Resistivity vs. Temperature of a Superconductor

1.0 Expected Learning Outcomes

• Calibrate a transducer (silicon diode) for low-temperature measurements and measure temperatures down to that of boiling liquid nitrogen.

• Accurately measure very low voltages, including techniques for reducing or compensating for electronic noise.

• Identify the source of and compensate for systematic error in measurements.

• Examine the behavior of a high-$T_c$ superconductor sample during the transition between the superconducting and normally-conducting states.

• Report the results of your measurements in a format appropriate for submission to a journal for publication.

2.0 Introduction

WARNING

Take extreme caution when handling the superconductor lead wires.

• Gently push the cable through the brass cap, being careful that you don’t damage the connectors on the end of the cable and that you don’t kink or unnecessarily flex the cable.

• Put a thin coating of the anti-seize compound on the threads of the superconductor assembly before you screw it into the aluminum heater block. This will ease the process when you eventually remove the superconductor from the heater block.

• Screw the superconductor assembly into and out of the heater block using a large screwdriver in the back slot. Never twist it in or out by the wires. The small yellow or gray screwdrivers are not appropriate for inserting or removing the superconductor from the block.
• It is usually good to use the screwdriver to hold the superconductor assembly stationary with respect to the room and rotate the aluminum heater block when you assemble or disassemble them. This will avoid the problem of wrapping the wires around the screwdriver and reduces the likelihood that you will break one or more wires during the assembly/disassembly process.

• *Never, never, NEVER,* bend the superconductor lead wires when frozen.

In this lab, you will measure the resistivity of a high-critical-temperature superconductor as a function of temperature. These amazing materials have a resistivity that drops to zero below a certain critical temperature. The high-critical-temperature material we will examine is (Bi,Pb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_{\delta}$ or Bi$_{2-\epsilon}$Pb$_\epsilon$Sr$_2$Ca$_{y-1}$Cu$_y$O$_{\delta}$. This is often written as just BiPbSrCaCuO or BSCCO (pronounced “bisco”) for short. This material has a critical temperature about 30 K above the temperature of boiling liquid nitrogen, a very high critical temperature. This high critical temperature makes it quite technologically relevant. Several companies are producing commercial products from this material including mammoth electrical motors for the Navy.

3.0 Equipment

In addition to the equipment used for the Thermal Measurements Lab, the following equipment will be available.

Each laboratory group will be given:

A superconductor assembly including a sample of BSCCO material, a 1N4148 temperature measurement diode, and color-coded connection wires. The assembly is mounted on an aluminum post using heat shrink tubing to hold it place. The post has 3/8-24 NF threads to facilitate mounting inside the aluminum heater block. You will retain the same assembly throughout the experiment.

In the storage cabinet in the lab:

Superconductor interface cables with three color-coded BNC connectors on one end and a DB-9 connector on the other end to accept the connection pins on the superconductor assembly. Connector pin assignments are shown in Figure 2 and Figure 3.

4.0 Grading

• Build a constant current circuit for resistivity measurements – 10 pts.
• Calibrate the diode in the superconductor assembly for low-temperature measurements – 10 pts.
• Measure the resistivity of the superconductor as a function of temperature – 20 pts.
• Lab notebook – 40 pts.

You will also write a formal report, (also on Learning Suite in Content ⇒ Superconductivity Measurements Lab ⇒ The Formal Paper ⇒ Formal Superconductor Report instructions) similar to a journal article, of your results. This report will count as 10% of your final grade broken up as follows.

• Annotated bibliography – 10%
• First (not rough) draft – 40%
• Final draft – 50%

The points for each of the report drafts will be divided roughly as follows.

• Content: completeness, relevancy, and insight – 50%
• Style: clarity, flow, and appropriateness for genre – 30%

4.1 Literature search

When performing scientific research, you should always start with a literature search. This means that you will search the journals for similar measurements or calculations to see what has been previously done. This will both guide your research as well as help you avoid unnecessarily repeating previous research.

The material we are using is very well developed and commercially produced by several companies. However, for the purposes of our exercise we will assume that we are working on a relatively new material and examine the literature from the period around 1990 as this material was being initially investigated.

