Superconductivity
Rochester Institute of Technology

PHYS-316 Advanced Lab*

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Goals

In this experiment, you will observe the perfect magnetic flux exclusion, known as the Meissner effect, from a small superconducting disc. You will use a four-wire technique to simultaneously measure the voltage across and current through two samples: one is superconducting and the other is a normal conductor. You will calculate the resistance and determine the critical temperature T_c for the superconducting sample, and determine its identity.

Introduction

Do not remove the probes from their sand-filled buckets. Do not handle the superconducting samples directly with your hands. Wear gloves.

Be careful with the liquid nitrogen. Do not leave the dewar sitting on top of a table or bench.

Read the safety section and follow careful lab practices.

Do not exceed 200 mA of current through the samples. Make certain you set the limits appropriately on the power supply, and use the 91-Ω power resistor in-line.

Superconductivity is one of the quintessential topics of condensed matter physics. Like ferromagnetism, it is an example of the emergent behavior of strongly-correlated electron systems. As such it is a deeply quantum mechanical phenomena, and one that is only partially understood.

Superconductors are characterized by a critical temperature, T_c, below which they have zero resistance. In the broadest terms, there are two classes of superconductors. There are those with low values for T_C, typically 10 K or less, and those with much higher T_C. Some of the high-T_C materials at atmospheric pressures can get up to 150 K while still being superconductors.

For low-temperature superconductors there exist very elegant theories that capture the be-

*Prepared by L. Barton, M. Pierce, and A. McGowan havior astonishingly well. There are many native elements that are superconductors at low enough temperatures. More than half of all the transition metal elements have been shown to be superconductors.

High-temperature superconductors present many additional challenges. While they are easier to observe experimentally, in the case of the high- T_C materials there is no satisfactory theory of their behavior. It is likely that whomever develops a complete theory of high-T_C superconductivity will win a Nobel prize. The high-T_C superconductors are typically brittle ceramics that cannot easily be formed into wire, and are difficult to attach regular wire to.

The high-T_C superconductors are superconducting at liquid nitrogen temperature (77 K). This is an enormous improvement over the transition metals, that need more expensive liquid helium (4 K) to cool them. The next great challenge with superconductor material development is to find materials that demonstrate stable superconductivity at room temperature and pressure. This would be a major breakthrough, regardless of whether the behavior can be understood theoretically. Recently, sulfur hydride was demonstrated to possess a T_C of over 200 K. However the pressures involved were extreme at 150 GPa (1.5×10^{11} Pascals).

One of the major limitations in modern energy technology involves the losses due to transmission which could be mitigated through high-T_C materials. Being able to transmit electricity without significant power loss would be an incredibly huge technological development.

Background

There are several key characteristics of a superconductor:

1. There is a range of temperatures for which there is effectively no loss of current through the material. Researchers have demonstrated that a closed loop of superconducing material maintains a steady current for years without any decrease in current.

2. Magnetic fields do not penetrate into the material beyond a thin skin depth. In essence, as long as external magnetic fields are not too strong, a superconducting material will behave as a perfect diamagnet. The magnetic field $\vec{B}$ within the superconductor is exactly $\vec{0}$.

3. There is a critical temperature T_C below which the system behaves as a superconductor, and above which it will display normal properties.

The high-T_C material you will examine in this experiment is one of two different cuprates. The first is $YBa_2Cu_3O_7$. In this material the stoichiometric ratio of Y, Ba, and Cu is 1,2,3 and hence it is sometimes called a "123" superconductor. The number of Oxygen atoms present is key to the superconducting properties of the sample. It is also known as "YBCO". The structure is related to that of perovskites.

The other sample is Bi-Sr-Ca-Cu-oxide. Several different compounds are present in the sample, but the one giving rise to the properties we will observe is $BiSr_2Ca_2Cu_3O_{10}$. It will show superconducting properties at even higher temperatures than the YBCO sample. The abbreviation BSCCO frequently gets pronounced as "Bis - co".

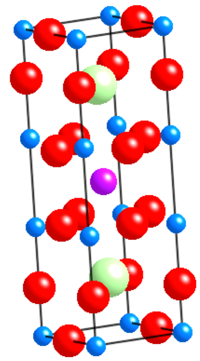

Figure 1: The unit cell of YBCO.

