beyond using interatomic forces to image topography on the nanometer scale. The ability to measure intermolecular forces and see atoms was scientifically tantalizing. Soon after, other material properties could be imaged and measured, including capacitance [2], magnetic forces [3], and surface potential [4].

Over the course of 30 years, AFM instrumentation has evolved and moved from basic research into product development laboratories and manufacturing lines. Capabilities such as resistance and conductance measurements using AFM are now available and are beneficial for a variety of applications. Two areas of interest that require electrical characterization include solar cells and microelectronics. Examples of these applications are included in this article to illustrate the capabilities for quantitative nanoscale electrical characterization using AFM.

Previous limitations of electrical AFM measurements include friction interference, lack of quantitative measurements, difficulties measuring soft materials, and limited resistance range. These drawbacks are resolved with the ResiScope module for the Nano-Observer AFM (Concept Scientific Instruments, France). The ResiScope has a large dynamic range covering 10 orders of magnitude for resistivity and can provide quantitative resistance and current measurements on many different materials.

Soft polymers like those used for organic solar cells can be characterized using a new Soft ResiScope mode. Available soon, the Soft ResiScope mode uses a semi-oscillating technique to eliminate friction or damage to the sample. The Soft ResiScope mode is currently the only commercially available method for quantitative resistance measurements of compliant samples. Concurrent operation of the Nano-Observer AFM with the ResiScope provides simultaneous imaging and material property measurements.

Materials and Methods

AFM imaging modes. Different imaging modes are referred to throughout this article and are briefly described here. All involve the basic premise of an AFM probe being raster-scanned across a surface. In contact mode, typically the probe is held at a constant force while the cantilever deflection is monitored through a feedback loop to yield a topographic image. Dynamic force mode, often referred to as either tapping, oscillating, or intermittent-contact, is an imaging technique that oscillates the cantilever so the tip is touching the surface intermittently. Oscillating imaging is beneficial for soft or compliant samples. Note that AFM images do not have any color information. Color scales are chosen by the user from the instrument software.

Electric force microscopy (EFM) measures the electrical field across a sample, usually in a dual-pass mode where the tip is close to the surface to image topography then retracted about 100 nm above the sample to measure the field. A conductive probe is required for this method. Some AFM systems also require additional software to enable this feature. Magnetic force microscopy (MFM) is a similar technique as it uses a second pass to measure the magnetic field a distance from the surface. MFM requires a probe with a magnetic coating.

Kelvin force microscopy (KFM) measures the surface potential of a sample, also requiring a second pass and conductive tip. An option for the Nano-Observer AFM is a high-definition mode (HD-KFM) that acquires data in a single pass at the sample surface, yielding higher spatial resolution than the standard KFM mode. HD-KFM is an option that requires additional hardware for the Nano-Observer AFM.

ResiScope module. The ResiScope module works in combination with the Nano-Observer AFM. The Nano-Observer AFM is capable of advanced modes and integrates the latest technology for high-resolution imaging. A patented flexure-guided stage with three low-voltage piezoelectric devices is mounted in a massive platform and is combined with a low-noise laser and electronics. Environmental controls for temperature, liquid, and gas are available. For this work, the Nano-Observer AFM modes used included contact, oscillating, and KFM in conjunction with the resistance and current measurements provided by the ResiScope. Conductive probes were also used for these samples.

Resistance measurements. The ResiScope module can provide resistance measurements simultaneously with other dynamic modes like EFM, MFM, or KFM. Resistance data from 10^2 to 10^{12} ohms can be obtained. In addition to the resistance data shown in this article, current measurements and current/voltage spectroscopy data can be acquired over an equivalent range.

