UNIVERSITI PUTRA MALAYSIA

EFFECT OF MAGNETIC NANOPARTICLE ADDITION ON THE SUPERCONDUCTING PROPERTIES OF Bi-Pb-Sr-Ca-Cu-O

HUSSEIN ABDULLAH HUSSEIN BAQIAH

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By

HUSSEIN ABDULLAH HUSSEIN BAQIAH

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

September 2009

DEDICATION

To my wife, my daughter and my son for
Their love, understanding and support......

To my mother, my father and family
For their concern and support.....

Abstract of thesis presented to the Senate of University Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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September 2009

Chairman: Professor Dr. Abdul Halim Shaari, PhD

Faculty: Science

The effect of magnetic nanoparticle additions on the $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta})_{1-x}$-(nano M)$_x$ with M= Sm_2O_3, Ho_2O_3, Nd_2O_3 and x= 0.0-0.05 systems, sintered at 850°C for 30 hours were investigated by X-ray diffraction techniques, critical temperature measurement, scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX). Magnetic nanoparticles, M= Sm_2O_3, Ho_2O_3 and Nd_2O_3 with 14.8 nm, 18 nm and 49-64 nm particle sizes respectively, were mixed with $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ precursor powder prepared by solid state reaction method before the final step heat treatment process. The phase purity, lattice parameters, superconducting properties, surface morphology and grain size were found to be dependent on the magnetic nanopatricles concentration in the sample.

The XRD result indicate that the dominant high T_c (Bi2223) phase decrease due to the increase of low T_c phase (Bi2212) with the presence of magnetic nanoparticles with $0<x\leq0.02$ and the later phase become significant for

further addition. The lattice parameters calculated from XRD data show a slight decrease in the *c*-axis while *a*-axis increase for initial nanoparticale addition. Lattice parameters decrease monotonically with $x \geq 0.02$.

The scanning electron microscopy viewing shows platelets like-grain for all samples which is a signature of Bi2223 and Bi2212 phases. The existence of large oriented platelet-like grains that have been observed in pure sample surface, are maintained for sample with $0 < x < 0.02$. Further more the previous samples have small, randomly oriented platelet-like grains that increase with the increase in magnetic nanoparticles content. For $x \geq 0.02$ the sample surface becomes more porous with large amount of randomly oriented platelet grains. The elemental analysis by EDX measurement of sample with $x = 0.05$ reveals the existence of nanoparticles that homogeneously distributed in BSCCO matrix. The chemical formula of sample's elements composition that has been estimated from EDX measurements is in good approximation to that of Bi2223 system with noticeable excess in oxygen ratio which may be due to the existence of magnetic oxide nanoparticles in the sample.

All samples exhibit normal metallic behavior above superconducting transition temperature. Zero resistivity temperature $T_{c\ (R=0)}$ which is around 102 K for pure sample was found to gradually decrease to lower temperature with magnetic nanoparticle additions and decrease to that of the low-T_c(2212) with $x \geq 0.02$. The onset transition temperature T_c is almost the same for sample with $0.005 \leq x \leq 0.02$ and become lower with higher concentration of addition. The superconducting transition width becomes wider with increase in the magnetic nanoparticles addition.

The hole concentration, *p*, of pure sample under preparation condition is around 0.13. The introduction of magnetic nanoparticles causes a decrease in the hole concentration of Bi2223 system. This decrease characterize by two steps. For initial addition of magnetic nanoparticle, the reduction of hole concentration per change in magnetic nanoparticles addition, $\Delta p/\Delta x$, is more than when $x>0.02$ for Ho_2O_3 and Nd_2O_3 and at $x>0.03$ for Sm_2O_3 addition.

Abstrak tesis yang dikemuakan kepada senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN PENAMBAHAN BUTIRAN NANO MAGNET KEATAS SIFAT SUPERKONDUKTOR Bi-Pb-Sr-Ca-Cu-O

Oleh

HUSSEIN ABDULLAH HUSSEIN BAQIAH

September 2009

Pengerusi: Profesor Abdul Halim Shaari, PhD

Fakulti: Sains

Kesan penambahan butiran nano magnet keatas sistem $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta})_{1-x}$(nano M)$_x$ dengan M= Sm_2O_3, Ho_2O_3, Nd_2O_3 dan x= 0.0-0.05 yang disinter pada 850°C selama 30 jam dikaji dengan teknik XRD, pengukuran suhu genting (T_c), mikroskopi elektron imbasan dan serakan tenaga sinar-X (EDX). Butiran nano magnet dicampur dengan serbuk pelopor $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ disediakan dengan kaedah keadaan pepejal sebelum langkah terakhir proses rawatan haba. Ketulenan fasa, parameter kekisi, sifat superkonduktor, morfologi permukaan dan saiz butiran dipercayai bergantung kepada kepekatan butiran nano magnet.

