UNIVERSITI PUTRA MALAYSIA

EFFECT OF La0.67A0.33MnO3 (A= Ba, Ca, Sr) MANGANITES ON THE SUPERCONDUCTING PROPERTIES OF YBa2Cu3O7-δ

NUR FADILAH BINTI BAHARUDDIN PALLAN

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By

NUR FADILAH BINTI BAHARUDDIN PALLAN

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

June 2013

DEDICATION

I dedicate this thesis to my family especially my beloved parents (Baharuddin Pallan Bin Abdullah and Kathijah Abdul Kader), my beloved siblings (Faizal, Fairuz, Faizah), sister in-law (Farizah, Imani), nieces (Farisya, Fatin, Filza), and also to all my friends.

Abstract of thesis presented to Senate of Universiti Putra Malaysia in fulfillment of
the requirements for the degree of Master of Science

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June 2013

Chairman: Professor Abdul Halim Bin Shaari, PhD

Faculty: Science

The discovery of $YBa_2Cu_3O_{7-\delta}$ (YBCO) high temperature superconductors (HTS) has attracted worldwide researchers to study its ability to trap large magnetic flux. In this research, the effects on structural, phases, transport properties, and microstructural properties were investigated after addition of manganites in YBCO superconductor. Characterizations of samples were carried out by using x-ray diffraction (XRD), AC susceptometer (ACS), and scanning electron microscope (SEM).

The samples that have prepared were pure YBCO and for the addition of manganites in YBCO superconductor, the manganites were $La_{0.67}Ca_{0.33}MnO_3$ (LCMO), $La_{0.67}Ba_{0.33}MnO_3$ (LBMO), and $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) with 0.2 wt%, 0.4 wt%, 0.6 wt%, 0.8 wt%, and 1.0 wt%. The polycrystalline samples of pure $YBa_2Cu_3O_{7-\delta}$ and addition of manganites in YBCO were prepared by the solid state reaction method.

The crystal structure for pure $YBa_2Cu_3O_{7-\delta}$ is orthorhombic with space group Pmmm. XRD patterns showed the orthorhombic structure were retained for addition of LCMO at 0.2 wt% - 0.6 wt%. The structure changed to tetragonal after added with

0.8 wt% - 1.0 wt% of LCMO. The crystal structure was tetragonal after addition with 0.2 wt% - 1.0 wt% of LBMO. The crystal structure was tetragonal after addition with 0.2 wt% - 1.0 wt% of LSMO.

AC Susceptometer measurement for manganite addition in YBCO revealed that the diamagnetic onset temperature, T_{c-on} decreases as the wt% of addition was increased. The temperature dependence ac susceptibility data for real part (χ') showed T_{c-on} shifting towards lower temperature on all types of manganites addition as the weight percentage increased. The imaginary component, χ'' showed decreases in the phase lock-in temperature (T_p) towards lower temperatures as the weight percentage of manganites increased. For pure YBCO, T_{c-on} is 90.2 K. The first part manganite addition is LCMO, highest T_{c-on} is 91.1 K for addition 0.2 wt%. The highest T_{c-on} is 91.0 K obtained from sample addition 0.8 wt% of LBMO. Addition 0.4 wt% of LSMO showed T_{c-on} 90.3 K as the highest T_{c-on} for the third part of manganites addition. Overall, the highest T_{c-on} obtained when used low ac field, 0.1 Oe for all types of manganites..

Addition of LCMO, LBMO, and LSMO in YBCO showed that the grains size decreases as the weight percentage of addition increased. The morphology of fractured surface and grains started to agglomerate for manganites in the range of 0.6 wt% until 1.0 wt%. When the amount addition of manganites increased, it's changed the crystal structure because of oxygen deficiency and lattice parameter decreased. When the samples are not fully oxygen loaded it's gave effect decreases of T_{c-on}. Role oxygen content in YBCO related with the dependence of T_{c-on} and lattice parameters of the YBCO compound. The decreases in T_{c-on} were due to poor connectivity among grains in the samples.

