Automatic Resistance Measurements on High Temperature Superconductors

Introduction

Since the discovery of high-temperature superconductors, research on the subject has increased rapidly. Lower cost of the test setups and materials (liquid nitrogen vs. liquid helium) also allows more laboratories to engage in materials and process research.

Many laboratories are working with small material samples, transducer applications, or thin-film sensors. These types of samples are usually tested at lower current densities (milliamps vs. tens of amperes). Since small test currents are used in conjunction with extremely low resistances, measured voltages are very small. Even ordinarily insignificant errors can seriously degrade overall system accuracy. For example, thermal voltage errors from system wiring can be greater than the measured voltage, and noise from electrical or magnetic fields can reduce system sensitivity, making repeatable measurements impossible.

This lab note addresses these issues and describes a system that:

- Measures and cancels thermal voltage errors.

- Controls noise from both electrical and magnetic fields found in a typical laboratory environment.

- Is automated to handle the details of the measurement.

- Has a system sensitivity of $1\mu\Omega$ and a maximum resolution of $0.01\mu\Omega$.
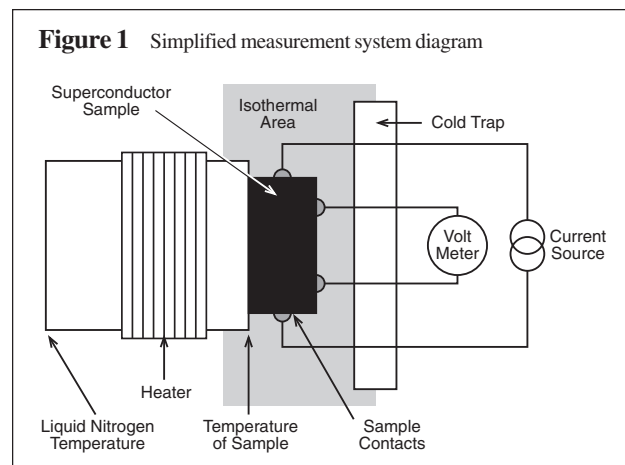
Basic Measurement Technique

The typical superconductor measurement ranges from a few ohms down to (ideally) zero ohms. These low values of resistance require using a 4-wire measurement technique to eliminate the effects of lead resistance. Even with these precautions, errors from thermally induced voltages can still cause errors in the measurement loop.

Thermal voltages occur whenever a thermal gradient appears across a junction of dissimilar materials. Most of the test system can use copper-to-copper connections to prevent the formation of dissimilar junctions. However, the connection to the sample will inevitably create a thermocouple junction. Some control of the thermal gradient is possible by using a "cold trap," which will help keep all the wires and the sample at the same temperature. Given the low measured voltages and the high thermal gradients, further thermal voltage error correction is required.

Some systems use an AC signal to measure the sample resistance. Since the thermal voltages are DC, they are rejected from the measurement. However, the AC measurement technique results in errors caused by system stray inductance and capacitance, and these stray effects can become significant unless very low frequency AC signals are used.

An alternative approach is the quasi-DC technique using the simplified system shown in **Figure 1**. A voltage measurement (V_1) is taken with positive test current; the current is then reversed, and another voltage measurement (V_2) is taken. If both measurements are taken before thermal gradients change, the thermal voltages cancel in the final calculation.

Figure 1   Simplified measurement system diagram

Superconductor Sample

Isothermal Area

Cold Trap

Volt Meter

Current Source

Heater

Liquid Nitrogen Temperature

Temperature of Sample

Sample Contacts

The model of the thermal voltages is shown in **Figure 2**. Only the thermocouples in the measurement loop are shown since these are the only ones that affect system accuracy. In a properly wired system, all junctions are made of copper-to-copper connections, including the connections to the meter.

In this manner, the only thermocouples junctions that form are the connections between the copper wires and the sample.

If both thermocouples are made of the same material and are at exactly the same temperature, the thermal voltages completely cancel. In practice, however, this ideal situation rarely occurs. Any slight temperature variation or difference in the materials prevents thermal voltages from canceling, and it may not be possible to use only copper-to-copper connections in some cases.