Keputusan XRD menunjukkan fasa dominan (Bi2223) berkurang berdasarkan pertambahan fasa (Bi2212) dengan kehadiran butiran nano magnet pada $0.0<x\leq 0.02$ dan kemudian fasa tersebut menunjukkan perbezaan besar untuk penambahan seterusnya. Pengiraan parameter kekisi dari data XRD menunjukkan sedikit pengurangan pada paksi-c manakala

penambahan pada paksi-a untuk penambahan awal butiran nano. Parameter kekisi berkurang secara monoton dengan $x \geq 0.02$.

Mikroskopi elektron imbasan menunjukkan kepingan seperti butiran untuk semua sampel yang menunjukkan kehadiran fasa Bi2223 dan Bi2212. Kehadiran kepingan butiran yang besar dan terjajar dapat diperhatikan dalam permukaan sampel tulen, hanya pada sampel $0 < x \leq 0.02$. Sampel yang terkemudian mempunyai butiran yang kecil, kepingan butiran terjajar bertambah dengan penambahan kandungan butiran nano magnet. Untuk sampel $x \geq 0.02$, permukaannya menjadi lebih poros disebabkan kandungan butiran kepingan rawak yang banyak. Analisis unsur dengan pengukuran EDX pada sampel $x=0.05$ menunjukkan kehadiran butiran nano yang homogen didalam matrik BSCCO. Formula kimia untuk komposisi elemen sampel yang telah dianggar dari pengukuran EDX menunjukkan sistem Bi2223 lebih peratusan oksigen yang ketara yang disebabkan oleh kehadiran butiran nano magnet oksida di dalam sampel.

Semua sampel menunjukkan sifat logam selepas suhu transisi superkonduktor. Suhu rintangan sifar T_c (R=0) pada 102 K untuk sampel tulen ketara berkurang ke suhu yang lebih rendah dengan penambahan butiran nano magnet dan berubah menjadi (Bi2212) pada $x \geq 0.02$. Permulaan suhu peralihan T_c, adalah hampir sama bagi kesemua sampel $0.005 \leq x \leq 0.02$ dan menjadi lebih rendah dengan pertambahan kepekatan. Lebar peralihan kesuperkonduksian bertambah dengan pertambahan nanozarah. Kepekatan lohong, *p*, sampel tulen semasa penyediaan adalah pada sekitar 0.13.

Pertambahan butiran nano magnet menyebabkan pengurangan kepekatan lohong pada sistem Bi2223. Pengurangan ini ditunjukkan dengan dua langkah. Penambahan awal butiran nano magnet telah mengurangkan kepekatan lohong setiap perubahan penambahan butiran nano magnet $\Delta p/\Delta x$, lebih daripada langkah kedua dimana $x>0.02$ untuk Ho_2O_3 dan Nd_2O_3 dan $x>0.03$ untuk pertambahan Sm_2O_3.

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May Allah Bless You All

I certify that a Thesis Examination Committee has met on 3rd September 2009 to conduct the final examination of Hussein Abdullah Hussein Baqiah on his thesis entitled " Effect of Magnetic Nanoparticles Addition on the Superconducting Properties of Bi-Pb-Sr-Ca-Cu-O " 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 Master of Science.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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DECLARATION

I declare that the thesis is my original work except for quotations and citation which have been duly acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

Hussein Abdullah Hussein Baqiah

Date:

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LIST OF SYMBOL AND ABBREVIATION

T	Temperature
T_c	Critical temperature
$T_{c\ onset}$	Onset critical temperature
$T_{c\ (R=0)}$	Zero resistance temperature
HTS	High temperature superconductors
LTS	Low temperature superconductors
BSCCO	Bi-Sr-Ca-Cu-O system
GL theory	Ginzburg-Landau theory
YBCO	Y-Ba-Cu-O system
k	Kelvin
BSC	Bardeen, Cooper, and Schrieffer theory
B	Magneticfield
H_c,H_{c1},H_{c2}	Critical magnetic field
e	Electron charge
h	Plank constant
φ	Magnetic flux
k_B	Boltzman constant
ξ	Coherence length
λ	Penetrating depth
R	Resistance
a,b,c	Lattice parameter

Bi2201 Phase member in $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with n=1

Bi2212 Phase member in $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with n=2

Bi2223 Phase member in $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with n=3

RE Rare earth elements

Sm_2O_3 Samarium Oxide

Ho_2O_3 Holmium Oxide

Nd_2O_3 Neodymium Oxide

A° Angstrom

φ Spatially varying phase

n_c Cooper pair density

ψ Quantum wave function

2Δ Width of energy gap

V_p Electron –phonon interaction factor

ω_D Phonon cut-off Debye frequency

k_e Elastic constant

STM Scanning tunneling microscopy

AFM Antiferromagnetic

p Hole concentration

M Isotope mass

θ Bragg angle

hkl Miller index

SEM Scanning Electron Microscope

ICDD International Center for Diffraction Data

XRD	X-Rays Diffraction
n_p	Magneton number
EDX	Elemental Compositional Analysis
FESEM	Field Emission Scanning Electron Microscope
ΔT	Superconducting transition width
Δp	Reduction of hole concentration
Δx	Changing of magnetic nanoparticles addition

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