UNIVERSITI PUTRA MALAYSIA

STRUCTURAL AND ELECTRICAL PROPERTIES OF YBCO ADDED WITH Nd_2O_3, Gd_2O_3 and Sm_2O_3 NANOPARTICLES

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By

AIMA BINTI RAMLI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in fulfilment of the Requirements for the Degree of Doctor Philosophy

September 2015

To my Father & Mother:

Haji Ramli Abu Bakar & Hajah Safiah Mohd Yusof

My beloved Husband & Children:

Mohd Najmuddin Ibrahim, Nur Alesya, Muhammad Naufal & Nur Athirah

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Doctor of Philoshophy

STRUCTURAL AND ELECTRICAL PROPERTIES OF YBCO ADDED WITH Nd_2O_3, Gd_2O_3 AND Sm_2O_3 NANOPARTICLES

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September 2015

Chairman : Professor Abdul Halim Shari, PhD
Faculty : Science

It is well known that the superconducting properties are strongly dependent on the synthesis technique and processing conditions. Coprecipitation method is frequently used in sample synthesis and thus, chosen in this project due to high homogeneity, low reaction temperature, fine and uniform particle size with non-agglomerate particles, easy set-up and economical, and time saving processing. The Yttrium Barium Copper Oxide (YBCO) samples have been prepared by coprecipitation of metal ion oxalates method added with Nd_2O_3, Gd_2O_3 and Sm_2O_3, x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0 wt%. Phase formation and volume fraction of each sample were examined using X-ray diffraction and Rietveld refinement technique. All samples show predominantly Y-123 with non-superconducting phase, Y-211 and impurities, Nd_2O_3, Gd_2O_3 and Sm_2O_3 with orthorhombic structure and *Pmmm* space group. The volume fractions of non-superconducting phase, Y-211 abruptly increase in all systems, might be due to the local differences in the size of Y-211, thus, affect the T_c and J_c in the Y123 system. The microstructure scanning electron microscope (SEM) revealed that the average grain sizes calculated from the Image J, decreased in all systems as the addition of magnetic nanoparticles, Nd_2O_3, Gd_2O_3 and Sm_2O_3 increased indicating that the poor grain connectivity due to the porosities and weak links. The transport measurement of resistance dependence, $T_{c\text{-onset}}$ was measured by using standard four point probe technique. T_c for pure sample is about 92 K. However, T_c decreased to 74 K, 80 K and 88 K for Nd_2O_3, Gd_2O_3 and Sm_2O_3, respectively. The suppression on $T_{c\text{-onset}}$ was attributed to the lowering oxygen content in samples. Since YBCO is granular in nature, AC susceptibility is used as an effective tool to characterize granular of this system. The inter-granular vortex was investigated with different applied field, H_{ac}, 0.005 – 3.0 Oe at fixed frequency 123 Hz. The matrix critical current density, J_{cm} was calculated in the framework of Bean's critical state model. Flux creep activation energy is determined in vortex dynamics exhibited by frequency dependence of AC susceptibility in the range of 123 – 6000 Hz. Sample with x = 0.6 wt% Nd_2O_3 shows maximum value of J_{cm}, 5.77 x 10^{-5} A/cm^2 and E_a, 9.212 x 10^{-19} J indicating at this point it has a optimum pinning centre. As a conclusion, Nd_2O_3 nanoparticles acting as flux pinning centres in matrixes of superconductors Y123 which gave the best result in term of J_{cm} value as compared to Gd_2O_3 and Sm_2O_3.

