

## PREPARATION OF THE 1-2-3 SUPERCONDUCTING PELLET

### Advanced Topic

#### Materials:

- Dust mask and safety goggles
- Mortar and pestle
- 50 ml acetone
- 1.00g yttrium oxide, Y(2).O(3)
- 2.11g cupric oxide wire, CuO
- 3.50g barium carbonate BaCO(3)
- Alumina boat, 90x17x11.5 mm
- Tongs
- Heat-proof gloves
- Tube furnace, 1 in. diameter
- Pellet press
- Quartz tube (24 mm o.d.) equipped with air-tight connections made at one end for attachment to an oxygen tank and the other end for attachment to an oil-filled bubbler.
- Tank of oxygen, with regulator and tubing
- Krylon No 11303, acrylic resin spray

#### Procedure for Pellet Preparation

Wear a dust mask and safety goggles at all time, and conduct grinding operations in a fume hood. In the mortar and pestle, grind together the yttrium oxide, copper oxide, and barium carbonate with enough acetone to make a thick slurry. Allow the acetone to evaporate completely in air; at this point the mixed powder will flow freely. Place the powder in the alumina boat and heat in the tube furnace at 950°C for one hour. Wearing heat-proof gloves and tongs, remove the boat containing the mixed oxides from the tube furnace. Allow the mixture to cool to room temperature. It will be black or green-black at this time.

When cool, regrind the dry powder in the mortar and pestle (do not use acetone). Return the powder to the boat, and heat it in the furnace for five hours at 950°C. Again, cool it to room temperature. Grind the now black powder for a third time in the mortar and pestle, this time using acetone. Allow the acetone to evaporate completely.

Use a pellet press to form pellets at about 50,000 lb/in<sup>2</sup> pressure. The pellets should be approximately 13 mm in diameter and 3 mm thick. There should be enough mixed oxide to make three pellets.

Place the pellets in the alumina boat, and place the boat inside the quartz tube. Place the assembly in the tube furnace and heat at 950 for one hour in order to sinter (heat just below the melting point to increase strength and density and to promote intergranular bonding) the pellets. Allow the furnace to cool to 500-600°C for the crucial "sensitization" step. At this point the ceramic is **oxygen deficient**. This means that there is not enough oxygen in the crystalline structure to superconduct. Pass pure oxygen through the quartz tube, over the pellets, at a rate of about 10ml/min for 3 hours. At the conclusion of the 3 hour period turn off the furnace and allow it to cool to room temperature while maintaining the flow of oxygen over the pellets. Oxygen will enter the YBCO compound as it **slowly**

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cools, between 600 and 400<sup>0</sup> C. By allowing oxygen to circulate in the oven as the sample cools your sample will become oxygenated. Several annealing procedures, heating and cooling cycles, seem to improve the quality of 1-2-3 ceramic superconductors.

Spray coat the pellets with an acrylic resin such as Krylon No 1303. This will protect the pellets from chemical decomposition, which can occur from prolonged exposure to water or water vapor.

Once cooled, the final material should be hard and black in color, any green coloration will indicate that you have an insulator rather than a superconductor. If the material exhibits the Meissner effect, it is a true superconductor. Remember the production of superconductors is still very much an experimenters game.