UNIVERSITI PUTRA MALAYSIA

EFFECTS OF NANO-(Pr_6O_{11},CeO_2) ADDITION AND Pr, Ce DOPING IN Bi-2223 SUPERCONDUCTOR

NUR AMALINA HAFIZA BINTI AHMAD NIZAR

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By

NUR AMALINA HAFIZA BINTI AHMAD NIZAR

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

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DEDICATION

To my husband, and my daughter 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 Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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The effect of doping and addition of Pr and Ce in Bi-2223 system sintered at 850 ^{o}C for 48 hours were investigated by X-ray diffraction techniques (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDX) and critical temperature measurement. $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Pr_xCu_3O_\delta$ and $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Ce_xCu_3O_\delta$ with x=0.00-0.10; $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta)_{1-x}$(nano Pr_6O_{11}) and $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta)_{1-x}$(nano CeO_2) with x=0.00 - 0.03 were prepared by solid state reaction method. The phase purity, lattice parameters, superconducting properties, surface morphology and grain size were found to be dependen on Pr and Ce concentration in the sample.

The XRD results patterns show that all Pr and Ce samples contain 2212 peaks which correspond to the low-superconducting phase. The intensity of these peaks increases towards higher value, as the Pr and Ce concentration increases. The volume of high 2223 phase decreases gradually as the Pr and Ce concentration increases. The lattice

parameters calculated from XRD data show a slight decrease in the *c*-axis while *a*-axis increase with the increase of Pr and Ce concentration.

The scanning electron microscopy viewing shows platelets like-grain for all samples which is a signature of high 2223 and low 2212 phases. The elemental analysis by EDX measurement of sample reveals the existence of Pr and Ce that are homogeneously distributed in BSCCO matrix. The chemical formula of sample elements composition that has been estimated from EDX measurements is in good approximation to that Bi2223 system with noticeable excess in oxygen ratio which may be due to the existence of Pr and Ce in sample.

The study shows that Pr and Ce do not improve the T_c of BSCCO system. This is due to the existence of low 2212 phase which weakened the coupling of grains.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains.

KESAN-KESAN NANO-(PR_6O_{11},CEO_2) PENAMBAHAN DAN PR,CE PENDOPAN DALAM Bi-2223 SUPERKONDUKTOR

Oleh

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Kesan pendopan dan penambahan Pr dan Ce di dalam system Bi-2223 yang disinter pada suhu 850 ^{o}C selama 48 jam dikaji dengan teknik XRD, miskroskopi electron imbasan, serakan tenaga sinar-X (EDX) dan pengukuran suhu genting T_c. $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Pr_xCu_3O_\delta$ dan $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Ce_xCu_3O_\delta$ dengan komposisi x=0.00 - 0.10; $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta)_{1-x}$(nano Pr_6O_{11}) dan $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta)_{1-x}$(nano CeO_2) dengan komposisi x=0.0-0.03 disediakan dengan kaedah keadaan pepejal. Ketulenan fasa, parameter kekisi, sifat superkonduktor, morfologi permukaan dan saiz butiran dipercayai bergantung kepada kepekatan Pr dan Ce di dalam sampel.

Keputusan XRD menunjukkan kesemua sampel Pr dan Ce mengandungi puncak-puncak fasa 2212 dan ini menunjukkan kehadiran fasa rendah. Keamatan puncak-puncak ini bertambah apabila kepekatan Pr dan Ce bertambah. Isipadu fasa tinggi 2223 menurun berperingkat-peringkat mengikut peningkatan kepekatan Pr dan Ce. Pengiraan parameter kekisi dari data XRD menunjukkan sedikit pengurangan pada paksi-*c* manakala penambahan pada paksi-*a* mengikut penambahan kepekatan Pr dan Ce.