Abstrak thesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

SIFAT STRUKTUR DAN ELEKTRIK BAHAN YBCO DICAMPUR NANOZARAH Nd_2O_3, Gd_2O_3 DAN Sm_2O_3

Oleh

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Telah diketahui bahawa sifat mensuperkonduksi sangat dipengaruhi oleh teknik sintesis dan keadaan pemprosesan. Maka, kaedah pemendakan logam sentiasa digunakan dan dipilih di dalam kajian ini disebabkan oleh kehomogenan yang tinggi, suhu tindakbalas yang rendah, saiz butiran yang halus dan sekata tanpa sebarang gumpalan, mudah untuk disediakan dan murah serta menjimatkan masa pemprosesan. Kesemua sampel Yttrium Barium Copper Oxide (YBCO) disediakan melalui kaedah pemendakan ion logam oxalate ditambah dengan Nd_2O_3, Gd_2O_3 dan Sm_2O_3, x = 0.0, 0.2, 0.4, 0.6, 0.8 dan 1.0 peratus berat. Pembentukan fasa dan peratusan pecahan isipadu setiap fasa sampel dikaji dengan teknik Pembelauan Tenaga Sinar-X dan pemurniann Rietveld. Kesemua sampel menunjukkan pra-mendominasi fasa Y-123 dan fasa tidak mensuperkonduksi, Y-211 dengan kewujudan bendasing Nd_2O_3, Gd_2O_3 dan Sm_2O_3, dengan struktur ortorombik dan kumpulan ruang *Pmmm*. Pecahan isipadu fasa mensuperkonduksi, Y-211 maningkat secara mendadak adalah disebabkan oleh perbezaan saiz penempatan Y-211 yang mempengaruhi T_c dan J_c di dalam sistem Y-123. Struktur mikro daripada Mikro Pengimbas Elektron mendedahkan bahawa saiz zarah yang dikira melalui Imej J berkurangan apabila penambahan nanozarah bermagnet Nd_2O_3, Gd_2O_3 dan Sm_2O_3 meningkat, menunjukkan bahawa bahan tersebut mempunyai ikatan butiran yang lemah berikutan kewujudan keadaan berliang dan lohong. Pengukuran perubahan rintangan dilakukan dengan teknik penduga empat titik. Tc untuk sampel tulen ialah 92 K. Walaubagaimanapun, T_c semakin berkurangan kepada 74 K, 80 K dan 88 K masing-masing untuk Nd_2O_3, Gd_2O_3 and Sm_2O_3 Didapati $T_{c\text{-onset}}$ semakin berkurangan disebabkan oleh kandungan oxigen yang semakin rendah di dalam sampel. Memandangkan superkonduktor YBCO bersifat butiran secara semulajadi, pengukuran keupayaan arus ulang alik digunakan sebagai alat yang efektif untuk mencirikan sifat butiran dalam sistem ini. Vorteks antara butiran diukur dengan medan yang dikenakan antara 0.005 Oersted hingga 3.0 Oersted pada frekuensi tetap, 123 Hz. Manakala ketumpatan arus kritikal matriks, J_{cm} dikira berdasarkan rangka Model Keadaan Kritikal Bean. Pengaktifan tenaga pergerakan fluks, E_a pula ditentukan di dalam dinamik vortex yang bersandarkan perubahan frekuensi di dalam keupayaan arus ulang-alik iaitu di dalam rangkuman 123 Hz hingga 6000 Hz. Sampel dengan nilai peratusan berat, x = 0.6 Nd_2O_3 mempunyai nilai J_{cm}, 5.77 x 10^{-5} A/cm^2 dan E_a, 9.212 x 10^{-19} J yang maksimum menunjukkan sampel ini mempunyai pusat pengepinan yang

optimum. Sebagai kesimpulan, nanozarah Nd_2O_3 bertindak sebagai pusat pengepinan fluks di dalam matriks superkonduktor, telah memberikan keputusan yang terbaik di dalam J_{cm} jika dibandingkan dengan system Gd_2O_3 dan Sm_2O_3.

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I certify that a Thesis Examination Committee has met on 14 September 2015 to conduct the final examination of Aima binti Ramli on her thesis entitled "Structural and Electrical Properties of YBCO Added with Nd_2O_3, Gd_2O_3 and Sm_2O_3 Nanoparticles " in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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Name of Member of Supervisory Committee: Associate Prof Dr. Chen Soo Kien

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LIST OF SYMBOLS AND ABBREVIATIONS

