



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 $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano M})_x$ with $M = \text{Sm}_2\text{O}_3, \text{Ho}_2\text{O}_3, \text{Nd}_2\text{O}_3$ 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, $M = \text{Sm}_2\text{O}_3, \text{Ho}_2\text{O}_3$ and Nd_2O_3 with 14.8 nm, 18 nm and 49-64 nm particle sizes respectively, were mixed with $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ 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 nanoparticle 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 $0 < x \leq 0.02$ 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 nanoparticle addition. Lattice parameters decrease monotonically with $x \geq 0.02$.

The scanning electron microscopy viewing shows platelet-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 dikemukakan 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

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Kesan penambahan butiran nano magnet keatas sistem $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano M})_x$ dengan $M = \text{Sm}_2\text{O}_3, \text{Ho}_2\text{O}_3, \text{Nd}_2\text{O}_3$ 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 $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ 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 terajar dapat diperhatikan dalam permukaan sampel tulen, hanya pada sampel $0 < x \leq 0.02$. Sampel yang terkemudian mempunyai butiran yang kecil, kepingan butiran terajar bertambah dengan penambahan kandungan butiran nano magnet. Untuk sampel $x \geq 0.02$, permukaannya menjadi lebih porous 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 $\Delta\rho/\Delta x$, lebih daripada langkah kedua dimana $x > 0.02$ untuk Ho_2O_3 dan Nd_2O_3 dan $x > 0.03$ untuk penambahan Sm_2O_3 .

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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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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:

LIST OF TABLES

Tables	Pages
1:1 Some important HTS and their approximate critical temperature.	6
1.2 Lattice parameters of superconducting phases in Bi–Sr–Ca–Cu–O system and of Pb substituted Bi2223	9
2.1 Summary of critical temperature of pure and added $\text{Bi}_{1.7}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_{1.1}\text{Cu}_{2.1}\text{RE}_x\text{O}_y$ system where RE=(La, Ce, Pr, Nd, Sm, Gd, Dy, Yb)	16
3.1 The critical temperatures of some superconducting phases in two HTS systems with different number of CuO_2 plane	31
5.1 Summary the lattice parameters of both Bi2223 and Bi2212 phases of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$	42
5.2 Summary the lattice parameters of both Bi2223 and Bi2212 phases of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$	45
5.3 Summary the lattice parameters of both Bi2223 and Bi2212 phases of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$	50
5.4 Superconducting transition parameter, T_c , ΔT of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$	75
5.5 The value of $d\rho/dT$ peak against temperature of and the peak width at half maximum $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hour	79
5.6 Superconducting transition parameter, T_c , ΔT of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$	82
5.7 The value of $d\rho/dT$ peak and width at half maximum of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hour	85



- 5.8 Superconducting transition parameters, T_c , ΔT of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$ 89
- 5.9 The value of $d\rho/dT$ peak and width at half maximum of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hour 93



LIST OF FIGURES

Figures	Page
1.1 A typical normal to superconducting transition at $T_c (R=0)$ curve	3
1.2 The Magnetic field-Temperature phase diagram of type I superconductors	4
1.3 Phase diagram of type II superconductors & schematic diagram of single vortex	5
1.4 Schematic crystal structures of the homologous series of $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$ superconductors with $n = 1$ ($\text{Bi}_2\text{Sr}_2\text{CuO}_6$, abbreviated as Bi2201), $n = 2$ ($\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ – Bi2212) and $n = 3$ ($\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ – Bi2223)	8
3.1 The energy gap symmetry above Fermi surface for LTS(a) & HTS(b)	28
3.2 Generic phase diagram of cuprate superconductors over hole doping in CuO_2 plane	29
4.1 Flow chart for preparation pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ superconductors	33
4.2 Schematic diagram of the four point probe technique	36
5.1 X-rays diffractions patterns of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ samples, sintered at 850°C for 30 hours, with $x=0.0-0.05$ Sm_2O_3 nanoparticle addition	40
5.2 Volume fraction of Bi2223 and Bi2212 phases against Sm_2O_3 nanoparticle addition	41
5.3 Unit cell volume of both Bi2223 and Bi2212 phases of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ samples with $x = 0.0-0.05$	42
5.4 X-rays diffractions patterns of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples, sintered at 850°C for 30 hours, with $x=0.0-0.05$ Ho_2O_3 nanoparticles addition	44
5.5 Volume fraction of Bi2223 and Bi2212 phases against Ho_2O_3 nanoparticle addition	44
5.6 Unit cell volume of both Bi2223 and Bi2212 phases of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples with $x = 0.0-0.05$	46
5.7 X-rays diffractions patterns of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples sintered at 850°C for 30 hours, with $x=0.0-0.05$ Nd_2O_3 nanoparticle addition	48



