University of Michigan **Physics 441-442**
February 22, 2006 **Advanced Physics Laboratory**

"High Temperature" Superconductors

1. Introduction

In this experiment you will investigate some of the properties of a superconductor that becomes superconducting in liquid nitrogen. Until about 1986 the only superconducting materials required temperatures below 35°K to operate. Starting in 1986 new classes of superconductors were discovered with transition temperatures above 90°K; the highest so far is a compound of thallium, strontium, barium, copper and oxygen with a transition temperature of 125°K.

As a practical matter, 77°K, the boiling point of liquid nitrogen, is an important milestone because liquid nitrogen is relatively cheap and readily available. Thus the possibility of large-scale use of superconducting devices can be envisaged. However, the high-temperature materials found so far are all hard and brittle ceramics, and it is difficult to fabricate them into wire required for large magnets. It also appears that the materials are not robust and can lose their superconducting properties if exposed to moisture or even air.

In this experiment you will use a "kit" produced by Colorado Superconductor Inc. (CSI) containing high temperature superconductors and rare earth permanent magnets to study some essential properties. The experiments and considerable background information are provided in the CSI manual, which also includes a table to convert the thermocouple readings to temperatures.

2. Safety Considerations and handling precautions

Liquid nitrogen can cause serious damage to the eyes and skin. Always wear glasses or safety goggles when working with it. If the safety goggles are not with the kit, ask for them. Always handle the superconductors with the tweezers provided.

Most heavy metal compounds are poisonous; this includes, in particular, the bismuth used in some of the superconductors. Wash you hands when you are through.

The superconducting samples are easily damaged by moisture. When you are finished for the day, dry them gently and thoroughly with a hair dryer or heat gun. Then wrap them tightly in the plastic bag containing the desiccant.

3. The Meissner Effect

At a temperature below its critical temperature T_c, a superconductor will not allow any magnetic field to enter it. This is called the Meissner effect, and is due to magnetic dipoles induced on the surface of the superconductor. The induced field repels the source of the applied field. If it is a small permanent magnet it can be made to "float" over the surface of the superconductor. This provides a dramatic demonstration of superconductivity and allows a simple means of measuring the critical temperature.

The kit contains two black discs which are the superconducting samples. One of these is embedded in a brass and aluminum housing with 6 wires coming out of it; this is used for the resistance measurements described below. The kit also contains two small rare earth permanent magnets; one is cubical, the other cylindrical. These are easy to lose, so tape them to the case when not in use.

First observe the Meissner effect. Carefully pour some liquid nitrogen into a shallow Styrofoam dish (the circular hole in the cover of the kit words fine). Use enough liquid to almost cover the disc. Use the tweezers to set the disc in the liquid. As it cools, the liquid with boil vigorously. After it settles down, use the tweezers to set one of the rare earth magnets on the center of the disc. Once the disc becomes superconducting the magnet will levitate over the disc. The cylindrical magnet will spin for a long time once it is set into rotation.

You can use the Meissner effect to measure the critical temperature. The red and blue wires on the disc with the wires are connected to a thermocouple attached to the underside of the disc. The translation between voltage out of the thermocouple and temperature in °K is given in the table in the CSI manual appended. The voltages are typically a few millivolts, so use a sensitive digital voltmeter such as Keithley 177 to measure them.

Fill a styrofoam cup or small thermos cup about 2/3rds full with liquid nitrogen and immerse the disc. Use the wire harness to handle it. Try not to flex the connections to the disc. After several minutes the voltmeter should read about 6.42 mV indicating a temperature of 77°K for liquid nitrogen boiling at atmospheric pressure. Set the disc on the styrofoam cover, superconducting side up. Using tweezers, set the small cylindrical magnet on the disc and watch it carefully. As the superconductor warms, less of it remains superconducting. Record the temperature when it just stops spinning and use this as an estimate of T_c. Repeat this a couple of times and try the cubical magnet as well (note questions 2 & 3).

4. Measuring Resistance vs. Temperature with the 4-point probe

The other 4 wires coming out of the housing are connections to a "4-point probe". These provide a means for measuring the resistance accurately by eliminating the effect of contact resistance (see the appendix for more information). For these measurements a current of approx. 0.5 A is introduced on the <u>black</u> leads; the voltage across the superconductor is read from the <u>yellow</u> leads. The resistance is obtained from the ratio of the voltage to the current. It is convenient to apply the current from a constant current source. Generally higher currents will yield higher accuracy, but currents >0.5 A can destroy the superconductor. Use a current source which can supply up to 0.2 A. Before starting, check the resistance between the two yellow leads and the two black leads with an ohmmeter. The resistance should be ~1 ohm; if it is much larger, the probe is no good. Hook the current source to the two black leads. First temporarily hook a digital current meter in series with the current source and set the current to 0.200 A. Once this is done the current should remain constant, and the current meter can be removed from the circuit. Use one digital voltmeter to monitor the

