Nanoscale Device & Material Electrical Measurements

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Electrical measurements on nanoscopic materials and devices are essential for the development of practical products, even those not intended for electronic applications. Using the right instruments and techniques can shorten test times and help assure collection of useful data.

ELECTRICAL measurements on nanoscale materials and devices reveal not only electronic characteristics, but also general properties like a nanoscopic particle's density of states. These fundamental properties can be used to predict and manipulate physical characteristics, such as tensile strength, color, and thermal conductivity. However, making meaningful measurements requires highly sensitive instruments and sophisticated probing techniques. Instrumentation designed specifically for nanotechnology research is increasing, but users must understand the types of measurement needed, and test system features that facilitate speed and accuracy.

Nanoparticle Characterization Methodologies

As a result of small particle sizes, the atoms and molecules of nanoscale materials often bond differently than they do in bulk substances. While the discovery of bulk properties remains important, measurements are needed to uncover quantum mechanic characteristics that are unique to nanoscale structures.

Particle size and structure have a major influence on the type of measurement technique used to investigate a material. Optical microscopic techniques have limited value for nanoscale materials. As particles shrink below micrometer sizes (referred to as mesoscopic), visual characterization can be done with a scanning electron microscope (SEM). For nanoscopic materials (particle sizes below100 nanometers), a scanning tunneling microscope (STM) can be used. Even smaller particles can be investigated with an Atomic Force Microscope (AFM).

When a particle has nanoscale dimensions, its physical behavior is fundamentally different from the bulk material. This dictates the use of non-visual measurement methodologies to uncover unique chemical and electrical properties. For many of these properties, the actual electrical quantity being measured is a low level current or voltage that is translated to another physical quantity. Direct electrical measurements are possible on many nanoscopic substances with probing instruments and nano-manipulators now available.

Electrical Property Measurements

If a particle becomes small enough, its physical size may approach the wavelength of the material's electrons. Because of quantum mechanical effects, the energy of its electrons cannot be predicted by the bonding normally associated with the bulk material.

For bulk macroscopic materials, electrons have thermal energies that lie within continuous energy bands. For nanoscale particles, the allowable energies within continuous bands can separate into discrete levels when the separation between levels approaches the thermal energy of the electrons. As this happens, the density of states of the material changes. The density of states is a measure of the number of energy options available to an electron as it falls into a lower energy level by giving up energy, or as it ascends to a higher energy level after absorbing energy. Since the density of states can be used to manipulate material properties, its characterization is a fundamental research activity.

Electron energy effects can be deduced from electrical measurements. One example is when a nanoscale substance is involved in an oxidation-reduction (REDOX) reaction, such as the chemical-electrical conversion

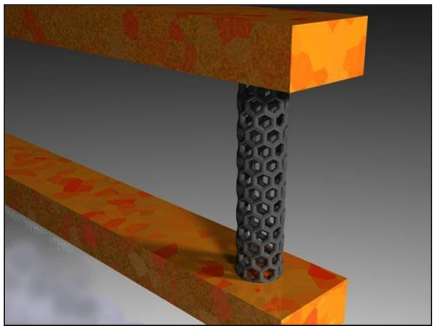


Figure 1. Representation of a carbon nanotube. These structures exhibit a wide range of characteristics, giving them unique properties that are useful in many types of electronic and physical structures.

that takes place in fuel cells or batteries. Electrical measurements of the number of electrons transferred from one species to another determine the reaction rate by tracking electrical current and potential with time. These measurements can be used to infer particle size, density of states, and other nanoscopic properties.

One of the important properties is the mean free path of an electron (distance traveled before it bumps into another atom), which approaches the same order of magnitude as a nanoscopic particle diameter. This characteristic affects the material's bandgap and DC resistance. More generally, it determines whether a particle is a conductor (bandgap < thermal energy of the electron), an insulator (bandgap > thermal energy of the electron), or a semiconductor. Furthermore, this characteristic can be altered dynamically.

An example of this is found in carbon nanotubes (CNTs). (See ***Figure 1***.) Typically, when CNTs are made, both conducting and semi conducting forms occur. When the two forms are separated, the conducting nanotubes can be used, for instance, as field emission display emitters. Semiconducting nanotubes can be used to make transistor switches. This is illustrated in ***Figure 2a***, where a semi-conducting CNT is connected between two electrodes that function as a drain and source. A third electrode, an insulated gate (***Figure 2b***), is placed directly under the entire length of the CNT channel. The introduction of an electric field through the channel (by increasing the voltage on the gate) can change the CNT from its semi-conducting state to its insulating state. Decreasing the gate voltage will transition the device into a conducting state.

