

Resistivity of a Superconductor

PURPOSE: To investigate the resistive properties of a superconductor as a function of temperature. To gain experience doing measurements at cryogenic temperatures using the 4-point resistance technique and to gain experience using a lock-in amplifier to measure small signals.

Introduction to the 4-point resistance technique.

Low resistance samples are difficult to measure using an ohmmeter. Using the ohmmeter setting on a multimeter means that you will measure the resistance of the sample plus the connecting leads to the sample, as shown in Figure 1.

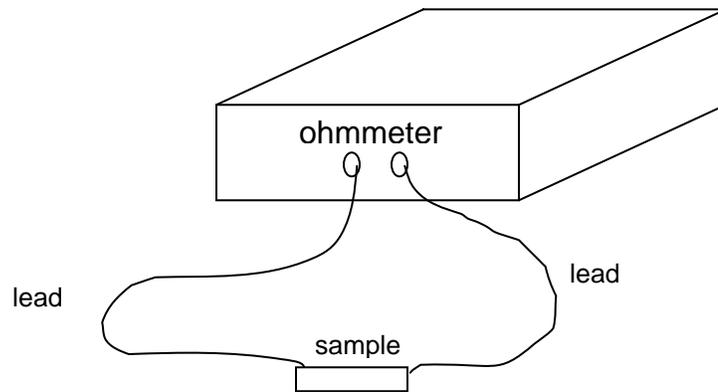


Figure 1. Using an ohmmeter to measure resistance.

To measure the resistance of the sample *only*, it is necessary to use the 4-point resistance technique. In the 4-point method, as shown in Figure 2, one sends a current through the ends of the sample and measures the voltage across two points on the sample and determines the resistance using Ohm's law. In this case, the resistance is measured between the two points on the sample where the voltage contacts are located.

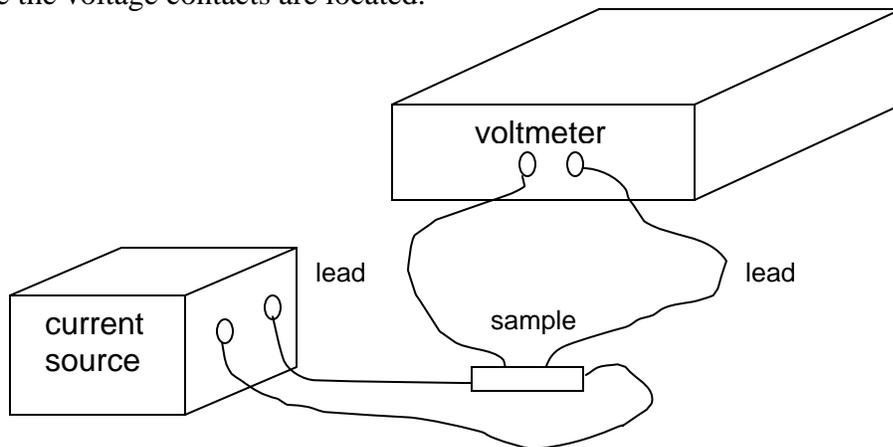


Figure 2. 4-point resistance method.

The 4-point method alleviates interference from the lead resistance (and resistance of the lead contacts to the sample) because of the very high resistance of the voltmeter. An equivalent circuit is shown in Figure 3. The small current that travels through the voltmeter is determined almost entirely by the resistance of the voltmeter and the sample resistance. The voltage read by the voltmeter is determined by this small current, which ultimately determines the measured resistance.

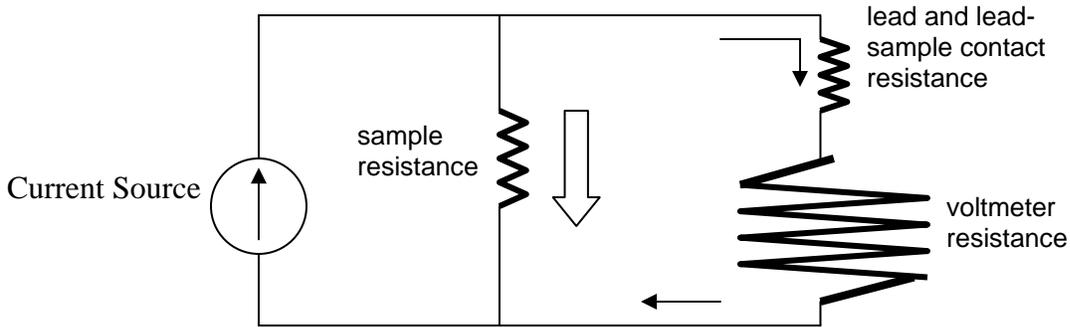


Figure 3. The 4-point equivalent circuit.

