

Physics 338 Advanced Physics Laboratory
Superconductivity
Due Tuesday, May 7, 2024, 5:00 p.m.

Safety

Liquid Nitrogen

You will use liquid nitrogen in this experiment. You can **seriously** injure yourself with liquid nitrogen. Here are a few specific cautions, but more generally, you should follow good safety practices, pay attention to your surroundings, and use common sense.

- Wear insulating gloves at all times when handling liquid nitrogen.
- Wear safety glasses at all times while near containers with liquid nitrogen.
- Do not let the liquid nitrogen, or objects that have recently been in liquid nitrogen, touch your skin or clothing.
- Be careful not to splash the liquid nitrogen when pouring. Pour it very slowly.
- Do not store liquid nitrogen in a container with a tight-fitting lid.
- Do not pour the liquid nitrogen on any surfaces except those designed for it. Most surfaces will be damaged by the extreme cold.
- Do not touch the brass sample holder with your bare skin. It will stay dangerously cold for a long time even after you pull it out of the liquid nitrogen.

Chemical Safety

The superconducting disks are made from materials that are toxic if ingested. You should not need to touch the disks at all, but if you do, you should wash your hands.

1 Introduction

High T_C superconductors, with transition temperatures above the liquid nitrogen liquification point of 77 K, have opened the door to classroom demonstrations of superconducting phenomena. We will observe the transition to the superconducting state in two cuprous oxide ceramics, $\text{YBa}_2\text{Cu}_3\text{O}_7$ (YBCO) and $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_9$ (BSCCO). (n is typically 2 or 3, depending on the manner in which the material is made. A typical polycrystalline sample will contain a mixture of both types.) More information about the chemistry and fabrication of these materials is in Colorado Superconductor's "Experiment Guide for Superconductor Demonstrations."

2 The Four Point Probe

The two superconducting samples have been outfitted with a four point current probe which eliminates the effects of contact resistance in measurements of the resistance of the superconducting sample. The probe is shown schematically in Figure 1. A DC power supply causes a constant current to flow the length of the sample through (black) probes labeled 1 and 4 in the figure. The voltage drop across the sample is read from (yellow) probes 2 and 3 (in reality, probe 2 is as close as possible without touching to probe 1, and probe 3 is similarly close to probe 4). Then the high impedance of the voltmeter prohibits any significant current flow through the leads attached to probes 2 and 3 so that their resistance does not contaminate the measurement.

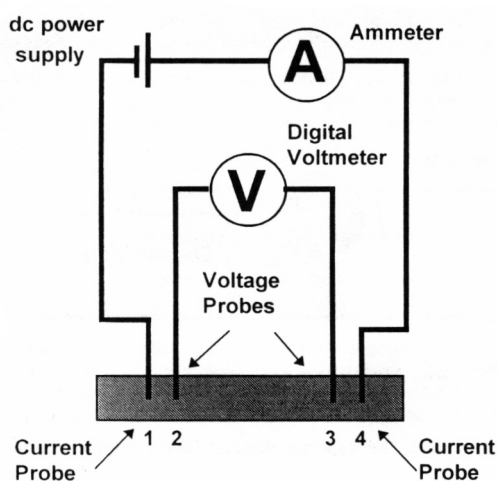


Figure 1: Schematic of Four Point Probe

The four point probe devices are encapsulated in rugged brass casings. On one side of the casing, the superconductor disk is visible. An aluminum end cap has been inserted into the back side of the brass casing to seal and to protect the probe connections to the superconductor. Please do not attempt to remove the end cap. A matched thermocouple has also been attached to the superconductor in this casing. The $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_9$ superconductor has the larger casing and is stamped "B" while the $\text{YBa}_2\text{Cu}_3\text{O}_7$ superconductor is stamped "Y". The pair of black wires are current leads connected to probes 1 and 4, while the yellow wires are attached to probes 2 and 3 for the voltage measurement. The red and blue wires are leads for the thermocouple which is used to measure the temperature of the sample.

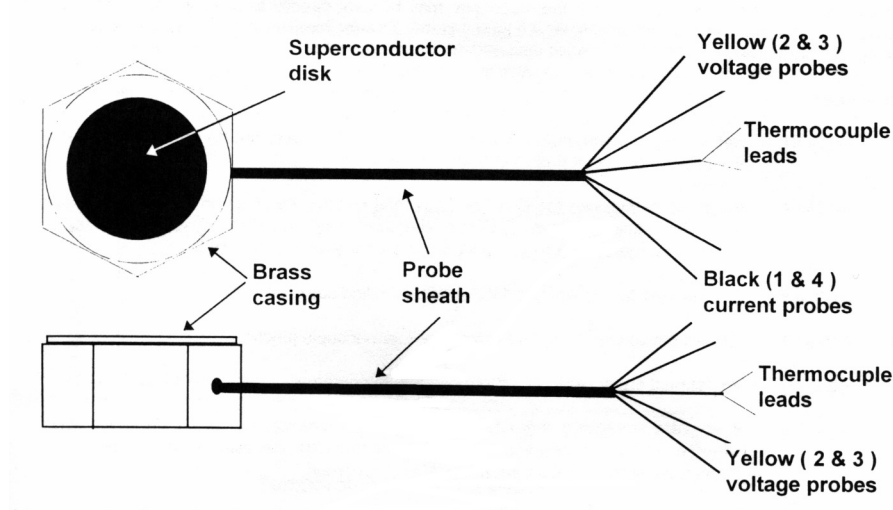


Figure 2: Leads for the Four Point Probe

3 Measuring Temperature With a Thermocouple

A thermocouple consists of a mechanical junction of two dissimilar metals. This junction generates a small electrical potential, the value of which depends upon the temperature of the junction. With an appropriate choice of metals and proper calibration, one can obtain a thermometer for the desired temperature range. For the range of interest here, room temperature (300 K) down to the liquification point of nitrogen (77 K), a type T, or Copper-Constantan, thermocouple is used. A digital millivoltmeter attached to the red and blue leads reads the potential difference across the junction, and that reading can be converted to temperature using a conversion table. The Keithley DMM 2000 multimeters have several standard conversion tables built in, so the meter can display the temperature directly.