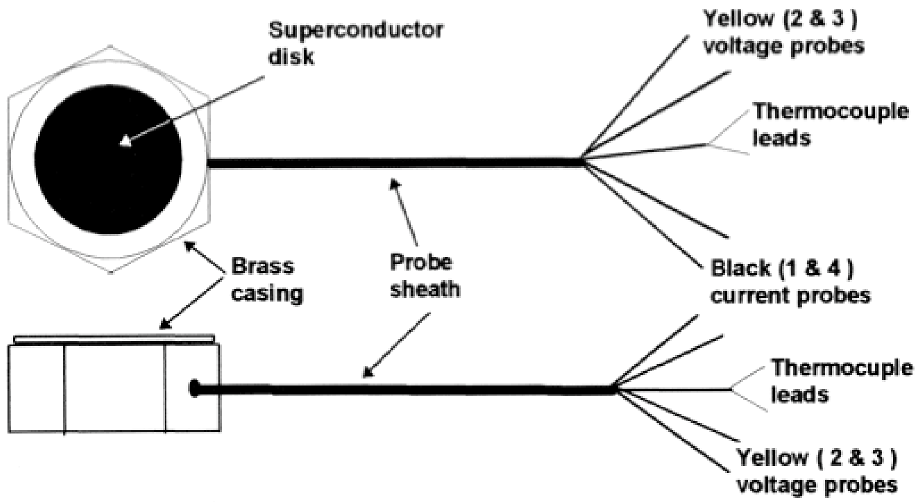

Figure 1 – The CSI "4-point probe"

temperature using the red-blue leads as before. Use a second one to measure the voltage across the superconductor V_{23} with the yellow leads.

At room temperature the temperature voltmeter should read very close to 0 mV and V_{23} should be approx. 0.3 mV with a current of 0.200 A. Note that these voltages are very small so care must be used in measuring them. Set the zero on the voltmeter by reversing the input leads to the meter and adjust the zero so that you get the same reading with either orientation. Note that you are not using a second thermocouple in an ice water bath to compare with the thermocouple in the probe, so that your temperature measurement will be affected somewhat by the temperature at the terminals of the DVM, so try to maintain these at constant temperature.

Immerse the superconductor in liquid nitrogen. After it settles down measure the voltages. Now V_{23} should be very close to zero, indicating the very low resistance characteristic of superconductivity. Remove the sample from the nitrogen and set it on the styrofoam cover. It is not a bad idea to cover the disc with a paper towel to help maintain a more nearly constant temperature over the whole disc and reduce the rate at which the temperature rises. Monitor the voltages as the disc warms up and until the temperature reading gets back down to 5 MV or so. The most interesting data comes in the first few seconds so repeat the procedure a couple of times and refine your technique.

Use the thermocouple readings and the calibration table from the CSI manual to determine the temperatures. Make a plot of resistance vs. temperature and from it estimate the critical temperature.

5. Determining T_c with the Magnetic Susceptibility Probe

Another way to observe the transition to superconductivity is to study the magnetic susceptibility of the material. This measurement is based on the expulsion of a magnetic field by the superconducting sample (the Meissner Effect). A current introduced into the coil will generate a magnetic field. When cooled below the critical temperature, the sample expels the induced field, which can be seen by a distinct change in the inductance of the coil. Analysis of the data reveals a sharp transition point at the critical temperature, T_c, of the sample, and can also be used to compute the magnetic susceptibility of the superconductor.

The susceptibility probe consists of a 0.5-inch long coil of wire (approx. 400 turns) around a 0.5-inch diameter superconductor rod. A thermocouple is also attached for temperature measurement.

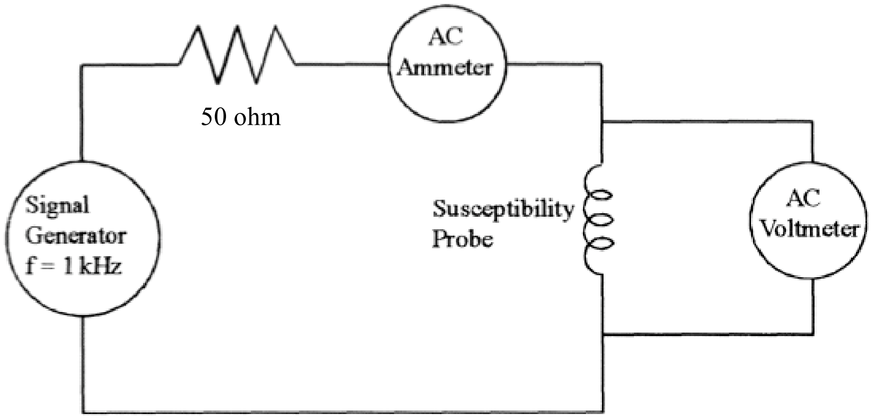

Figure 2 – Schematic for magnetic susceptibility measurement

Set up the apparatus as shown in Figure 2. The current can also be monitored by measuring the voltage across the 50 ohm resistor, rather than the AC ammeter. Set the signal generator to give a sine wave at a frequency of approximately 1 kHz (integer multiples of 60 Hz should be avoided). Carefully straighten the thermocouple leads. Attach them to a dc voltmeter with a precision of at least 0.01 mV. .

