The Effect of Inorganic Nanocrystals on the Properties of High –Temperature Superconductors

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Abstract: The great increase of magnetical and electrical properties of 1-2-3 superconductors were observed with inorganic nanocrystals as starting materials.Nanocrystalline copper and barium oxide powders are obtained with the average particle size ~ 30nm by refluxing hydrated barium and copper hydroxide precipitate. The results show that crystals of a finite size may assume a modified morphology and /or crystal structure so that the atoms, especially those at the surface,can adopt the most stable equilibrium configuration of minimal energy.
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1. Introduction

Superconductors are materials whose resistivity drops to zero at sufficiently low temperatures , in contrast with metal conductors whose resistivity decreases as the temperature falls but remains finite even at the lowest temperatures[1].The temperature below which the resistivity of a superconductor goes to zero and the material becomes superconducting is known as the critical temperature ,T_c[2] .Below T_c a superconductor can sustain a current indefinitely with no loss due to resistance,which is an electrical analog of friction[3,4].

As current passes through a circuit containing metal conductors and semiconductors ,current is lost to resistive heating [5].A second property possessed by all superconducting materials is an ability to exclude magnetic fields from their interiors at temperatures below T_c. In the superconducting state ,these materials are perfectly diamagnetic.This phenomenon,called the Meissner effect,was discovered in 1933 by Meissner and Ochsenfeld[6,7] .The Meissner effect is responsible for the ability of superconductors to levitate magnets[8].Heike Kamerlingh Onnes discovered the phenomenon of superconductivity in 1911,whemn he observed that the resistance of mercury abruptly fell to zero when cooled to 4K by liquid helium.Subsequently ,other materials were discovered that exhibited superconductivity; however, the superconductivity state could only be accessed through cooling with liquid helium,which is expensive and requires special handling[9] .Consequently ,the practical applications of superconductors have been limited and highly specialized.A notable application of superconductors requiring liquid helium cooling is in the magnets that produce the high fields required for nuclear magnetic resonance(NMR)spectroscopy and magnetic resonance imaging (MRI); the coils of these magnets are constructed from Nb-alloy superconductors[10].

Superconductivity occurs in a wide variety of materials ,including simple elements like tin and aluminium, various metallic alloys and some heavily – doped semiconductors.Superconductivity does not occur in noble metals like gold and silver , nor in pure samples of ferromagnetic metals[11] .In 1986 the discovery of a family of cooperate-perovskite ceramic materials known as high temperature superconductors,with critical temperatures in excess of 90 kelvin,spurred renewed interest and research in superconductivity for several reasons.As a topic pure research ,these materials represented a new phenomenon not explained by the current theory.Inaddition,because the superconducting state persists up to more manageable temperatures ,past the economically – important boiling point of liquid nitrogen(77 Kelvin),more commercial applications are feasible ,especially if materials with even higher critical temperatures could be discovered[12].

2. Materials and methods

2.1 Materials

All of materials are reagent grade from Fluka Chemicals . Oxalic acid ,ammonium hydroxide was purchased from Merck Chemicals .

2.2 Preparation of copper oxide nanocrystals

A copper oxalate precipitation method was applied to prepared the nanocrystalline copper oxide powders. A copper alcohol solution (0.5M) was added at a controlled speed to an oxalic acid solution in alcohol to form the copper oxalate complex precipitate.Oxalic acid was strongly stirred by a magnetic stirrer during addition of copper solution.The suspension was oven dried overnight at 100°C and then ground and sieved through a 180 μm sieve. The sieved complex powder was calcined at 250°C for 2 h.The resulting black powder was sieved again through a 180 μm sieve and stored in a desiccator.

2.3 Preparation of nanocrystal BaO_2 powder

$BaCl_2$ was diluted with ice-cold distilled water to form $BaOCl_2$ solution. To this solution ammonium hydroxide was added dropwise to precipitate barium as hydroxide.The hydrated barium hydroxide gel was thoroughly washed free of anions and transferred to flask fitted with a water condenser.The gel was continuously stirred for 6 h and temperature was maintained around 70-100°C .The solid mass after refluxing was found to be crystalline and free flowing .Then the crystalline powder formed was filtered and oven dried.