The general principle of the ResiScope is shown in Figure 1. Resistance measurements are made by applying a DC bias between the sample and a conductive AFM probe, holding the AFM tip at virtual ground. For standard measurements, the tip is scanned in contact mode using the laser deflection for AFM feedback. As an independent measurement, the ResiScope measures the sample resistance through a fast-response amplifier (HPA). During the resistance measurement, the digital signal processor (DSP) chooses the best gain in real time to optimize the measurement made by the HPA. This operating condition affords high sensitivity across the entire range of resistivity at a regular scan speed for AFM. Exclusive to the ResiScope, the https://doi.org/10.1017/S1551929515000991 Published online by Cambridge University Press

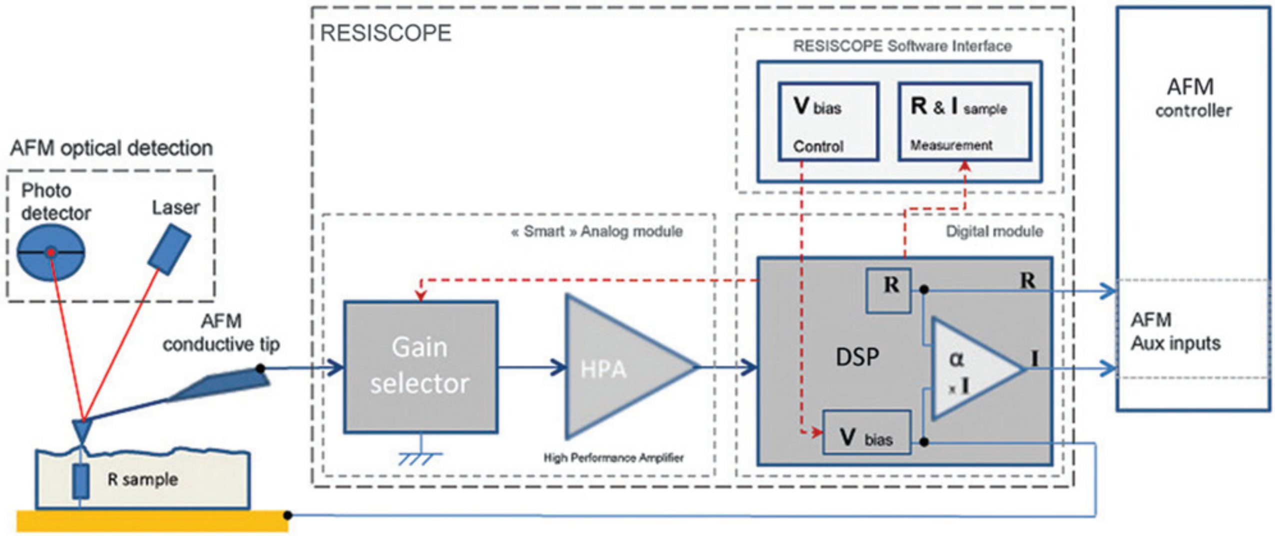

Figure 1: Schematic diagram showing how the ResiScope works. A DC bias is applied between the sample and a conductive AFM tip. The tip is held at virtual ground, and sample resistance is measured through a fast-response amplifier module (HPA), independent of the AFM imaging feedback signal. Unique to the ResiScope, the module will auto-calibrate when the system is started, using internal precision resistors as references to provide quantitative data.

current between the probe and the sample is limited as the module introduces a resistor in series to the sample resistor. This has the result of minimizing the local effect of oxidation or electrochemistry, thereby protecting the conductive probe from possible high-current damage.

ResiScope mode vs. conductive AFM. There are similarities and differences between conductive AFM mode and ResiScope mode. Both techniques require a conducting AFM tip and use contact mode imaging to scan a sample. However, conductive AFM covers only a few orders of magnitude, can only measure current, and does not limit damage to the sample. Conductive AFM data cannot be acquired simultaneously with other imaging modes.

ResiScope can operate in oscillating mode, over 10 orders of magnitude of resistance, and limits the current to minimize damage to the sample. The ResiScope measures resistance or current and can provide I/V spectroscopy data. Other imaging modes can be measured simultaneously or in the same location with ResiScope mode.