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

KESAN $La_{0.67}A_{0.33}MnO_3$ (A = Ba, Ca, Sr) MANGANIT KE ATAS SIFAT-SIFAT SUPERKONDUKTOR $YBa_2Cu_3O_{7-\delta}$

Oleh

NUR FADILAH BINTI BAHARUDDIN PALLAN

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Pengerusi: **Professor Abdul Halim Bin Shaari, PhD**

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Penemuan superkonduktor suhu tinggi (HTS) $YBa_2Cu_3O_{7-\delta}$ (YBCO) pukal telah menarik perhatian penyelidik di seluruh dunia untuk mengkaji keupayaan untuk memerangkap ketumpatan fluks magnet yang besar. Dalam kajian ini, kesan pada struktur, fasa-fasa, sifat pengangkutan, dan sifat-sifat mikrostruktur telah disiasat selepas penambahan manganit pada YBCO superkonduktor. Pencirian sampel telah dijalankan dengan menggunakan pembelauan sinar-x (XRD), AC susceptometer (ACS), dan mikroskop elektron imbasan (SEM).

Sampel YBCO tulen telah disediakan untuk manganit tambahan dalam superkonduktor YBCO terdiri daripada $La_{0.67}Ca_{0.33}MnO_3$ (LCMO), $La_{0.67}Ba_{0.33}MnO_3$ (LBMO), dan $La_{0.67}Sr_{0.33}MnO_3$ (LSMO) dengan penambahan 0.2% berat, 0.4% berat, 0.6% berat, 0.8% berat, dan 1.0% berat. Sampel polihablur tulen $YBa_2Cu_3O_{7-\delta}$ dan penambahan manganit dalam YBCO telah disediakan dengan kaedah tindak balas keadaan pepejal.

Struktur hablur $YBa_2Cu_3O_{7-\delta}$ tulen adalah otorombik dan mempunyai ruang kumpulan Pmmm. Corak XRD telah menunjukkan struktur otorombik kekal untuk

penambahan LCMO pada 0.2% berat - 0.6% berat. Sistem hablur telah berubah menjadi tetragon selepas ditambah dengan 0.8% berat - 1.0% berat LCMO. Struktur hablur adalah tetragon selepas penambahan dengan 0.2% berat - 1.0% berat LBMO. Struktur hablur adalah tetragon selepas penambahan dengan 0.2% berat – 1.0 % berat LSMO.

Pengukuran AC Susceptometer untuk penambahan manganit dalam YBCO menunjukkan penurunan suhu mula diamagnet, $T_{c\text{-}on}$ apabila peratusan berat tambahan semakin meningkat. Pergantungan suhu menunjukkan data kerentanan ac menunjukkan komponen sebenar (χ') beralih pada diamagnet $T_{c\text{-}on}$ kepada suhu rendah pada semua jenis penambahan manganit apabila peratusan berat meningkat. Komponen khayalan, χ'' telah menunjukkan pengurangan suhu fasa kunci dalam (T_p) terhadap suhu yang rendah apabila peratusan berat manganit meningkat. Untuk YBCO tulen, keputusan yang diperolehi $T_{c\text{-}on}$ adalah 90.2 K. Penambahan bahagian pertama ialah LCMO, suhu $T_{c\text{-}on}$ tertinggi adalah 91.1 K untuk 0.2% berat. Untuk bahagian kedua, suhu $T_{c\text{-}on}$ tertinggi adalah 91.0 K yang diperolehi daripada sampel tambahan LBMO 0.8% berat. Penambahan LSMO 0.4% berat memperolehi suhu tertinggi $T_{c\text{-}on}$ 90.3 K untuk bahagian ketiga penambahan manganit. Secara keseluruhannya, $T_{c\text{-}on}$ tertinggi diperolehi apabila menggunakan medan ac yang rendah iaitu 0.1 Oe ke atas semua jenis manganit.

