YBCO Synthesis and Characterization
(Electrical Transport Option)

http://education.qdusa.com/experiments.html

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In this Educational Module, students will attempt to synthesize and electrically characterize their own polycrystalline samples of $YBa_2Cu_3O_{7-\delta}$ (YBCO) superconductor, the first material discovered with a superconducting critical temperature (T_C) above liquid nitrogen's boiling temperature.

Materials List:

The following are tables of the supplies and equipment that can be used to perform this activity. The catalog numbers and suppliers listed are merely suggestions, and instructors should feel free to obtain whatever materials are within their means and accessibility. The chemical quantities mentioned should easily last a semester.

Sample Synthesis and Heat Treatment

Item	Quantity	Catalog Number	Supplier
Y_2O_3	50 g	205168-50G	Sigma Aldrich
$BaCO_3$	500 g	237108-500G	Sigma Aldrich
CuO	25 g	203130-25G	Sigma Aldrich
Mortar and Pestle	1	12-950AA	Fisher Scientific
Balance (.01 mg)	1	01-920-052	Fisher Scientific
Weighing Paper	1 pack	S402871 or 02-202-100	Fisher Scientific
Spatula/Scoopula	1	14-357Q	Fisher Scientific
Hydraulic Press	1	13-872	Fisher Scientific
13 mm Press Die	1	SDS13	Across International
Tube Furnace	1	10-472-53	Fisher Scientific
Air supply line	1		
Combustion Boat	1	07-680a	Fisher Scientific

Sample Mounting

Item	Quantity	Catalog Number	Supplier
Zoom Stereo Microscope	1	12-070-850	Fisher Scientific, Zeiss
Calipers	1	14-648-17	Fisher Scientific
25 μm diameter Pt Wire	5 m	10292	Alfa Aesar
Lighter	1		
Scissors	1		
Leitsilber 200 Silver Paint	30 g	16035	Ted Pella
Tweezers	2	17-447-102	Fisher Scientific
Thin stick or needle			
Apiezon H Grease	25 g	FAPGH00025	Fisher Scientific

Sample Measurement

Item	Quantity	Catalog Number	Supplier
Multimeter	1		
VersaLab	1	1300-001	Quantum Design
VersaLab ETO Module	1	1084-700	Quantum Design

YBCO Synthesis:

A successfully performed synthesis mixed the chemicals in proportions such that the atomic ratios of yttrium, barium, and copper were 1:2:3: $0.5Y_2O_3$ + $2BaCO_3$ + 3CuO + → $YBa_2Cu_3O_7$ + CO_2.

Y_2CO_3 = 0.2823g
$BaCO_3$ = 0.9868g
CuO = 0.5966g

A successful heat treatment cycle is:

Ramp up to 900 C in 4 hours.
Dwell at 900 C for 10 hours.
Cool to room temperature in 8 hours.

Re-grind the sample.

Ramp up to 930 C in 4 hours.
Dwell at 930 C for 15 hours.
Cool to room temperature in 10 hours.

Re-grind the sample and press into a pellet.

Anneal the pellet by ramping up to 955 C in 3 hours.
Dwell at 955 C for 5 hours.
Cool to room temperature in 7 hours.
Ramp the pellet one last time to 500 C in 2 hours.
Dwell at 500 C for 14 hours in order to set the oxygen content.
Cool to room temperature in 4 hours.

The samples should be deep black in color. If the color is still somewhat grayish, or if the sample does not superconduct, then repeating the final 35 hour annealing process beginning with the ramp to 955 C will likely increase the quality of the sample.

<u>YBCO Sample Preparation and Mounting:</u>

Whole pellet samples may be mounted onto the puck, or for a little more efficiency, two samples can be mounted. Contacts to the sample can be made using indium instead of silver paint, and connection to the puck pads can also be made using solder instead of silver paint. If thick wire is soldered to the puck pads, bear in mind not to apply too much force to the wire, or else the pads can be ripped off.

Ideally, the contact resistances to the sample should be less than 100 Ω. If the resistances are higher, the wiring should be redone. The room temperature resistivity of YBCO along the *c*-axis has been reported up to 21 mΩ·cm [1], such that any initial reading should be < 1 Ω at the dimensions discussed here.

Sample Measurement Using the ETO:

In MultiVu, you may run the measurement using a 10 mA excitation at a frequency between 15 Hz and 21 Hz. You may refer to ETO measurement parameter guidance for current, frequency, and autoranging in the transport newsletter:
http://www.qdusa.com/sitedocs/newsletters/Applications_Newsletter_Fall_2013.pdf