Soft ResiScope mode for compliant samples. For the soft mode, the AFM probe only stays in contact with the sample during a short period of time. This is done with a constant force control, which allows the ResiScope module to measure the resistance and the current in the best conditions for quantitative measurements. The tip is then retracted and moved to the next point. Applied as an intermittent contact mode, there is no friction with this technique. Because the measurement is done at constant force, the measurements are quantitative.

To show there is no difference between the standard and Soft ResiScope modes for non-compliant samples, images were acquired of a static random access memory (SRAM) chip sample. Figure 2 shows topography and resistance images acquired in each mode. For each image, the displayed upper half was acquired in the Soft ResiScope mode with intermittent contact. The lower half of each image was taken in the standard ResiScope mode with contact mode imaging. The cross-sectional analysis below the images demonstrates nearly identical results between the standard and Soft ResiScope modes. The red line corresponds to a line scan from the Soft ResiScope image. The blue line

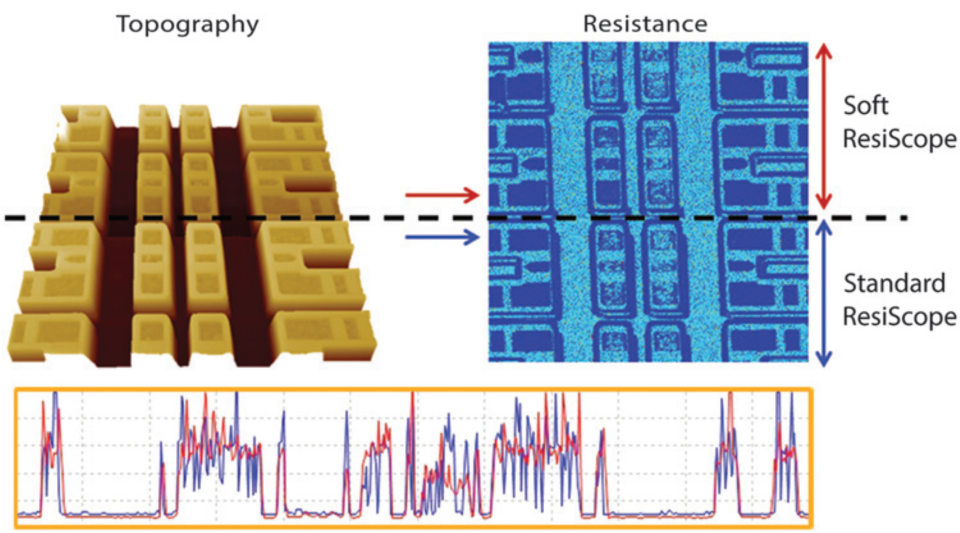

Figure 2: Standard ResiScope imaging versus Soft ResiScope imaging for an SRAM chip, indicating equivalent results for topography (left) and resistance (right). The displayed upper half of each image was measured in Soft ResiScope mode and is comparable to the displayed lower halves that were acquired with the standard ResiScope imaged using contact mode. The cross-section analysis shows similar results between the two techniques for the same sample, with the red line for Soft ResiScope and the blue line corresponding to the standard ResiScope data. Image width = 30 µm.

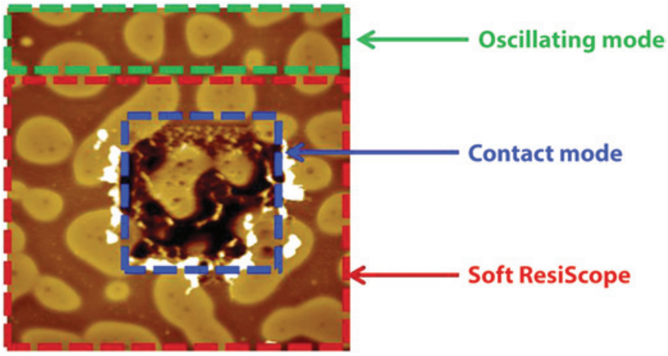

Figure 3: Image of a soft polymer that compares oscillating and contact mode imaging to Soft ResiScope mode. The area defined by the blue square was imaged using contact mode. The red square outlines the region imaged using Soft ResiScope and compares well to the area outlined in green for oscillating mode imaging. Final image width = 50 μm.

corresponds to data taken at the same locations in the standard ResiScope mode.