Note that the thermocouple leads must be connected to the voltmeter with wires of the same material, and the connection between those wires and the thermocouple leads must be at room temperature for the calibration to be correct. This calibration will be one of the main sources of error in this experiment, but a proper calibration procedure would be significantly more involved than we need for this lab.

4 Superconducting Critical Temperature

The critical temperature can be measured by observing the point at which the sample resistance becomes immeasurably small while cooling, or, equivalently, by measuring the

temperature at which the resistance suddenly jumps upon warming. As a practical matter, it is easier to warm slowly in a controlled manner, so that is the transition we will use.

Follow the following procedure for each superconductor sample:

1. Connect a constant-current power supply the black leads. Any supply with a current-limiting mode ought to work fine. If the power supply does not have a digital current readout, use a Keithley multimeter set to DC I to read the current. The maximum safe current is 0.5 A, but a value of half that is plenty for this experiment. Set the current knob to zero and turn the voltage knob about 1/4 of the way up. *Slowly* increase the current knob until a current of about 0.25 A flows. The value will fluctuate a little as the power supply warms up, but ought to settle down.
2. Connect the yellow leads to a Keithley 2000 DMM. Set this to read DC Volts. If you get a negative voltage, swap the leads. In order to read the values on the computer, you may need to use the front panel to enter the GPIB menu (use the **Shift** key), set the address to 14, and the **Lang** to SCPI. (Move within a menu entry with the left and right arrow keys, change values with the up and down arrow keys, and press **Enter** to enter a value.)
3. Connect the thermocouple leads to a second Keithley 2000 DMM. Connect the red lead to the copper (blue) wire and the black lead to the silver (red) wire. Set the DMM to read Temperature. Use the **Shift Tcouple** front panel keys to set the units to Kelvin, the **Type** to T, and set **Junc Sim** to 294. (Replace this value with a better estimate of room temperature.) In order to read the values on the computer, you may need to use the front panel to enter the GPIB menu (use the **Shift** key), set the address to 15, and the **Lang** to SCPI.
4. Gently place the whole device in a small dewar. Place the dewar inside a larger stainless steel can (to contain spills).
5. On the computer, launch the **superconductivity** LabView program. Enter the current in the input current box.
6. It will record the resistance and temperature repeatedly. You can change the delay between data points by changing the **delay** box. A value of 0.5 s works well.
7. Select a **.txt** file name to store the final data.
8. Wear safety glasses and gloves whenever handling liquid nitrogen. *Slowly and carefully* pour nitrogen into the dewar. It will boil vigorously. That's ok, but add the liquid *slowly and carefully* to try to avoid splashing. Do *not* cover the sample with liquid nitrogen—it will take too long to all boil off!

Watch the temperature. Once it gets below about 85 K, the sample is sufficiently cold.

9. Check that the current I_{14} is still constant and that the value entered into the computer program is still correct. Wait until the boiling subsides. There should still be a small amount of liquid at the bottom of the dewar.
10. While still wearing the gloves, add some foam insulation to the top of the dewar and press it down gently.
11. The thermocouple should read 77 K, but likely will not because we have not calibrated our thermocouple junction carefully. That's ok; record the temperature actually reported by the thermocouple.
12. Start the computer program to record the temperature and resistance. You start the program by pressing the small arrow on the top toolbar. The program will run as long as the big green button is green. When you press the green button, the program will stop and prompt you for a place to store the output file, unless you have already entered the filename above. To start the program again, you have to press the big button again and then press the small run arrow on the top toolbar.
13. There will be a fairly sharp jump in resistance when the sample warms above the transition temperature. Since the sample is not a single uniform crystal, there actually will be a small range of transition temperatures. In addition if there are any temperature gradients in the sample, not all parts will go through the transition temperature at the same time. In this experiment, the transition typically takes place over a range of about 5 K.
14. Put on the gloves and pull the sample out of the dewar and place it in the tupperware with the dessicant beads.
15. Once it is closer to room temperature, carefully dry the sample with a hair dryer and place it back in the tupperware with the dessicant beads.
16. Repeat the procedure for the other sample.

5 Analysis

1. Plot resistance vs. temperature for the warming portion of your data. (Delete any extraneous data before making your final plot.)
2. In this experiment, the transition typically takes place over a range of about 5 K. Make your best estimate for the transition temperatures for each of your samples. How do they compare with published values?
 - (a) You can sometimes improve your estimate of T_c by fitting a straight line to curve shortly above the transition and extrapolating back to $R = 0$.

- (b) Note that the value for $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_9$ depends on the n value. The samples supplied by Colorado superconductor are designed to be mostly $n = 3$, but likely have a modest amount of $n = 2$ as well. This will likely lead to a much broader transition. Is that consistent with your observations?
- (c) Though you can search for published values through the “Web of Science” through the Lafayette library home page, you may find it difficult to wade through all the citations. The Wikipedia references for “BSCCO” and “YBCO” are pretty helpful. A relatively recent *CRC Handbook of Chemistry and Physics* (available in room HSC-033) also has tabulated values for many superconducting compounds. Finally, the “Experiment Guide” posted on Moodle has many more details.
3. Initially, the sample warmed very slowly. However, as soon as it reached the transition temperature, the temperature rose more rapidly. Why do you think that is?