I. Preparation of $YBa_2Cu_3O_{7-x}$

$YBa_2Cu_3O_{7-x}$ will be prepared by sintering a pellet containing Y_2O_3, BaO_2, and CuO. The pellet is heated to 930 C to form bonds among the constituents, and then cooled to 500 C to allow reaction with oxygen to obtain a nonstoichiometric compound with $x \approx 0.1$. The reaction (with only the metal ions balanced) is given by:

$$Y_2O_3 + 4\ BaO_2 + 6\ CuO + O_2 \rightarrow 2\ YBa_2Cu_3O_{7-x}$$

Precautions

Barium salts are toxic. Before handling the compounds, read over the Materials Safety Data Sheets in the Appendix. Wear a lab coat, vinyl gloves, and goggles; dust masks are also available. Avoid creating or breathing dust. Avoid eye contact and excessive skin contact. With the exception of the pellets, handle all materials in the laminar flow hood. Wash hands thoroughly after handling. Dispose of waste pellets and powders in the specially marked waste bottle. Contaminated weighing paper, gloves, etc. should be placed in the trash can beside the laminar flow hood. **Clean up your mess!**

Calculate the weights of BaO_2 and CuO required to react stoichiometrically with 0.30 g of Y_2O_3 to produce $YBa_2Cu_3O_{7-x}$. Weigh out the compounds on folded weighing paper, and transfer to a small, dry beaker. Mix the compounds with a spatula and transfer to the mortar. Mix and grind with a pestle for about 10 minutes, periodically scraping the caked material off the sides of the mortar with the spatula. The final mixture should be uniform gray in color, with no black or white spots or streaks visible, and no large particles.

Scrape the mixture out of the mortar onto a folded piece of weighing paper. Check the punch and die of the pellet press for cleanliness, and clean as necessary. Make a pellet from the mixture using the following procedure, which is described with illustrations in the Parr pellet press notes in the Appendix:

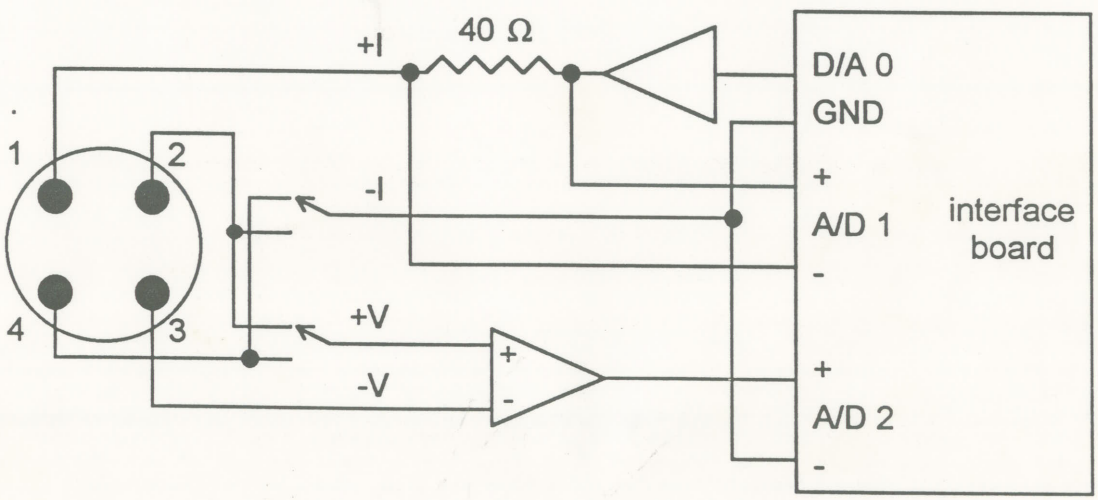
- **Fill the die.** Set the die into its holder, with the beveled edge of the die cavity facing upward, and with the bottom of the die resting on the flat surface of the reversible holder. Pour the powder mixture into the die cavity. Tap the holder gently on the bench to settle the powder.

- **Compress the powder.** Place the die and its holder in place on the anvil of the press. Push the lever down to compress the powder into the pellet. *Don't push too hard!* The lever should require a firm push as it moves through its full stroke. If a full stroke is not obtained with reasonable force, rotate the anvil to lower the die slightly. If the lever moves through the full stroke without encountering sufficient resistance, rotate the anvil to raise the die slightly.