Safety Notes for Handling Liquid Nitrogen

Please read the general Liquid Nitrogen Hazard document, Section 1, before doing this experiment. If you will be working with liquid nitrogen in the future, then it is worthwhile to read the remainder of that document as well.

Liquid nitrogen (LN2) is at 77 K. It can seriously burn (freeze) you, or someone near you, if it is mishandled. It will adhere to your skin when circumstances are favorable.

Never leave a dewar of liquid nitrogen sitting on top of something like a lab bench. This leaves the chance that it can tip over, spilling the contents suddenly.

Always use proper cryo gloves to handle the dewar and eye protection to prevent boil-off drops from splashing into your eyes. It will boil violently when something warm (anything not at 77 K) is placed in contact with the LN2!

Do not use metallic tongs to transfer samples into or out of LN2. You can burn yourself and very likely drop the sample.

Ask questions if you are unsure of proper handling of the LN2.

Observing the Meissner Effect and Diamagnetism

The expulsion of magnetic fields from a superconductor, property 2 above, is known as the Meissner effect. Essentially the superconducting material becomes a perfect (or nearly perfect) diamagnet with a magnetic susceptibility of $\chi = -1$. In normal diamagnetic materials, the orbital spin of the electrons align in a way to oppose any applied field (following Lenz's Law), partially canceling or reducing the field inside the material. In a superconducting material it is quite different. Persistent screening currents manifest that oppose the external field and cancel it. Due to the perfect conductance, the screening currents can become as large as needed, thus completely eliminating the magnetic field from inside the superconducting material[1].

[1]This only works to a point. If you apply a large enough magnetic field, you can "break" the superconductor and force it back across the transition to a normal material. Observing the spontaneous formation and/or expulsion of the magnetic field from a material is a good way to detect the superconducting transition.

To begin, you will observe the Meissner effect for the unwired samples.

1. Place a small superconductor disc in a Styrofoam cup. Carefully pour just enough liquid nitrogen to cover the superconductor.

2. Wait until the boiling stops.

3. Remove the disc from the cup using the **plastic tweezers**.

4. Place the superconducting disc over the collection of magnets provided and observe what happens. Describe what you see. Sketch or take a photo for your notebook.

You can try the same thing with the pryolitic graphite sample (thin, shiny gray flakes) at room temperature. Graphite is an extremely good diamagnet even at room temperature, but not a perfect diamagnet like the superconductors. You should be able to observe the graphite to float, just barely, over the magnets.

Look up the relative permeability of pyrolytic graphite, and cite your source. What is the theoretical value for your cold disc in its superconducting state?

Measuring the Critical Temperature T_c

The basic high-T_c superconductor probe you will use looks like Figure 2. It is a small disc of ceramic material, pre-wired and protected in a brass holder. There are a total of six wires: two are thermocouple wires to measure temperature, two are black wires to run a small amount of current through, and two are yellow wires to measure the voltage drop across the sample. The same wiring is used in the standard conductor sample in the other bucket.

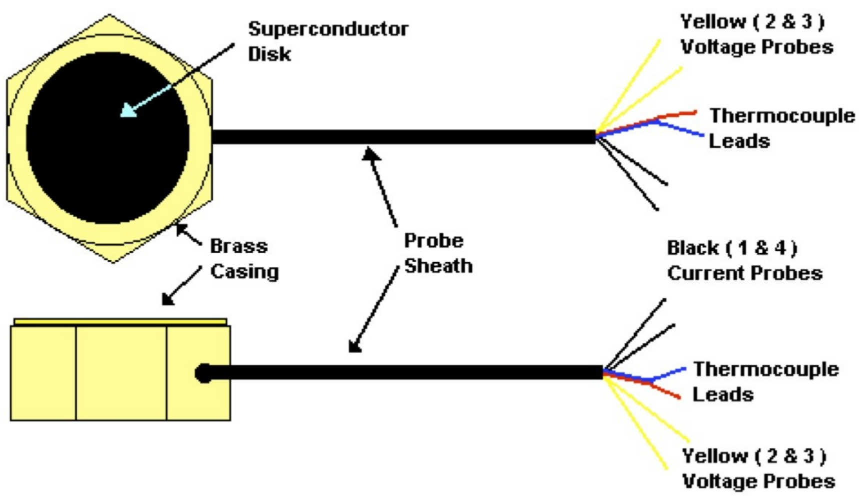

Figure 2: The superconducting discs have six leads attached: the red and blue leads of a type-T thermocouple, the two black leads for the input current ($<$ 200 mA), and the two yellow leads for sensing voltage.