The quasi-DC technique cancels any remaining thermal voltages in the system. The final resistance calculation is:

$$R_{DUT} = \frac{V_1 - V_2}{|2I|}$$

where: R_{DUT} = resistance of device under test
V_1 = first measured voltage
V_2 = second measured voltage
I = test current (2 currents, exactly equal and opposite)

This relationship holds true only if the temperature difference between the two thermocouples does not change between the two voltage measurements. In a real system, the thermal gradients are changing with time, and it is seldom possible to allow the system to reach full thermal equilibrium before taking a reading. In this case, the thermal voltages do not completely cancel, and an error term enters the calculation. The error term is a function of the change in temperature difference between the two thermocouples over time.

A mathematical model that includes the thermal voltage and the change in this voltage is given as follows:

$$V_{1(measured)} = V_1 + V_{th}$$

$$V_{2(measured)} = V_2 + V_{th} + \delta V_{th}$$

where: V_{th} = thermal voltage when V_1 is measured

δV_{th} = the change in the thermal voltage (V_{th}) between the first and second voltage readings.

Combining terms and letting $V_2 = -V_1$, we have:

$$R_{DUT} = \frac{(V_1 + V_{th}) - (-V_1 + V_{th} + \delta V_{th})}{|2I|}$$
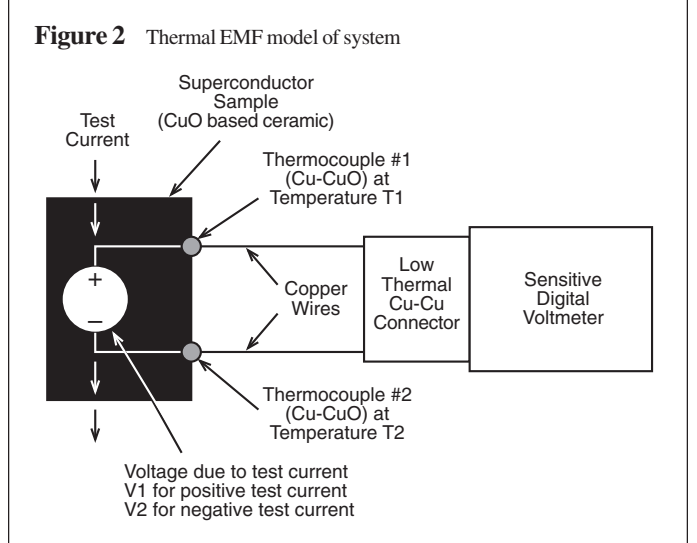
Simplifying further:

$$R_{DUT} = \frac{V_1 + (\delta V_{th})/2}{|I|}$$

Note that δV_{th} is a function of temperature given by:

$$\delta V_{th} = k(\delta T),$$

where δT is the change in temperature difference $(T_1 - T_2)$ since the first reading, and k is the thermoelectric voltage coefficient.

Since δT represents a change in temperature between the two thermocouples after a period of time, it is an implicit function of time. In this case, we can take the differential of resistance, where

Figure 2 Thermal EMF model of system

time (t) is the domain of the function. Allowing for approximation, we obtain:

$$\Delta R_{DUT} = \frac{k}{2|I|} \frac{d(\delta T)}{dt} \Delta t$$

This error term can be minimized by making sure the two voltage readings are taken close together in time (small Δt). Also, if the cryostat and cold trap are properly designed, δT can be kept to a very small fraction of the sample cooling rate.
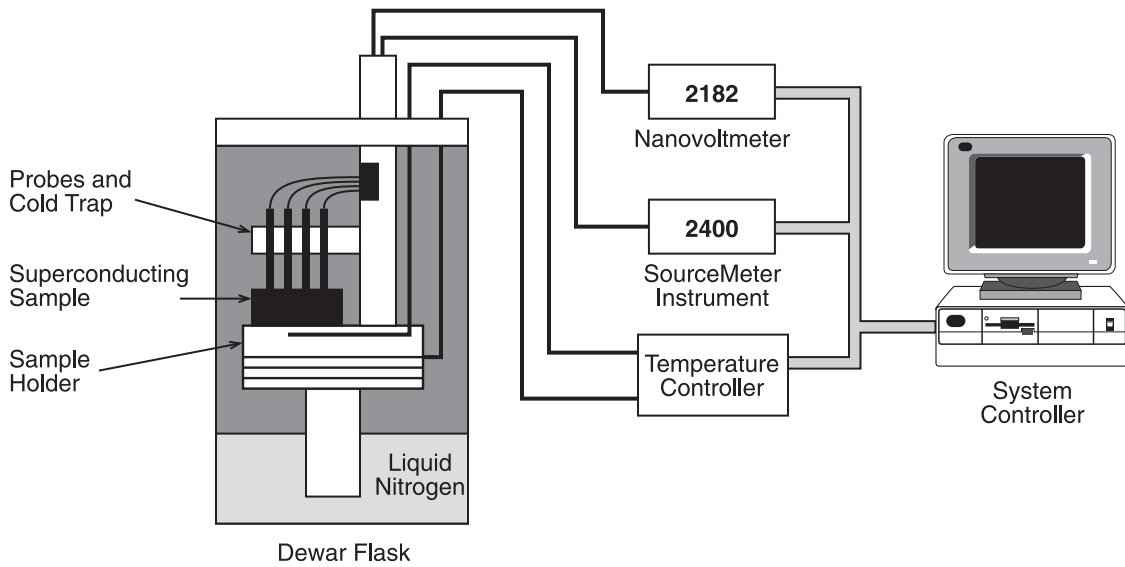
Practical Measurement Setup

Figure 3 shows a basic superconductor resistance measurement test system using the combination of a Model 2182 Nanovoltmeter and a Model 2400 SourceMeter instrument for measuring the resistance. The voltage leads should be made of a material with a low Seebeck coefficient with respect to the sample. The sensitivity of the Model 2182 Nanovoltmeter is crucial to obtaining precision measurements because the application demands the ability to measure extremely low voltages. If the application requires picovolt resolution, the Model 1801 Nanovolt Preamplifier with the Model 2001 or 2002 DMM can be used for even greater sensitivity. The Model 2400 SourceMeter instrument can source bipolar currents up to 1A.

The value of the test current must be high enough that a significant change in resistance can be detected, yet not so high that it equals or exceeds the critical current of the sample. If the test current exceeds the critical current for the material, the resistance of the sample will increase rapidly. If the current becomes too high, the power dissipated may damage the sample and the cryostat.

Assuming thermal voltages are completely canceled, measurement accuracy is limited by the instruments used. The accuracy of the Model 2182 Nanovoltmeter and the Model 2400 SourceMeter instrument define the resistance resolution and accuracy, while the characteristics of the temperature controller define temperature accuracy. Error term calculations are as follows:

Figure 3   Resistance measurement system

Resistance Accuracy

Model 2400, 100mA output accuracy: $\cong 0.066\%$

Model 2182, 10mV range accuracy: ±54ppm (full-range input)

System accuracy and resolution depend on the accuracies of both the current source and the voltmeter:

System accuracy: $\cong \pm0.0714\%$ for a 100mA test current and 10mV voltage measurement.

System resolution (no filtering):
$\Delta R = \Delta V/I \leq 1nV/100mA = 10n\Omega \,(10^{-8}\Omega)$