Place the pellet between the two coils, inserting the thermocouple between the pellet and one of the coils. Attach the stainless steel cover to the sample holder, and place the holder in the vacuum dewar. Fill the insulated container with ice water and insert the reference thermocouple. Verify the lock-in settings listed in the Appendix. Adjust the Phase of the lock-in to obtain a maximum positive output. Start the program HTCMAG (in the directory HTC). Gradually add liquid nitrogen to the dewar. HTCMAG will record the lock-in ouptut voltage versus temperature as the sample cools from room temperature to 77 K. Save your data on disk, and use EXCEL or some similar program to analyze and plot the data. Estimate the superconducting transition temperature, the width of the transition, and the fraction of your sample that is superconducting. Can you estimate the temperature dependence of the superconducting order parameter from your data?

3. Resistance versus temperature

A schematic of the apparatus used to measure the resistance is shown below. The D/A converter provides an output voltage which is converted to a current by the amplifier and series resistor. (The amplifier is necessary because the D/A converter has a maximum output current of only 5 mA.) The sample voltage is amplified and measured using the A/D converter. The resistance is measured using a four-probe configuration. The switch allows two different combinations of current and voltage leads to be used.



To make contacts to your pellet, cut four thin slices from the indium tear drops, and press them using a glass slide onto one face of your pellet. The indium pads should not touch, and should be placed to match the contact pin pattern in the sample holder. Put a small amount of N-grease onto the other side of the pellet, place the thermocouple onto the copper plate, and press the pellet onto the thermocouple and plate. Assemble the sample holder, and carefully tighten the screws until the contact pins touch the indium pads. *Don't overtighten: the contact pins are very delicate!* Attach the stainless steel cover to the sample holder, and insert the holder in the dewar.

- **Reverse the die holder.** Raise the lever while holding the die so the punch pulls out of the die cavity. Slide the die and holder out of the press. Turn the holder over to put the deep cavity on top and place the die in the holder over the cavity. Replace the die and holder on the anvil.

- **Eject the pellet.** Bring the lever down gently to eject the pellet into the cavity of the holder. Raise the lever and remove the holder from the press. Carefully remove the pellet with nylon forceps and place it in the alumina crucible.

- **Clean the die and holder.** Remove the die from the punch and clean loose powder from the die cavity by tapping on a piece of weighing paper. Shake any loose material out of the die holder onto the paper and dispose of it in the waste bottle. Then clean the die and punch with fine grit sandpaper.

Repeat this procedure to make a second pellet.

Place the crucible containing the pellets in the quartz flow tube, and push the crucible down the tube to the middle of the furnace. Gently rotate the quartz end cap onto the ground glass end of the tube until a good seal is made. Open up the valve on the oxygen cylinder and the small valve. Adjust the regulator pressure to roughly 2 psi. Purge the tube for ten minutes with a large flow rate, and then reduce to 0.3 cfh.

A wiring diagram for the furnace and controllers, the furnace temperature profile calibration, and instructions for programming the controllers are given in the Appendix. The furnace has two independently controlled zones which allow temperature gradients, needed in the growth of many materials, to be produced. Sintering of the pellets requires a uniform temperature, so both zones in this experiment will be heated to the same temperature. The two temperature controllers are programmed to provide the following time-temperature sequence:

Step	Temperature	Time
1	0 → 930 C (0 → 1706 F)	12 hours
2	930 C (1706 F)	12 hours
3	930 → 500 C (1706 → 932 F)	12 hours
4	500 C (932 F)	12 hours
5	Furnace turned off	

To run the program, press the START/STOP keys on both controllers. If the green MAN light comes on continuously, you are in Manual mode, and need to switch to Auto (program) mode. Press START/STOP, then press AUTO/MANUAL, then press START/STOP again. To check on the status of the program (actual temperature, power output, temperature at end of current program segment , time left in current segment), press the DISPLAY button. Pressing this button repeatedly will loop through the program status information. If you want to experiment with growth conditions, feel free to enter your own program. The furnace has limit controllers, so you can't melt it. (Don't change the settings on the limit controllers!)

Remove the pellets from the furnace when the temperature has dropped below 150 °F, and press the START/STOP buttons on both controllers to end the program. The finished pellet should be dark gray to black. A dark green material is a second, non-superconducting phase.