2.4 Preparation of high –temperature superconducting materials using Oxalate coprecipitation procedure

Stoichiometric amounts(as shown in Table 1) of the reagent grade Y_2O_3 obtained from Fluka Chemicals and prepared BaO_2 and CuO nanocrystalline dissolved separately in 10-40 mL dilute nitric acid.The three solutions were mixed followed by filteration to obtain a clear solution.

Table 1- Weight (gms)of reagent grade starting materials used for each component to obtain (Y)-Ba-CuO

Sample No.	Stoichiometry	Y_2O_3	BaO_2	CuO	Oxalic Acid
1	$Y_{1.0}Ba_2Cu_3O_7$	1.1291	3.9470	2.3862	14.8132

An excess of 10% solution of oxalic acid was then added to obtain light blue precipitate of mixed oxalate.The precipitate was then centrifuged and dried at 80°C for 10 hours. The light blue powder was first fired for 28 hours at 860°C in air using an alumina crucible with three intermediate grindings.During the heating procedure the color of the powder changed from green to black. The resulting black powder was then fired for 15 hours at 900°C in air with two intermediate grindings. The powder was then compressed into pellets(1.0 cm diameter and 0.25mm thick)under a pressure of about 20,000 psi using a hydraulic press.Often at this stage the material is deficient in oxygen,and is usually semi-conducting or sometimes even non-conducting. The pellets need to be sintered for at least 12 hours at 900°C in oxygen atmosphere and then cooled down at a cooling rate of 25°C/15 minutes over a period of 8-10 hours. Slow annealing under oxygen atmosphere from the elevated temperatures to room temperature is essential for producing the orthorhombic superconduting phase.

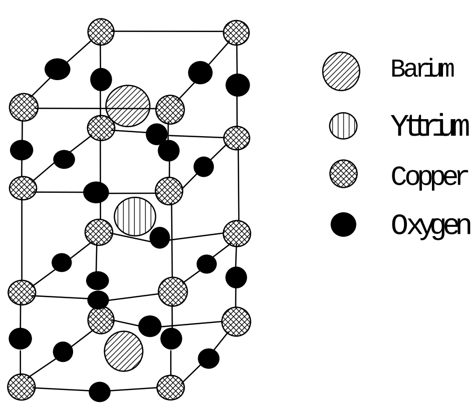

Figure 1- Unit cell of $YBa_2Cu_3O_7$

3. Results

3.1 Heating –cooling sequence for synthesis of the 1-2-3 superconductor

Generally, $YBa_2Cu_3O_7$ superconductor is readily prepared by heating an intimate mixture of yttrium oxide,barium peroxide and cupric oxide at approximately 930°C for 10-12 hours.Figure 1 shows the crystalline structure of $YBa_2Cu_3O_7$ superconductor .

In this stage the crystalline structure is formed by the inter diffusion of ions ,but it has a deficit of oxygen.By cooling the materials to 500 °C and annealing at this temperature for 10-12 ,allows it to react further with oxygen from the air,reducing d to less than 0.1.This compound is often called the 1-2-3 materials from ratios of Y:Ba:Cu.The heating –cooling synthesis sequence is shown in Figure 2.