5.8	Volume fraction of Bi2223 and Bi2212 phases against Nd ₂ O ₃ nanoparticle addition	49
5.9	Unit cell volume of both Bi2223 and Bi2212 phases of (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Nd ₂ O ₃) _x samples with x= 0.0-0.05	50
5.10	Atomic ratio percentage of element composition Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ} -(nano Sm ₂ O ₃) _x sample with x= 0.05	57
5.11	Atomic ratio percentage of element composition Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ} -(nano Ho ₂ O ₃) _x sample with x= 0.05	64
5.12	Atomic ratio percentage of element composition (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Nd ₂ O ₃) _x sample with x= 0.05	71
5.13	Normalized resistance–temperature of (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Sm ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30 hours.	74
5.14	Hole concentration dependence-critical temperature T _c (K) for (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Sm ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30 hours	76
5.15	Hole concentration – Sm ₂ O ₃ nanoparticle addition graph of (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Sm ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30 hours	77
5.16	Derivative of resistance dp/dT against temperature graphs of (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Sm ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30 hours	79
5.17	Normalized resistance –temperature of (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Ho ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30hours.	81
5.18	Hole concentration-critical-temperature T _c (K) dependence for (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Ho ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30 hours	83
5.19	Hole concentration – Ho ₂ O ₃ nanoparticle addition graph of (Bi _{1.6} Pb _{0.4} Sr ₂ Ca ₂ Cu ₃ O _{10+δ}) _{1-x} (nano Ho ₂ O ₃) _x samples with x= 0.0-0.05, sintered at 850°C for 30 hours	84



5.20	Derivative of resistance dp/dT against temperature graphs of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hours	86
5.21	Normalized resistance –temperature of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, after final sintering at 850°C for 30hours	88
5.22	Hole concentration -critical temperature $T_c(\text{K})$ dependence for $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hours	90
5.23	Hole concentration – Nd_2O_3 nanoparticle concentration graph of of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hours	91
5.24	Derivative of resistance dp/dT against temperature graphs of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ samples with $x= 0.0-0.05$, sintered at 850°C for 30 hours	93



LIST OF PLATES

Plates	Pages
4.1 X'Pert HighScore diffractometer	37
4.2 Scanning Electron Microscopy (SEM) model (JEOL: JSM-6400)	38
5.1 SEM micrograph of Pure $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ superconductors sintered at 850°C for 30 hours	51
5.2 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ with $x=0.005$ superconductors sintered at 850°C for 30 hours	53
5.3 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ with $x=0.01$ superconductors sintered at 850°C for 30 hours	53
5.4 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ with $x=0.02$ superconductors sintered at 850°C for 30 hours	54
5.5 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ with $x=0.03$ superconductors sintered at 850°C for 30 hours	54
5.6 Areas of X-rays spectrum in the same micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ with $x=0.05$	56
5.7 a)The distribution of Sm_2O_3 nanoparticles in $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ sample with $x=0.05$ from cross section viewer, (b) inset mapping of Sm ions.	57
5.8 The distribution of Sm_2O_3 nanoparticles in $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Sm}_2\text{O}_3)_x$ sample with $x=0.05$ from surface viewer,(b) inset mapping of Sm ions.	58
5.9 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ with $x=0.005$ superconductors sintered at 850°C for 30 hours	60
5.10 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ with $x=0.01$ superconductors sintered at 850°C for 30 hours	60
5.11 SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ with $x=0.02$ superconductors sintered at 850°C for 30 hours	61