Delta Measurement Mode

A Delta measurement mode is built into the 2182. This mode provides the measurements and calculations for the quasi-DC current-reversal technique to cancel the effects of thermoelectric EMFs. Each Delta reading is calculated from two voltage measurements on Channel 1; one on the positive phase of an alternating current source, and one on the negative phase.
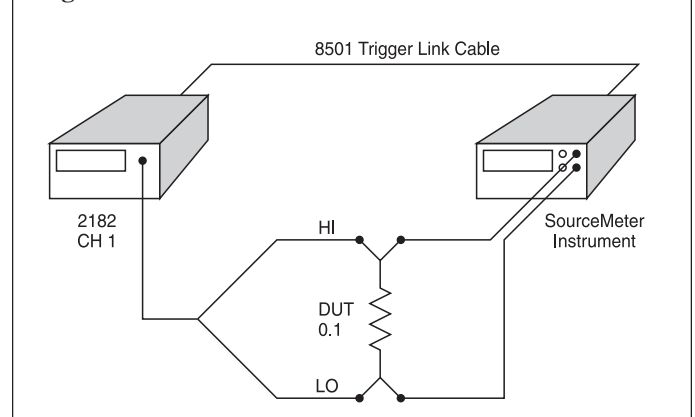
The Model 2182 is optimized to provide low-noise readings when measurement speed is set from 1 to 5 PLC. At 1 PLC, current can be reversed after 100ms. At 5 PLC, current can be reversed after 333ms. At these reading rates, noise induced by the power line should be insignificant. Filtering can be used to reduce peak-to-peak reading variations.

Delta measurements by the Model 2182 require the use of an alternating polarity source. The source must have external triggering capabilities that are compatible with the external triggering capabilities of the Model 2182. The following technique shows how to use a Keithley Model 2400 with the Model 2182 to perform Delta measurements.

A Keithley SourceMeter instrument (Model 2400, 2410 or 2420) can be used as a bipolar source by configuring it to perform a custom sweep. In general, a custom sweep is made up of number of specified source points. To provide current reversal, the positive current value(s) are assigned to the even numbered points, and the negative current value(s) are assigned to the odd numbered points. Applications that use Delta measurements require either a fixed current or a growing amplitude current. When a fixed current is required, the SourceMeter instrument can be configured to output a bipolar 2-point custom sweep. That sweep can be run a specified number of times or it can run continuously. For example, if a fixed current of 1mA is required for the test, the two bipolar sweep points for the custom sweep would be +1mA and –1mA. When a growing-amplitude current is required, the custom sweep can be configured to include all the current values required for the test.

The connection for the Model 2182 Nanovoltmeter, the Model 2400 SourceMeter instrument, and the DUT is shown in **Figure 4**.

Figure 4   Delta Measurement connections

This application also requires the connection of the Model 8501 Trigger Link Cable from the 2182 to the 2400.
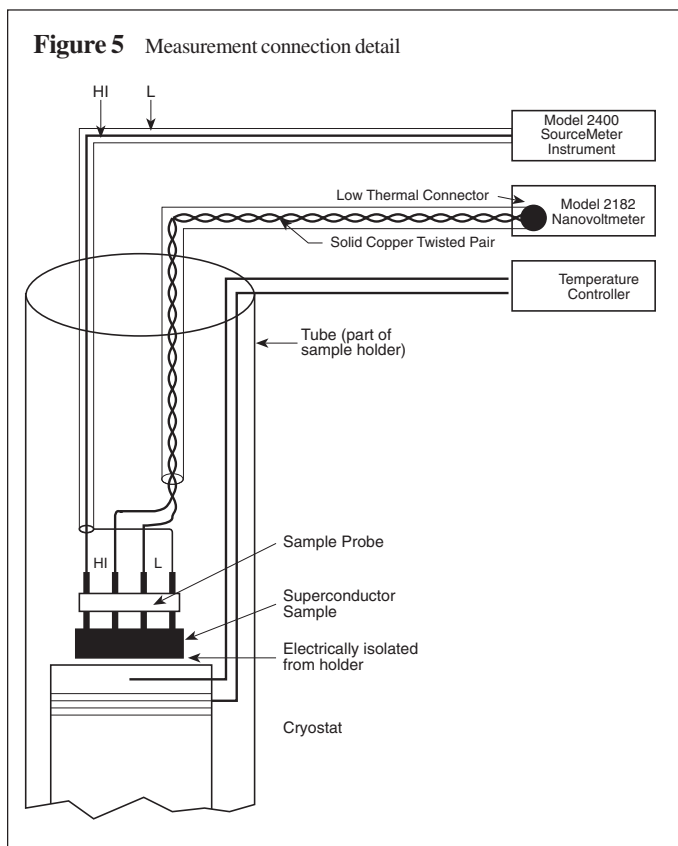
System Noise Control

Another issue in low-level measurements is system noise and noise control. In this application, all lines are low impedance, which minimizes the need for shielding. However, given the low voltages to be measured, and the level of electrical noise found in most laboratories, some shielding will probably be required in most applications.