YBCO	Yttrium Barium Copper Oxide
NdBCO	Neodymium Barium Copper Oxide
Y123	Yttrium Barium Copper Oxide
Pr123	Praseodymium Barium Copper Oxide
REBCO	Rare-earth Barium Copper Oxide
HTS	High Temperature Superconductor
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
AC	Alternating Current
DC	Direct Current
RE	Rare Earth
A	Ampere
T_c	Critical temperature
Bi (2201), Bi (2212), Bi (2223)	Bi-based superconducting family
MRI	Magnetic Resonance Medical Imaging
β	Critical Index
α, α_o	Pinning Strength Parameters
H_c	Critical Field
H_{c1}	Lower Critical Field
H_{c2}	Upper Critical Field
F_L	Loretnz Force
ϕ_o	Vector
F_p	Opposing Pinning Force
V	Electro-motive Force

v	Velocity
d	Distance
R	Rate
f_o	Characteristic Frequency
E_a	Activation Energy
k_B	Boltzmann's Constant
J_c	Critical Current Density
H_{ac}	Applied Field
H_o	Field at 0 K
m	Magnetic moment
m_{rev}	Reversible Magnetic Moment
M	Magnetisation
M_o	Magnetisation at 0 K
W	Hysteresis loss per unit volume per field cycle
χ_{Ac}	Complex Susceptibility
χ'	Real Part of Susceptibility
χ''	Imaginary Part of Susceptibility
χ_m	Partial Susceptibility of Matrix
χ_g	Partial Susceptibility of Grains
P	Critical Current Parameter
f_g	Effective Volume Fraction of Grains
a/b	Sample Size
Calcination	Heating Process where the solid state reaction occur
Sintering	Heating Process for Improving Material Properties
2θ	Diffraction Angle
θ	Glancing Angle, Angle in Degree

d_{hkl}	Reciprocal d Vector
h k l	Miller Indices
(rms)	Root Mean Square
T_{cm}	Extrapolated Temperature for Matrix Susceptibility
T_p, T_{pm}	Extrapolated Peak Temperature for Matrix Susceptibility
J_{cm}	Inter-granular Critical Current Density
a,b,c	Lattice Parameters
f	Frequency (Hz)

CHAPTER 1

INTRODUCTION

1.1 Introduction

The discovery of high temperature superconductivity (HTS) in the copper oxide based materials by Bednorz and Müller in 1986 resulted in worldwide interest in these materials. This breakthrough discovery is very significant because liquid nitrogen can be used as a coolant for materials to superconducting at higher temperature and this is referred to as high temperature superconductor. Intense research efforts into superconductivity have been undertaken during the last two decades in order to search for a new superconducting materials with higher critical temperature.

Recently, there are so many applications on cuprate based superconductor that have greatly contributed to the society. The fundamental technologies for applications are categorized into superconducting bulks, superconducting tapes and superconducting devices. Materials for superconducting bulks are $REBa_2Cu_3O_7$, where RE is either Nd, Sm, Gd or Y. In bulk, the pinning force of magnetic flux in the superconducting state is very strong and at 77 K the critical current density is more than 10^4 A/cm^2 (Tanaka, 2006). As the pinning force of the magnetic flux is strong, the applied magnetic field cannot penetrate the bulk in the superconducting state. This results in a strong levitation force when the bulk is close to an ordinary permanent magnet, and at 77 K this force usually reaches 15 kg/cm^2. However, when the bulk is in the normal state a magnetic field applied from outside is uniformly distributed throughout the bulk. But after the bulk is cooled below the critical temperature, the magnetic field is quantized and quantized flux is pinned by strong pinning centres. Then the quantized flux is remained inside when the external field is removed and behaves like a permanent magnet. The strength of the remained magnetic field in $GdBa_2Cu_3O_7$ bulk reaches more than 2 T at 77 K and more than 3.5 T at 30 K. This application has been successfully used in water cleaning system by using magnetic separation effect (Tanaka, 2006).

In HTS the first generation of superconducting tape is bismuth-based superconductor, BSCCO. This multifilament oxide-powder-in-tube (OPIT) tapes with high current transmission cable carry large amount of electric power to be sent to central part of city. The second generation of superconductor tape is YBCO conductor which has been grown either by biaxially textured substrate (RABiTS) method, ion-beam-assisted deposition (IBAD) or inclined-substrate deposition (ISD) technique. YBCO with $J_c >$ 10^6 A/cm^2 is able to maintain a high current-carrying capacity in fields up to several tesla compare to BSCCO tape the current densities begin to decrease at field well under 1 T (Owens & Poole, 2002).

