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Techniques for Accurate Nanotech Electrical Measurements

Jonathan Tucker, Keithley Instruments, Inc.

The science of nanotechnology, by its extended variety of applications, is driving researchers to develop new materials and components using carbon nanotubes, chemical molecules, quantum dots, and even polymers. Characterizing these nanoscale components and materials is far from trivial as many exhibit low current, low resistance, high resistance, and low power electrical properties.

This article will offer insight into electrical measurements required for nanotechnology, with examples given for carbon nanotube-based materials and electronics, molecular electronics, and materials. The sources of measurement uncertainty that affect such sensitive measurements will be discussed, together with a discussion of available test equipment solutions.

THE science of nanotechnology is pulling in researchers from many different disciplines from electronics to chemistry to biology. In each discipline, a variety of potential applications and products are being researched and developed that can have a significant impact on a large number of industries. Anything from sensors, drug delivery systems, stronger, lighter materials, faster and smaller electronic components, to more efficient energy systems will be the new innovations from nanotechnology research.

To meet the challenges of nanoscience, researchers must conduct a variety of measurements, including current versus voltage (I-V) characterization, resistance, resistivity and conductivity, transport, and optical spectrum and energy measurements to unravel the complexities of matter at the nanoscale and to make reliable electronic devices based on nanomaterials. To reach this end, nano researchers require sensitive electrical measurement tools and an understanding of electrical measurement principles.

There are a number of nanoscale electrical measurement challenges that the researcher must understand before they can attempt to make sensitive measurements. These are:

- Measurement fundamentals
- Learning curves
- Sensitivity and resolution
- Sources of generated error
- Cables, connection, & probes
- Metrology standards

We will discuss these in more detail and then examine some real applications where the principles apply.

Measurement Fundamentals

Electrical measurements—volts, ohms, and amps—on nanoscale components present unique difficulties, with their own potential for error. Working with materials and components on a quantum scale puts limits on what one can measure electrically. The theoretical limit of sensitivity in any measurement is determined by the noise generated by the resistances present in the circuit. Voltage noise is proportional to the square root of the resistance, bandwidth, and absolute temperature. High source resistances limit the theoretical sensitivity of the voltage measurement. While it is certainly possible to measure a 1 microvolt signal that has a 1 ohm source resistance, it is not possible to measure that same 1 microvolt signal level from a 1 teraohm source. Even with a much lower 1 megohm source resistance, a 1 microvolt measurement is near theoretical limits, and it would thus be very difficult to make using an ordinary digital multimeter (DMM).

In addition to having insufficient voltage or current sensitivity, many DMMs have high input offset currents when measuring voltage, and lower input resistance compared to more sensitive instruments meant for low-level DC measurements that are often required for nanotechnology. These characteristics add more noise to the measurement, disturb the circuit unnecessarily, and cause errors in the measurement.

Once the system is assembled its performance must be verified and potential sources of error eliminated. Errors sources can

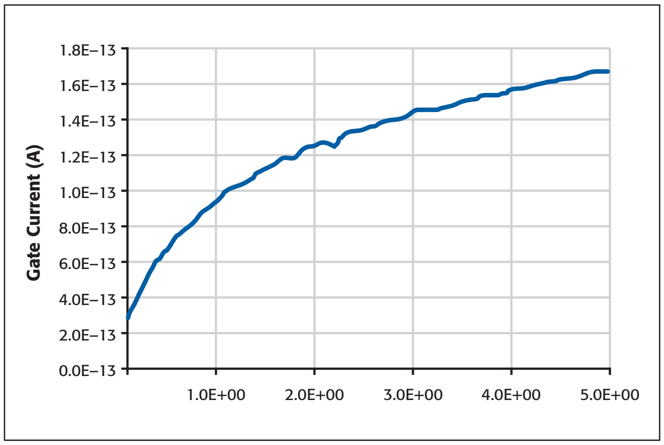

Figure 1: Gate leakage current in a nanoscale MOSFET ranges from 30 femtoamps to about 170 femtoamps.