Penambahan LCMO, LBMO, dan LSMO dalam YBCO menunjukkan bahawa saiz butiran berkurangan apabila peratusan berat tambahan meningkat. Morfologi permukaan yang berderai dan butiran yang mula menggumpal berlaku apabila kenaikan manganit dalam julat 0.6% sehingga 1.0% berat. Apabila jumlah penambahan manganit meningkat, ia mengubah struktur kristal kerana kekurangan oksigen dan parameter kekisi menurun. Apabila sampel tidak sepenuhnya dimasukkan oksigen ia memberi kesan penurunan pada $T_{c\text{-}on}$. Peranan kandungan oksigen dalam YBCO berkaitan dengan pergantungan $T_{c\text{-}on}$ dan parameter kekisi kompaun YBCO itu. Penurunan $T_{c\text{-}on}$ adalah kerana rangkaian yang lemah di kalangan butiran dalam sampel.

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I certify that a Thesis Examination Committee has met on **21 JUNE 2013** to conduct the final examination of Nur Fadilah Binti Baharuddin Pallan on her thesis entitled "**Effect of $La_{0.67}A_{0.33}MnO_3$ (A= Ba, Ca, Sr) Manganites on the Superconducting Properties of $YBa_2Cu_3O_{7-\delta}$**" 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 this 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, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

NUR FADILAH BAHARUDDIN PALLAN

Date: 21 June 2013

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LIST OF ABBREVIATIONS

δ	Differentiation
θ	Diffraction angle
λ	Wavelength
μ	Micron
π	= 180º
$T_{c\text{-}on}$	Transition temperature onset
T_p	Phase lock-in temperature
χ'	Real part of susceptibility
χ''	Imaginary part of susceptibility
Ω	Ohm
Å	Angstrom unit
~	Approximately
Ac	Alternating current
Dc	Direct current
ACS	Alternating current susceptometer
Hz	Hertz
I	Current
K	Kelvin
R	Resistance
SEM	Scanning electron microscope
T	Absolute temperature (Kelvin)
V	Voltage
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background and history of superconductivity

Superconductors are materials that allow dc current to flow without any resistance. The field of superconductivity changes after the discovery of high temperature superconductivity (HTS) in the cooper oxide based materials which were later found to superconduct above the boiling point of liquid nitrogen in 1986. They act as perfect diamagnetic when applied magnetic field. Superconductors do not show any dc electrical resistance below transition temperature (T_c). In superconductors, the charge carriers from pairs which are also known as the Cooper pairs.

Remarkable behaviour in superconductivity discovered by Kamerlingh Onnes (1911) by liquefying helium. The first superconducting element which exhibited a dramatic drop in resistivity at 4.2 K from 0.03 Ω to 3×10^{-6} Ω within a temperature range of 0.01 K was Mercury. The measurement consisted in applying a voltage V across the mercury, recording the current *I* that flowed, and calculating the resistance R by dividing the voltage by the current, (R = V/I). Onnes noticed that while the mercury was at 4.3 K if he turned off the voltage, the current stopped flowing, as expected. He also noticed that the current continued to flow if he turned off the voltage below 4.2 K.

Superconducting state known as resistance is zero. Zero resistance is the first characteristic property of a superconductor, the second such property is magnetic in nature. Other metallic elements, metals that contain only one type of atom, such as aluminum and zinc, were found to superconduct at temperatures below that of mercury. Two years later the element lead was found to be superconducting at 7.2 K, and 17 years later in 1930, niobium was found to superconduct at 9.2 K. Bednorz and Muller (1987) discovered high temperature superconductivity in the copper oxide based materials resulted in worldwide research on these materials. $YBa_2Cu_3O_{7-\delta}$ is one of the example for copper oxide based materials were found to have T_c higher than the boiling point of liquid nitrogen (77 K).

The German professor Alexander Meissner and his graduate student R. Ochsenfeld (1933) took 22 more years to discover the second characteristic property of the superconducting state, a property that became known as the Meissner effect. When a superconducting metal is placed in a magnetic field and then cooled below the transition temperature, the magnetic field is expelled that discovered by Meissner and Ochsenfeld. A phenomenon known as the Meissner effect when it turns out that no magnetic field is allowed inside a metal when it is in the superconducting state.