1.2 Early Discovery of Superconductivity

On July 1908, in Leiden University, Netherland, the great Dutch physicist, Heike Kamerlingh Onnes was became the first person successfully liquefied helium. Three years later, on 8 April 1911 Kamerlingh Onnes and one of his assistants showed that the resistance of superconductivity of mercury approached practically zero as the superconducting transition temperature dropped to 4.2 K. Infinite conductivity indicates that if a current were passed through the material during its superconducting phase, the current would flow forever without any dissipation. They studied mercury because very pure samples could be easily prepared by distillation. In 1933, the German Physicist, Walter Meissner and Robert Ochsenfeld discovered that the superconductors are more than a perfect conductor of electricity as they are able to expel the applied magnetic field if cooled to below its transition temperature, T_c. It will exhibit a perfect diamagnetism. This phenomenon is known as the Meissner effect in which the magnet could levitate on the superconducting material

The understanding of superconductors gradually got sophisticated and solving this long-standing puzzle required a special set of talents and proficiencies. Only in 1957 the understanding of basic mechanism of superconductivity was solved by John Bardeen, Leon Cooper and Robert Schrieffer. They introduced the Bardeen-Cooper-Schrieffer (BSC) theory which involved the electron-phonon interaction to form Cooper pairs (Bardeen *et al*., 1957).

In 1973 Nb_3Ge alloys was found by Gavaler with A15 phase structure described by chemical formulae of A_3 B and critical temperature, T_c of 23.2 K. It held the record as the highest critical temperature superconductor since no other elements with higher T_c was obtained for more than 10 years. But nevertheless, in 1986 Johannes Georg Bednorz and Karl Alex Muller, the Swiss researchers from IBM Zurich Laboratory, Switzerland discovered a new ceramic material that can achieve superconductivity at higher critical temperature and it was barium-lanthanum-copper oxide with perovskite structure and T_c up to 35 K (Bednorz & Muller, 1987). They have inspired other researches all over the world to search for new ceramic superconducting materials at temperatures more than four times higher than the earlier ones.

Just a year after Muller and Bednorz's breakthrough, Paul Chu and his colleagues at University of Houston, Texas found high temperature superconductor (HTS) of $YBa_2Cu_3O_{7-\delta}$ with T_c = 92 K which above the boiling point of liquid nitrogen 77 K. Using liquid nitrogen as a cooling medium give benefits to researchers because it is cheaper, more efficient, easy handled coolant than liquid helium. Then a year later, Maeda and his group at the National Research Institute for Metals, Tsukuba Laboratory in Japan working on HTS and they found Bi-Sr-Ca-Cu-O system without any rare earth element with T_c about 115 K (Maeda *et al*., 1988).

Shortly afterwards, the thallium system Tl-Ba-Ca-Cu-O had been discovered by Sheng and Hermann at University of Arkansas. The T_c was about 120 K (Sheng & Hermann, 1998). Because of it is highly toxicity the exploration of thallium system has not widely studied. Besides that, in 1993 Schilling and his co-workers discovered Hg-Ba-Ca-Cu-O system with T_c of 134 K. In January 2001, Nagamatsu and his colleagues, group from Japan reported MgB_2 with T_c about 39 K (Nagamatsu *et al*., 2001). This was the highest

T_c for a non-copper-oxide bulk superconductor. Since then, until July 2001 more than 260 studies about this superconductor have appeared (Buzea & Yamashita, 2001). Figure 1.1 shows the time evolution of the transition temperature of superconductors.

