UNIVERSITI PUTRA MALAYSIA

EFFECTS OF MgO, MgB_2 AND YBCO ADDITION ON FORMATION, MICROSTRUCTURE AND SUPERCONDUCTING PROPERTIES OF $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta$ CERAMIC

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By

ARLINA BINTI ALI

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

July 2015

DEDICATION

To my mom and dad who encourage me
AISHAH BINTI OTHMAN
ALI BIN NAYAN

Beloved husband
RAJA ZULKIFLI RAJA HUSAIN

Beloved daughter
RAJA NUR INTAN ARIANA BINTI RAJA ZULKIFLI

Sibling
ANOR ARDI ALI
ANOR FIRDAUS ALI
MOHD ANIFF ALI
MOHD NAQIB ALI
SITI FARHAH ALI

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirements for the degree of Doctor of Philosophy

EFFECTS OF MgO, MgB_2 AND YBCO ADDITION ON FORMATION, MICROSTRUCTURE AND SUPERCONDUCTING PROPERTIES OF $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta$ CERAMIC

By

ARLINA BINTI ALI

July 2015

Chairman: Professor Abdul Halim Shaari, PhD.
Faculty : Science

The superconducting properties of $Bi_{1.6}(Pb_{0.4})Sr_2Ca_2Cu_3O_\delta(M)$(BPSCCO) added with M=MgO, MgB_2 and YBCO have been studied so as to acquire further information related to the effect of superconducting and non-superconducting impurities as pinning centers in the bulks BPSCCO. Samples of $(Bi_{1.6}(Pb_{0.4})Sr_2Ca_2Cu_3O_\delta)_{1-x}(M)_x$ at x=0.00-0.10 were prepared using solid state reaction method. The samples were calcinations for 3 times at 800^{o}C for 20 hour, 820^{o}C for 10 hours and 840^{o}C for 4 hours then sintered in air at 855^{o}C for 96 hours followed by annealing at 830^{o}C for 30 hours. The transport properties of the samples were measured using x-ray diffraction (XRD), Scanning electron microscope (SEM), Energy Dispersive X-ray (EDX), four-point probe electrical resistance measurement and magnetic properties were measured by using Alternating Current Susceptibility (ACS).

X-ray diffraction (XRD) technique showed that all samples are in polycrystalline phase with some of the peaks belonging to Bi-2223 and Bi-2212 as determined from Reitveld Refinement Technique. The structural properties such as volume fraction and lattice parameter were calculated using volume percentage equation and Bragg's Law. The XRD peaks increased when x=0.02 (MgO, MgB_2 and YBCO) for all samples and dominated by Bi-2223 (about 76%). However, the volume fraction of Bi-2223 phase decreased gradually as the addition of materials increased. The sample was still dominated by Bi-2223 phase with slight increase in Bi-2212 phase. The lattice parameters calculated from XRD data show a slight decrease in the *c*-axis while *a*-axis and *b*-axis increases. The decrease at the lattice of *c*-axis may be due to the increase in oxygen content. The reason that *a*-axis and *b*-axis increases may be due to extra electrons valence on Cu so that these is an increase of Cu-O bond length.

Scanning Electron Microscope (SEM) images showed the flaky plate-like grain structure samples, in which the grains size become smaller with higher addition concentration. The elemental analysis by EDX measurement of samples reveals the existence of additives materials that are distributed in Bi-2223 matrix.

The resistivity measurement showed that all samples exhibit normal metallic behavior in the normal state. Critical temperature $T_{C(R=0)}$ which is around 98 K for pure sample while it was found gradually to increase to higher temperature with x=0.02 (100 K, 99 K and 102 K), the $T_{C(R=0)}$ decreases gradually to lower temperature. The holes concentration of pure sample is around 0.127 and this value decreases as the addition concentration increase.