(If a suitable amount of energy is absorbed (> bandgap) then electrons can jump from the valence band into the conduction band.)

Density of States Measurements

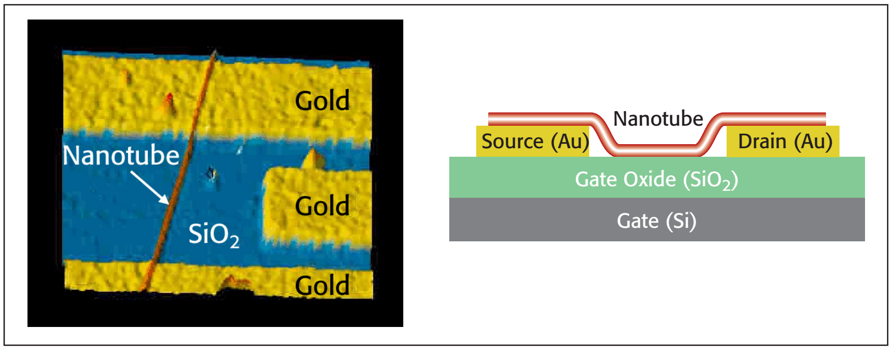
Density of states corresponds to the density of a material's energy levels. Highly conductive materials possess a greater density of states because of an abundance of free energy levels in the conduction band (i.e., more allowed energy levels per unit of energy). Insulating materials have an electronic structure with a scarcity of energy levels in the conduction band.

The three dimensional density of states as a function of energy can be expressed as:

$$\rho(E) = \frac{dn_S}{dE} = \frac{4\pi(2m)^{3/2}}{h^3}\sqrt{E}$$

In this equation the quantity $\rho(E)$ is expressed as the derivative of ns, the density of states per unit volume with respect to energy, E. Thus, $\rho(E)$ represents the number of electron states per unit volume per unit energy at energy E (electron orbital location expressed in electron volts). In the expanded equation, m represents the effective mass of the particle, and h is the Plank constant.

While the result is independent of volume (can be applied to any size particle), this equation is of limited value if the particle size/structure is unknown. However, there are other ways to determine density of states experimentally. X-ray spectroscopy is frequently used, but a material's density of states can also be deduced from electrical impedance and conductance measurements. Prior art has used a scanning tunneling microscope (STM) to tunnel a current through a nanoscopic device. The density of states is found through differential conductance measurements.



(a) *(b)*

Figure 2. A CNT being used to create a new type of transistor switch. (Courtesy of IBM Corporation)

Differential conductance is simply (di/dv)/(i/v). The quiescent current vs. voltage (I-V) characteristics are established through the STM's high resistance contact, with a low level AC modulation on top of the quiescent operating point to measure di/dv. This is divided by the quiescent conductance, I/V, and plotted against applied voltage.

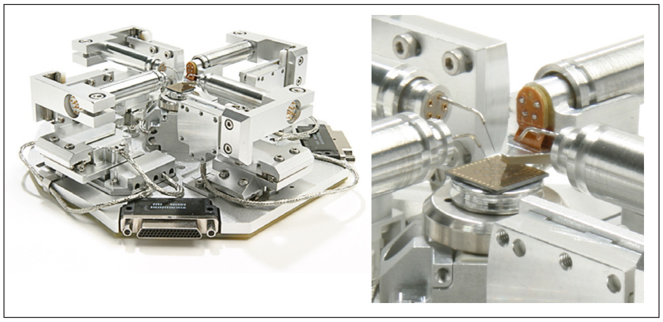
Other Means of Direct Electrical Measurements

For reasons of cost, convenience, and speed, alternatives to the STM are desirable for direct electrical measurements. An STM and its high resistance contact can be replaced with a nano-manipulator that creates a low resistance contact to the nanoparticle. Nanomanipulators, such as the one shown in ***Figure 3a***, have as many as four positioners that grasp, move, and optimally position a nanoscale sample along four axes. This permits simultaneous manipulation, imaging and electrical probing of the sample (***Figure 3b***).