Another cause of skepticism resulted from mathematical calculations made by some theorists during 1960s and 1970s that indicated that the Bardeen-Coopers-Schrieffer (BCS) theory sets an upper limit of 30 K on superconducting transition temperatures. Many experimental physicists believed these theoretical calculations, and as a result quite a few researchers had given up working in the field of superconductivity.

All known superconductors operated at temperatures far below the boiling point of liquid nitrogen during the mid-1980s, which is 77 K. This is equivalent to -199°C or -326°F. Each material superconducts below a characteristic transition temperature denoted by the symbol T_c, and it becomes a normal metal at higher temperatures. Niobium (Nb), with $T_c = 9.3$ K, has the highest T_c of any element. The compound niobium-germanium, with the formula Nb_3Ge and $T_c = 23.2$ K, was then the highest of all materials. The fact that this transition temperature is less than a third of the liquid nitrogen boiling point 77 K caused most specialists in the field to believe that the possibility was indeed very remote of ever finding a material to superconduct above, or even close to, the magic value of 77 K.

IBM’s Zurich research laboratories, the Swiss researchers Bednorz and Muller were not convinced by these arguments. Muller had spent many years working on crystal structure changes, in particular, oxide materials known as perovskites, the prototype of which is the compound strontium titanate. The behaviour of this class of materials that led him to believe that crystals of this type would display superconductivity at much higher temperatures.

Claude Michel and co-workers (1985) at the University of Caen in France synthesized a copper oxide compound and in their article they noted that it exhibited an unusual metallic like electrical behaviour. Bednorz and Muller tried the measurements on similar materials. They tested a lanthanum, barium copper oxide compound, they observed to their astonishment the phenomenon of superconductivity in the temperature range of 30-35 K, over 10 degrees above the niobium-germanium value, and almost halfway to the liquid nitrogen temperature.

Paul Chu, a professor of physics at the University of Houston who had spent much of his professional research life in search of a higher temperature superconductor material. Chu set himself the task of trying to raise the superconducting transition temperature T_c in this material. He knew that some known superconductors had exhibited higher transition temperatures under pressure, so his approach was to apply pressure to the material. By doing this he was able to raise T_c by another 10 degrees.

At the same time a group in Japan headed by Dr. Koichi Kitazawa, an MIT-educated scientist also repeated the result with the lanthanum compound.

Since 1988 much work has been done to synthesize materials that become superconducting at higher temperatures. In 1994 superconductivity was observed in a new class of copper oxide materials containing mercury. These materials became superconducting at 133 K, but subjecting one of these mercury compounds to high pressure raised this value to 147 K.

(Bednorz, 1987) of the IBM Zurich Laboratory were awarded the Nobel Prize in physics for their discovery of the new class of superconducting materials. Although the discovery of this latter much higher temperature yttrium compound provided the potential for many applications and initiated extensive publicity, it was nevertheless the original discovery of the 35 K lanthanum material that started the search for operation above 77 K and soon led to the subsequent synthesis of the 90 K yttrium compound.

1.2 High Temperature Superconductors

High temperature superconductor are great potential for a new class of materials and widely use in technological applications. News media of the world were filled with accounts of a major discovery in material science in March 1987. A superconductor is a metal that conducts electricity without a loss in energy, hence without any cost to the user. Examples of high temperature superconductors are YBCO, BSCCO, TBCCO and HBCCO systems. The highest T_c for YBCO system belongs to YBCO (123) phase, that is $T_c = 92$ K, while for BSCCO (2223) phase, $T_c = 110$ K, while for TBCCO (2223), $T_c = 125$ K and for HBCCO (1223) $T_c = 153$ K as stated by Alecu

(2004). Synthesis also plays a major role in maintaining superior superconducting properties.

Two importance things to understand the transport and magnetic behaviors of these high temperature superconductor materials. First, it could lead to a better understanding of the basic phenomenon of superconductivity. Second it could provide ways to improve the magnetic quality of the presently known materials by enhancing flux pinning in a controllable manner. Figure 1.1 showed the time evolution of the superconducting critical temperature since the discovery of superconductivity in 1911.