Figure 3 illustrates the differences between contact, intermittent, and Soft ResiScope modes for a compliant polymer sample. A small area of the sample was first imaged in contact mode. Then a larger area was imaged, initially in oscillating mode and switched to Soft ResiScope mode for imaging the remainder of the scan area. Damage from the original contact mode is evident, but no damage and no image differences are observed between the oscillating and Soft ResiScope modes.

Results

Organic solar cells. New research developments seeking inexpensive renewable energy sources often employ polymer materials [5]. Electrical characterization of conjugated polymer-based organic solar cells made from poly(3-hexylthiophene), referred to as P3HT, is of interest for this application. Conductive AFM would damage the organic polymer. Because these samples are soft, the Soft ResiScope mode was used to obtain resistance data for these samples. Figure 4 shows topography and resistance data for a P3HT organic solar cell. There is no AFM imaging-induced damage to the sample. The resistance image indicates variation across the polymer sample and is a great demonstration of the sensitivity of the Soft ResiScope mode. The resistance image shows the conductivity across the P3HT layer and is independent of topography. Understanding differences in resistance and polymer distribution may help to improve solar cell efficiency.

The images in Figure 4 are also an excellent illustration of why there are different AFM imaging modes. For soft samples, contact mode imaging is not possible without damaging the surface. Topography imaging alone does not always provide the answers that researchers need. Further, topography data often does not correlate to the material properties. For these reasons, advanced imaging and Soft ResiScope modes have been developed to provide quantitative data on a variety of surfaces.

Semiconductor SRAM. Static RAM for the semiconductor industry is commonly used for memory. The p-n junctions of the SRAM are clearly defined by resistance measurements as shown in Figure 5. The color scale for the resistance image (right) indicates that resistance in this sample ranges from