Because of the complexity involved in connecting individual instruments to nanomanipulators, it is best to use an integrated source-measure system with a suitable interface and application software. The source-measure units (SMUs) in these systems have the added advantage of being able to dynamically alter their measurement mode to adapt to the impedance state of a nanoscale material, which can range from highly conductive to highly resistive in the case of CNTs.

There are two possible measurement modes for an SMU: source current/measure voltage, or source voltage/measure current. When considering the measurement of low impedance materials and devices (less than 1000 ohms), the source current/measure voltage technique generally yields the best results. When measuring high impedance (greater than 100,000 ohms), the source voltage/measure current technique is best. The SMU can switch modes automatically as a material's conductive state changes, and measurement resolution can be as good as femtoamps and nanovolts.

Specialized SMU systems are available with software written specifically for nanoscale testing. This shortcuts many measurement tasks by providing common routines for collecting electrical data on a nanotech device, such as a CNT, bio-device, molecular



(a) *(b)*

Figure 3. Nanomanipulator for conducting direct electrical measurements on nanoscale structures. (Courtesy of Zyvex Corporation)

electronic component, or a nanowire. Typically, these routines take measurements, plot I-V curves, and have the ability to make differential conductance measurements for determining density of states.

Connection and Accuracy Issues

A major issue in nanoscale electrical measurements is making reliable connections at the right location. At the nanoscopic level, it may be necessary to connect the device under test (DUT) back to pads that can be reliably probed. One example of this is particle self-assembly from silicon to silicon, where conventional photolithographic techniques are used to make electrical connection pads for probing. Particles that are long enough to straddle such pads (for example, carbon nanowires) can be connected to the pads through externally generated electrostatic fields.

In any case, connections to the DUT must not affect measurement accuracy. This is particularly important in low resistance measurements on nanowires and sheet resistivity measurements on films. Typically, low resistance measurements require a four-point probe (Kelvin) technique to eliminate the effects of lead resistance and ensure accuracy. The two most commonly used four-point techniques for sheet resistivity are the collinear probe method and the van der Pauw method [1]. SMU-based test systems may include these test routines and associated calculations in their application software.

Test signal integrity depends on a high quality probe contact, which is directly related to contact resistance. During the course of their use, probe needles wear and may become contaminated, resulting in increased contact resistance and measurement errors. The best way to enhance long-term performance of probe tips is to incorporate periodic cleaning procedures in the test protocol. Some automated test systems have software that includes probe maintenance routines.

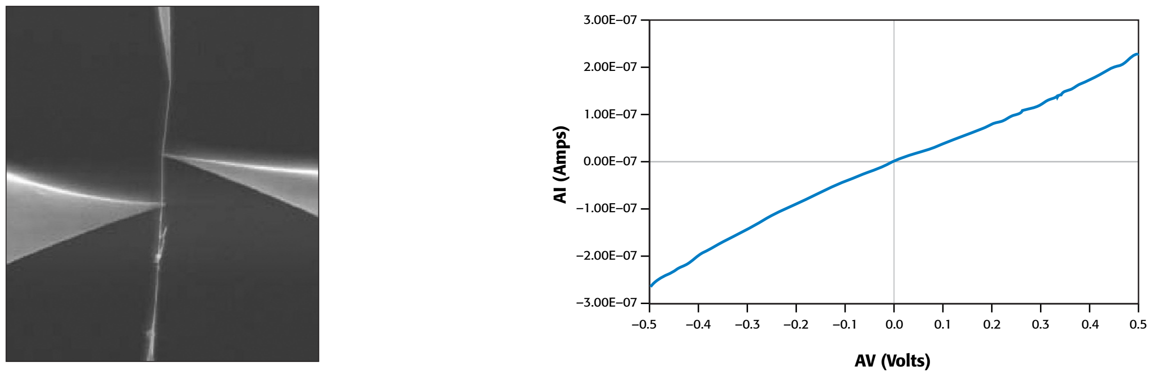
Probing any nanomaterial or device requires care to avoid non-ohmic contacts. Non-ohmic contacts create a potential difference that is not linearly proportional to the current flowing through them. A typical method for determining ohmic contact on the DUT is to perform an I-V sweep with the SMU and verifying that it crosses through zero. If the IV curve does not cross through zero, then ohmic contact is highly unlikely. Another method is to change measurement ranges. Changing ranges, especially when measuring resistance, can change the test currents. Ohmic contact would be indicative of the same reading but with higher or lower resolution depending on whether the range went up or down. Different readings on different ranges may indicate non-ohmic contact.