Noise caused by magnetic fields is another important factor in this application. Any loop in the voltmeter's leads will pick up small magnetic field changes that will induce a current in the leads. This current will show up as noise in the voltage readings. Twisting the leads together and keeping the leads as short as possible will significantly reduce this noise problem. If there are large and varying magnetic fields in the laboratory, special magnetic shielding (mu metal) may be needed.

Figure 5 shows the detail of system wiring used in this example. Electrostatic shielding and twisted pair wires (to reduce magnetic field effects) are included, but mu metal magnetic shields are not used.

Another level of noise control can be introduced by adding filtering to the system software. An averaging filter, for example, can reduce the noise by the square root of the number of samples averaged. However, the cost of extra filtering is a slower measurement cycle. Note that filtering should always be applied to the calculated resistance values — not to the voltage readings. Filtering the voltage will reduce the voltage reading rate, increasing thermal voltage errors because of the increased time period between the two voltage readings of each measurement cycle.

System Software

Once the details of hardware configuration and connections are complete, it is a fairly simple task to design and implement the software driver. Since the measurement technique is now known, the steps to implement it are straightforward. For further reference on the methodology, refer to the 2182 manual section on Ratio and Delta titled the "Delta Measurement Procedure Using a SourceMeter."

Key considerations with any software design are the system timing reading rates. The Model 2182 can send a low voltage reading over the IEEE-488 bus roughly every 10-15ms, while the Model 2400 can output a programmed current within 100-500ms depending on the range and value sourced. Since two voltage readings are required for each resistance calculation, each complete cycle could take between 20ms to 30ms. The bus transfer rate of the 2182 for ASCII readings is the limiting factor. However, storing data to the internal data buffer of the 2182 and recalling that data later will speed up the total measurement cycle time. This computation does not take into effect the time required for the temperature controller and any temperature measurements recorded and transferred across the bus.

Figure 6 shows a sample set of subroutines that could be used to program the 2182 and the 2400, including recall of the measurements. This is only one example of obtaining a resistance measurement using the Delta mode. Please refer to Section 5: Ratio and Delta in the Model 2182 Users Manual for additional examples for testing superconductor materials.

The subroutines shown in **Figure 6** are designed to set up the instruments, as well as initiate and obtain readings from the 2182. The *SetupInst* routine should be called at the beginning of the program to initialize the instruments. Note that there are settings within this routine that can be changed based upon specific needs. If there's a need to program the 2182 for a measurement range other than 10mV, this can be easily modified, as well as the reading rate. The same holds true for the 2400. The code can be modified for the particular current sourcing range that best suits the current requirements.

The *TakeReading* routine sends the two current values to the 2400 that will be outputted. One must be positive and the other negative. The code illustrates the use of ±100mA output. However, the code can be modified to meet specific needs. Once the 2400 has the desired current values, the 2182 SRQ (Service Request) is enabled for when a reading is available and its triggering function is initialized. Finally, the 2400 output is turned on and its triggering

Figure 5 Measurement connection detail

HI L

Model 2400 SourceMeter Instrument

Low Thermal Connector

Model 2182 Nanovoltmeter

Solid Copper Twisted Pair

Temperature Controller

Tube (part of sample holder)