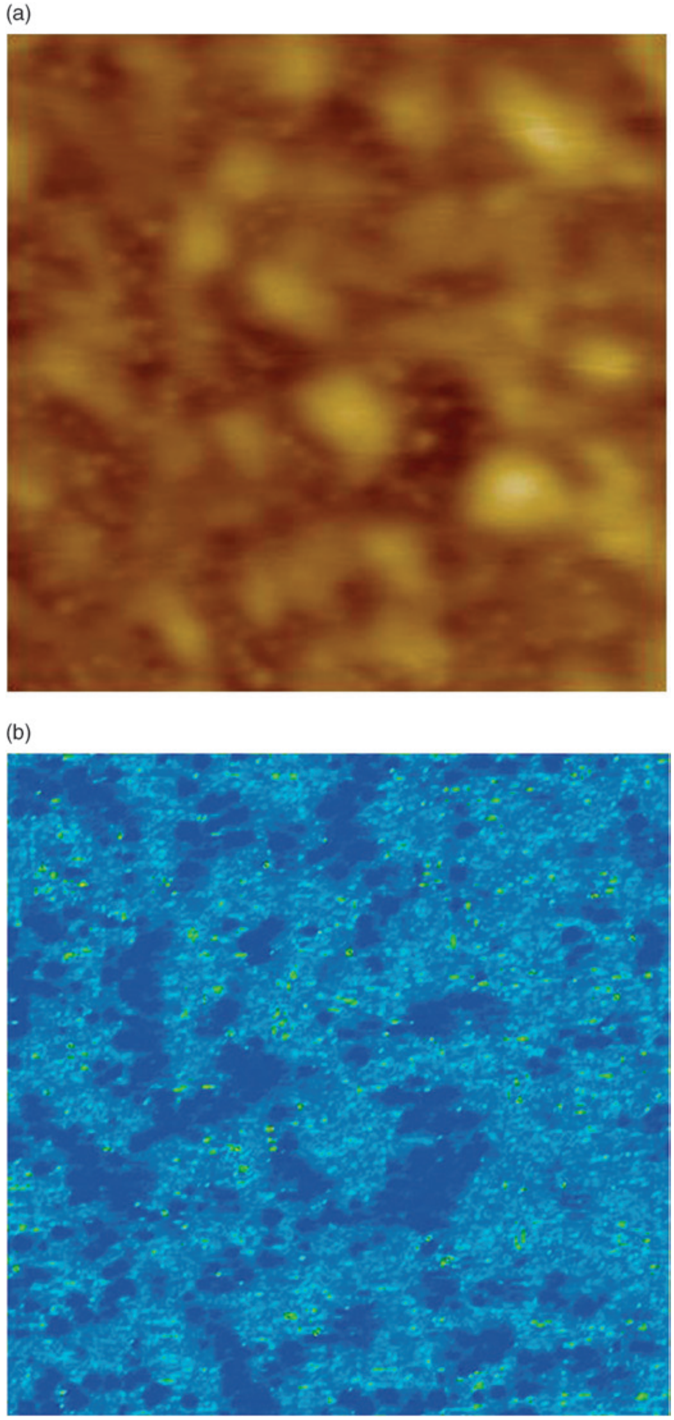

Figure 4: Topography (a) and resistance (b) data using Soft ResiScope mode for an organic solar cell sample of poly(3-hexylthiophene). Image width = 3 μm.

10^6 to 10^{12} ohms. These images were acquired using a doped diamond probe with a sample bias of -1.0 V. The regions in yellow and green have lower resistivity and correspond to the n-regions that are rich in electrons and have greater conductivity. The p-regions bordering n-regions have higher resistance, as indicated by the discrete blue regions in the resistance image.

Multiple imaging modes. An advantage of using the ResiScope module with the Nano-Observer AFM is the ability to obtain data from multiple modes at the same sample