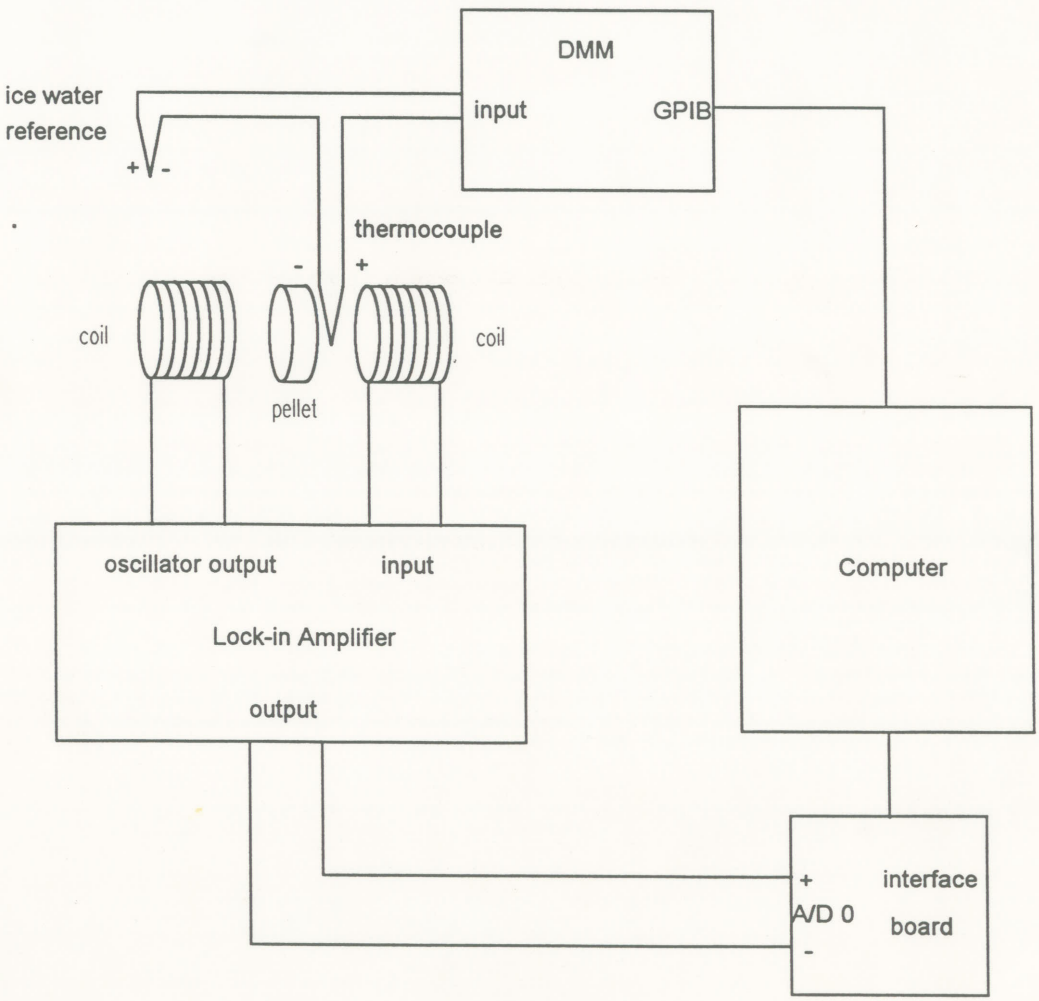
II. Measurements

1. Qualitative test for superconductivity

Place the pellet in a cut-down styrofoam cup and then place a small magnet on top. Gently pour liquid nitrogen into the cup, adding more as it boils off, until the pellet is completely immersed. If your pellet contains any phases which are superconducting at 77 K, the magnet should levitate above the pellet.

2. Susceptibility versus temperature

A schematic of the apparatus used to measure the susceptibility of the pellet versus temperature is shown below. The 33 Hz oscillator output of the lock-in amplifier drives one coil, and the magnetic flux produced passes through the sample and induces a voltage in the second coil. This voltage is measured by the lock-in amplifier, and the lock-in's output is read by an A/D converter connected to the computer. The thermocouple voltage is recorded by the voltmeter, which is connected by a GPIB interface to the computer.



