Experiment 7:High-Temperature Superconductivity

From PhysicsLab

This is a materials science experiment, with a primary purpose of making a specimen of the ceramic superconductor yttrium barium copper oxide (YBCO). This compound belongs to the group of materials discovered in 1986 by G. Bednorz and K. Muller at the IBM Zurich Research Lab. These superconductors have the important property that their superconducting transition temperature is high enough (T_c = 93K) that they only need to be cooled in liquid nitrogen, which is cheap and readily available. In the one week version of this lab, the test for superconductivity that you will carry out is the ability of a superconductor to exclude a magnetic field (the Meissner Effect). If a YBCO pellet is cooled below 93K, it should be possible to suspend a small strong permanent magnet a few millimeters above the cold superconducting pellet. In the two week version of this lab, you will also make an electrical measurement to test for the change in resistance associated with superconductivity. This second test also requires additional sample preparation.

Pre-lab tasks

1. Before the first class, summarize the toxic properties and describe the safety precautions necessary to handle Y_2O_3, $BaCO_3$, and CuO, the main ingredients of $YBa_2\ Cu_3O$?.
2. Calculate the percentage composition of each ingredient in order to make up YBa_2Cu_3Ox. Start with 1 g of Y_2O_3 and the other precursors in the correct proportions. This should yield enough material to press three pellets of 2-3 g each in the Carver 1 / 2 inch pellet press.

Procedure

Day 1:

1. The mortar and pestle must first be thoroughly cleaned by grinding a slurry of a small amount of sea sand and a small quantity of hydrochloric acid.
2. Weigh the materials carefully to 2 decimal place accuracy and grind them together using a mortar and pestle for about 30 minutes.
3. Press the materials into a pellet under about 4000 Psi of pressure using the large hydraulic press, placing the powder in a Carver pellet press with ½ inch diameter bore. Be sure to have anvils and the plunger stacked in the correct orientations. The square-cornered sides of the anvils should face towards the powder (beveled edges away from the powder), and the beveled end of the plunger should be outside the cylinder, to be pressed on by a block in the hydraulic press. (The square end will "flare" if the plunger is used backwards, and might irreparably gall the inside of the cylinder the next time it is used the right way around.
4. Hold the pressure at 4000 Psi for at least 30 seconds (you may have to pump slightly more to maintain the pressure, as the pellet compresses and/or the hydraulic fluid backflows).
5. Carefully remove the pellet using the pellet ejector base and the smaller hydraulic press.
6. Between samples it is important to thoroughly clean the pellet press in order to minimize the abrasive action of leftover powder. This can be done using alcohol and a dowel wrapped with an emery cloth.
7. The pellets will then be heated to 900 °C and held there for 20 hours. This will be done in the Physics Oven

in Room 3-44. Small crystals of the YBCO compound should form throughout the pellet during this step ("calcining").

8. The oven temperature slowly ramps down (over about 12 hours) and must be near room temperature before the pellets can be removed.

Day 2:

9. At this point you will cool your pellets in liquid nitrogen and test whether or not they will levitate a small permanent magnet above them (indicating the Meissner effect, the best "litmus test" for superconductivity).
10. Record your observations as you work.
11. Using the Hall probe, measure the magnetic field strength of the permanent magnet along the axis of the magnetic dipole. (there is only one setup for this, just as there is only one pellet press. most groups should be able to make the magnetic field measurements while waiting a turn on the hydraulic press.)
12. Now you must grind some pellets back into powder, using the mortar and pestle again. This might take about 45 minutes of effort, to obtain a reasonably fine powder for re-pressing into a new pellet (again at 4000 Psi). Aim for a thickness of the new pellet between 2 and 3 mm.
13. The new pellets will be placed in the oven again, this time cooked at 950 C, in order to fuse ("sinter") together the YBCO crystallites created the first time around.

Day 3:

14. Meet in Room CEB 2-25 at 2:00; the class will walk over to the thin films lab at CCIS, in order to learn about the process of having metal electrodes evaporated onto the sintered pellets (preparation for the electrical resistance measurement). The metal film will make much more intimate electrical contact to the pellet than can be achieved simply by clipping an electrode on without such a pad. Sintered pellets should only be handled using clean tweezers and/or while wearing clean nitrile gloves.

