

# Application Note Series

#### Number 2481

# I-V Measurements of Nanoscale Wires and Tubes with the Model 4200-SCS and Zyvex S100 Nanomanipulator

## Introduction

It is difficult to characterize very small circuit elements electrically in current-generation semiconductors, as well as in next-generation nanoscale electronics and materials such as Single Wall Carbon Nanotubes (SWNT). Many prober systems can have unacceptably large metal pad requirements, and manual mechanical probe stations with optical microscopes can't resolve fine features. With standard gate dimensions of less than 90nm and space budgets shrinking continuously, the smallest probe pad dimensions required for most prober systems remain fixed at about 50 microns. This limitation is largely the result of the inaccuracy of probe movements and the size of the probe tips.

These difficulties can be eliminated by combining a Keithley Model 4200-SCS Semiconductor Characterization System with an advanced nanomanipulation system, such as the Zyvex S100 Nanomanipulation System (shown in *Figure 1*). This combination offers 5nm movement precision with probe tip diameters of less than 20nm and current-measuring capability better than 1pA.





## Methods and Techniques

Unlike general-purpose measurements and I-V curve generation on macro- and micro-scale components and materials, measurements on molecular wires and carbon nanotubes require special care and techniques. General-purpose resistance measurements and I-V curve generation are often performed using a two-point electrical measurement technique. However, when the resistance to be measured is relatively low (as may be the case with molecular wires, semiconducting nanowires, and carbon nanotubes) or when the resistance of the probes or the contacts is relatively high, a four-point probe will yield more accurate results.

## Theory of Two-Point Measurements

In order to determine a resistance, Ohm's law is used: R = V/I. A known current is sourced and flows through the unknown resistance. The voltage that develops across the resistance is measured and then the resistance is determined by dividing the measured voltage by the sourced current. A problem that occurs when using a two-wire setup is that the voltage is measured not only across the resistance in question, but includes the resistance of the leads and contacts as well (see *Figure 2*). When using an ohmmeter to measure resistances greater than a few ohms, this added resistance is usually not a problem. However, when measuring low resistances on nanoscale materials or components, or when contact resistance may be high, obtaining accurate results with a two-wire measurement may be a problem.





Figure 1: Keithley Model 4200-SCS Semiconductor Characterization System and the Zyvex S100 Nanomanipulator

## Theory of Four-Point Measurements

A solution to the problem of two-point measurements that measure the lead and contact resistance along with that of the device under test (DUT) is the four-wire or "Kelvin" measurement. With Kelvin measurements, a second set of probes is used for sensing. Negligible current flows in these probes; therefore, only the voltage drop across the DUT is measured (see *Figure 3*). As a result, resistance measurement or I-V curve generation is more accurate.



Figure 3: Four-Point Measurement

#### Test System Configuration for Four-Point Measurements

Combining the Keithley Model 4200-SCS Semiconductor Characterization System with the Zyvex S100 Nanomanipulation System creates an automated instrument system for performing I-V and C-V characterization for nanoscale and semiconducting devices, test structures, and other deep sub-micron devices. With a combined system noise floor less than 55fA rms, current measurements better than 1pA are readily achievable.

Tests are easily and quickly configured and executed from the Keithley Interactive Test Environment (KITE). KITE is an application program designed and developed for characterizing semiconductor devices and materials, but very applicable for nanoscale devices. Source and measurement functions for a test are provided by Source-Measure Units (electronic instruments that source and measure DC voltages and currents). Test capabilities are extended with support from a variety of external components. An example setup for a Carbon Nanotube I-V test is shown in *Figure 4. Figure 5* illustrates an I-V Curve on a Carbon Nanotube using the Keithley 4200-SCS, KITE, and the S100.