The behavior of weak link and intergranular coupling was studied using Alternating Current Susceptibility (ACS) technique. Diamagnetism onset temperature $T_{C\text{-}ON}$ was observed from real part (χ'), where a two-step transition is related to the superconducting intra and intergrain coupling. The intra and intergranular AC loss peaks as observed from imaginary part, (χ'') move to lower temperature and the height and the width of the peaks increase showing the extend of the AC loss. Analysis of the temperature dependence of the ACS near the transition loss peak temperature T_p has been done by using Beans's Critical State Model. The higher percentage of additives increase the behavior of weak link and intergranular coupling. It was found that the Bi-2212 phase and additions of additives on the grain boundaries play the role in influencing the intergranular coupling and weak links. The calculated value of Josephson current, I_o obtained from the AC susceptibility data showed a much higher value I_o=123.21 μA for addition with YBCO (x=0.02) as compared to the pure samples with I_o=119.83 μA. The diamagnetic behaviour is decreased and shifted toward the lower temperature due to the improvement of weak link connectivity.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

KESAN DARIPADA PENAMBAHAN MgO, MgB_2 DAN YBCO KEPADA FORMASI, MIKROSTRUKTUR DAN SIFAT SUPERKONDUKTOR SERAMIK $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta$

Oleh

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Sifat superkonduktor $Bi_{1.6}(Pb_{0.4})Sr_2Ca_2Cu_3O_\delta(M)$(BPSCCO) ditambah dengan M=MgO, MgB_2 danYBCO telah dikaji untuk mengetahui lebih lanjut informasi berkaitan dengan superkonduktor dan bendasing bukan superkonduktor sebagai pusat penyematan dalam bahan BPSCCO. Sampel untuk $(Bi_{1.6}(Pb_{0.4})Sr_2Ca_2Cu_3O_\delta)_{1-x}(M)_x$ dengan x=0.00-0.10 telah disediakan menggunakan kaedah tindak balas bahan pepejal. Bahan akan melalui proses pengkalsinan sebanyak 3 kali pada suhu 800^{o}C selama 20 jam, 820^{o}C selama 10 jam and 840^{o}C selama 4 jam dan persinteran menggunakan relau yang terdedah dengan udara pada suhu 855^{o}C selama 96 jam diikuti dengan pemanasan sepuh-lindap pada suhu 830^{o}C selama 30 jam. Sifat bahan telah diukur menggunakan Spektrum Pembelauan Sinar-X (XRD), Mikroskop Pengimbas Elektron (SEM), pengukur elektrik empat titik mengukur kerintangan dan sifat magnet menggunakan Kerentangan Arus Ulang Alik (ACS).

XRD menunjukkan bahawa semua sampel adalah merupakan fasa polihablur di mana sebahagian daripada puncak adalah Bi-2223 dan Bi-2212 seperti ditentukan menggunakan teknik pemurnian Rietveld. Sifat struktur seperti jumlah pecahan dan parameter kekisi dikira menggunakan persamaan jumlah peratusan puncak dan hukum Bragg's. Puncak XRD bertambah pada x= 0.02 (MgO, MgB_2 and YBCO) untuk semua sampel dan didominasi oleh Bi-2223 (sekitar 76%). Walau bagaimanapun jumlah pecahan fasa Bi-2223 berkurang dengan penambahan bahan ditambah. Tetapi sampel masih didominasi fasa Bi-2223 dan dengan perlahan penambahan fasa Bi-2212. Parameter kekisi dikira dari data XRD menunjukkan pengurangan pada paksi-*c* manakala paksi-*a* dan paksi-*b* bertambah. Pengurangan pada kekisi paksi-*c* kemungkinan disebabkan penambahan dalam lebihan oksigen . Paksi-*a* dan paksi-*b* bertambah berkemungkinan disebabkan oleh lebihan elektron valensi pada Cu menyebabkan pertambahan ikatan pada Cu-O.

Imej SEM menunjukkan struktur butiran sampel berbentuk serpihan plat butiran, di mana saiz butiran menjadi semakin kecil dengan kepekatan bahan ditambah. Unsur elemen di analisis dengan pengukur EDX menunjukkan kehadiran sampel bahan ditambah bertaburan dalam butiran Bi-2223.

Pengukur kerintangan menunjukkan semua sampel merupakan sifat metalik pada keadaan normal. Suhu kritikal $T_{C(R=0)}$ untuk sampel asal adalah 98 K dan kemudian bertambah kepada suhu yang lebih tinggi dengan x=0.02 (100 K, 99 K and 102 K), manakala dengan x>0.02, $T_{C(R=0)}$ menurun perlahan-lahan kepada suhu yang lebih rendah. Kepekatan lohong untuk sampel asal adalah 0.127 dan nilai ini berkurang dengan penambahn kepekatan bahan ditambah.