Mikroskopi electron imbasan menunjukkan kepingan seperti butiran untuk semua sampel yang menunjukkan kehadiran fasa tinggi 2223 dan fasa rendah 2212. Analisis unsure dengan pengukuran EDX menunjukkan kehadiran Pr dan Ce homogen di dalam matrix BSCCO. Formula kimia untuk komposisi elemen sampel yang telah dianggar dari pengukuran EDX menunjukkan system Bi2223 lebih peratusan oksigen yang ketara yang disebabkan kehadiran Pr dan Ce di dalam sampel.

Kajian ini menunjukkan bahawa kesan Pr dan Ce tidak meningkatkan suhu genting T_c bagi system BSCCO. Ini adalah disebabkan oleh pembentukan fasa rendah 2212 yang melemahkan gandingan butiran.

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In the Name of ALLAH, the most Merciful and Beneficent

First and foremost, I bow my head before Allah Almighty Who Blessed me with good health and vital resources to complete this project. I am extremely thankful to Prof. Dr. Abdul Halim Shaari for his support, guidance and kindness that help me to complete this project. He is always very helpful and share his knowledge whenever I encounter project problems. I am grateful for all his advices and guidance.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment 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 hereby declare that the thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Putra Malaysia or any other institutions.

NUR AMALINA HAFIZA AHMAD NIZAR

Date: 20 June 2013

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LIST OF SYMBOLS AND ABBREVIATIONS

T	Temperature
T_c	Critical Temperature
$T_{c\ onset}$	Onset critical temperature
$T_{c(R=0)}$	Zero Resistance Temperature
HTS	High Temperature Superconductor
LTS	Low Temperature Superconductor
BSCCO	Bi-Sr-Ca-Cu-O system
k	Kelvin
BCS	Bardeen, Copper and Schrieffer Theory
B	Magnetic Field
H_c,H_{c1o},H_{c2o}	Critical Magnetic Field
a,b,c	Lattice Paremeter
R	Resistance
Bi2201	$Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with n = 1
Bi2212	$Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with n = 2
Bi2223	$Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ with n = 3
Pr_6O_{11}	Praseodymium Oxide
CeO_2	Cerium oxide
XRD	X-Rays Diffraction
SEM	Scanning Electron Microscope
ΔT	Superconducting Transition Width
AFM	Antiferromagnetic

CHAPTER 1
INTRODUCTION

1.1 Historical Review

Superconductivity was first observed in mercury in 1911 by Dutch physicist Heike Kamerlingh Onnes of Leiden University. In 1908, Onnes had become the first person to liquefy helium. He noticed that below 4.2 K, the electrical resistance of mercury dropped to a value below the resolution of his instruments while he was investigating the electrical properties of various substances.

The element lead (Pb) was found to be superconducting at 7.2 K in 1913 and after 17 years, the element niobium (Nb) was found to be superconducting at 9.2 K. In subsequent decades other superconducting metals, alloys and compounds were discovered. The A15 type compounds Nb_3Sn, Nb_3Ga and Nb_3Ge was given an attention because they exhibit T_c above 17 K. Nb_3Ge had the highest T_c of 23.2 K known up to 1987. Nb compound has been used to fabricate superconducting wires that was used to produce strong magnetic field and his many practical applications. However, the major problem is due to the high cost and maintenance of liquid helium and difficulty in its preparation. The developing materials with higher transition temperatures progress very slow before 1986. As seen from Table 1.1, that in only 10 years since 1986, the transition temperature was raised progressively from 23 K to 35, 52, 95, 110, 125 and 133: applying a pressure of 30 GPa raised the T_c of the mercury superconductor to 147K (Owens and Poole, 2002).

Table 1.1: Progress in Rising the Superconducting Transition Temperature T_c Since the Discovery of Cuprates in1986, (Source: Owens and Poole, 2002)

Material	T_c (K)	Year
$Ba_2La_{5-x}Cu_3O_9$	30 - 35	1986
$(La_{0.9}Ba_{0.1})_2Cu_4O_{4-x}$ (at 1-GPa pressure)[a]	52	1986
$YBa_2Cu_3O_{7-x}$	95	1987
$Bi_2Sr_2Ca_2Cu_3O_{10}$	110	1988
$Ti_2Ba_2Ca_2Cu_3O_{10}$	125	1988
$Ti_2Ba_2Ca_2Cu_3O_{10}$ (at 7-GPa pressure)	131	1993
$HgBa_2Ca_2Cu_3O_{8+x}$	133	1993
$HgBa_2Ca_2Cu_3O_{10}$ (at 30-GPa pressure)	147	1994

[a]A pressure of 1 GPa is about 10,000 atm.