HI L

Sample Probe

Superconductor Sample

Electrically isolated from holder

Cryostat

Figure 6 Software Driver Listing

```
Sub SetupInst()

        'Send Reset command to 2182 and 2400. 2182 at Address 16, 2400 at Address 24.

        Call Send(16, "*RST", status%)
        Call Send(24, "*RST", status%)

        'Set 2182 Voltage Function, 10mV range, reading format, and triggering functions.

        Call Send(16, ":Sens:Func Volt", status%)               'Set 2182 reading mode to volts
        Call Send(16, ":Sens:Volt:Chan1:Range 0.010, status%)   'Channel 1 at 10mV range
        Call Send(16, ":Sens:Volt:NPLC 1", status%)             '1 Power Line Integration rate
        Call Send(16, ":Sens:Volt:Delt On", status%)            'Delta Mode on
        Call Send(16, ":Sens:Volt:Dig 6", status%)              '6 1/2 digit resolution
        Call Send(16, ":Trig:Sour Ext", status%)                'External triggering
        Call Send(16, ":Trig:Count 2", status%)                 'Trigger count of two
        Call Send(16, ":Form:Elem Read", status%)               'Return readings only
        Call Send(16, ":Stat:Meas:Enab 32", status%)            'Set to SRQ on Reading Available

        'Set 2400 for 100mA range, 2V maximum compliance
        Call Send(24, ":Sens:Func 'Volt'", status%)             'Set 2400 reading mode to volts
        Call Send(24, ":Sens:Volt:NPLC 0.01", status%)          '.01 Power Line Integration rate
        Call Send(24, ":Sens:Volt:Prot 2", status%)             'Voltage Compliance at 2 volts
        Call Send(24, ":Arm:Coun 1;Sour Imm;Tcon:Dir Sour", status%)

'Set ARM layer triggering
        Call Send(24, ":Trig:Sour TLIN", status%)               'Set TRIGGER layer for TLINK
        Call Send(24, ":Trig:Dir Sour", status%)                'Trigger Source bypass
        Call Send(24, ":Trig:Tcon:Dir Sour", status%)
        Call Send(24, ":Trig:Outp Sour", status%)               'Output Trigger after source
        Call Send(24, ":Trig:Coun 2", status%)                  'Trigger count is two
        Call Send(24, ":Trig:Del 0", status%)                   'No trigger delays
        Call Send(24, ":Sour:Func:Mode Curr", status%           'Source Current
        Call Send(24, ":Sour:Curr:Mode List", status%)          'Current List mode
        Call Send(24, ":Sour:Curr:Rang 100E-3", status%)        '100mA Range
        Call Send(24, ":Sour:Del 0", status%)                   'Source delay is 0

End Sub

Sub TakeReading()

        'Send to the 2400 the positive and negative current values
        PosCurrent = 0.1: NegCurrent = PosCurrent * -1
        Call Send(24, ":Sour:List:Curr "+ str$(PosCurrent) + "," + str$(NegCurrent), status%)
                                                                'Set alternating values
        Call Send(16, "*SRE 1", status%)                        'Enable 2182 SRQ
        Call Send(16, ":Init", status%)                         'Initialize 2182 triggering

        'Turn on 2400 output and initialize sequence
        Call Send(24, ":Output On", status%)                    'Turn on 2400 Output
        Call Send(24, ":Init", status%)                         'Initialize 2400 Triggering

        'At this point, the program waits for the SRQ from the 2182.
        'When the SRQ is identified, execute the following code:

        Call Send(24, ":Output Off", status%)                   'Turn off 2400 Output

        Call Send(16, ":Fetch?", status%)                       'Query 2182 for Delta Reading
        Call Enter(Delta$, 20, l%, 16, status%)                 'Get delta reading

        Resistance = Val(Delta$) / PosCurrent                   'Compute resistance

        'In order to reset the instruments and clear the SRQ,
        'the following commands need to be sent to the 2182 and 2400:
        Call Send(16, ":Stat:Meas?", status%)                   'Query 2182 for poll value
        Call Enter(poll$, 20, l%, 16, status%)                  'Get poll value
        Call Send(16, ":Abort", status%)                        'Reset Triggering in 2182
        Call Send(24, ":Abort", status%)                        'Reset Triggering in 2400
        Call Send(16, ":Trig:Clear", status%)                   'Clear all pending 2400 triggers

End Sub
```

is initialized. The routine then waits for the SRQ from the 2182. Once the SRQ is identified from the 2182, the 2400 output is turned off and the Delta reading is acquired. With the reading and the current known, the resistance can be calculated. At the end of the routine, reading the measurement status register clears the 2182 SRQ. Finally, trigger control for both instruments is reset and any pending triggers in the 2400 are cleared. If a temperature controller is being run, any commands used to control the temperature, as well as obtain any temperature readings, should be incorporated into this routine.