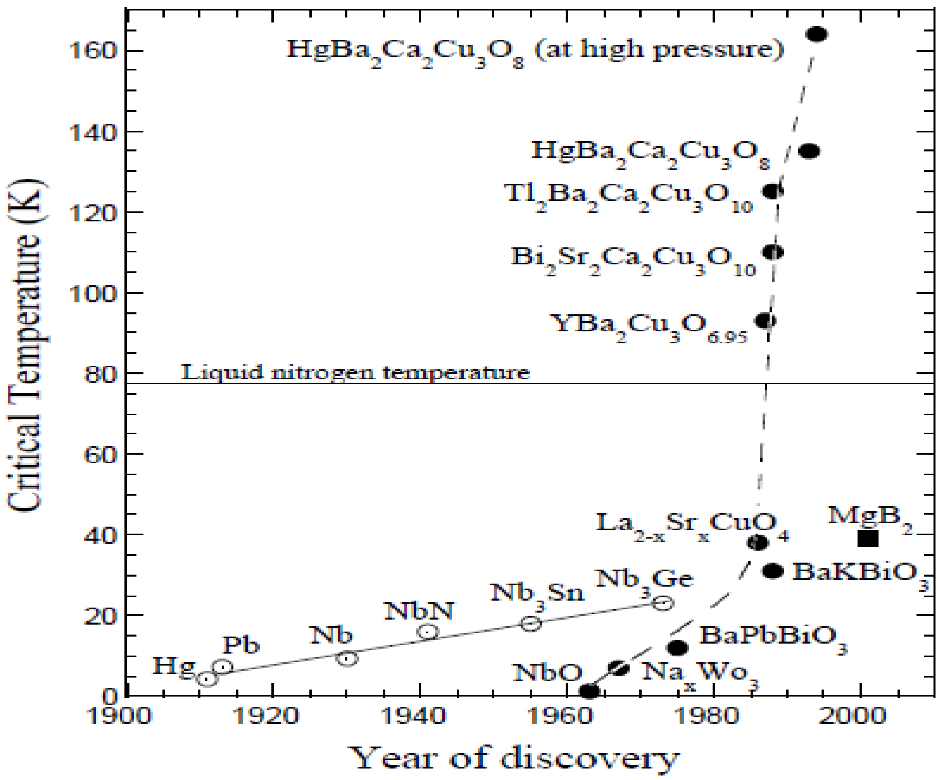

Figure 1.1: The time evolution of the superconducting critical temperature since the discovery of superconductivity in 1911. The solid line shows the T_c evolution of metallic superconductors, and the dashed line marks the T_c evolution of superconducting oxides (Source: Room Temperature Superconductivity, Andre Marouchkine, Cambridge International Science Publishing)

1.3 Y-Ba-Cu-O System

1.3.1 Crystal Structure

Y123 was discovered in attempt to replace La in LBCO with another rare earth, the 4d transition element Y. The Y-Ba-Cu-O compound can form several phases such as $YBa_2Cu_3O_{7-\delta}$ (Y123), $Y_2Ba_4Cu_7O_{14-\delta}$ (Y124) which shows high temperature superconductivity. The Y123 material was discovered by Wu et al. (1987). In this material, Y can generally be replaced with most of the rare earth elements except for Ce, Tm, and Pr and still shows superconductivity near 90 K. These materials have complex perovskite-like structure with oxygen defects playing an important role for superconductivity. The orthorhombic and tetragonal structures of Y123 are shown in Figure 1.2.