The Zyvex S100 Nanomanipulator is a positioning and testing tool for micro- and nano-scale research and development applications. It accommodates up to four positioners (three-dimensional stages) with 5nm positioner resolution that grasp, move, test, and optimally position micro- and nano-scale samples in Scanning Electron Microscopes (SEMs) and Focused Ion Beam Systems (FIBS). For a four-point measurement on a nanoscale wire or tube, all four positioners of the test system are used. Each positioner controls a single probe. *Figure* 6 illustrates the connec-



Figure 4: I-V Curve Setup for Testing a Carbon Nanotube with the Keithley 4200-SCS KITE Application Program







Figure 6: Schematic Diagram of Zyvex S100 Test Head with Keithley 4200-SCS

tions of the Keithley 4200-SCS with the S100. *Figure* 7 illustrates actual contacts to a Carbon Nanotube.

To implement a four-point measurement on nanoscale materials and wires, the outer and inner probes that are closest to each other on the DUT connect to the same Source-Measure Unit (SMU). The outer probe connects to the force terminal on the SMU and the inner probe connects to the sense terminal on the SMU. KITE on the Keithley 4200-SCS is used to run the test. The "four-point" interactive test module, which is found in the KITE folder titled "two wire resistor," can be used to generate



Figure 7: SEM Picture of a Carbon Nanotube Attached to the S100 Probes

results for the I-V curve. This is a pre-configured routine that implements four-point measurements using four cables and two SMUs as described previously. It's also possible to create a project based upon the "two wire resistor" and use a bitmap that looks like a Carbon Nanotube, as shown in *Figure 4*.

## Typical Sources of Error

Significant errors can be introduced into low voltage and low resistance measurements by offset voltage and noise sources that can normally be ignored when measuring higher signal levels. The following paragraphs discuss factors that can affect low voltage and low resistance measurement accuracy.

#### **Offset Voltages**

Ideally, when a voltmeter is connected to a relatively low impedance circuit in which no voltages are present, it should read zero. However, a number of error sources in the circuit may be seen as a non-zero voltage offset. These sources include thermoelectric EMFs, offsets generated by rectification of RFI (radio frequency interference) and offsets in the voltmeter input circuit. Steady offsets can generally be nulled out by shorting the ends of the test leads together, then enabling the instrument's zero (relative) feature. Note, however, that cancellation of the offset drift may require frequent re-zeroing, particularly in the case of thermoelectric EMFs.

#### **Thermoelectric Voltages**

Thermoelectric voltages (thermoelectric 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. Constructing circuits using the same material for all conductors minimizes thermoelectric EMF generation.

Allowing test equipment to warm up and reach thermal equilibrium in a constant ambient temperature also minimizes thermoelectric EMF effects. Any remaining thermoelectric EMF, provided it is relatively constant, can be compensated for by using the instrument zero feature. To keep ambient temperatures constant, equipment should be kept away from direct sunlight, exhaust fans, and similar sources of heat flow or moving air. Wrapping connections in insulating foam (e.g., polyurethane) also minimizes ambient temperature fluctuations caused by air movement.

Measurements of sources at cryogenic temperatures pose special problems because the connections between the sample in the cryostat and the voltmeter are often made of metals with lower thermal conductivity than copper, such as iron, which introduces dissimilar metals into the circuit. In addition, because the source may be near zero Kelvin while the meter is at 300 Kelvin, there is a very large temperature gradient. By matching the composition of the wires between the cryostat and the voltmeter and by keeping all dissimilar metal junction pairs at the same temperature, very low voltage measurements can be made with good accuracy.

#### Non-Ohmic Contacts

Non-ohmic contacts are evident when the potential difference across the contact is not linearly proportional to the current flowing through it. Non-ohmic contacts may occur in a low voltage circuit as a result of oxide films or other non-linear connections. A non-ohmic connection is likely to rectify any radio frequency energy (RFI) present, causing an offset voltage to appear in the circuit. There are several ways to check for non-ohmic contacts and a number of methods to reduce them.

When using a micro-ohmmeter, digital multimeter (DMM), or Source-Measure Unit to make low resistance measurements, check for non-ohmic contacts by changing ranges. By changing the measurement range, the test current is usually changed as well. A normal condition would indicate the same reading but with higher or lower resolution, depending on whether the instrument was up or down ranged. If the reading is significantly different, this may indicate a non-ohmic condition.