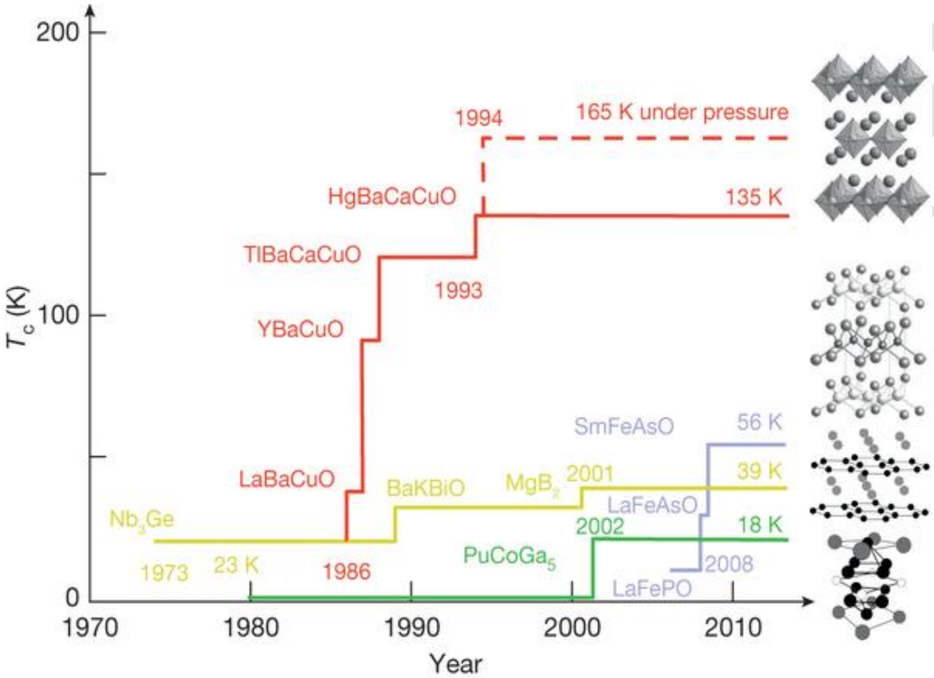

Figure 1.1: The evolution of T_c of superconductors (Keimer *et al*. 2014).

1.3 Problem Statement

Flux pinning is very crucial in HTS where supercurrent can flow without any energy dissipative. However, since polycrystalline superconductor samples are granular in nature, the superconducting properties are limited by weak links effect and inhomogeneity of the samples. In order to improve the homogeneity and weak links effect, coprecipitation method was chosen due to low reaction temperature, fine and uniform particle size, easy set-up and economical, and time saving processing. Besides, the nanoparticles of Nd_2O_3, Gd_2O_3 and Sm_2O_3 were selected because these magnetic rare earth substitutions are compatible to the size of pinning centre thus, may increase the critical current densities, J_c.

1.4 Objectives

The objectives of this work are:
1) To synthesize the $YBa_2Cu_3O_{7-\delta}$ via co-precipitation method.
The advantages of co-precipitation method over solid-state method are small particle size, high purity and homogeneity because of atomic scale mixing and ability to control size and shape of particles.

2) To measure and study the relative effect of nano – Nd_2O_3, Gd_2O_3 and Sm_2O_3 addition with different weight percentage (0.0 – 1.0 wt%) on superconducting transition temperature, T_c of $YBa_2Cu_3O_{7-\delta}$ by measuring the resistance as a function of temperature using the standard four point probe set up with a close-cycle helium cryostat from 30 K – 300 K to determine the value of $T_{c\text{-onset}}$ and $T_{c\text{-offset}}$.

3) To study the phase formation and microstructure evolution of YBCO added by Nd_2O_3, Gd_2O_3 and Sm_2O_3 using X-ray diffraction and analysed by Rietveld refinement method and SEM.

4) To study the effect of Nd_2O_3, Gd_2O_3 and Sm_2O_3 addition on J_c of YBCO and the pinning mechanism of superconducting properties by using Bean's model.

1.5 Outline of Thesis

The thesis is outlined as follows. Chapter 1 consists of general introduction to the research conducted, early discovery of superconductivity, problem statement and the objectives of research. In Chapter 2, the basic theory and literature review of basic properties of superconductor, crystal structure and the effect of doping and adding technique for various types of elements on the superconducting properties of YBCO superconductor are briefly discussed. A detailed description of the sample preparation and experimental methods to perform the measurements are stated in Chapter 3. While Chapter 4 contains all the results that obtained from the measurements and discussion of data analysis of thermogravimetric, crystal structure, resistance dependence of temperature, morphological images and AC susceptibility at field and frequency dependence. Finally, the relation between phase, structure and superconducting properties is concluded and suggestions for future works are stated in Chapter 5.

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