To convert this resistance value into a resistivity, equation 1 can be used provided that the diameter of the sample is significantly less than its length.

$$R = \frac{l}{A} \rho \quad (1)$$

Here, R is the resistance, ρ the resistivity, A the cross sectional area of the sample and l is the distance between the voltage contacts.

Measuring small signals, the lock-in amplifier

A lock-in amplifier is an instrument that is capable of retrieving an extremely small signal buried in noise. The instrument works by “tagging” the signal of interest with a small ac voltage of a specific frequency and a specific phase. The lock-in then measures this reference signal along with any external noise. However, the signal of interest is amplified considerably while the external noise (of a different frequency and phase from the reference) is averaged to zero.

For our 4-point resistivity measurements, we would like to use small currents to avoid sample heating. This practice will be especially necessary for measuring the superconductor since its properties are affected by both temperature and magnitude of the current. If small currents are used, small voltages will be measured, which will necessitate a very sensitive measuring instrument, the lock-in amplifier.

Figure 4 illustrates the configuration of the lock-in amplifier for our 4-point resistivity measurements. Like the voltmeter, the lock-in amplifier has an extremely large input resistance

which eliminates interference from the lead resistance. The lock-in has several other unique features that the voltmeter does not have. The oscillator output of the lock-in places a small ac current through the sample. The source impedance of this oscillator output is high, about 600 ohms; therefore changes in the resistance of the sample or the contact lead resistance during the experiment have little effect on the current through the sample. The small ac voltage across the sample is measured as the input to the lock-in. By using differential input mode, the signal is measured as the *difference* between the A and B inputs, which means that common mode noise, imparted from outside sources to each of the input wires, is rejected from the measurement. In addition, as mentioned, only ac voltages at the same frequency and in-phase with the oscillator sine wave will be amplified, further decreasing influences from external noise.

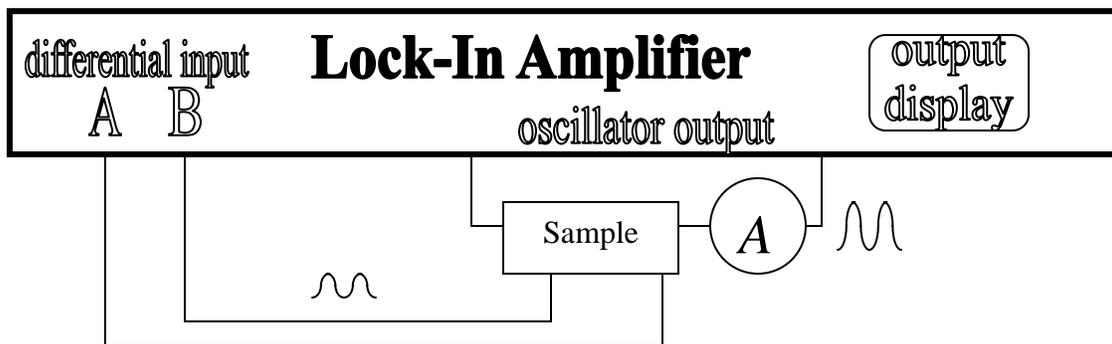


Figure 4. Configuration of the lock-in amplifier for our 4-point resistivity measurements. The circle with the A denotes a separate ammeter to facilitate current measurements.

Superconductivity

Superconductors are materials that lose their electrical resistance below a certain temperature known as the critical temperature. As long as the temperature remains below the critical temperature, the resistance is zero and the material is said to be in the superconducting state. If the temperature is above the critical temperature, the resistance is finite and the material is said to be in the normal state, since it behaves like a normal metal.

In this experiment, we will be using the 4-point resistance technique to measure the resistivity of a superconductor as a function of temperature. To measure temperature, we will use a thermocouple. Thermocouples are temperature sensors that consist of two dissimilar metals. Two different metal wires are connected at one end and this end is placed next to the sample. The other ends of the wires are attached to a voltmeter at room temperature. A voltage is generated when the end of the thermocouple next to the sample differs in temperature from the end of the thermocouple attached to the voltmeter.

PROCEDURE:

Measurement of Copper at Room Temperature

1. Measure the resistance of the copper rod using just two connecting wires and the ohmmeter setting on the multimeter.
2. Now measure the resistance of the connecting wires themselves, that is, without the copper rod between them. What can you say about the reliability of your measurement from step 1? Do a calculation of the resistance of the copper rod (based on its dimensions and the known room temperature resistivity of copper $\sim 1.7 \times 10^{-8} \Omega\cdot\text{m}$) and comment on whether it is possible to measure this resistance using the ohmmeter.
3. Now use the four-probe resistance technique. That is, to determine the resistance, put a current through the ends of the copper rod and measure the voltage across the copper rod using the lock-in configuration from figure 4. The following lock-in settings will facilitate these measurements:

Oscillator level:	2 V
Oscillator frequency:	500 Hz
Reference:	Internal
Input mode:	A-B, Float

An oscillator level of 2 V is the maximum output and will ensure the largest current and therefore largest possible voltage signal for the measurement. Still, the current will be small enough to avoid sample heating. Considering the 600 ohm source resistance of the oscillator, the current will be at most, $I = V/R = 2 \text{ V}/600 \Omega \sim 3 \text{ mA}$. The fairly high oscillator frequency will ensure that significantly many sine wave cycles will be averaged in a reasonable amount of time. For example, in 1 s, 500 cycles would be averaged which will ensure an accurate measurement of the signal and also allow elimination of external noise. You will need to employ the fluke multimeter in ammeter mode to determine the current through the rod.

After you have made all appropriate connections between the copper rod, the lock-in and the ammeter, push the AUTO button and then the MEASURE button on the lock-in. This action will cause the lock-in to cycle through the various voltage ranges and find the one most appropriate to maximize the signal. In addition, it will select a time constant that determines the number of cycles to average for noise reduction. If the output display is somewhat unstable once the procedure is finished, you may wish to increase this time constant to further stabilize the output.

The output display should be set to read %FS (percent of full scale). The voltage is then determined by multiplying this percent by the sensitivity (or range) of the measurement, read on the left side of the front panel of the lock-in. Record this voltage and the measured current on the ammeter, then determine the resistance of the rod.

4. Make all necessary length measurements to determine the resistivity of the copper rod. Keep in mind that you measured the resistance of the rod between the location of the voltage lead connections. Determine the resistivity and compare to the accepted value.

Measurement of a Superconductor

5. Measure the dimensions of the sample and distance between contacts since you will need this information to convert your data into resistivity units. As an initial check, using the ohmmeter, measure the resistance between the current leads and then between the voltage leads for your sample. These measurements are made at the top of the probe and are denoted as I and V. The value of your resistances is almost entirely determined by the lead-sample contact resistance and this number should be less than 50 ohms for the experiment to proceed properly. If it is larger, get assistance from the instructor.
6. Make the appropriate connections between the sample probe, the lock-in and the ammeter as you did for measuring the copper rod. The T connection at the top of the probe denotes the thermocouple voltage and should be measured by a second Fluke multimeter.
7. The idea behind this experiment is to gradually lower the tail of the sample probe into the liquid nitrogen and **slowly** cool the sample from room temperature to the temperature of boiling nitrogen (77 K). As the sample cools, you will measure the thermocouple voltage, the sample voltage (lock-in output), and the sample current. To determine when to take data, first locate the calibration table for your particular thermocouple (indicated on the box on the top of the probe) in a CRC handbook. You should collect data at about 50 - 100 temperature values. By examining the approximate range of voltages from the CRC handbook, you can then decide at which thermocouple voltages to collect your readings.
8. You will first record all three data values at room temperature (before the tail of the probe touches the liquid nitrogen). To begin, use the same AUTO then MEASURE procedure that you employed for the copper rod. Once you have performed this step, the lock-in will have adjusted to the proper range for recording data for the rest of the experiment. Do not employ the AUTO-MEASURE feature again. You may wish to set the time constant to a fairly high value (1 s) that will ensure a stable signal but will still allow measurements to proceed at a moderate pace. Once you have recorded your room temperature data, lower the tail of the probe into the nitrogen so that about ¼" is submerged in the liquid. You will keep the tail at this height as you record thermocouple readings and corresponding sample current and voltage values. After the temperature stops changing, lower the probe down another ¼" and record another series of data.
9. Once the sample is completely submerged into the liquid nitrogen, record all three data values and you will use this thermocouple voltage along with your thermocouple voltage at room temperature as calibration points for the thermocouple. Assuming a linear voltage vs. temperature response (which is very roughly the case for a thermocouple), you can determine an equation to convert all of your thermocouple voltages into temperature values.

ANALYSIS:

You should convert all of your voltage and current values appropriately in order to make a plot of resistivity vs. temperature. From your plot, you should be able to determine several important properties of your sample and use this information to determine the composition of your sample and how it compares to other superconductors and to copper, for example. These properties include: (1) the critical temperature and from this temperature the most likely composition of the material; (2) the shape of the normal state resistivity vs. temperature curve and how this compares to a metal; (3) the value of the resistivity at room temperature and how this compares to a metal (for example, copper).

EQUIPMENT:

1. EG & G 5209 Lock-In amplifier
2. Two BNC to banana plug connectors (to facilitate differential input mode)
3. One banana plug extender (to facilitate connecting BNC cable to multimeter)
4. Two Fluke 45 multimeters
5. 1-liter dewar
6. Liquid nitrogen
7. Appropriate BNC and banana plug leads
8. Small copper rod
9. Calipers
10. Hardware for mounting probe to table