Find and identify the thermocouple reader, power supply, and voltmeter you will use for the experiments. Verify that the thermocouple reader is set to the same probe type as your thermocouple plug label (type K or T).

The probe apparatus itself is kept in a sand-filled jar, which in turn is kept in a bucket filled with some insulation material. The thermocouple itself is a junction of two dissimilar metals, in this case Copper and Constantan. At the junction there is a small voltage difference of around -0.16 mV. As the temperature changes, this voltage difference changes. This combination, known as a type-T thermocouple, is fairly reliable in the range of 77 K up to room temperature.[2]

The reason to use a 4-point probe, where we both apply a current and measure a potential drop, is to eliminate the effects of contact and lead resistance. Normally contact resistance, and the resistance of the leads going to the device, is too small to present a noticeable effect. Due to the nature of superconductors it would produce a sizable effect, obscuring the physical phenomena we wish to study. You may want to ask yourself why the voltage probe itself does not appreciably impact the measurements.

Because you will measure small changes to an already small voltage, you should be using one of the precise Keithley multimeters. A standard DVM is sufficient to measure the current. Likewise you should verify that all wiring connections are secure. Loose or lightly-touching wires can lead to spurious results when you measure small voltages.

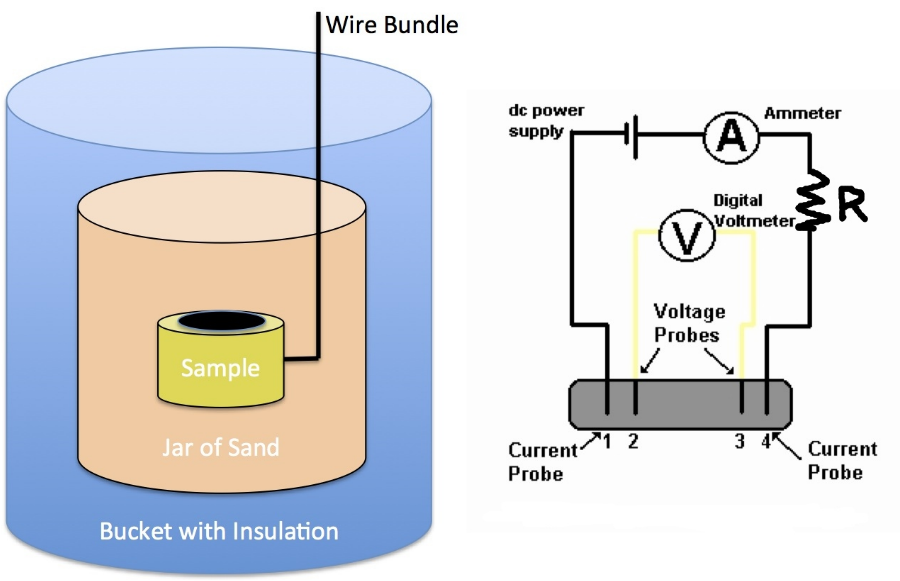

Figure 3: The circuit you need to build has an ammeter and a power resistor in series with the wired sample. This will help you keep the current to < 200 mA. The two yellow leads are for sensing voltage.

Your goals are:

1. Measure the profile of the resistance versus temperature of both samples.

2. Identify which sample is the superconductor.

[2]It is important not to confuse the settings on the thermocouple reader, or the temperature units you are measuring. The thermocouple of you have is type-T (blue), while the most common in a physics lab is type-K (yellow). Be sure your thermocouple reader is indeed set to "T", and that you know your temperature scale (C or K). Don't confuse reading a type-T thermocouple in Kelvin, with having the thermocouple reader set to read a type-K probe.

3. Estimate the critical temperature T_c for this sample (the bottom of the S-shaped curve, where resistance is zero for colder temperatures, and nonzero for higher temperatures), research high-T superconductors, and identify our sample; as described above, it is either YBCO or BSCCO.

Week 1: Video/Manual Data Collection

You should collect at least two trials of cooling and warming data for each bucket. **It is recommended that you record a video of all measurement devices for all trials of your experiment, as the temperature changes may be too rapid to manually record temperature, voltage, and current simultaneously.**

Check the temperature of both samples before you begin; it is advantageous to start with a cold sample if a previous lab group cooled it recently. Using your chosen sample, wire things together, being very careful not to exceed 200 mA of current. Also verify that the thermocouple is reading in units of C (or K) and is reasonable.

