Class Demonstration: Measuring the Loss of Electrical Resistance in YBCO with the 4-Probe Method

First Draft
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The high temperature superconducting material provided in the kit goes by the acronym, YBCO. These letters stand for the four elements contained in the crystalline structure: yttrium, barium, copper, and oxygen. The formula for the material is commonly given as $YBa_2Cu_3O_7$. In the fabrication of the ceramic, the material may deviate from this stoichiometric ratio and/or the pellet itself most likely does not have a homogeneous structure throughout.

Objectives:

- Students observe the relationship between temperature and resistance for a high temperature superconducting material and create a resistivity vs. temperature curve.
- Students evaluate the critical temperature of YBCO.
- Students apply Ohm's Law to the experimental data.
- Students recognize the limitations of the precision of the instruments available in the high school laboratory.

Equipment and Materials:

YBCO pellet modified with 4-point contacts
Type E thermocouple with a temperature meter
Constant current power supply
Digital Multimeter that can measure millivolt range
Small Styrofoam cooler
Liquid Nitrogen
Cryogenic gloves
Goggles

Handling Liquid Nitrogen

Please read these cautions before doing the experiment, especially if you have not worked with liquid nitrogen before.

- Liquid nitrogen is inexpensive; it can be obtained from local universities or welding shops. The problem is transport and storage. You should obtain a Dewar bottle with a capacity of 4 liters or less. These bottles are expensive; you might be able to borrow one from your provider. The lid must be loose at all times. Never screw down a top on a bottle of liquid nitrogen. The pressure can build until the container explodes.
- The liquid nitrogen is at a temperature of -196°C (77 K). Even short term exposure can cause severe skin burns. Always wear cryogenic gloves when handling liquid nitrogen.
- Whenever you pour liquid nitrogen into a room temperature container, it will break into a rapid boil. Pour slowly and wear goggles. Point the bottle away from

your students or keep them at a distance so they will not be splattered during the pouring.

- Any item that has been immersed in liquid nitrogen and is allowed to cool should not be handled until it has had considerable time to warm back up to room conditions.
- Liquid nitrogen is nontoxic but it could cause asphyxiation if allowed to displace oxygen from the immediate area. Be sure to work in a well ventilated place.
- The best way to dispose of excess liquid nitrogen is to allow it to evaporate slowly from an open container. During this process, the container should be in a safe, well-ventilated area where it will not be disturbed. It could also be poured outside on unused soil; be careful to avoid splattering during outdoor disposal.
- Liquid nitrogen demands respect in the laboratory. Be sure to model responsible behavior in front of your students.

Taking Care of Your YBCO Superconducting Samples

Please read these cautions before doing the experiment. High temperature superconducting (HTS) materials are ceramics; therefore, they are quite brittle. To extend their lifetime, the following handling suggestions should be heeded.

- Do not drop or hit with a sharp blow. You may crack or shatter the pellet.
- The pellets have been coated with GE varnish to eliminate absorption of water that would weaken the pellet over time. Do not expose the pellet to water. Store the pellet in a box with a desiccant packet.
- Do not expose the pellet to high temperatures (over 100°C).
- After immersion in liquid nitrogen, the pellet will accumulate condensed water vapor as it warms up. Gently wipe the frost from the pellet. You can also use a hair dryer on a low setting to gently evaporate the moisture. Do not overheat the pellet!

<u>Teacher Background on 4-point contacts</u>

The loss of resistance below the critical temperature can be measured by measuring the voltage drop across the pellet in a circuit with constant current passing through the pellet. The voltage drop is proportional to the resistance drop (Based on Ohm's Law, V = IR). For best results, it is best to observe the warming of the pellet after it has thermally equilibrated to the temperature of its liquid nitrogen bath. When the YBCO pellet falls below approximately 90 K, it is in a superconducting state and R = 0; therefore, V = 0 and no voltage drop should be measured. The four-point contact technique is used to eliminate contact resistance. Although superconductors have the marvelous ability to carry large currents, a major problem is getting the current into the ceramic material via the contact points. If there is too much resistance at the contact points, it will dissipate power as heat and limit the current flow. To lower the effect of contact resistance, a constant current enters at the two outer contact points while a voltage drop is measured at the two inner contact points. Then the resistance in the ceramic material itself, if it is assumed that all four contacts have equivalent resistance, is the ratio of the measured voltage to the output current of the power supply, a nice application of Ohm's Law, R = V/I. "A Teacher's Guide to Superconductivity for High School Students" by Robert Dull of Largo (Florida) High School gives a detailed discussion of electrical contacts on