Insert the susceptibility probe into a styrofoam cup. Fill the cryostat with enough sand or Drierite to completely cover the probe. Add liquid nitrogen slowly until the temperature is below 80 °K. The thermocouple should read about -0.16 mV at room temperature and about +6.42 mV in liquid nitrogen.

Allow the sample to warm slowly. Periodically record the current (I), the voltage across the coil (V_L), and the thermocouple temperature, until the sample warms to about 120 °K. As the sample reaches the critical temperature, the voltage across the inductor (V_L) will increase dramatically. A slower warming rate at this point is advantageous in order to obtain accurate measurements.

The resistance of the coil (R_L) is temperature dependent, therefore we need to determine R_L as a function of temperature. This can be accomplished by simply measuring the DC resistance of the coil vs. temperature using an ohmmeter with the same procedure as above.

ANALYSIS OF DATA

The critical temperature (Tc) at which the superconducting transition takes place can be determined by plotting the inductance of the coil vs. temperature. The impedance of the coil (ωL) can be computed using the following equation:

$$\omega L = [(V_L/I)^2 - R_L^2]^{1/2}$$

where V_L is the voltage across the inductance (the ac voltage measured across the black coil leads), and R_L is the resistance of the coil, which was determined by DC measurements. Make sure you measure V_L and I consistently (*i.e.,* peak or rms values).

An ideal superconductor screens the B-field completely at B-fields lower than the critical field. This makes a superconductor perfectly diamagnetic and thus the magnetic susceptibility (χ) is equal to -1. If the coil contains a sample with χ not equal to -1, the magnetic flux through the coil will change, resulting in a change in the inductance of the coil. The inductance of a coil measured in a medium of susceptibility χ is given by:

$$L = L_0(1+\chi)$$

where L_0 is the inductance of the coil in a vacuum. The susceptibility is small when the sample is warm, so that L_0 is approximately the inductance you find when the probe is warm.

This equation holds for any sample provided that the sample occupies all the space in which the coil produces a field. Since our sample occupies most of the volume (sample diameter = 1.26 cm, coil diameter = 1.36 cm) we can approximate the inductance (L) by adjusting the above equation to account for this difference:

$$L = L_0(1+f\chi)$$

where f is the fraction of the coil volume occupied by the sample. It follows that χ is given by the equation:

$$\chi = (L/L_0 - 1)/f$$

Calculate χ with the data collected in the previous exercises and plot χ *vs.* T. You will notice that the plot yields the same type of graph as ωL, as well as the same critical temperature.

Although the superconducting transition is clearly visible, the susceptibility in the superconducting state is not exactly -1. This is due in part to geometrical corrections that were neglected in our equation for χ, which can result in an error in susceptibility.

6. Effect of Magnetic Fields on the Superconducting Transition

If the external magnetic field is sufficiently large, the field will penetrate into the material. This limits the superconducting regime to environments with sufficiently low temperature and lowish magnetic fields known as Meissner phase. The magnetic field at which the Meissner effect breaks down is known as the critical field, H_C. Type II materials, such as the high T_C superconductors, exhibit partial Meissner breakdown at the critical field and allow partial field penetration, which increases with field strength.

Polycrystalline samples of $YBa_2Cu_3O_7$ contain grains of material with lower critical fields so that not all of the material will become superconducting at the normal critical temperature.

This can be verified by repeating the magnetic susceptibility measurements in the presence of a magnetic field. A large ferrite permanent magnet is available in the lab. Repeat the susceptibility measurements with the probe set as close as possible to the center of the ferrite slab. Afterwards, use a gaussmeter to measure the magnetic field at the approximate position of the probe. Compare your susceptibility curves to those without the magnetic field.

Questions

1. Why are "high temperature" superconductors potentially of great practical importance? What are some of the reasons this potential has not been realized?
2. When you set a magnet on the center of the superconducting disc it usually sits there in a stable equilibrium, while if you set it near the edge of the disc it gets pushed aside. Try to explain these effects.
3. You may have observed droplets of what looks like liquid nitrogen being attracted to the small rare earth magnets yet liquid nitrogen is not paramagnetic. Try to explain this effect (hint: this effect is more prominent after the liquid nitrogen is boiling for a while).
4. The discs only develop a layer of frost after the liquid nitrogen boils away. Why is this?
5. Why is the transition in resistance gradual at the critical temperature?
6. Are your results for the magnetic susceptibility consistent with $\chi = -1$ below T_C? If not, discuss reasons why they may not be.
7. Compare your magnetic susceptibility curves with and without the external magnetic field. Search the web for data on H_C for YBCO samples and compare this with the magnetic field measured in your configuration.
8. Magnetic susceptibility probes have many practical uses. Do a bit of research and briefly discuss their practical applications

References

1. W. Hiller and K. Kopitzki, Physica **C174**, 467 (1991) (Experimental data and discussion of inverse AC Josephson effect in high temperature superconductors).
2. J.T. Chen, *et al.*, Phys. Rev. **B5**, 1843 (1972) (Theoretical explanation of inverse AC Josephson effect).