When you are ready, have your instructor verify your circuit setup, and pour the liquid nitrogen into the jar of sand to begin cooling. During this process you should notice the temperature and resistance drop. There may be significant thermal gradients present within the sample and jar that prevent you from taking meaningful data right from the start. Eventually, after a few doses of LN2, the temperature will get below 80 K reliably. At this point you will need to wait for it to begin warming.

To take data, record the voltage drop across the sample, and current flowing through the sample, as a function of temperature. You will find a region with almost no change in voltage vs. temperature, a region with a rapid rise, and a region of slowly rising resistance with temperature. You should record the resistance in small steps, especially through the area of rapidly rising resistance. The full range you should include may need to be up to around 150 K, but you will get a sense of when the resistance graph is no longer interesting and you can begin cooling with LN2 again. Collect data for both the warming and cooling phases.

You will need to repeat these cycles a few times. The first time you take data things may not have equilibrated enough and you may notice some variation from the subsequent attempts.

Once you have enough data for the first sample, switch to the sample in the other bucket, usually on your second day of the experiment.

Week 2: GPIB Data Collection

The computer and the multimeters will use a GPIB communication protocol (the IEEE 488 standard). This is an older method of electronic device communication, but is reasonably robust and still sees use today. While it is relatively slow (by computer standards) it does allow many tens of instruments to all be connected and talk to each other, without the need for ethernet or USB.

For GPIB to work properly, each instrument should have a unique ID associated with it. This ID is usually manually set on the device itself by the user. The most important thing is that no two devices claim the same ID number. You should verify that all Keithley multimeters use a different ID.

The measurement of the temperature is only slightly more complicated than that of resistance. Again you are measuring voltage, but this time the voltage value will be used to interpolate a temperature. There are several phenomenological equations that can be used to describe the relation between voltage and temperature. The Keithley 2000 multimeters have built-in calibration constants for the T-type thermocouples that we use most often.

Ask your instructor to give you an introduction to the wonders of GPIB and how to get started collecting data using the attached computer.

Data collection will be much faster using this method. Only collect data for the superconducting sample bucket, and you should collect at least five cyles of cooling and warming. Do not cool all the way to 80 K, and do not warm all the way to room temperature! You should learn along the way how to narrow in on the transition region and only cool(warm) just below(above) that region.

Analysis

Experimentally, T_c is most often taken to be at the transition point of the resistance vs. temperature plot - the onset of the transition (if you are warming up from LN2 temperature) back to normal resistance behavior. This temperature must be determined as accurately as possible. You must understand the limitations of the system and determine a reasonable uncertainty in your temperature results. When you compare your numbers to those available elsewhere (cite your sources), you must realize that our samples may be somewhat different and not look exactly like those published elsewhere. Lastly, even in the best samples and experiments there appears to be a natural width of a few degrees Kelvin which corresponds to the onset of superconductivity. This itself is not entirely understood and a bit at odds with the (easier to explain) low-temperature superconductors, which typically exhibit a "sharper" transition.

1. For each trial of each sample, plot the resistance versus temperature.

2. Consider various analysis methods for identifying the important features of your plots. You may choose to take one or more numerical derivatives to locate the temperatures of interest. You may also choose to fit an appropriate analytical function to your data, to allow extraction of appropriate fit parameters.

3. Determine the onset temperature of the transition where the resistance begins to increase from zero, with uncertainty.

4. Compare your temperature to literature values for your superconducting sample. The onset temperature value should agree within reasonable uncertainties, but there may be differences. One plausible explanation for this relates to the size of the ceramic superconducting disk and that the temperature across it may not be uniform. Try to articulate why this might produce an effect in your measurements. Ultimately, you should be able to tell if our sample is YBCO or BSCCO.

5. Estimate your uncertainty in all derived temperatures. Consider the variation over all trials that were collected, and whether there are differences between heating and cooling

data.

One deep mystery is why the high temperature superconductors have a transition over a large range of temperature (even when the temperature gradient is not large across the sample during warming or cooling). In low-temperature systems, the transition often occurs much more sharply, making the identification easier within the data and more theoretically well-developed when understood as a phase transition.

At higher temperatures above the transition, what does the resistance vs. temperature curve remind you of (from a previous lab)?