Sifat untuk ikatan lemah dan ikatan antara-butiran dikaji menggunakan teknik Kerentangan Arus Ulang Alik (ACS). Suhu peralihan diamagnet $T_{C\text{-}ON}$ telah dikaji dari bahagian sahih (χ'), dimana dua langkah peralihan merupakan ikatan superkonduktor dalam butiran dan antara butiran. Puncak kehilangan AC pada butiran dalam dan antara butiran dikaji dari bahagian maya (χ'') berubah kepada suhu rendah, dan tinggi dan lebar puncak bertambah menunjukkan semakin banyak kehilangan AC. Analisis untuk pergantungan suhu ACS berhampiarn peralihan suhu puncak kehilangan T_p telah dikaji oleh Model Keadaan Kritikal Bean. Peningkatan bahan ditambah menambahkan sifat ikatan lemah dan pengurangan ikatan antara butiran. Didapati fasa Bi-2212 dan pertambahan bahan ditambah pada sempadan butiran telah menyebabkan kesan pada ikatan lemah dan ikatan antara butiran. Pengiraan nilai untuk arus Josephson, I_o daripada kerentanan arus ulang alik menunjukkan nilai tertinggi adalah I_o=123.21 μA untuk penambahan YBCO (x=0.02) jika dibandingkan dengan bahan asal sampel dimana nilainya adalah I_o=119.83 μA. Sifat diamagnet berkurang dan berubah kepada suhu yang rendah kerana peningkatan pada penyambungan ikatan lemah.

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Arlina Ali

I certify that a Thesis Examination Committee has met on 3 July 2015 to conduct the final examination of Arlina binti Ali on her thesis entitled "Effects of MgO, MgB_2 and YBCO Addition on Formation, Microstructure and Superconducting Properties of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta$ Ceramic" 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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- the research conducted and the writing of this thesis was under our supervision;
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$T_{C(R=0)}$	Critical Temperature
$T_{C\text{-onset}}$	Onset temperature
p	Hole concentration
$\delta R/\delta T$	Derivative of resistance
H_{ac}	Critical magnetic fields
χ	Susceptibility
ACS	Alternating Current Susceptibility
I_o	Josephson current
χ'	Real Part
χ''	Imaginary Part
J_C	Critical current density
J_{cm}	Intergranular critical current density
J_{cg}	Intragranular critical current density
T_p	Temperature peaks
T_{cj}	Phase lock-in temperature
$T_{c\text{-on}}$	Diamagnetic onset temperature
BPSCCO	Bi-Pb-Sr-Ca-Cu-O
BCS theory	Barden, Cooper dan Schrieffer theory
LBCO	La-Ba-Cu-O system
YBCO	Y-Ba-Cu-O system
Y-123	Family member in $YBa_2Cu_3O_{7-x}$
Bi-2201	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$, n=0
Bi-2212	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n,}$ n=1
Bi-2223	Family member in $Bi_2Sr_2Ca_nCu_{n+1}O_{6+2n}$ n=3
TBCCO	Tl-Ba-Ca-Cu-O system
HBCCO	Hg-Ba-Ca-Cu-O system
Calcination	Heating process where the solid state reaction occur
Sintering	Heating process yielding for more compacting of the sample grains and improve its properties
MRI	Magnetic Resonance Image

SQUID	Superconducting Quantum Interference Device
XRD	X-ray diffraction
SEM	Scanning Electron Microscope
HTSC	High Temperature Superconductor
hkl	Miller indices
a, b, c	Lattice parameter

CHAPTER 1

INTRODUCTION

1.1 Brief Historical Review

Superconductors are materials that allow direct current to flow without any resistance below critical temperature $T_{C(R=0)}$. Superconductivity was discovered in 1911 by Heike Kamerlingh Onnes in Leiden, and later got Nobel Prize in Physics for his contribution of properties of materials at low temperature in 1913. Onnes found that below 4.2 K, the electrical resistance of mercury drops to immeasurable value. The second distinct property of superconductors appeared in 1933 when Meissner and Ochsenfeld noticed that magnetic flux is expelled from the interior of the superconductor which cooled below the critical temperature in weak external magnetic field.