Figure 4a illustrates a nanomanipulator making a four-wire connection to a CNT ‘wire’. Upper and lower probes are used to inject a current through the CNT, while the left and right probes measure voltage across a segment of it. Note that the resulting I-V sweep (***Figure 4b***) does cross zero, indicating ohmic contact.

Another source of error is self-heating due to excessive electrical current through the DUT. Such currents may even lead to catastrophic failure of the sample. Therefore, instrumentation must automatically limit source current during device testing. Programmable current and voltage compliance circuits are a standard feature of most SMUs. In some systems, pulsed current sources are available, which may be required to avoid self-heating of some low resistance structures.

For high resistance applications, the DUT stimulus typically is a voltage, and the response current is measured, which can be as low as a few femtoamps. Therefore, instrumentation must provide this level of sensitivity and adequate resolution.

Regardless of measurement mode, external sources of error must be minimized. These errors can arise from stray magnetic fields, electrostatic charges, cable connections, thermoelectric EMFs, and currents generated by triboelectric and electrochemical effects. To protect nanoscale samples from electrostatic charge and magnetic fields, as well as maintaining the integrity of the measurement, a



(a) *(b)*

Figure 4: (a) Nanomanipulator Kelvin connections to a CNT. (b) Ohmic contact to the CNT is indicated by the I-V sweep crossing zero.

Faraday cage can be used. Guarded cable connections help eliminate parasitic capacitance and cable leakage effects. There are many good references available on how to minimize external measurement errors [2].

Errors Arising from Test Procedures and Programming

Since electrical characterization of a nanoscale structure is essentially a source-measure procedure, signal timing is important. There may be a significant amount of capacitance associated with the DUT and its test connections, including external cables. This could lead to an extended settling time in the DUT's response to an applied stimulus, particularly in high resistance materials. If the DUT response measurement is made too soon after the source signal is applied, the response may not have settled to a stable value (i.e., the quantity measured is wrong).

To avoid such errors, preliminary measurements on the DUT to establish the test system settling time should be taken (***Figure 5***). The settling time is then used to program the SMU test system with an adequate delay between the end of the source application and the response measurement.

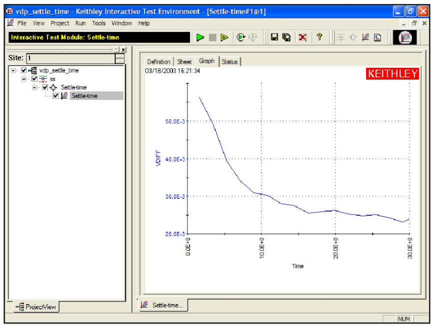


Figure 5. Settling time of an SMU-based test system performing four-point van der Pauw resistivity measurements.

In a similar vein, SMU mode switching must be considered when making measurements on a nanoscale device that changes state during the course of a test. As noted earlier, structures can rapidly change from being an insulator to become a conductor with the application of an electric field. An SMU can automatically alter its measurement mode when it detects this change in the DUT, but the mode switching is not instantaneous. Depending on the SMU model, the switching time can range from 100ns to 100µs. Although such switching speeds are not fast enough to track a nanoparticle as it changes state, the time is short enough to allow accurate measurements of both states while limiting DUT power dissipation to acceptable levels.

In general, an overall test objective is to minimize measurement noise while maximizing measurement speed and accuracy. To accurately characterize the impedance of a nanoscale material, the instrumentation and measurement techniques must allow for an appropriate sample rate. Furthermore, the measuring instrument must have a stable time base in order to compute the impedance mathematically. Ease of use and programming (or no programming at all) are also important considerations in the selection of a nanoscale test system.

References

[1]. Keithley Application Note #2475, "Four-Probe Resistivity and Hall Voltage Measurements with the Model 4200-SCS."

[2]. *Low Level Measurements Handbook*, 6th Edition, 2004, Keithley Instruments, Inc., Solon, OH.

About the Author

Jonathan Tucker is the Lead Industry Consultant for Nanotechnology at Keithley Instruments in Cleveland, Ohio. He is currently involved with measurement solution business development for nanotechnology applications requiring electrical characterization. He is also responsible for new test & measurement application development in the Research & Education market segment. Jonathan has over 18 years of experience in Test & Measurement since receiving his Bachelors of Electrical Engineering from Cleveland State University and his MBA from Kent State University.

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