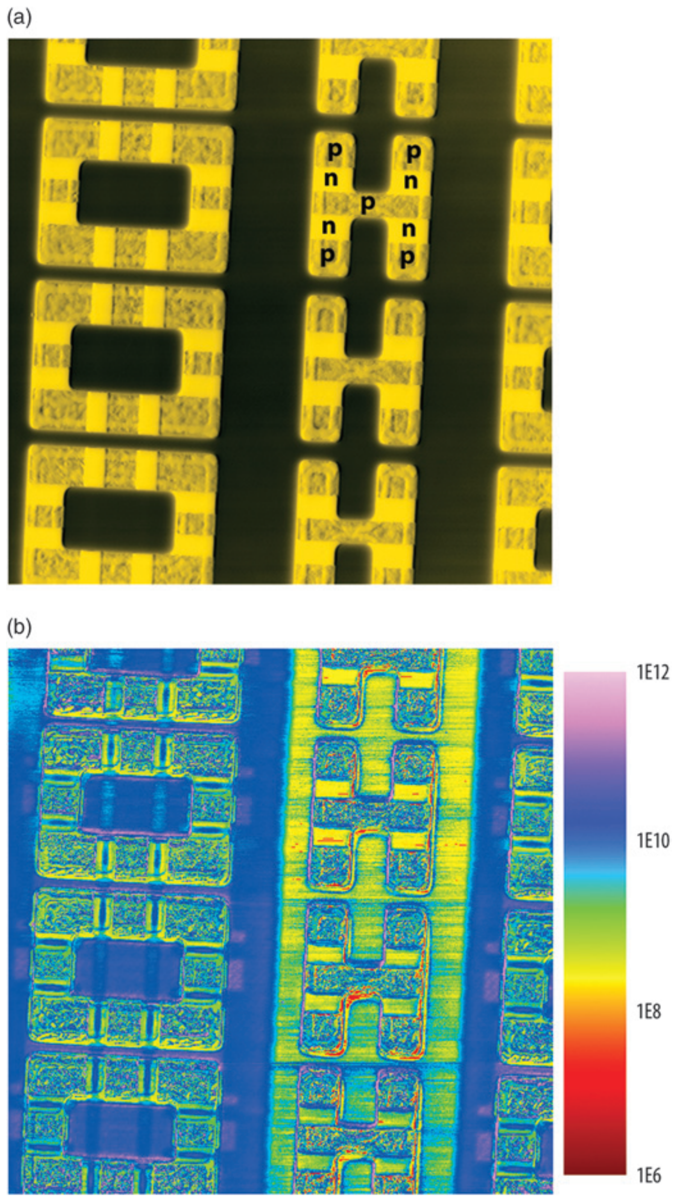

Figure 5: Topography (a) and resistance (b) data for an SRAM sample. Some of the n-doped regions and p-doped regions are labeled in the topography image. These correspond to the clear delineation between the n- and p-areas in the resistance image. Image width = 30 μm.

location. Many AFM systems cannot image the same location using as many imaging modes as the Nano-Observer AFM. For the Nano-Observer AFM, the tip holder can be used for multiple modes and the controller can switch between techniques, providing data for different properties at the same sample location. This is illustrated by the example in Figure 6 of a standard RAM sample. Oscillating, KFM, and ResiScope resistance modes were applied consecutively. Using KFM, the surface potential can be mapped for a sample and compared to resistance data from the ResiScope. The areas of greater conductivity (dark areas in the resistance image) have a lower surface potential. This is another illustration of how the material properties do not correspond to the topology of a sample. For the example in Figure 6, the KFM data were acquired in a dual pass mode.