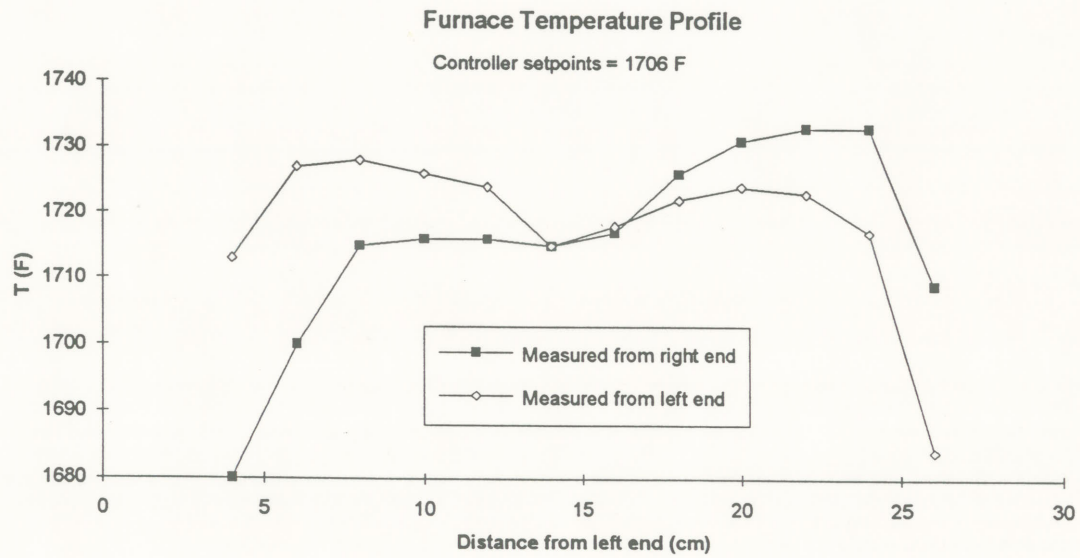
Start the program HTCRES. Measure the room-temperature resistance of your sample for two different current/voltage lead combinations, and record the results. Measure an I-V curve. Is the sample ohmic? Calculate the conductivity of your sample using Van der Pauw's method, discussed in the Appendix. Compare your value with the value quoted in Figure 4 of Batlogg's article.

Next, add liquid nitrogen to the dewar, and record the resistance versus temperature as the sample cools to 77 K. What is the transition temperature and the width of the transition? Why does the R(T) curve have the shape you measure? When the temperature stabilizes at 77 K, measure an I-V curve for your pellet. Can you determine the critical current?

Programming the Furnace Temperature Controllers

To check/change the program, press in sequence TUNE - LAST - YES; the green TUN indicator will illuminate on the display when pressed correctly. Press the PARAM CHECK key repeatedly to step through the Tune loop. Ignore the first few displays, and when RAMP AND SOAK? comes up, press YES.

Press the PARAM CHECK key to step through the time-temperature program. The temperature setpoints and segment durations Press the up and down arrow keys (⇑ ⇓) to change the temperature setpoints and segment durations. At the end of the Tune loop program, the display will read END OF TUNE. Hit the RETURN key to exit Tune mode. If you change the program, please change it back to the original program listed in the front of this manual when you're done.

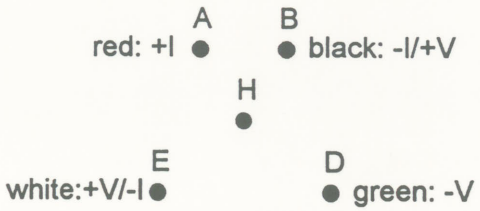


Lock-in Amplifier Settings

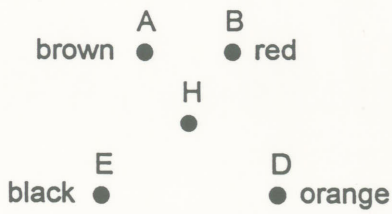
Offset:	+	**Off**	-
Filter:		On	
DC:		On	
20 dB post:		On	
Gain:	20 dB	Off	**40 dB**
Mode:	**Int**	Ext	
Inputs/Outputs:	**Shield**	Isolate	
Detection:	**Normal**	Quadrature	
	2f	**Off**	