Resistance vs. Resistivity

The actual parameter measured using this system is the sample resistance. The resistance (measured in ohms) is a function of the material as well as of sample size and shape. Volume resistivity (measured in ohm-cm), however, is a function only of the material.
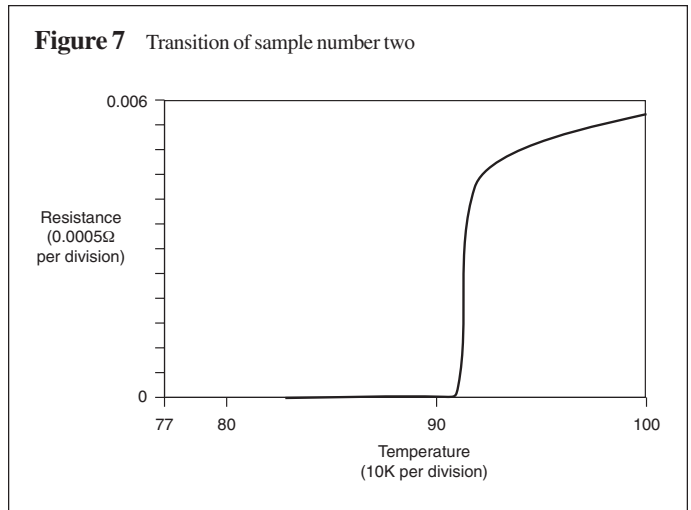
Resistance comparisons among different samples are valid as long as the sample shapes and sizes are the same. The placement and spacing of the probe must also be the same for each sample. A sample holder and a collinear probe can take care of the spacing and placement.

The test methods and calculations for resistivity measurements are generally easy to implement, and these methods are regularly used by the semiconductor industry. Test methods and geometric corrections have been calculated for many different sample shapes.

If the sample is a circular disk, a four-point collinear probe can be used. For bar or bridge (parallelepiped) samples, a two-point or other multiple contact method may be used. If the sample is similar, the van der Pauw technique may be used. The van der Pauw technique can also determine if the sample resistivity is inhomogeneous.

The specifics of these methods and the correction factors are described in detail in the *Annual Book of ASTM Standards*, volume 10.05, standard numbers F43, F76, and F84.

In cases where the sample does not meet standard requirements, the system can be "calibrated" using a sample of a material with a known resistivity. The known material should have a resistivity similar to the test samples and be the same size and shape. The known sample resistance is measured and the correction factor (CF) calculated from CF = (resistivity)/R. The superconducting sample resistivity can then be calculated by (resistivity) = R × CF.

Figure 7   Transition of sample number two

Conclusion

This system was developed to demonstrate the DC measurement technique and to show a method of automating superconductor resistance and temperature measurements. The system was relatively easy to implement. The greatest difficulty was connecting the sample to the test leads. Since only three samples were available, fine copper wire and silver paint were used to connect to the samples.

As shown in **Figure 7**, the resistance can be plotted vs. temperature as the sample temperature is changing.

For determining the critical current, the Model 2182 and the Model 2400 can be used together to produce a precision I-V curve over a range of currents. When operated in sweep mode, the Model 2400 can source current from hundreds of picoamps to 1A.

This system can also be applied to measure different parameters. Critical density vs. temperature is a typical measurement that can be performed with this system. If higher current densities are required, a higher power source (with pulse capability and/or a quench detector) can be used instead of the Model 2400 SourceMeter instrument, such as the Model 2430 1kW Pulse SourceMeter instrument.

Other tests such as Hall effect and van der Pauw resistivity can be added to this system. These tests require some signal switching in the system, and the switches must be designed for low thermal voltages to prevent signal degradation. The Keithley Model 7168 Nanovolt Scanner Card and the Model 7001 or 7002 Switching Mainframe are ideal for this purpose.

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