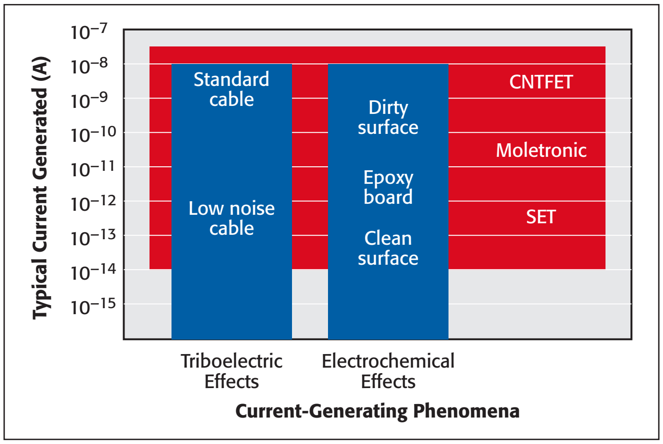

Figure 2: Triboelectric currents are generated by charges created between a conductor and an insulator due to friction. Free electrons rub off the conductor and create a charge imbalance that causes the current flow, as for example when insulators and conductors rub together in a coaxial cable. Error currents also arise from electrochemical effects when ionic chemicals create weak batteries between two conductors. Etching solution, flux or other contamination on a PC board can generate currents of a few nanoamps between conductors.

include cables, connections, probes, contamination, and thermals. Ways to reduce some of these errors will be discussed in the next sections.

Safety must be an ever-present concern Electrical measurement tools can source dangerous or lethal voltage and currents. Knowing when these situations can occur with the instrument is important so one can take the proper safety precautions. Read and follow the safety instructions provided with the tools.

Many of the measurement fundamentals and concepts for making sensitive electrical measurements are discussed in Keithley's *Low Level Measurement Handbook*, which can be ordered at no charge at www.keithley.com/handbooks or downloaded in PDF form at www.keithley.com/servlet/Data?id=9538.

Learning Curves

Programming chores and arcane details of instrument operations can be a distraction to busy researchers. Many electrical characterization tools are very complex, and their data transfer mechanisms are tedious and require extensive amounts of storage media. Graphical analysis takes too long. Learning and programming steals time better devoted to research.

User-friendly instruments are important, both to researchers and to the design engineers and manufacturing specialists who take new discoveries and convert them into practical products. State-of-the-art electrical characterization systems should be PC-based with the familiar point-and-click, cut-and-paste, and drag-and-drop features of the Windows™ operating system. These system features make test setup, execution, and analysis more time efficient by shortening the learning curve.

Sensitivity/resolution

The third measurement challenge is sensitivity and resolution. The sensitivity of an instrument is normally characterized by its lowest range divided by the resolution. Resolution is the smallest portion of the signal that can be observed. The importance of this is apparent in the graph in ***Figure 1***, a measurement of gate leakage current from a nanoscale metal-oxide semiconductor field effect transistor (MOSFET). The current measured ranges from 30 femtoamps to about 170 femtoamps. This measurement requires a current sensitivity on the order of 100aA (100E-18 amps). In all cases the level of sensitivity required depends on the application.

Sources of Generated Current Errors

Making low current measurements requires an understanding of the potential sources of error that could result in unwanted measurement errors. Two very common sources of error that can affect measurements on a variety of nanoelectronic devices are triboelectric effects and electrochemical effects (***Figure 2***).

Triboelectric currents are generated by charges created between a conductor and an insulator due to friction. Free electrons rub off the conductor and create a charge imbalance that causes the current flow. A typical example would be electrical currents generated by insulators and conductors rubbing together in a coaxial cable, as shown in the diagram. "Low-noise" cable greatly reduces this effect. It typically uses an inner insulator of polyethylene coated with graphite underneath the outer shield. The graphite provides lubrication and a conducting equipotential cylinder to equalize charges and minimize charge generated by frictional effects of cable movement. However, even low-noise cable creates some noise when subjected to vibration and expansion or contraction, so all connections should be kept short, away from temperature changes (which would create thermal expansion forces), and preferably supported by taping or tying the cable to a non-vibrating surface such as a wall, bench, or rigid structure. Other solutions to movement and vibration problems include:

- Removal or mechanical decoupling of the source of vibration. Motors, pumps, and other electromechanical devices are the usual sources.
- Stabilization of the test hookup. Securely mount or tie down