The theory of phenomenon superconductivity was discovered by John Bardeen, Leon Cooper and Robert Schrieffer in 1957. BCS phenomenon was explains superconductivity at a state of zero resistance. The pair up electrons known as Cooper pairs are formed by electron phonon interaction (Mourachkine, 2001). According to the theory, the lattice will be distorted as a negative charged electron passes a positive charged ion in the lattice of a superconducting material.

Superconductivity was discovered in some metal, several compounds and alloys. An attention was given to A15 compound such as Nb_3Sn, Nb_3Ga and NbGe which exhibit $T_{C(R=0)}$ above 17 K, the highest transition temperature. But the complication is due to the high cost of liquid helium and the difficulty in its preparation.

1.2 High Temperature Superconductivity

High Temperature Superconductor (HTSC) in new oxide ceramics containing lanthanum (La-Ba-Cu-O) with a critical temperature of 35 K was discovered by Bednorz and Miller. This led to the discovery of superconductivity in several cuprate oxides with critical temperature more than 77 K (the boiling point liquid nitrogen), which offers a cheaper and an easier cooling than that used in low temperature superconductor by liquid helium. HTSC have layered crystal structures which are commonly consisted of conductive CuO_2 planes separated by other A elements and oxygen, A_mO_n layers. Table 1.1 gives some examples of the important systems in HTSC.

It is now well established that Bi-based cuprates superconductors belong to the homologue series $Bi_2Sr_2Ca_{n-1}Cu_nO_{Q}$ /with specific critical transition temperature T_c(s), ranging from 20 K (Bi-2201 phase), 80 K (Bi-2212 phase) and 110 K (Bi-2223 phase), for n=1,2 and 3, respectively. The structural difference between two consecutive phases is the addition of CuO_2-Ca-CuO_2 slab packed along the *c*-axis of the unit cell.

Table 1.1: Some important HTSC with their superconductor critical temperature (Maple, 1998).

HTSC system	Acronym	Critical Temperature $T_{C(R=0)}$(K)
$YBa_2Cu_3O_7$	Y-123	92
$YBa_2Cu_4O_8$	Y-124	80
$Bi_2Sr_2Ca_2Cu_3O$ /	Bi-2223	110
$Bi_2Sr_2CaCu_3O$ /	Bi-2212	80
$Tl_2Ca_2 Ba_2Cu_3O_{10}$	Tl-2223	125
$Hg_2 Ba_2Ca_2Cu_3O_{9-/}$	Hg-123	153
$Hg_2 Ba_2Ca_3Cu_3O_{9-/}$	Hg-1234	134

However the highest T_c(Bi-2223) phase in the series is not easy to prepare in a single phase form(Togana et. al, 1988). Partial substitution of lead Pb for Bi was found to increase the growth rate of the Bi-2223 phase (Fukushima, 1989). It was found, that substitution or addition of elements having different character can change the properties of the Bi-Pb-Sr-Ca-CuO material (Sasakura et al., 1998). Thus, the superconducting properties of the material are enhanced, suppressed or destroyed depending on the amount and the outcome of the additive or the substituent in the structure.

Structural and transport properties of superconductors such as superconducting phases, lattice parameters, grain shape and critical temperatures were generally characterized by X-ray diffractometer (XRD), Scanning Electron Microscopy (SEM) and the Four Point Probe devices under specific criteria in order to produce superconductors suitable for practical applications.

The complex AC susceptibility ($= $¶± *i*$¶¶ $ the response of a superconductor from the motion of flux lines and can be quantitatively studied to an AC magnetic field. The ACS technique has been widely used for characterization of superconductivity based on response field. It is generally accepted that the magnetic response of a high-temperature superconductor is associated with pinning of Josephson vortices formed along the grain boundaries at as well as pinning of Abrikosov-like vortices in the superconducting grains at sufficiently large fields (intregranular fields). AC susceptibility data are analyzed and interpreted in terms of %HDQV /DZ¶V WR XQGHUVWDQG WKH QDWXUHteRI FRXS some important physical parameter and temperature variation on the critical current density. Extensive studies of the theoretical and experimental aspect of ac losses were based on the critical state models for granular superconductivity.