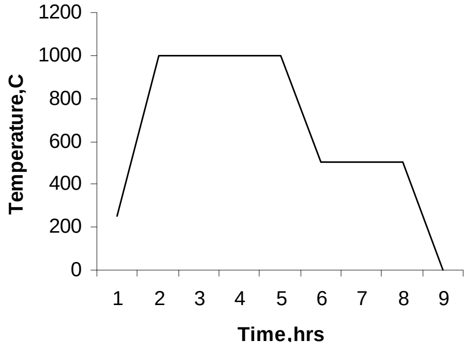

Figure 2 – The heating - cooling synthesis sequence of superconductor

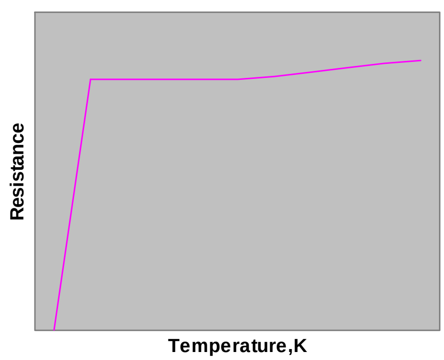

Figure 3- Electrical resistance of prepared superconductor

3.2 Determination of the magnetic properties of the 1-2-3 superconductor(Meissner effect).

The Meissner effect is distinct from this because a superconductor expels all magnetic fileds,not just those that are changing .Suppose we have a material in its normal state,containing a constant internal magnetic field . When the material is cooled below the critical temperature ,we would observe the abrupt explusion of the internal magnetic field ,which we would not expect based on Lenz's law.Therefore,we have studied the magnetic properties of prepared superconductor by a magnet levitating above it , cooled with liquid nitrogen. Persistent electric current flows on the surface of the superconductor,acing to exclude the magnetic field of the magnet (the Meissner effect). The results have shown that copper and barium nanocrystals have increased the meissner effect .Therefore , prepared 1-2-3 superconductor from above nanocrystals are stronger electromagnet.

3.3 Determination of T_c .

At ordinary temperatures ,metals have some resistance to the flow of electrons ,due to the vibration of the atoms which scatter the electrons .As the temperature is lowered,the atoms vibrate less and the resistance declines smoothly,until the material's critical temperature,T_c,is reached.At this point ,the resistance drops abruptly to zero.The recent literatures show that 1-2-3 superconductor have critical temperature about 89K .the prepared superconductor from nanocrystals has electrical critical temperature 126K. These results show that copper and barium nanocrystals increase T_c about 34K(Figure 3).

3.4 Loss of electrical resistance by 4-probe technique(demonstration by TA)

The loss of resistance below the critical temperature can be measured by measuring the voltage drop across the pellet in a circuit.The 4-probe apparatus used is shown schematically in Figure 4.

A battery generates a current,I, which passes through the pellet.The voltage drop,$V = I \times R$,along the pellet due to the pellet's resistance,R, at room temperature is measured with the voltmeter. When the material becomes superconducting , R = 0 , so V = 0 and no voltage drop should be measured.

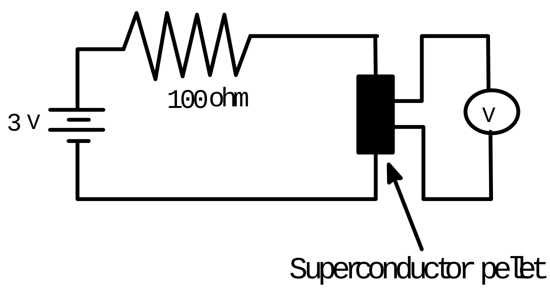

Figure 4- The 4-probe conductance test circuit.

4. Discussion

The major aim of this research is the study of effects of inorganic nanocrystals on the properties of superconductors.It is well-known that inorganic nanocrystals are a benchmark model for nanotechnology ,given that the tenability of optical properties and the stabilization of specific phases are uniquely possible at the nanoscale. The understanding of size effects of inorganic materials in the superconductors structure is also of interest due to the possibility of tuning its properties.The results show that crystals of a finite size may assume a modified morphology and /or crystal structure so that the atoms , especially those at the surface,can adopt the most stable equilibrium configuration of minimal energy.Consequently , some materials ,which are less stable in the bulk and do not exist in ambient atmosphere,may become substantially more stable at the nanoscale.This is largely due to the large contribution of surface energy which can stabilize the origin of unique phases. Nanoscale CuO and BaO_2 is observed to be relatively stable with respect to bulk CuO and BaO_2 , which can readily convert to other state in the experimental conditions.The reason for this morphological and stoichiometric control is still a subject of discussion in the literature,although crystal structure is believed to play a part.The role of inorganic nanocrystals in the superconductor structure and its properties are very important .We observed a increase of Tc and Meissner effect of prepared superconductor with a decrease of copper and barium oxides particle size from bulk to nanoscale.

4. Conclusions

The great increase of magnetical and electrical properties of 1-2-3 superconductors were observed with inorganic nanocrystals as starting materials.Nanocrystalline copper and barium oxide powders are obtained with the average particle size ~ 30nm by refluxing hydrated barium and copper hydroxide precipitate . The results show that crystals of a finite size may assume a modified morphology and /or crystal structure so that the atoms , especially those at the surface,can adopt the most stable equilibrium configuration of minimal energy.

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