

Experiment 17

SYNTHESIS OF SUPERCONDUCTOR

Objective

The purpose of this experiment is to synthesize a high-temperature superconductor, $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ ($x \leq 0.25$) by solid-state reaction, and investigate its properties.

Lab techniques

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

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, which could levitate a bar magnet.

In this experiment, yttrium oxide, barium carbonate, and copper oxide are used as starting materials to synthesize $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ (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 (77 K) to show the Meissner effect (Fig. 17-1), which indicates positive superconductivity.

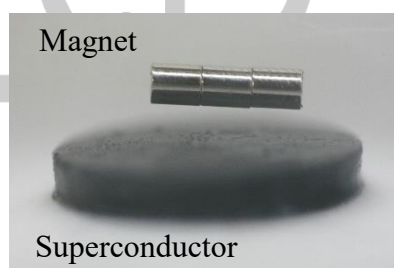


Figure 17-1 Y-123 superconductor levitates a magnet at liquid nitrogen temperature

Apparatus

I. Preparation:

plastic spatula, alumina crucible lid, and face mask (self-prepared).

Shared: electronic balance, agate mortar and pestle, hydraulic press, dies, plastic

tweezers, cellulose sponge, and box furnace.

II. Test:

Shared: plastic tweezers, bar magnet (Nd-Fe-B), petri dish, Dewar flask, cryo-gloves, and hair dryer.

Chemicals

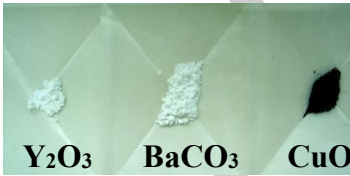
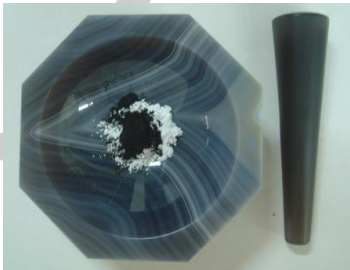
Yttrium oxide, Y_2O_3

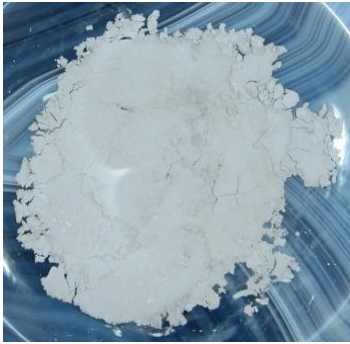




Barium carbonate, BaCO_3

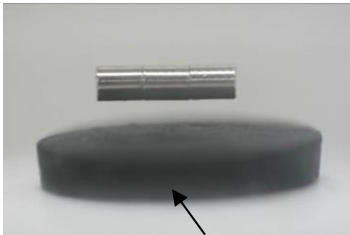

Copper(II) oxide, CuO

Liquid nitrogen, $\text{N}_2(\text{l})$

Procedure

Procedure		Illustration
I. Synthesis of superconductor		
1.	<p>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 $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$.</p> <p>Note 1: Include the calculations in the prelab report.</p> <p>Note 2: The high-purity chemicals used in this experiment are very expensive. Avoid wasting.</p>	
2.	<p>Measure precisely the required amounts of Y_2O_3, BaCO_3, and CuO. Transfer them into an agate mortar.</p> <p>Note 1: Wear a face mask while mixing and grinding powders to avoid inhaling chemical powders.</p> <p>Note 2: The agate mortar is also quite expensive. Handle it with care.</p>	

3.	<p>Use a plastic spatula to mix the starting materials, then grind the chemicals for at least 10 min, until a homogeneous fine powder is obtained.</p> <p>Note 1: Use a plastic spatula to avoid scratching the agate mortar.</p> <p>Note 2: Wash the agate mortar with water and sponge, then dry it with paper tissues after use.</p>	
4.	<p>Transfer the mixed powder to a weighing paper. Use the hydraulic press and dies to press the powder under a pressure of $1 \text{ ton}\cdot\text{cm}^{-2}$ for 1 min, then a round ingot with diameter of 2 cm and thickness of about 0.3 cm is obtained.</p> <p>Note 1: The pressure should not exceed $1 \text{ ton}\cdot\text{cm}^{-2}$ to avoid cracking during calcining.</p> <p>Note 2: After use, wash the pressing dies thoroughly with water and sponge to remove powders stuck to its surface and then wipe-dry.</p>	
5.	<p>(1) Place the sample on an alumina crucible lid with plastic tweezers, and put it into the box furnace.</p> <p>(2) Allow it to calcine at 930°C for 10 h in the air by raising and lowering the temperature at a rate of $5^{\circ}\text{C}\cdot\text{min}^{-1}$.</p> <p>(3) After cooling down to room temperature, take the product out.</p> <p>Note: Record the position of the crucible to identify your product. No need to label it since it will be carbonized at high temperature.</p>	 
II. Test of the Meissner effect		
6.	<p>(1) Place the obtained product in a petri dish, and use plastic tweezers to place one small bar magnet on it to check the interaction.</p> <p>(2) Pour some liquid nitrogen into petri dish, immerse and cool the product.</p> <p>(3) Place one small bar magnet on it, examine the</p>	

	<p>levitation of the magnet, and measure the height of the levitation.</p> <p>(4) Increase the number of bar magnets to investigate the Meissner effect.</p> <p>Caution: Avoid direct contact with liquid nitrogen, which can cause frostbite.</p>	 <p>Superconductor</p>
7.	<p>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.</p>	

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 (2024/07/31).