From the research literature (i.e., journals, not web pages) find at least five articles that discuss measurements of resistivity vs. temperature on this superconductor (BSCCO). These articles will be used in your annotated bibliography discussed in “Superconductor Formal Report” and on Learning Suite under Content ⇒ Superconductivity Measurements Lab ⇒ The Formal Paper.
You should note the behavior of the resistivity, \( \rho(T) \), and the temperatures and conditions involved. There is some terminology that varies between papers. Be sure you know how a particular author has used the terms “transition temperature” and “critical temperature.” This will give you an idea of what to expect in your measurements and how other groups have been measuring the transition temperature of this material.

5.0 Preparation for the measurement

There are several steps that need to be taken before you can begin your measurements. It is strongly recommended that you examine and follow, the checklist given in “Some important hints for making the superconductor measurements” and found in Learning Suite under Content ⇒ Superconductivity Measurements Lab. It will help ensure that you will have valid data after completing your measurements – and will help you avoid damaging your sample.

5.1 Preparing your sample

Your superconductor samples are mounted on aluminum sample holders. A diode is mounted next to the superconductor to measure temperature. Fig. 1 illustrates the arrangement of the superconductor sample and the temperature measurement diode as they are placed on the aluminum mounting post. Fig. 2 illustrates the connections of the leads to the sample and the diode. Fig. 3 shows the pin assignments on the adapter cable.

5.2 Calibrating the diode for temperature measurement

You will need to calibrate the diode mounted with your superconductor sample using the same technique you used in the previous labs. You can use the same LabVIEW VI to acquire the calibration data as you used in the Thermal Lab.

The calibration constants will be near those found in the Thermal Lab but not exactly since you are using a different diode. Note that you DO NOT put the superconductor sample in boiling water. You will only calibrate using ice water and boiling liquid nitrogen. Do not put the superconductor sample assembly directly in water – it must be in a plastic bag or you will have to wait for all the water to evaporate out of the assembly before continuing.
Figure 1: Photograph of a superconductor sample. The sample is a rectangular prism with four leads to facilitate the measurement of the resistance using the four-lead measurement technique. The diode is in close thermal contact with the superconductor sample to allow reasonably accurate measurement of the sample temperature.

5.3 A constant current supply for measuring the resistance

If you have followed the hints for the superconductor measurements, you will notice that the measured resistance of the sample is difficult to determine with an ohmmeter. To make a real measurement of the resistance, you will need to use the four-lead method. Put a measured current through the outer two leads (white and black), measure the voltage across the inner two leads (orange and red), and compute the resistance. This removes the problem of contact resistance and contact voltage.

To make this measurement, you need to have a known current in the sample. Measuring current with LabVIEW is a little involved because most data acquisition hardware can only measure voltages. Using a known constant current will make it unnecessary to use LabVIEW to monitor that current.

5.3.1 Building your own constant-current supply

You have already learned how to make a constant-current source. It is fairly simple
to **add a second constant-current supply** to the existing $\approx 100 \mu A$ source you already built for measuring the temperature using a diode. You will still need the first supply to measure the temperature using the diode packaged inside the superconductor assembly. This second supply should provide a current somewhere in the $10 – 50 \text{ mA}$ range. Do not exceed 100 mA or you will burn out the 2N3906 transistor in the supply.

The second current supply can use the other half of the TL3472 op amp (you only used one of the two amplifiers for the 100 $\mu A$ supply you already built). You can also just use a jumper to connect the same voltage divider for the reference voltage on both supplies.

You will have **two separate output stages** consisting of R3, a 2N3906 transistor, C3, and C4, and the output BNC connector. The only shared components between the two supplies are the TL3472 op amp, C1, and the voltage divider for the reference voltage. The value of R3 will be different for the two supplies to provide the different output currents.
Figure 3: The connections for the adapter cable. The notation in parenthesis on each connection indicate whether it is the center conductor or the shield on the attached coaxial cable. The shield is the outer conductor which is connected to the outer shell on a BNC connector. The constant current for the four-lead measurement of the resistance is applied to the white/black cable. The voltage is measured across the orange/red cable. The yellow/violet cable is used to measure the temperature using the 1N4148 diode attached to the superconductor.

5.3.2 Using an external constant-current supply

If you have access to an external power supply that can be operated in constant-current mode, it is possible to use that supply to provide the current for the four-lead resistance measurement. You should verify the stability of the output current by monitoring it for a period roughly equal to that required to complete the temperature scan on your sample.