1.3 Problem Statement

Superconductor is one of the materials that can provide high current for usage of various devices. High current usage include power transmission cables, high-efficiency generator, transformers and motors, current fault limiters and superconducting magnetic technology, example MRI scanners, levitated train system and energy storage systems (SMES). Some of the important features of superconducting tape are very high currents per unit cross-sectional area and negligible electric losses. Essentially, superconductors have zero electric resistance and perfect diamagnetism (Bay et al. 2004).

The problem of Bi-2223 is to appear as single phase since Bi-2212 and Bi-2201 would appear as secondary phases. Doping Pb at Bi site with 0.4 mol facilitates the formation of Bi(Pb)-2223 (Gul et al., 2006). Enhancing flux pinning and critical current density (Jc) is the most important goals of the research in the field of the high temperature superconductors for their use in practical application. This weakness that defines in the materials is low flux pinning strength and low critical current density (J_C). However, Bi-Sr-Ca-Cu-O (BSCCO) materials have several limitation to coherence length and penetration depth. This causes a drastic drop of critical current density across the grain boundary itself. The grain boundary problem is more serious as it affects the flux pinning and hence increases the weak link.

It is known that addition at small amount of magnetic materials (Huseyin et al., 2007) could produce defects within superconducting grains as dislocation or stacking faults, which enhance the flux pinning and critical current density (Annabi et al., 2004). In this work, addition of MgO, MgB_2 and YBCO in BPSCCO were studied. Pure Bi-2223 samples will separated by weak links grains (Goldfarb et. al., 1992). Addition with MgO is isolated by a non-superconducting matrix grain boundaries and contributed to the increase in the transition sharpness of the susceptibility (Bruneel et al., 1999), increasing the superconducting volume fraction in the samples and decreasing the total width of the transition (Christova et al., 2002). Addition with MgB_2 increased the flux pinning strength because intermetallic materials have longer coherence length without suffering the weak links. (Matsushita et al., 2010). The high levitation force, high trapped fields and self-stabilized levitation of high quality YBCO bulk make it possible to enhance flux pinning (Yang et al., 2013). This could shed more light in understanding the role and influence of grain boundaries to the superconducting properties in pinning effect.

1.4 Research Objective

The main objective of this study is to determine the effect of composite (MgO, MgB_2, YBCO)$_x$ addition in $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{/})_{1-x}$ superconducting system at various concentration x=0.00, 0.02, 0.04, 0.06, 0.08 and 0.10 prepared via conventional solid state reaction method. The composite also from alkaline earth, transition series element and rare earth element were added into $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{/})_{1-x}$ to play a role in change of superconducting mechanism. The addition of MgO is the potential candidate to enhance the pinning centers, MgB_2 is the most stable superconductor and YBCO is to identify the effect of same properties with the different system. The breakdown of these objectives can be given as follows:

i. To identify the phases formation of Bi-2223 system and its lattice parameter with MgO, MgB_2 and YBCO composite addition
ii. To investigate the effect of MgO, MgB_2 and YBCO composite addition on the superconducting properties of Bi-2223
iii. To identify the elemental composition and the distribution of MgO, MgB_2, YBCO in the added samples
iv. To study the effect of MgO, MgB_2, YBCO addition on the electrical properties of Bi-2223 systems such as critical temperature $T_{C(R=0)}$ and transition width, ûT
v. To study the response of magnetic field in intergranular properties of the samples at different magnetic fields and estimate the intergranular J_c X V L Q J % H D Q ¶ V 0 R G H O

1.5 Overview of the Thesis

Extensive background of literature and the current status of physical properties of Bi-based high temperature superconductor added with various materials is given in Chapter 2. The review of the AC susceptibility studies also included in the same chapter. 7 K H E D V L F W K H R U H W L F D O G H I L Q L W L R Q V R I V X S H U the AC susceptibility from the viewpoint of the critical state model are given in Chapter 3. A detailed description of the sample preparation and experimental methods which are used to perform the measurements are accounted in Chapter 4. Chapter 5 is devoted to results and discussion. The results consist of crystal structure, morphological images, temperature dependence of electrical properties, transport and the magnetic response of the samples. Finally, the relation between structure, microstructure, and transport properties of the samples is concluded and suggestions for possible continuation of the work are given in Chapter 6.

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