ceramic YBCO pellets. This guide is available at http://www.ornl.gov/info/reports/mlornlm3063r1/contents.html.

Making 4-point contacts on YBCO pellet

You have been provided a YBCO pellet with the four-point contacts already installed. This procedure explains how it was made and is for your information only.

1. Make a groove from the center of the pellet out to the edge using an Exacto knife scraping firmly and gently on the ceramic material. The groove must be deep enough to allow the thermocouple wire (Omega type E, 5SRTC-GG-E-30-36) to lie flush with the face of the pellet. To establish proper thermal contact with the ceramic material, the tip of the thermocouple should be bent slightly against the pellet surface.
2. Place a little amount of superglue into the groove to hold the insulated wire of the thermocouple in place**.**
3. Use GE varnish to fill the remaining gap between thermocouple and pellet.
4. A piece of G-10 (approx. 2.5 cm x 7.0 cm) is used as sample mount. After slightly sanding the surface of the G-10 sample to improve biding, the sample is mounted using cryogenic grade, alumina filled epoxy resin (Emerson Cumings Stycast 2880).
5. Measure out 10 g of Stycast 2880 and add 0.75 g of LV 24 activator. Stir well for about a minute.
6. Apply some Stycast on the sanded side of the G-10 piece and press the pellet with the thermocouple facing down into the Stycast and fix the assembly with some sticky tape. The thermocouple is now sandwiched between the pellet and the G-10.
7. Cut two approx. 1.5 m of formvar-insulated, twisted copper litz (27 AWG is sufficient). Scrap off the insulation of the ends of the wires carefully. Label the wire pairs with *I* for current and *V* for voltage.
8. Tape the wire bundles onto the G-10 to hold them in place. They should run along the G-10 in the same direction as the thermocouple wire. The ends of the wires must extend over the diameter of the pellet. To serve as strain relief apply Stycast to bind the copper wires and the thermocouple wire onto the G-10 surface. Allow the entire piece to dry for 12 to 24 h.
9. Position the copper wires ends across the diameter of the pellet surface. Space them using forceps so that they are about equally spaced across the pellet space. The current leads end at the two outer contact points. The voltage leads end at the two inner contact points. Hold the four wires down tightly on the pellet surface. You can do this by placing a strip of rubber cut from a wide rubber band across the wires and clamping down with a medium-size file clamp. Do this carefully to prevent cracking the ceramic pellet.

<better use photo of the whole assembly and labels>

10. Mix a small amount of silver-epoxy in equal parts. Sharpen the end of a wooden applicator to a point or use a toothpick to apply the epoxy to the contact points. Avoid too much epoxy bridging between the wire ends and the pellet surface.
11. Place the entire arrangement in a lab oven set for 83 ^{o}C for 90 min.
12. Apply GE-varnish to the entire pellet surface to reduce exposure to moisture, which will degrade the pellet properties over time.
13. Attach banana plugs to the free ends of the copper wires.