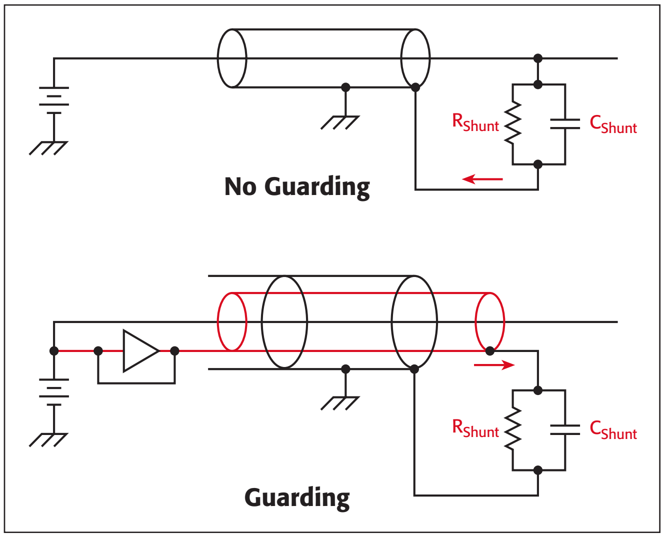

Figure 3: Currents generated by triboelectric and electrochemical effects can be well within the range needed to characterize a device such as a carbon nanotube FET, or a molecular electronic component, or even a single electron transistor SET.

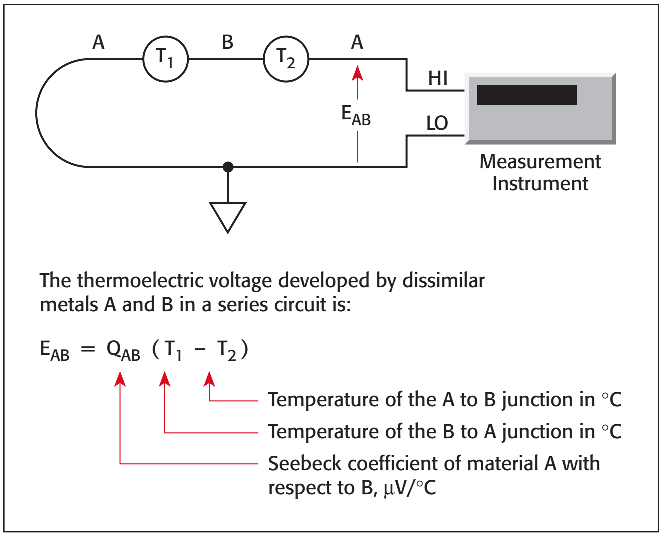

Figure 4: The shunt resistance and capacitance path across the inner conductor and the shield of a coaxial cable will allow leakage currents to flow. In addition, the shunt R and shunt C form an RC circuit that can greatly slow down a low current or high resistance measurement.

electronic components, wires, and cables. Shielding should be sturdy.

Triboelectric effects can also occur in other insulators and conductors that touch each other. Therefore, it is important to minimize contact between insulators as well as conductors in constructing test fixtures and connections for low current and high impedance.

Error currents also arise from electrochemical effects when ionic chemicals create weak batteries between two conductors. For example, commonly used epoxy printed circuit boards, when not thoroughly cleaned of etching solution, flux or other contamination can generate currents of a few nanoamps between conductors. Insulation resistance can be dramatically reduced by high humidity or ionic contamination.

To avoid the effects of contamination and humidity, select insulators that resist water absorption, and keep humidity to moderate levels. Also be sure that all insulators are kept clean and free of contamination. If insulators become contaminated, a cleaning agent such as methanol should be applied to all interconnecting circuitry. It is important to flush away all contaminants once dissolved in the solvent so they are not re-deposited. Use only very pure solvents for cleaning; lower grades may contain contaminants that leave an electrochemical film.

Figure 3 illustrates the ranges for which these two sources of error can generate unwanted currents. Each of these unwanted currents can be well within the range needed to characterize a device such as a carbon nanotube FET, or a molecular electronic component, or even a single electron transistor (SET).

The Importance of Guarding

Improper use of cables can result in very long measurement periods. A coaxial cable provides an inner signal carrying conductor and a shield. Across the inner conductor and the shield is a shunt resistance and capacitance path that will allow leakage currents to flow (***Figure 4***). In addition to a path for leakage current to flow, the shunt R and shunt C form an RC circuit that can greatly slow down a low current or high resistance measurement, and can mean a wait of five RC time constants to achieve an accurate reading. Measuring very high resistances—gigaohms and higher—can take seconds to minutes before the reading will settle to within 1% of the final value.