Day 4:

15. Meet again in Room 2-25 to try to measure the disappearance of electrical resistance when the pellet is cooled through the superconducting transition. The resistance is very small and must be tested using a 4-terminal measurement. You will use a "van der Pauw" configuration of voltage and current leads. (The original reference is "A method of measuring specific resistivity and Hall effect of discs of arbitrary shape". L.J. van der Pauw, Philips Research Reports, vol. 13, pp. 1-9 (1958).)
16. Wear nitrile gloves so you do not get any finger oils or other contaminants on the pellets when you are handling them. Clean the contacts of the alligator clips before you connect them to the specimen. (You can use the edge of a paper towel dampened with alcohol; clamp the clip on and pull the towel out.)
17. Gently attach the alligator clips to the pellet, for the two pairs of leads in a van der Pauw arrangement. The solder mound inside the jaw should connect to the silver electrode. The teflon attached to the lower jaw electrically isolates it from the back of the sample.
18. The current will be drawn from a constant-current power supply. With the current knob dialed to zero, run the voltage knob up about half way. You should then be able to control the current smoothly using the current knob. If you run into a current limit below 1 amp, you will need to increase the voltage setting some more.
19. Do not increase the current beyond 1 amp. Be very careful not to touch the live leads.
20. In order to get more sensitivity for the measurement of voltage drop, you must run the signal through the preamplifier module in the upper left hand corner of the Teachspin signal processor. A gain of 100X is

recommended (+ input, DC coupled; - input, grounded). Read the output of the preamp with a Fluke digital voltmeter. If the voltage doesn't seem as stable as you would like, you can also run the preamp output through the low-pass filter amplifier module in the lower right hand corner, to average the voltage with a longer time constant than that of the Fluke meter. Recommended settings: 1 second time constant, 6 dB/octave rolloff, gain between 1 and 10. (There is no need to have the maximum signal at 1 amp current exceed the 200 mV range of the most sensitive voltage scale of the Fluke meter).

21. First at room temperature, take voltage readings for currrents from 0 to 1 amp, in 0.1 amp increments. Determine the effective resistance from the slope. Don't forget to divide the signal by the total gain of the Teachspin processor, to determine the corresponding voltage at the input to the preamp.
22. Repeat the previous step for two additional sample temperatures: dry ice (methanol slurry), and liquid nitrogen. At the cold temperatures there may be a thermocouple effect (electromotive force induced by the temperature difference between the ends of the leads). It is important to analyze your data via the slope in order to remove the effect of any such DC offset.
23. What limit can you place (upper bound) on the resistivity of the pellet at liquid nitrogen temperature, as a fraction of the room temperature resistivity?

<u>Setup photos: overview; clipped pellet; signal processor front panel.</u>

Fig 1: overview of the electrical measurement setup (here with Wavetek meter instead of Fluke meter).