5.12	SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ with $x=0.03$ superconductors sintered at 850°C for 30 hours	61
5.13	Areas of X-rays spectrum in the same micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ with $x=0.05$	63
5.14	(a) The distribution of Ho_2O_3 nanoparticles in $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ sample with $x=0.05$ from cross section viewer, (b) inset mapping of Ho ion.	64
5.15	(a) The distribution of Ho_2O_3 nanoparticles in $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Ho}_2\text{O}_3)_x$ sample with $x=0.05$ from surface viewer, (b) , inset mapping of Ho ions .	65
5.16	SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ with $x=0.005$ superconductors sintered at 850°C for 30 hours	67
5.17	SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ with $x=0.01$ superconductors sintered at 850°C for 30 hours	67
5.18	SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ with $x=0.02$ superconductors sintered at 850°C for 30 hours	68
5.19	SEM micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ with $x=0.03$ superconductors sintered at 850°C for 30 hours	68
5.20	Areas of X-rays spectrum in the same micrograph of $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ with $x=0.05$	70
5.21	(a) The distribution of Nd_2O_3 nanoparticles in $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ sample with $x=0.05$ from cross section viewer, (b) , inset mapping of Nd ions.	71
5.22	The distribution of Nd_2O_3 nanoparticles in $(\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta})_{1-x}(\text{nano Nd}_2\text{O}_3)_x$ sample with $x=0.05$ from surface viewer, inset mapping of Nd ions .	72



LIST OF SYMBOL AND ABBREVIATION

T	Temperature
T_c	Critical temperature
$T_{c \text{ onset}}$	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	Magnetic field
H_c, H_{c1}, H_{c2}	Critical magnetic field
e	Electron charge
h	Planck constant
ϕ	Magnetic flux
k_B	Boltzmann constant
ξ	Coherence length
λ	Penetrating depth
R	Resistance
a, b, c	Lattice parameter



Bi2201	Phase member in $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$ with $n=1$
Bi2212	Phase member in $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$ with $n=2$
Bi2223	Phase member in $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$ with $n=3$
RE	Rare earth elements
Sm_2O_3	Samarium Oxide
Ho_2O_3	Holmium Oxide
Nd_2O_3	Neodymium Oxide
Å	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



TABLE OF CONTENTS

	Page
DEDICATION	ii
ABSTRACT	iii
ABSTRAK	vi
ACKNOWLEDGEMENT	ix
APPROVAL SHEETS	x
DECLARATION	xi
LIST OF TABLES	xii
LIST OF FIGURES	xiv
LIST OF PLATES	xvii
LIST OF SYMBOL AND ABBREVIATIONS	xix
 CHAPTER	
1 INTRODUCTION	1
1.1 Brief Historical Review	1
1.2 Superconductivity : a Brief Overview	3
1.3 High Temperature Superconductors	5
1.4 BSCCO System	7
1.5 Research Objectives	9
 2 LITERATURE REVIEW	 11
2.1 Effect of Nanoparticle addition on BSCCO system	11
2.2 Effect of Rare Earth on BSCCO System	14
2.2.1 Effect of Rare Earth on Bi2212 Phase	14
2.2.2 Effect of Rare Earth on Bi2223 Phase	16
2.3 Effect of Normal powders Addition on BSCCO System	22
 3 MICROSCOPIC THEORY AND HIGH TEMPERATURE SUPERCONDUCTIVITY	 25
3.1 BCS Theory	25
3.2 Superconductivity In HTS	27
 4 METHODOLGY	 32
4.1 Chemical preparation	32
4.1.1 Pure Sample Preparation	32
4.1.2 Addition Of Nanoparticles	34
4.2 Characterization techniques	35
4.2.1 Resistivity Measurements	35
4.2.2 X-rays Diffraction Measurements	36



4.2.3	Microstructure Analysis	38
5	RESULTS AND DISCUSSION	39
5.1	X-Rays Diffractions Measurements	39
5.1.1	Effect Of Sm ₂ O ₃ Nanoparticle Addition	39
5.1.2	Effect of Ho ₂ O ₃ Nanoparticle addition	43
5.1.3	Effect of Nd ₂ O ₃ Nanoparticle Addition	47
5.2	Microstructure and EDX analysis	51
5.3.1	Pure Sample Morphology	51
5.2.1	Effect of Sm ₂ O ₃ Nanoparticle Addition	52
5.2.2	Effect of Ho ₂ O ₃ Nanoparticle Addition	59
5.2.3	Effect of Nd ₂ O ₃ Nanoparticle Addition	66
5.3	Resistivity Measurements	73
5.3.2	Effect of Sm ₂ O ₃ Nanoparticle Addition	73
5.3.3	Effect of Ho ₂ O ₃ Nanoparticle Addition	80
5.3.4	Effect of Nd ₂ O ₃ Nanoparticle Addition	86
6	CONCLUSION	94
	RECOMMENDATION FOR FUTURE WORK	96
	REFERENCES	99
	APPENDICES	102
	BIODATA OF STUDENT	106
	LIST OF PUBLICATIONS	109