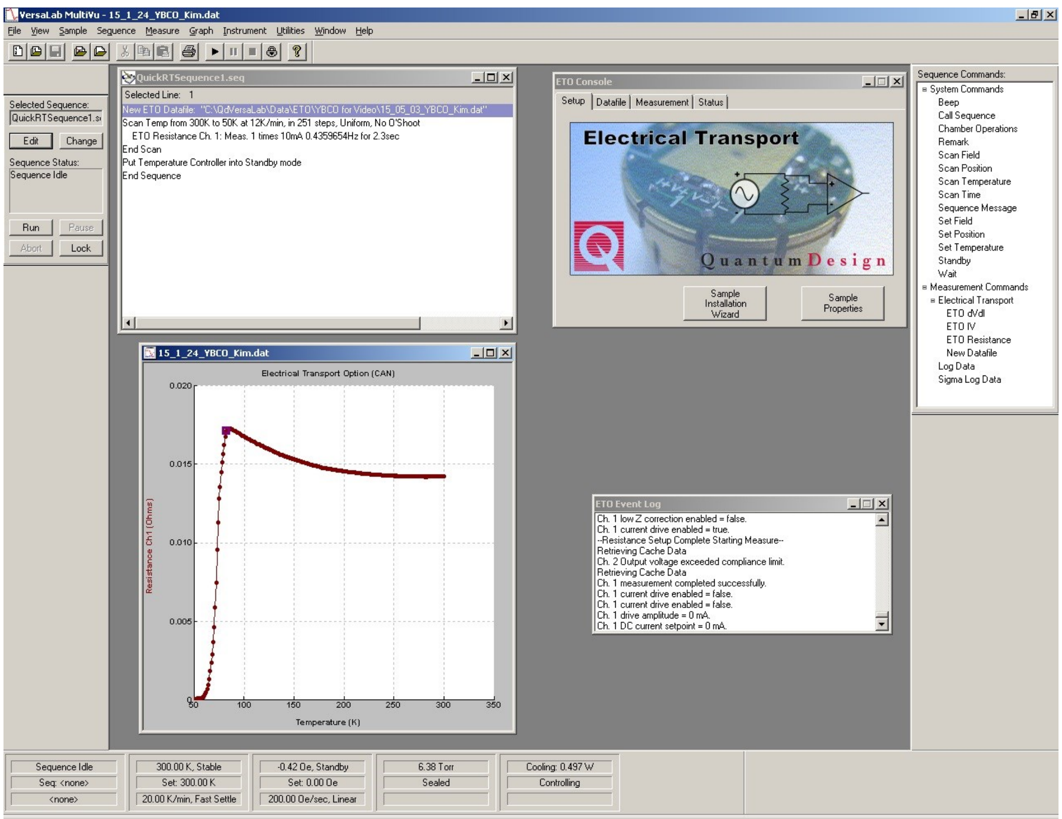

Figure 1: MultiVu interface for performing the measurement

Data and Discussion:

Ideal YBCO samples will possess a fairly sharp transition temperature ~ 93 K.

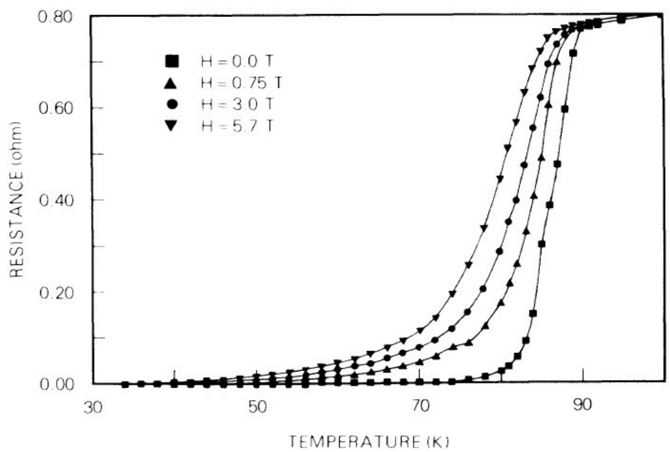

Figure 2: Resistance vs. temperature plots of YBCO under application of various magnetic fields

It is possible to obtain sub-optimal superconducting samples with lower T_C and very broad transitions. Even if superconductivity is not achieved, then the students can learn about the impact of synthesis conditions and insulating properties. It is also possible to obtain mixed phase samples that show indication of superconducting domains buried in a semiconducting matrix. As such, this is a rich activity that illustrates the challenges of materials synthesis and engages the students in trying to explain the behaviors of their samples. The possibility of failure can be exciting for the students, and instructors can make a competition out of this laboratory project. Most importantly, students should leave this activity well versed in fundamental techniques of condensed matter research.

Potential questions and further investigations for your students may include:

- You may consider asking the students to plot the first derivative of their data with respect to temperature in order to determine the onset of T_C in a more precise manner than just by eye.
- The students could apply magnetic fields up to 3 T in magnitude with the Versalab and look for an effect on T_C. Similarly, Resistance vs. Applied Magnetic Field above and below T_C can reveal magnetic interaction that does not occur in the normal state.

- If the samples are very high impedance (>10 MΩ), you may ask the students why the 4-wire technique would fail and discuss the ranges and limitations of various measurement techniques.
- The students may measure a semiconducting sample and a metal sample in order to observe the difference resistance vs. temperature behaviors, which could be fit to theory. This could facilitate discussion of different conduction mechanisms.
- Several low resistance measurements can be performed at a fixed temperature in order for the students to perform an exercise in error analysis.
- You may ask the students to dig deeper into the cryogenic operation of the Versalab and the measurement principles of the ETO.

<u>Additional Reference:</u>

[1] C.P. Poole, H.A. Farach, and R.J. Creswick. *Superconductivity*, Academic Press 1995, pp. 28-29.