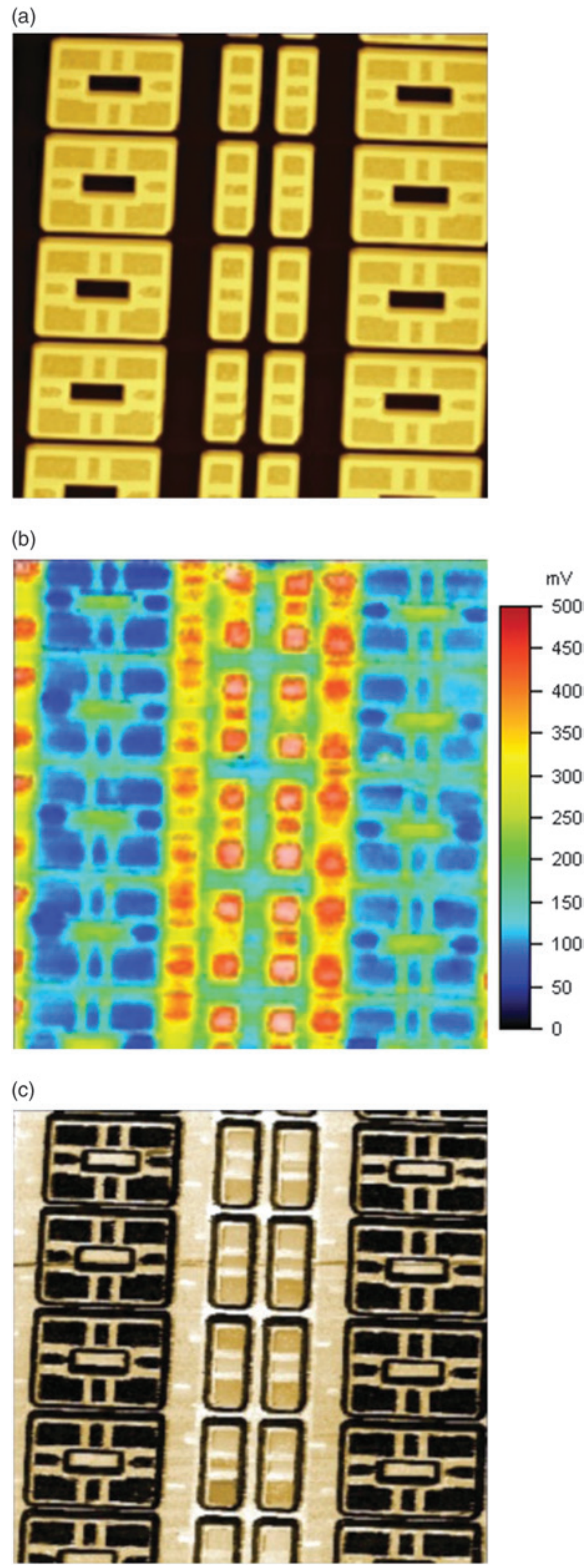

Figure 6: Topography (oscillating mode) (a), KFM surface potential (b), and ResiScope mode (c) data acquired simultaneously for a RAM chip sample. Image width = 50 μm.

Other modes such as EFM or MFM can be measured consecutively with the ResiScope mode as needed.

Discussion

The ResiScope mode for AFM resistance measurements provides an unprecedented range, spanning ten decades from 10^2 to 10^{12} ohms. Because of the system configuration, the current between the sample and probe is limited, minimizing current leakage and local oxidation. Typically conductive AFM techniques only use amplifiers to measure a current with a constant bias applied. The ResiScope module is configured to auto-calibrate when the system is started, using internal precision resistors as references. No other system has this capability.

The Soft ResiScope mode is the only commercial system providing quantitative electrical characterization on soft or compliant samples. Other systems would require a higher force and cause damage. In the Soft ResiScope mode, the tip is held at a point long enough to make an electrical measurement so that the data is quantitative. It is also semi-oscillating so the sample is not damaged. Using the Nano-Observer AFM with the ResiScope module, data is acquired with a constant and controlled force while taking quantitative resistance and current measurements.

Conclusions

Quantitative resistance and current data can be measured using a ResiScope module with a Nano-Observer AFM. The ResiScope overcomes previous limitations of electrical AFM measurements including friction interference, lack of quantitative measurements, difficulties measuring soft materials, and limited resistance range. Because the current between the sample and probe is limited, the conductive probe is protected from high-current damage. Soft or compliant samples cannot be imaged successfully in contact mode, whereas the Soft ResiScope mode for electrical characterization can image these materials. Hard samples were imaged to confirm that data from the standard and Soft ResiScope modes are equivalent. A soft polymer was imaged and measured to illustrate the effectiveness of the Soft ResiScope mode. It is also possible to take multiple measurements simultaneously or consecutively when the ResiScope and Nano-Observer AFM are used in combination. Analyses of multiple measurements provide an excellent illustration of why there are different AFM imaging modes. Topography imaging alone does not always provide the answers that researchers need, and the data often do not correlate to the material properties. For these reasons, advanced imaging and Soft ResiScope modes have been developed to provide quantitative data on a variety of surfaces.

Acknowledgements

Thanks for fruitful discussions and data from Louis Pacheco at Concept Scientific Instruments.

References

[1] G Binnig et al., *Phys Rev Letters* 56 (1986) 930–33.
[2] RC Barrett and CF Quate, *J Appl Phys* 70(5) (1991) 2725–33.
[3] U Hartmann, *Journal of Magnetism and Magnetic Materials* 157 (1996) 545–49.
[4] M Nonnenmacher et al., *Appl Phys Lett* 58 (1991) 2921–23.
[5] S Gunes et al., *Chem Rev* 107 (2007) 1324–38.

MT