1.2 Bi-Sr-Ca-Cu-O Superconductors

After the discovery of high-T_c superconductors in the Bi-Sr-Ca-Cu-O system by Maeda et al., (1988), Tallon et al., (1988) reported that Bi-compounds (BSCCO) posses three superconducting transitions at 110 K (2223 phase), 80 K (2212 phase) and 10 K (2201 phase), with triple, double and single Cu-O layer respectively. The high-T_c (2223) phase is extremely difficult to prepare in single phase (Subramaniam et al., 1988). Structure analysis indicated that the compounds have an orthorhombic or tetragonal distorted perovskite structure.

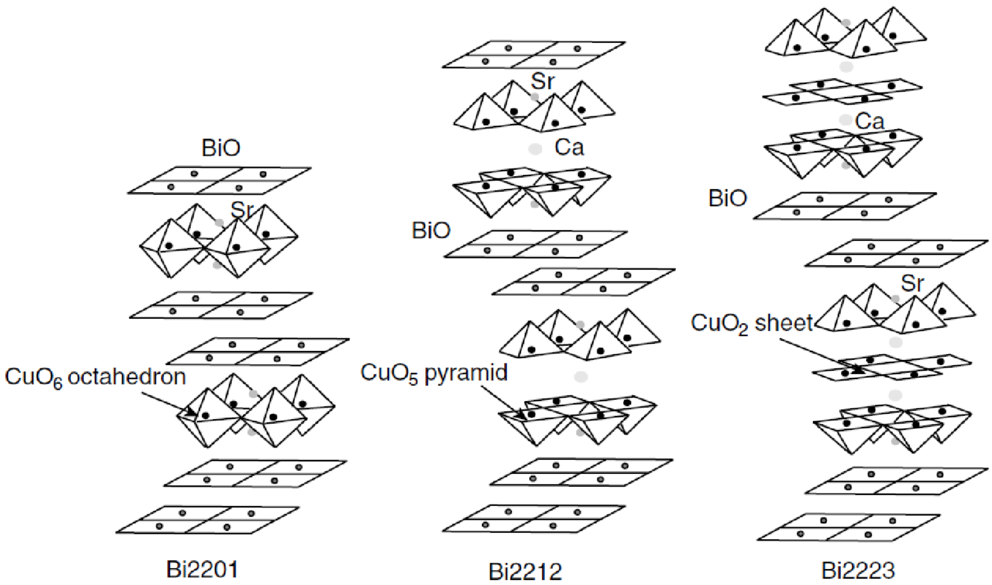

Figure 1.1: Crystallographic structure of $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4}$ superconductors system with n =1 ($Bi_2Sr_2CuO_6$, abbreviated as Bi2201), n = 2 ($Bi_2Sr_2CaCu_2O_8$ - Bi2212) and n = 3 ($Bi_2Sr_2CaCu_3O_{10}$ - Bi2223). Oxygen atoms are not drawn for simplicity. (Source: Kamimura et al., 2005)

The crystallographic structures for 2201, 2212 and 2223 phases of the BSCCO system are shown in Figure 1.1. All the phases contain double sheets of corner sharing square plane CuO_2. When n = 0 in 2201 phase, there is only one CuO_2 plane and no Ca. In 2212 phase with one Ca sandwiches with two CuO_2 planes and 2223 phase, two layer of Ca interleaves three layers of CuO_2. The spacing from CuO_2 to Ca layer is 1.66 Å. There exist BiO double layers, which are not exactly stacked over each other. Thus, the lattice parameter *c* changes by a factor of about one-half the *c* parameter in corresponding members of the layered series. The lattice is pseudo-tetragonal (a = b γ c) with space group 14/mmm (Tarascon et al., 1989). The length of lattice parameters for single phases in BSCCO system is shown in Table 1.2 (Debsidkar, 1989 and Bourdillon, 1993).

