

New Techniques for Nanoscale Electrical Contact Resistance Measurements

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Nanoscale Electrical Properties

Discovering the electrical properties of nanoscale materials often involves a combination of probing and microscopy to definitively execute the measurement at the position of interest. However, an additional variable that must be considered is the effect of the applied probe force on the testing results, as many materials exhibit a pressure dependence that can cause a drastic change in the material's electrical characteristics.

Now, a novel measurement technique reveals electrical and mechanical properties as a function of applied probe force, revealing nanoscale phenomena not previously seen. This nanoscale electrical contact resistance measurement tool (nanoECR[™] by Hysitron Inc., Minneapolis, MN) can simultaneously obtain in-situ electrical and mechanical property measurements under highly controlled load or displacement contact conditions. The technique provides a time- based correlation of multiple measurements, including force, displacement, current, and voltage to greatly augment the wealth of information one can obtain from traditional nanoscale probe measurements. Such measurements are the basis for extracting multiple parameters from many classes of nanoscale material volumes and devices.

The discovery, understanding, and control of the unique properties present at the nanoscale are some of the primary research interests in modern day science. The correlation between the mechanical properties, electrical properties, and deformation behavior is of paramount importance in designing next- generation materials and devices. The nanoECR system facilitates research in these areas and can be used to investigate pressure induced phase transformations, diode behavior, tunneling effects, piezoelectric response, etc. of nanoscale volumes of material.

New Measurement Methodology

The diversity of nanotech applications presents a unique set of challenges in the coupling of mechanical and electrical measurements while achieving high levels of accuracy, repeatability, and probe positioning. Depending on the probe/sample contact conditions, current levels may range from pA to mA, voltages from μ V to V, applied probe forces from nN to mN, and probe displacements from Å to μ m. Additionally, the unique geometries and size of nanoscale contacts present numerous technical difficulties.

For these reasons, a system was developed that integrates a Hysitron TriboIndenterTM nanomechanical test instrument with a Model 2602 Dual Channel SourceMeter[®] instrument (Keithley Instruments Inc, Cleveland OH). The system also includes a conductive sample stage, patented capacitive (nanoECR) transducer, and a conductive indenter probe (see *Figure 1*). The transducer maximizes test accuracy and repeatability by allowing current flow without the need to attach an external wire to the probe. This "through



Figure 1. Block diagram of a test system for simultaneous measurement of mechanical and electrical properties of nanoscale materials and devices.

tip" measurement assures a secure contact and helps reduce potential sources of error.

The system also includes a fully integrated data acquisition system allowing for a realtime correlation between force-displacement and current-voltage measurements. Auxiliary instruments can be connected to this acquisition system for the real-time measurement and extraction of other desired parameters. The user interface allows for easy setup of all test variables under a wide range of load and displacement controlled conditions. This is facilitated by the SourceMeter instrument's on-board Test Script Processor, which can automatically run test sequences, provide synchronization for other hardware components, and minimize timing/control issues between various parts of the system.

System Operation

During a test, the probe is pushed into the sample surface while the displacement is continuously monitored. The sample's hardness and elastic modulus can be immediately calculated using the force and displacement data. For electrical parameters, the Keithley SourceMeter instrument applies a bias voltage to the conductive stage, to which the device under test (DUT) is electrically coupled. As the conductive indenter probe penetrates into the material, current, voltage, force, and displacement are measured continuously.

The force actuation/displacement sensing of the probe is obtained through an electrostatically actuated transducer and provides extremely low measurement noise and a high degree of sensitivity. The transducer/probe combination is mounted to a piezoelectric positioning system that allows for scanning probe microscopy (SPM) imaging of the sample topography and extremely accurate test positioning.

In a typical measurement, one channel of the SourceMeter instrument performs the source and measure operations while the other is utilized as a current to voltage amplifier, transmitting current data to the control computer. The control software is highly flexible, allowing a user to specify and measure the magnitude of the sourced current and voltage, and perform I-V sweeps over predefined force or displacement points. The user controls all SourceMeter instrument functions through the nanoECR software



Figure 2. Mechanical (force- displacement) and electrical (current- displacement) curves showing pressure induced phase transformations occurring during the nano-deformation of P-type silicon.

interface, which eliminates the need to manually change parameters on the meter itself. The software's flexibility and automated test routines allow hands-free operation and testing of even the most challenging samples. Test time is highly dependent on user defined variables, but a typical test sequence takes approximately one minute.

The Hysitron nanoECR system resolution, accuracy, and noise specifications are:

- Force Resolution: 1nN
- Force Noise Floor: 100nN
- Displacement Resolution: 0.04nm
- Displacement Noise Floor: 0.2nm
- Current Resolution: 5pA
- Current Noise Floor: 12pA
- Voltage Resolution: 5µV
- X-Y Positioning Accuracy: 10nm

Silicon Phase Change Example

Silicon is a well studied example of a material that undergoes pressure- induced phase transformations during probing (see references). As the probe force increases/ decreases during the probe loading/unloading cycle, a series of phase transformations occur in the nano-deformed region under the acting probe. During loading of the probe, Si-I (diamond cubic crystal structure) transitions to Si-II (metallic β -Sn) at an applied pressure of approximately 11-12 GPa. As the probe/ sample contact pressure is decreased during unloading, additional transformations occur from the Si-II to Si-III/XII crystal structures.

The applied force and measured current vs. probe displacement plots are shown in *Figure 2*. Upon loading into the silicon surface the force- displacement plot shows a relatively continuous curve, while the current-displacement plot exhibits a discontinuity at approximately 22nm of probe displacement, indicating the Si-I to Si-II phase transformation. During gradual unloading, the Si-II to Si-III/XII transformation is evident in both the force-displacement and current- displacement measurement. These transformations occur rather suddenly and are referred to pop- in and pop- out events, which are labeled in *Figure 2*.

The rate of probe loading/unloading can also impact the electrical characteristics of the material. For example, fast unloading from maximum load in silicon will form -Si and exhibit a completely different electrical signature. These types of measurements are critical in such applications as silicon-based MEMS and NEMS devices. In these devices, small forces on small structures can translate into large pressures and microstructural changes within the material, which can determine electrical and mechanical properties.

Conclusions

Successful development and implementation of nanoscale materials and devices depends heavily on the ability to quantitatively assess and tailor their electrical and mechanical properties. The nanoECR system provides a straightforward, convenient, and quantitative technique that enables researchers to discover material properties/behavior not possible through conventional means. In addition to silicon, this research tool can be

applied to study metallic glasses, piezoelectric films, organic LEDs, ITO thin films in solar cells and LCDs, plus a wide variety of other nanoscale solids. New insights can be gained in the areas of thin film fracture, dislocation nucleation, deformation transients, contact resistance, fatigue, diode behavior, tunneling effects, piezoelectric response, and more. **Remuter**

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