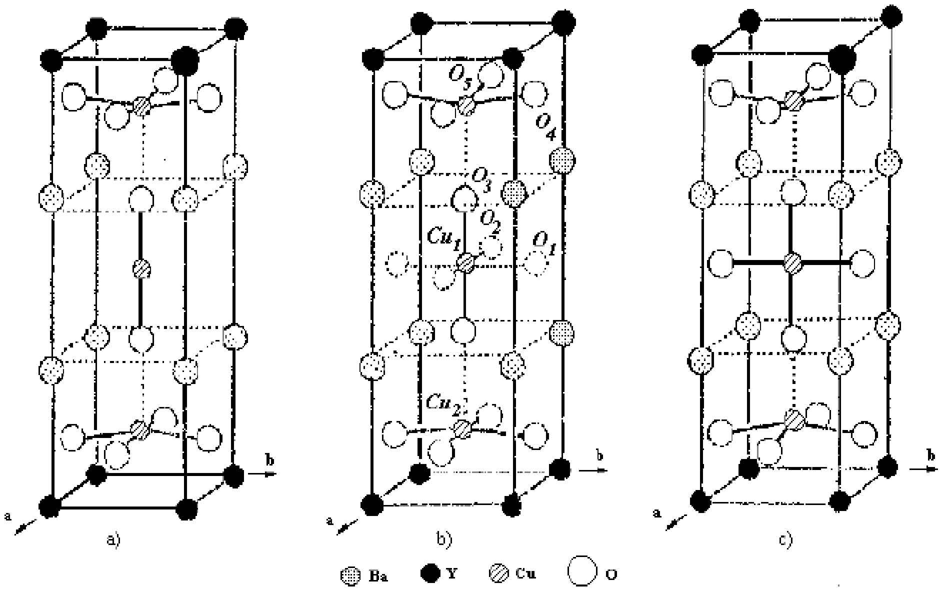

Figure 1.2: The oxygen deficient perovskite structures for YBaCuO system with oxygen content a) $\delta = 1$ b) $0.6 < \delta < 0.6$ c) $\delta = 0$ (Source: Romanian Reports in Physiscs, Volume 56, No. 3, P. 404 – 412)

The oxygen defects result in a mixed state copper valence and the chemical formula of this material can be written as $YBa_2Cu_2^{2+}Cu^{3+}O_{7-\delta}$. The Cu^{2+} ions occupy the CuO_2 plane and the Cu^{3+} ions occupy the CuO chains. For $\delta < 0.6$ the materials exhibit an orthorhombic structure and for $\delta > 0.6$ they exhibit a tetragonal structure. The O_7 material has a transition temperature of about 90 K and exhibits an orthorhombic structure a = 3.821 Å, b = 3.885 Å and c = 11.676 Å.

When the oxygen content is reduced the material undergoes a transition from the orthorhombic to tetragonal (O-T) structure with a = b = 3.857 Å and C = 11.819 Å for $\delta = 1$. As the oxygen content is reduced from 7 to 6 the normal state changes from metallic to insulator or semiconductor. For $\delta > 0.6$, no superconducting transition is observed. When doped with oxygen, which occupy the CuO chain, this material slowly becomes metallic because oxygen ions attract electrons from the CuO_2 layer. Optimum T_c (of about 92 K) occurs for oxygen content $O_{6.93}$. The oxygen content can be increased or decreased through a series of annealing steps and this process is reversible. The CuO_2 layer acts as hole conductors and CuO chain acts as charge reservoir. All attempts to prepare YBCO directly failed regardless of temperature and pressure (Sleight, 1995). The critical temperature T_c could be determined from the temperature resistance measurement.

1.4 Colossal Magnetoresistance (CMR) as the Addition Materials

Recently years there has been a lot of research study in manganites materials or also known as Colossal Magnetoresistance (CMR) effects in rare earth manganite perovskite with general formula $La_{1-x}A_xMnO_3$ (A = Ba, Sr, Pb and Ca) due to their potential technology application. These compounds are Mn^{3+} rich and doping of divalent atoms introduces mixture valency of $Mn^{3+}$and Mn^{4+} ions plays a major role in Double Exchange (DE) ferromagnetic interaction coupled with metallic resistivity. Double exchange effect is an exchange of electrons from neighbouring Mn^{3+} to Mn^{4+} ions through oxygen when their core spin are parallel and hopping is not favoured when they are anti-parallel.