Table 1.2: The length of lattice parameters for single phases in BSCCO system.

Bi-Sr-Ca-Cu-O Compound	**Phase**	**a (Å)**	**b (Å)**	**C (Å)**
$Bi_2Sr_2CuO_6$	2201	5.4	5.4	24.6
$Bi_2Sr_2CaCu_2O_8$	2212	5.4	5.4	30.7
$Bi_2Sr_2CaCu_3O_{10}$	2223	5.4	5.4	37.1

1.3 Application of Superconductors

Two important discoveries at that time led to two types of applications called small scale and large scale.

1.3.1 Small-scale applications

YBCO is widely used for the High Temperature Scale (HTS) small-scale devices because it has a high critical temperature and can accommodate high current densities. The best-known small-scale device is the Superconducting Quantum Interference Device (SQUID) magnetometer. A SQUID contains a ring of superconductor with one or more Josephson junction. When a current is introduced into the SQUID that is the larger than the critical current of the Josephson junction a voltage appears that is proportional to the magnetic flux through the SQUID ring. The SQUID is so exceedingly sensitive that it can detect magnetic field 100 billion times smaller than the Earth's field, it can also detect any other physical quantity that can be converted to magnetic flux.

1.3.2 Large-scale applications

Low-temperature superconductors, LTS, are already in quite common use for large-scale applications, but still have stability problems due to the complex and expensive refrigeration required. Large-scale applications for HTS materials present a major challenge to the materials scientist. Compared with the small-scale applications, a large-scale application generally means that much larger currents and lengths of superconductors are required in a working environment where the magnetic field may be several Tesla. The most important applications under consideration are magnets, power transmission cables, current leads, fault current limiters, transformers, generators, motors, magnetic resonance imaging (MRI), levitated trains (MAGLEV Trains) and magnetic energy storage. HTS superconductors would simply replace conventional conductors, e.g. a copper winding in an electromagnet is replaced by a BSCCO tape winding.Fault current limiters for electrical utilities depending on the fact that superconductivity is lost and resistance appears above a critical current. Under normal conditions the fault current limiter is superconducting and offers no impedance to the ordinary current. During a power surge the large fault current exceeds the critical current and is limited by the consequent resistance as the superconductor goes normal. The superconductivity returns after the current spike (Hassenzahl, 2000).

1.4 Problems Statement

Electrons in superconductors are known to join together in pairs in order to gain immunity from scattering, but exactly how this occurs in many materials has yet to be established. It is very difficult to prepare single phase of Bi2223. The Bi2212 phase appears only after a few hours of sintering at approximately 860–870 °C, but the larger fraction of the Bi2223 phase is formed after a long reaction time of more than a week at approximately 870 °C. Although the substitution of Pb in the Bi–Sr–Ca–Cu–O compound has been found to promote the growth of the high T_c phase, a long sintering time is still required. Doping and addition of rare earth oxide is expected to play an important role to improve the superconducting of BSCCO system. It has been rarely reported a doping and addition of BSCCO with rare earth.

1.5 Objectives

The main objectives is to study the effect of (Pr_6O_{11}, CeO_2) doping and addition on the superconducting properties of Bi2223 system. The break down of these objectives is explained as follows:

1. To prepare the $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}(Pr,Ce)Cu_3O_\delta$ and $(Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_\delta)_{1-x}$ (nano M)$_x$ where M=Pr_6O_{11}, CeO_2 samples using solid state method reaction and to identify the phases formation and determine its lattice parameters using X-ray diffraction techniques (XRD).
2. To investigate the surface morphology using scanning electron microscopy.
3. To investigate the electrical properties such as critical temperature $T_{c(R=0)}$, onset critical temperature $T_{c\ onset}$, and superconducting transition width, ΔT.

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