If using a separate current source and voltmeter to make low resistance measurements, each instrument must be checked for nonohmic contacts. If the current source contacts are non-ohmic, there may be a significant difference in the compliance voltage when the source polarity is reversed. If the voltmeter contacts are non-ohmic, they may rectify any AC pickup present and cause a DC offset error.

To prevent non-ohmic contacts, choose an appropriate contact material, such as indium or gold. Make sure that the compliance voltage is high enough to avoid problems due to source contact non-linearity. To reduce errors due to voltmeter non-ohmic contacts, reduce AC pickup by using shielding and appropriate grounding.

#### **Device Heating**

Device heating can be a consideration when making resistance measurements on temperature-sensitive devices such as nanoscale components or materials. The test currents used for low resistance measurements are often much higher than the currents used for resistance measurements in the normal range, so power dissipation in the device can be a consideration if it is high enough to cause the device's resistance value to change.

Recall that the power dissipation in a resistor is given by the formula  $P = I^2 R$ . From this relationship, we see that the power dissipated in the device increases by a factor of four each time the current doubles. Thus, one way to minimize the effects of device heating is to use the lowest current possible while still maintaining the desired voltage across the device being tested. In many cases, however, the test current is not adjustable. Most micro-ohmmeters and DMMs, for example, do not have provisions for setting the test current. In those cases, alternate means must be found to minimize device heating. One simple but effective way to do so is to use the instrument's one-shot trigger mode during measurements. While in this mode, the instrument will apply only a single, brief current pulse to the DUT during the measurement cycle, thereby minimizing errors caused by device heating. Source-Measure Units, such as those used in the Keithley 4200-SCS, offer complete programmability of the current to be sourced, making it easy to control device heating.

#### Contaminated Probes

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 the course of their 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 problems, 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. Another consideration is that too little cleaning adversely affects test yields.

How do you know that the probes are dirty? 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 that something is wrong, and reprobing does not change the failure rate. A microscope inspection of the probe tips can verify the diagnosis. Refer to the cleaning recommendations from the company that supplies the probes.

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# Test System Safety

Many electrical test systems or instruments are capable of measuring or sourcing hazardous voltage and power levels. It is also possible, under single fault conditions (e.g., a programming error or an instrument failure) to output hazardous levels even when the system indicates no hazard is present.

These high voltage and power levels make it essential to protect operators from any of these hazards at all times. Protection methods include:

- Design test fixtures to prevent operator contact with any hazardous circuit.
- Make sure the device under test is fully enclosed to protect the operator from any flying debris.
- · Double insulate all electrical connections that an operator could touch. Double insulation ensures that the operator is still protected, even if one insulation laver fails.
- Use high reliability, fail-safe interlock switches to disconnect power sources when a test fixture cover is opened.
- Where possible, use automated handlers so operators do not need to access the inside of the test fixture and do not need to open guards.
- Provide proper training to all users of the system so they understand all potential hazards and know how to protect themselves from injury.

It is the responsibility of the test system designers, integrators, and installers to make sure that operator and maintenance personnel protection is in place and effective.

### For More Information

For further reading on two- and four-wire resistance measurements, low voltage measurements, and typical sources of error, refer to Keithley's Low Level Measurements handbook.

#### Conclusion

The Keithley 4200-SCS Semiconductor Characterization System combined with the Zyvex Test System is an extremely effective measurement tool for I-V characterization of nanoscale components. With four-point probe capability, 1pA accuracy, and 5nm precision movement, the system offers a unique combination of features that is ideal for nanotechnology and semiconductor characterization.

## Acknowledgements

Keithley Instruments would like to thank Phil Foster, Manager of Applications/Sales Engineering, and Jeff Hochberg, Control System Engineer, of the Zyvex Corporation for contributing to this application note.

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