Jahn-Teller distortion (JT) could be responsible for the transport properties. The Jahn- Teller effects cause further degeneracy of the example, orbital of Mn^{3+} in MnO_6 octahedral and resulting electrical transport via hopping effect (Rama et al. 2008) and (Dagotto et al. 2001). The Magnetoresistance (MR) percentage of MR defined as [(RH-Ro)/Ro]*100 where RH is the resistance in the present of magnetic field and Ro is the resistance at zero field. In general, CMR effects of polycrystalline ceramic bulk exhibit two classes of Magnetoresistance (MR), the intrinsic and extrinsic MR. The former is referred to intragrain effect where its MR shows a maximum near paramagnetic-ferromagnetic transition temperature (T_c) and maximum electrical resistivity at metal-insulator transition temperature (T_p).

The latter is due to the intergrain effect where higher MR could be observed over a wider temperature range below T_c and is characteristic of a Low-Field MR (LFMR), which is believed to be due to Spin-Polarized Tunnelling (SPT) or Spin-Dependent Scattering (SDS) at grain boundary (Zhao et al. 2006; Ravi et al. 2007; Kameli et al. 2008). The effect of grain boundaries in the polycrystalline manganites has been studied intensively, where the grain's shape or microstructure will be changed with different preparation process (Wang, 2008) or dopant (Im et al. 2007; Venkataiah et al. 2007; Kalyana Lakshmi et al. 2008). It is found that substitution of divalent atoms with variance atomic radius and preparation process will influence the magnetic properties and electrical properties of the system (Kalyana Lakshmi 2008; Wang et al. 2008). Sintering the $La_{0.67}Ca_{0.33}MnO_3$, $La_{0.67}Ba_{0.33}MnO_3$, and, $La_{0.67}Sr_{0.33}MnO_3$ oxide at an optimized sintering temperature, 1300 °C, gives an optimal sinter ability, which dominates the electrical and mechanical properties of the oxide.

1.5 Problem Statement

This research proposes to trace the effect and compare the difference in diamagnetic transition temperature ($T_{c\text{-}on}$), phase lock-in temperature (T_p), and grain growth when lanthanium calcium manganese trioxide ($La_{0.67}Ca_{0.33}MnO_3$), lanthanium barium manganese trioxide ($La_{0.67}Ba_{0.33}MnO_3$), and lanthanium scrontium manganese trioxide ($La_{0.67}Sr_{0.33}MnO_3$) were added with amount of 0.2 wt% - 1.0 wt% to $YBa_2Cu_3O_{7-\delta}$ (YBCO) superconductor prepared by solid state reaction technique with intermediate grinding. Study effect addition of manganites in YBCO system are importance in the understanding and optimization of the properties as technologically potential material.

The choice of manganites material is motivated because it has manganese based which is supposed to play a role in the changes of superconductivity mechanism and to see the quality of sample produced as the best superconductor properties . YBCO is a crystalline material, and the best superconductive properties are obtained when crystal grain boundaries are aligned by careful control of annealing, sintering time, and suitable adding materials. Changes in crystal structures, transport properties, and morphology of LCMO, LBMO, and LSMO-added in YBCO superconductor were discussed. When amount of addition increasing, it effect the lattice parameter which changed the crystal structure, oxygen content influenced value of $T_{c\text{-}on}$, and finally gave impact on connectivity among grains in the samples. Findings are compared with available literature based on the main superconductor which is YBCO. Literature review related with this type of addition is little published, only present work have done by Park et al. (2011) on the substitution of LCMO in YBCO. Addition by using manganite which also known as colossal magnetoresistance never been studied. Previous and present studies showed elements and non-complex compounds only have been used as the addition materials in YBCO.

1.6 Objective of the Research

The main objective of this study was to determine the effect of manganites addition in YBCO superconductor at various weight percentages on the superconducting properties that were prepared via conventional solid state reaction method. This study focused on the effect of manganites addition on structural properties such as types of structure, lattice length, and phase identification, electrical properties, and surface morphology.

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