At present, the power supplies available in the Physics 240 lab are not sufficiently stable to be used unless you have your LabVIEW VI constantly monitor the current through as well as the voltage across your superconductor sample.

Be sure that you understand the grounding situation on this power supply so you can set the grounding switch on the data acquisition system correctly.
6.0 Acquiring the data

Create a LabVIEW VI (Virtual Instrument) that will simultaneously measure the temperature and the resistance of the superconductor as it warms up from the temperature of boiling liquid nitrogen to room temperature. Your program should take an average over a short period to reduce noise (of course). The voltage across the superconductor sample is very small, and you will hopefully be taking the sample to the superconducting state where this voltage is zero. You will need to be sure that the range specified in your analog input VI is appropriate for the signals. Your VI should also save the data into a spreadsheet file to allow for analysis and reproduction.

After calibrating the temperature-sensing diode, you are ready to make the resistivity measurement. Put the superconductor in the cryostat heater block (see warnings above) to reduce temperature gradients across the sample and to mechanically protect the sample and the leads.

You will use one of the Styrofoam cups to contain the liquid nitrogen. It will be necessary to submerge the heater block almost completely in the liquid nitrogen to get the sample sufficiently cold.

When you are allowing the sample to warm up (as you are taking data), you may want to apply a little heater power to reduce the time required for some portions of this measurement.

You may notice that the title of this handout includes the word “Resistivity” rather than “Resistance.” The results are usually given as resistivity rather than resistance because the resistance of an object depends on its geometry whereas the resistivity only depends on the intrinsic characteristics of the material. Resistivity (\( \rho \)) is related to the resistance (\( R \)) of the material by the equation \( R = \rho L/A \) where \( L \) is the distance between the voltage probe electrodes and \( A \) is the cross-sectional area of the sample. Your final results should be given in terms of resistivity.

The dimensions for the individual superconductor samples are not consistent. We used the dimensions from a collection of ten samples and found that the cross-section is approximately \( 3.01 \pm 0.04 \text{ mm} \) by \( 2.04 \pm 0.012 \text{ mm} \) with about \( 9.47 \pm 0.09 \text{ mm} \) between the inner contact rings. For example, the range for the distance between the contact rings varied from \( 9.35 \text{ mm} \) to \( 9.59 \text{ mm} \). You will need to assume that these values are appropriate for your sample.

1. Your first goal is to survey a large temperature range. Measure \( \rho(T) \) from \( 75 \text{ K} \) to \( 280 \text{ K} \). This does not need to be done by scanning slowly. You should be able to find roughly the superconducting transition temperature (where resistivity goes to zero).

It will likely be necessary to immerse the heater block/superconductor assembly completely in the liquid nitrogen to get below \( 80 \text{ K} \).
2. Now do a slow scan from about 75 K to about 130 K (you can start a little lower and go a little higher). The scan speed should be \( \leq 0.5 \text{ K/sec} \). You can do this by insulating the cryostat better, placing it in the vapor from the liquid nitrogen, or with whatever you method you can come up. Scanning slowly will reduce temperature gradients across the sample and reduce the temperature difference between the diode and the sample to improve the accuracy of your measurement. Measure the resistivity curve at least twice to check for reproducibility.

### 7.0 Analysis to prepare to write your formal report

Several items to consider as you analyze your results:

1. This superconductor is BiPbSrCaCuO, primarily in the 2223 phase although it may have some 2212 phase present. The “2223” specifies the rough ratio of the different elements in the superconductor (compare the formula given at the start of this handout). As far as we know these samples are doped with Pb to increase the fraction of the 2223 phase but the doping fraction is unknown.

2. How do your results compare with those given in the papers you consulted in your literature search? Can you determine anything about your sample by comparing your results with those from other groups?

3. An ideal conventional superconductor will have an abrupt change in \( \rho(T) \) at the transition temperature. Is the change in your sample abrupt? If not, do you think it is due to your measurement (such as not being able to resolve a fast change in \( \rho \), or change \( T \) slowly enough), or is it a true property of the sample? (Hint: As noted above, there are several different phases of BSCCO, and it’s possible that you have more than one phase present in your sample.)

4. What is the behavior of \( \rho(T) \) above the transition temperature? Does it behave like a metal or a semiconductor? Find a fit for the behavior in this region. It is typically not possible to fit an analytic solution to the superconducting transition region because there is not a good analytical model for that region.