Experiment 17

SYNTHESIS OF SUPERCONDUCTOR

Objective

The purpose of this experiment is to learn the solid-state reaction, to synthesize a high-temperature superconductor, $YBa_2Cu_3O_{7-x}$ ($x \leq 0.25$), and investigate its properties.

Lab techniques

- Weighing chemicals.
- Operating grinding, hydraulic pressing, and calcination.
- Handling of liquid nitrogen.

Introduction

A superconductor exhibits superconductivity, namely zero electrical resistance, and the Meissner effect at the superconducting critical temperature (T_c). The Meissner effect is the exclusion of a magnetic field from a superconductor during its transition to the superconducting state, that could levitate a bar magnet. In this experiment, yttrium oxide, barium carbonate, and copper oxide are used as starting materials to synthesize $YBa_2Cu_3O_{7-x}$ (abbreviated as Y-123), a superconductor with a T_c at ~95 K, by solid-state reaction. The Y-123 superconductor can levitate a bar magnet at liquid nitrogen temperature (Fig. 17-1) to show the Meissner effect, which indicates positive superconductivity.

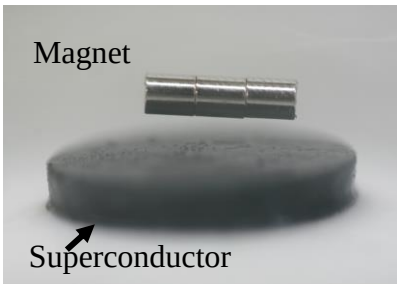

Figure 17-1 Y-123 superconductor levitates a magnet at liquid nitrogen temperature

Apparatus

I. Agate mortar and pestle, alumina crucible, hydraulic press, dies, plastic spatula, plastic tweezers, box furnace, and disposable mask (self-prepared).

II. Plastic tweezers, bar magnet (Nd-Fe-B), petri dish, Dewar flask, and hair dryer.

Chemicals

Yttrium oxide, Y_2O_3

Barium carbonate, $BaCO_3$

Copper(II) oxide, CuO

Liquid nitrogen, $N_2(l)$

Procedure

I. Synthesis of superconductor

1. Calculate the stoichiometric amount of Y_2O_3, $BaCO_3$, and CuO to make the molar ratio of Y : Ba : Cu atoms equal to 1 : 2 : 3 to synthesize 0.004 mol of $YBa_2Cu_3O_{7-x}$.

   Note 1: Include the calculation in the prelab report and verify the calculation results with the lab instructor.

   Note 2: The high purity chemicals used in this experiment are very expensive. While weighing them, you should avoid wasting.

2. Measure precisely the required amounts of Y_2O_3, $BaCO_3$, and CuO, and then transfer them into an agate mortar. Mix and grind the chemicals for at least 10 min, until a homogeneous fine powder is obtained.

   Note 1: The mixing or grinding of powders needs to be carried out in the fume hood or wearing a disposable mask to avoid inhaling chemical powders.

   Note 2: The spatula used in this experiment should be made of plastic to avoid scratching the agate mortar.

3. Use the hydraulic press and dies to press the powder into a pellet under a pressure of 1 $ton \cdot cm^{-2}$ for 1 min.

   Note: Wash the pressing dies thoroughly with water to remove any powder stuck to its surface and then wipe-dry or blow-dry them with a hair dryer.

4. Place the pellet in alumina crucible, and place it into the box furnace. Allow it to calcine at 930°C for 10 h in air by raising and lowering the temperature at a rate of 5$^{o}C \cdot min^{-1}$.

   Note: No need to label on the crucible. The label will carbonize at high temperature. Record the position of the crucible to identify your product.

II. Test of the Meissner effect

5. After the pellet cools to room temperature, immerse and cool the obtained product in liquid nitrogen. Use plastic tweezers to place one or more small bar magnets on the superconductor pellet. Observe the levitation of the magnet and then measure the height of the levitation. The levitation of the magnet is an evidence of superconductivity. You may record the magnetic levitation with digital camera.

Note: Avoid direct contact with liquid nitrogen, which can cause frostbite.

6. At the end of the magnetic-levitation test, dry the superconductor with a hair dryer to warm it to room temperature, remove any moisture on it, and put it in a zipper bag.

References

1. She, J. L.; Liu, R. S. *J. Chem. Educ.*, **2008**, *85*, pp 825-826.
2. Wu, M. K.; Ashburn, J. R.; Torng, C. T.; Hor, P. H.; Meng, R. L.; Gao, L.; Huang, Z. J.; Wang, Y. Q.; Chu, C. W. *Phys. Rev. Lett.* **1987**, *58*, 908.
3. https://en.wikipedia.org/wiki/Meissner_effect.