<u>Experimental Procedure</u>

1. Connect leads 1 and 4 to the constant current supply. Adjust the current to 0.20 A but do not run current through the pellet until step 8!
2. Connect leads 2 and 3 to the multimeter. Turn multimeter to the millivolt range.
3. Connect leads from the thermocouple to the thermometer meter. Make sure that the meter is set on a type E thermocouple. The meter reports the temperature in ^{o}C so students will have to convert Kelvin. Good for them!
4. Place the meters so that the students can see the digital display. Designate students to be data recorders.
5. Pour liquid nitrogen into your small Styrofoam cooler to a 2-3 inch depth.
6. Begin to lower the pellet toward the liquid nitrogen. Do not plunge the pellet into the liquid. Hold the pellet just above the surface and have the students watch the temperature drop on the temperature meter. Ask the students why the thermocouple was secured beneath the pellet rather than glued to its top surface. (Actually the thermocouple is sandwiched between the pellet and a piece of cryogenic material called G10.)
7. After the pellet has cooled to about -130°C, slowly immerse it into the liquid nitrogen. Have the students observe the boiling of the nitrogen from the pellet surface. Ask them why this is occurring. Eventually, the boiling will subside. (Why again?)
8. Once the pellet has come to thermal equilibrium with the liquid nitrogen (boiling stops), the students should observe a reading of -196°C on the temperature meter. Pass a current of 0.20 A through the pellet. Have the students notice that the volt meter reads 0.0 mV. Why? This is a great application of Ohm's Law. We have current but no voltage drop because the YBCO material is in a superconducting state, i.e., it gives zero resistance to the flow of current. If R = 0, then V must equal zero although I = 0.20 A.
9. Once the observation in step 8 has been quickly observed, raise the pellet out of the liquid nitrogen and allow it to slowly warm. During the warming, you want to collect simultaneous voltage and temperature readings. An excellent way to do this is to videotape the meter readings side by side and then replay the tape frame by frame after the experiment is over to record data.
10. What will you see? The voltage should remain 0.0 mV until the pellet reaches its critical temperature (T_c). You will then see the voltage climb. A set of sample data is included to help you to know what to expect. It is important to know that the T_c will vary from pellet to pellet because of nanostructure variations in the ceramic material.

11. Before the pellet warms up significantly, it would be wise to either switch off the current to protect the life of your pellet. Cool the pellet again to repeat the experiment in order to test the reliability of the T_c determination. This is a great opportunity to discuss the precision of measurement and how it influences the reproducibility of an experimental result.
12. Have the students fill in a data table with 4 columns for temperature in ^oC, temperature in K, voltage and resistance. They can calculate the corresponding resistances for each voltage reading using Ohm's Law (R = V/I). They should now prepare a graph with resistance in milliohms on the Y axis and temperature in Kelvin on the X axis.

Extensions

1. Repeat the experiment at different currents. **Do not exceed 0.50 A!** Ask students to predict the result before the trials are performed. Why might a higher current be desirable? With a higher applied current, the voltage drop should be greater (Ohm's Law again) when the ceramic rises in temperature above its critical temperature. Therefore, it might be suggested that the best evaluation of T_c occurs at a higher current – but not one that fries your pellet!
2. Have the students calculate the resistivity of the YBCO ceramic material in ohm-cm. Discuss with them the difference between resistance and resistivity.
3. Consider the effect of an external magnetic field on the properties of the YBCO pellet. For example, if the pellet were mounted in the core of a functioning electromagnet, would it behave differently? (This consideration initiates a discussion of critical magnetic field, B_c, for YBCO.

Discussion Points and Questions

1. Have students identify the T_c on the graph and write an explanation of the shape of the graph. What is the significance of the T_c value?
2. In the literature, the T_c for YBCO is reported to be 92 K. What is your experimental percentage of error? Identify the sources of error.
3. Why would a two-point contact measurement result in a non-zero value for resistance even below the critical temperature of YBCO?
4. How could the precision of the the T_c determination be improved? Would your suggestion require an equipment upgrade? If so, which devices used in this experiment do you think limit the precision most?

Sample Data

Temperature (^oC)	Temperature (K)	Voltage (mV)	Resistance (mOhm)
-196		0.0	
-195		0.0	
-194		0.0	
-193		0.0	
-192		0.0	
-191		0.0	
-190		0.0	
-189		0.0	
-188		0.0	
-187		0.0	
-186		0.0	
-185		0.1	
-184		0.2	
-183		0.2	
-182		0.3	
-180		0.4	