Wiring

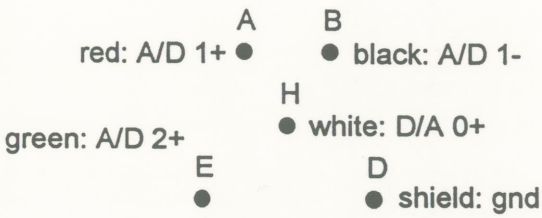
Sample Cable Connector



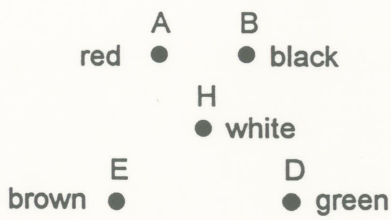
Amplifier Box Sample Connector



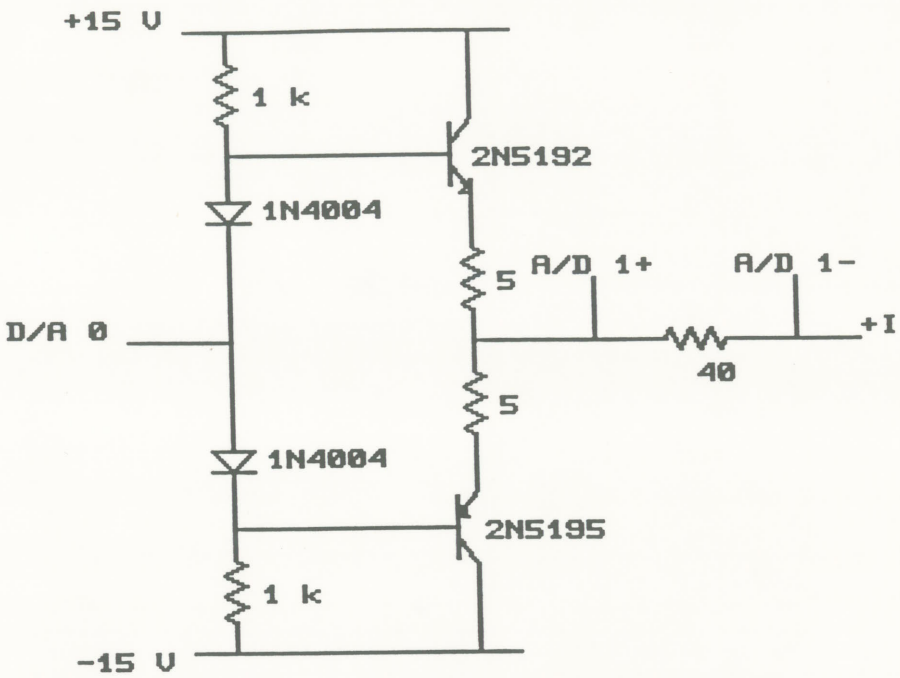
Interface Board Cable Connector



Amplifier Box Interface Board Connector



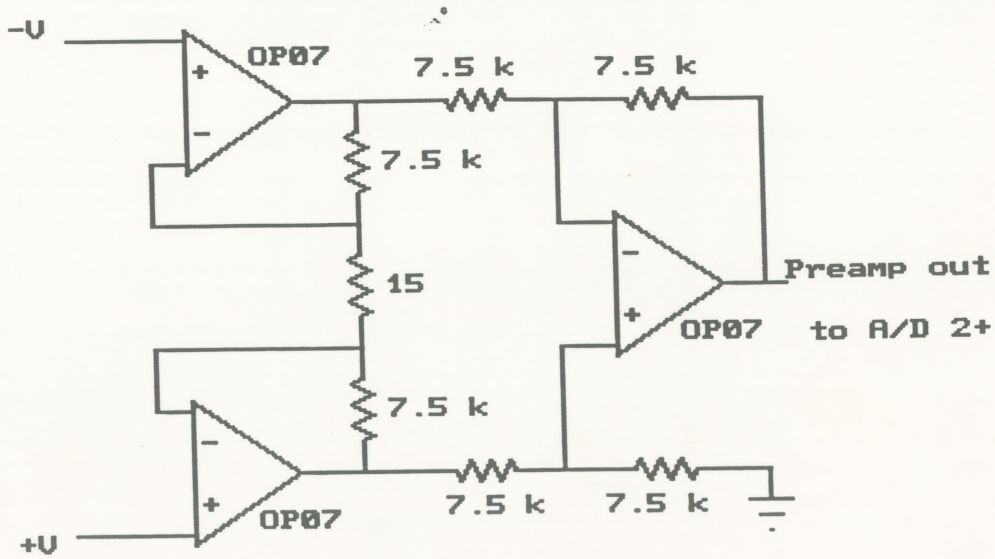
Current Booster Amplifier



SAMPLE VOLTAGE PREAMP

Gain = 1000

Supply voltages = +15 V / -15 V



Wiring Diagram for T-Controller
Mod#1138-M Tubular Furnace
Physics 510-Lab