We suggest the use of triaxial cable and guarding to eliminate leakage paths and the settling time issues. In the second configuration in ***Figure 4*** the cable is made up of an inner conductor, an inner shield, and an outer shield. The loading effects of cable resistance (and other leakage resistances) can be virtually eliminated by driving the cable inner shield with a unity-gain amplifier. Since the voltage difference between the inner conductor and the inner shield is now essentially zero, all the test current now flows through the inner conductor and on to the measurement instrument's input. The leakage current flowing through the inner shield-to-ground leakage path may be considerable, but that current is supplied by the low-impedance output of the unity-gain amplifier rather than by the current source.

By definition, a guard is a low-impedance point in the circuit that is at nearly the same potential as the high-impedance input terminal. In modern electrometers, the preamplifier output terminal is such a point, and can be used to reduce the effect of cable leakage. An additional benefit is that the effective cable capacitance is also reduced, making the response speed of the circuit much faster, improving measurement time.

Temperature Effects

Thermoelectric voltages or EMFs are the most common source of errors in low-voltage measurements. These voltages are generated when different parts of a circuit are at different temperatures and when conductors made of dissimilar materials are joined together, as shown in ***Figure 5***. The Seebeck coefficients of various materials with respect to copper are summarized in the table.

Constructing circuits using the same material for all conductors minimizes thermoelectric EMF generation. For example, connections

Paired Materials	Seebeck Coefficient, Q_{AB}, microvolts/°C
Cu-Cu	<0.2
Cu-Au	0.3
Cu-Pb/Sn	1–3
Cu-Si	400
Cu-CuO	1000

Figure 5: Thermoelectric voltages are generated when different parts of a circuit are at different temperatures and when conductors made of dissimilar materials are joined together.

made by crimping copper sleeves or lugs on copper wires results in cold-welded copper-to-copper junctions that generate minimal thermoelectric EMFs. Also, connections must be kept clean and free of oxides. For example, clean Cu-Cu connections may have a Seebeck coefficient of ±0.2 microvolt/°C, while Cu-CuO connections may have a coefficient as high as 1mV/°C.

Minimizing temperature gradients within the circuit also reduces thermoelectric EMFs. A technique for minimizing such gradients is to place all junctions in close proximity to one another and to provide good thermal coupling to a common, massive heat sink. Electrical insulators having high thermal conductivity must be used, but, since most electrical insulators do not conduct heat well, special insulators such as hard anodized aluminum, beryllium oxide, specially filled epoxy resins, sapphire, or diamond must be used to couple junctions to the heat sink. Additionally, allowing test equipment to warm up and reach thermal equilibrium in a constant ambient temperature also minimizes thermoelectric EMF effects. Some instruments even provide built in measurement modes that alternate test signal polarity to cancel out so that the thermal EMFs.

Making Contact with the DUT

In order to make contact with a nanoscale device or component fixturing, microscopes, and probing systems are required. Nano researchers today are using tools like atomic force microscopes, scanning electron microscopes, and focused ion beam tools to visualize, to make mechanical measurements, and to perform I-V electrical characterization on their devices. Tools such as nanomanipulators are needed for micro- and nano research, development and even production applications. These systems can have as many as four positioners that grasp, move, test, and optimally position nanoscale samples with four axes of movement. This permits simultaneous manipulation, imaging and electrical testing of nanoscale experiments.

Unfortunately, probe tips and the probing system can be a source of measurement error that can be larger than the electrical measurement tools themselves. Test signal integrity depends on a high quality probe contact, which is directly related to contact resistance. Probe contact resistance has become increasingly important as signal voltages drop, contact pressures decrease, and new device technologies in nanotechnology are researched.

During use probe needles can become contaminated, resulting in measurement errors. Probe tip wear and the resulting contamination that builds up on the tip can cause an increase in contact resistance. The best way to enhance long-term performance of probe tips is to incorporate periodic cleaning procedures in the test protocol. While regularly scheduled cleaning removes contaminants before they cause test yield loss due to higher contact resistance, this gain must be weighed against its cost. One major cost element associated with cleaning is reduced test throughput while the probe system is out of service. On the other hand, too little cleaning adversely affects test yields.

Any of the following may point to a contamination problem:

- The probe develops high contact resistance.
- There is yield fallout traceable to high contact resistance.
- Reprobing does not improve the test failure rate.
- A visual (microscopic) inspection reveals particles or a coating on the probe tip.

Usually, incorrect measurements are the first clue something is wrong, and reprobing does not change the failure rate. A microscope inspection of the probe tips can verify the diagnosis. Cleaning recommendations should be available from the company supplying the probes.