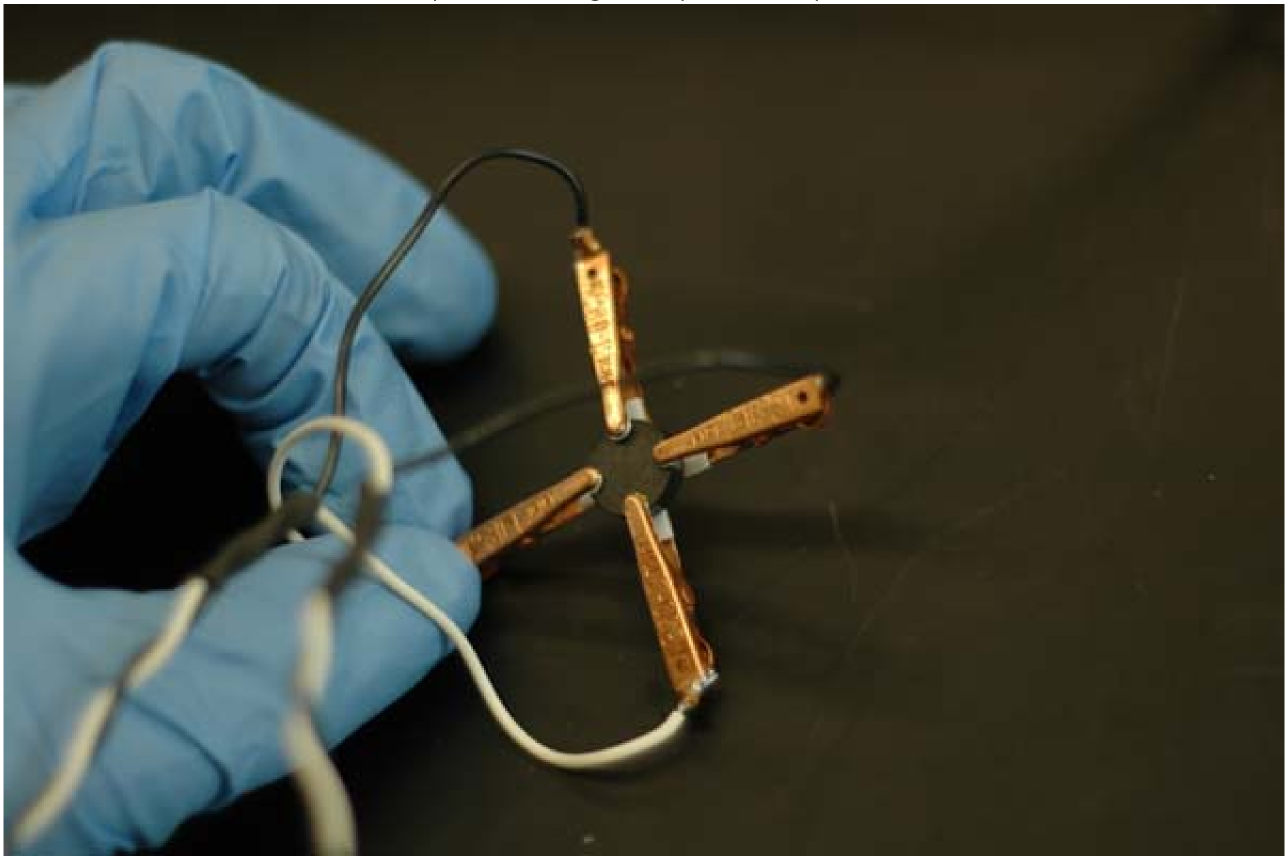

Fig 2: close-up of alligator clips attached to pellet (silver pads barely visible under copper jaws).

Fig 3: the signal processor front panel, with output of preamp connected to input of low-pass filter-amplifier in improvised fashion, using a too-short coax.

<u>Theory</u>

It is convenient to replace a small magnet with a simple magnetic dipole m, a vector quantity with units amp-m^2 . Such a dipole will produce a magnetic field B at a position r given by:

$$B = (\mu_0/4\pi r^5)[3\mathbf{r}(\mathbf{m}\cdot\mathbf{r}) - \mathbf{m}r^2]$$

where μ_0 is the permitivity of free space.

A superconductor acts as a magnetic mirror. A magnet in the vicinity of a superconductor is equivalent to two magnets one being a reflection of the other in the surface of the superconductor.

The force experienced by the dipole in a magnet field is given by

$$\mathbf{F} = -\nabla(\mathbf{m}\cdot\mathbf{B})$$

Question: Assume that when the permanent magnet is brought close to the superconductor an image magnetic dipole is present within the superconductor (along an axis which intersects the centres of both dipole moments). The direction of the image dipole is anti-parallel to that of the permanent magnetic dipole, and the two dipoles have equal magnitude. Calculate the force between the two dipoles as a function of the angle between the permanent dipole and the surface of the superconductor. Show that the maximum magnitude of the repulsive force that a dipole can exert on its image is given by $F = 3\mu_o m^2 / 4\pi z^4$ where z is the distance between the dipole and the image.

If this is for a short report (2-day version):

Hand in a report including the following components:

1. Your log book notes (including the pre-lab work) and observations.
2. A short account of the achievements to date within the materials community, with regards to the preparation of high T_c materials. What is the highest transition temperature that has been achieved? What about practical forms of the material (wires, for example) or single crystals (good for research)? (Include references to your sources of information.)
3. The measurement and calculation of the magnetic dipole strength of the magnet used, including a linearized plot of the magnetic field strength as measured along the axis of the magnet (hint: the expression for $\mathbf{B}$ simplifies along the axis, with $\mathbf{m}$ parallel to $\mathbf{r}$.)
4. A short discussion of the prospects for technological applications.