Examples

Carbon nanotubes offer properties that make them an excellent material to use for electronic components. ***Figure 6*** shows carbon nanotubes used in an FET structure. To develop some of the I-V characteristic curves of the device, instruments with low current measurement capability are suggested. A typical instrument used is the source measure unit (SMU), which can source either voltage or current and measure either current or voltage, respectively. By setting one SMU to sweep the gate voltage and a second SMU to control the source-to-drain voltage, the source-to-drain current for the device can be measured. Note that the levels of current being measured are in the nanoamp ranges. For

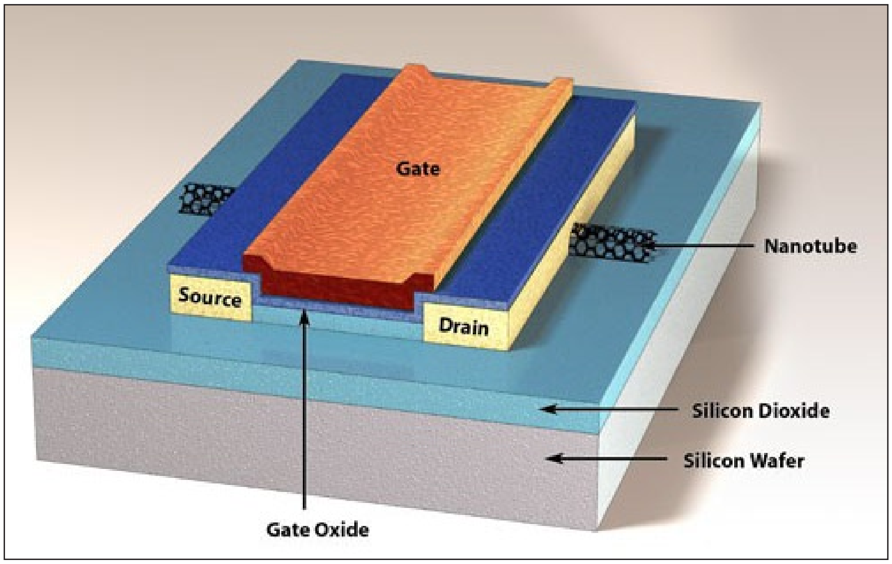

Figure 6: Carbon nanotubes offer properties that make them an excellent material to use for electronic components, as here in an FET structure. To develop some of the I-V characteristic curves of the device, instruments with low current measurement capability are suggested.

most measuring instruments, this is often trivial and noise is not often a problem, but there is a potential for errors as previously discussed.

Chemical self-assembly of electronic components represents a new paradigm for manufacturing electronics, and could lead to development of molecular diodes, switches, or memory. These devices use currents in the hundreds of nanoamps, a reasonable measurement to make, but under certain conditions currents being measured are in the hundreds of picoamps, which requires more care in measurement.

The single electron transistor is a new type of switching device that uses controlled electron tunneling to amplify current. The only way for electrons in one of the metal electrodes to travel to the other electrode is to tunnel through the insulator. Since the tunneling is a discrete process, the electric charge that flows through the tunnel junction flows in multiples of e, the charge of a single electron. When the gate voltage is set to zero, very little tunneling occurs. The opposition to tunneling is called the Coulomb blockade. The charge on the gate capacitor can be set to a non-integral number of electron charges because charge transfer in metals is continuous. This voltage controlled current behavior makes the SET's operation much like that of a FET, but on a much smaller scale.

Because of the behavior of SET devices, and that the movement of single electrons is involved, the current measurements can be very small. There are very discrete steps (the Coulomb staircase) in the current levels as the gate voltage is swept from at least –5mV to +5mV. These current measurements are in the pA range.

Clearly, this application requires very low current sensitivity and even low voltage sourcing capability with microvolt resolution. With the gate voltage being swept with very low voltage steps over a range that can easily be from –100mV to +100mV, this suggests the need for large data storage requirements in order to capture the many points along the I-V curve. To store more than 20,000 points is not unrealistic.

To perform I-V characterization on the three examples shown here, we recommend using the various commercial I-V characterization tools. Source measure units can also offer the same capabilities at a lower cost, offering the functionality of a compact single channel DC parametric tester.

Semiconducting nanowires are seen as a way to interconnect nanoelectronic components such as transistors or logic gates, with research under way on silicon, gallium arsenide, gallium nitride, and other 3-5 materials. Researchers need to understand the resistivity, overall uniformity, reliability, and current carrying capacity of these wires if they are to be useful. Because these device are so small, the measurements must be done at low current, which requires equipment capable of sourcing and measuring currents down to the nanoamp level. Resistance measurements at these low currents requires low voltage measuring instruments using a four-wire method to factor out lead resistances of the measurement system. Sourcing small values of current also minimizes the I^2R across the wire and the possibility of damaging it.

Considerable work is being done on the use of polymers for nanoscale interconnects. Since these are typically high resistance materials, their resistance is usually measured by sourcing voltage and measuring the resulting current, which may be in the femtoamp range. Accuracy demands precautions to reduce or eliminate any extraneous currents in the test system such as those generated by triboelectric effects, electrochemical effects, cable connection issues, and noise generated by electrostatic charges.

To protect the nanoscale samples from electrostatic charge, a Faraday cage made of sheet metal or mesh can be used. The electric field inside a closed, empty conductor is zero, so the object placed inside it is shielded from any atmospheric or stray electric fields.

There are a number of instruments available that will make the necessary resistance measurements. For low resistance measurements, source measure units with nanovoltmeters to supply current and to measure low voltage respectively are appropriate. For high resistance measurements, instruments such as electrometers and sub-femtoamp source measure units are suggested. State-of-the-art I-V characterization tools often have the ability to measure both low and high resistances, which is advantageous when working to increase the conductivity of high resistance materials by adding more conductive materials such as carbon nanotubes.

Speaking of materials, many nanoscience researchers are creating materials like special papers and films that require the need to understand their surface and volume resistivity and conductivity properties. Again, low current and high resistance are often the properties measured. These measurements require a test fixture designed for making the measurement on flat materials.

Measuring resistance, then converting to surface or volume resistivity by taking geometric considerations into account determines resistivity. The ideal way to measure the resistance of an insulating material is to apply a known potential to the sample and measure the resulting current with an electrometer or picoammeter.

Volume resistivity is a measure of the leakage current directly through a material. It is defined as the electrical resistance through a one-centimeter cube of insulating material and is expressed in ohm centimeters. When measuring volume resistivity the test sample is placed between two electrodes and a potential difference is applied between them. The resulting current is distributed through the volume of the test sample and is measured using a picoammeter.

Surface resistivity is defined as the electrical resistance of the surface of an insulator and is expressed in ohms (usually referred to as ohms per square). It is measured by placing two electrodes on the surface of the test sample, applying a potential difference between them and measuring the resulting current.

Conclusion

This article has touched on just a few of the many applications in nanotechnology and the measurements needed to understand more about the devices and materials being investigated. Every day, new ideas and innovations are coming out of research labs. With new ideas also come the needs for new and different measurements. For example, researchers are interested in testing the mechanical properties of materials while seeing what happens to their electrical characteristics. Visualization will continue to be needed so as to see what is happening at the atomic levels., Certain phenomena at the molecular and atomic level often happen very quickly. To characterize these events, current measurements will have to be made much faster and with low noise. Nanoscale and molecular electronics can be destroyed very easily by applying too much current. Instruments must be able to limit the power so that the Joule heating effects are minimized. And, when sourcing voltages to test devices, voltage stepping with resolution down to 1 microvolt will be important so that researchers can clearly see what is happening over a very small step change.

The following four steps to good nanotech measurements can add to confidence in results.

- Define measurement quality. Understand how much sensitivity, resolution, and accuracy will be needed.
- Design the measurement system. Select the appropriate tools, cables, probe systems, and fixturing.
- Build and verify the performance of the system. Understand the potential sources of error in your environment and reduce or eliminate those errors. Know what the system is capable of doing.
- Start taking measurements. KEITHLEY

About the Author

Jonathan Tucker is the Lead Industry Consultant at Keithley Instruments, Inc. in Cleveland, Ohio, where he is responsible for new application test and measurement development in the research and education market segments. His current efforts include product development for nanotechnology applications. He has 19 years of experience in test and measurement. He holds a BS in Electrical Engineering from Cleveland State University and an MBA from Kent State University.

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Keithley Instruments, Inc. 28775 Aurora Road • Cleveland, Ohio 44139 • 440-248-0400 • Fax: 440-248-6168
1-888-KEITHLEY (534-8453) • www.keithley.com

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