EFFECTS OF MULTIFERROICS BFO AND HMO NANOPARTICLES ADDITION ON STRUCTURAL, ELECTRICAL AND SUPERCONDUCTING PROPERTIES OF $YBa_2Cu_3O_{7-\delta}$

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By

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Chairman : Professor Abdul Halim Shaari, PhD
Faculty : Science

The $YBa_2Cu_3O_{7-\delta}$ (Y-123) superconductor is one of the materials that have been considered to have the potential of making a significant impact on technology, in particular on the issue of critical current density. The main problem that restricts the technology applications of bulk Y-123 is the low grain boundary conductivity due to weak links and poor flux pinning, causing low J_c in the presence of magnetic field. In this work two types of multiferroics, namely $BiFeO_3$ and $HoMnO_3$ having nano-sized particles were incorporated into Y-123 to form composites with the compositions; $(YBa_2Cu_3O_{7-\delta})_{1-x}$(BFO or HMO)$_x$ with x = 0.0025, 0.005, 0.0075, 0.01, 0.03 0.05, 0.07, and 0.10. The composites were fabricated by introducing sol-gel synthesized BFO and coprecipitation synthesized HMO during solid state reaction process of Y-123. Samples were characterized using XRD, FESEM, EDX, four-point probe, AC susceptometer, ESR and Impedance analyzer.

From the XRD results, single phase Y-123 having orthorhombic structure were observed. On the other hand, the BFO showed single phase of $BiFeO_3$ belonging to hexagonal structure ,while the HMO showed two mixed phases of $HoMnO_3$ in about 85% of the total phase with hexagonal structure and $HoMn_2O_5$ in about 15% with orthorhombic structure.

XRD patterns manifested that all composite samples, could be indexed to orthorhombic crystal structure, while Y-211 was detected as minor phase. Besides, YBa_2BiO_6 and YFe_2O_4 were detected in samples with BFO addition and phases $YBaMn_2O_5$, $YBaMn_2O_6$ and $HoBa_2Cu_4O_8$ were detected in HMO added samples. FESEM images showed that grain size of Y-123, BFO and HMO are 11.0, 0.065 and 0.085 μm respectively. The grain size decreased as additions increased. EDX analysis

showed accurate match for standard peak position for Y, Ba, Cu and O for Y-123. Spectra also showed the presence of agglomerated particulates related to Fe and Bi compounds embedded between Y-123 grains in BFO added samples. Similarly agglomerated particulates related to Mn and Ho phases were detected residing between Y-123 grains. The Y-123 sample exhibited good metallic behavior in the normal state. $T_{C(R=0)}$ for Y-123 was 92 K and $T_{C(onset)}$ was 97 K. The superconducting behavior of Y-123/BFO samples showed a decrease in $T_{c(R=0)}$ as the addition increased. The composite with x = 0.01 showed highest $T_{C(R=0)}$ of 90 K and degraded to 44 K at x = 0.10. When added with HMO the highest $T_{C(R=0)}$ was 91 K at x = 0.0025, then gradually decreased to 83 K for x=0.1.

The AC susceptibility measurement for the pure Y-123 showed sharp curve of diamagnetism transition in the real part (χ') which is exhibited by two-step transitions related to the intragrain and intergrain couplings. The imaginary part (χ'') demonstrates two peaks attributed to intrinsic behavior of intragrain and intergrain couplings. The intergranular critical current density, $J_C(T_P)$, and Josephson current, I_0, of pure Y-123 are 16.54 Acm^{-2} and 40.59 μA respectively. AC susceptibility curves in the real part (χ') for the added BFO samples manifested inclined transitions for all samples, whereas the samples with less BFO contents of x = 0.0025 and 0.005 showed the strongest intergrain couplings. While the AC susceptibility curves for the samples with HMO showed sharp transition for the samples with x = 0.0025, 0.005 and 0.0075. In both systems, I_0, and $J_C(T_P)$ of the composite samples were higher than that of pure Y-123. Maximum I_0 and $J_C(T_P)$ were observed when x = 0.0025 in both addition systems. The maximum $J_C(T_P)$ observed for BFO and HMO samples are 18.07 Acm^{-2} and 21.96 Acm^{-2} respectively.

Dielectric parameters ε_r' and ε_r'' decreased as the frequency increased for all samples in pure and composites. The ε_r versus frequency measurements showed an increase in ε_r' and ε_r'' values for all added samples as compared to Y-123. The highest values for ε_r' and ε_r'' were obtained when x = 0.1 with the highest loss at lower frequency. The highest values of ε_r' and ε_r'' for (Y-123), (Y-123/BFO) and (Y-123/HMO) are (2.05×10^2, 1.56×10^3), (3.67×10^3, 22.56×10^3) and (5.15×10^3, 24.66×10^3) respectively. Nyquist plot of complex impedance (Z′-Z″) were analysed where two semi arc circulars representing grain and grain boundary effects were deduced in both systems.

From ESR measurements all pure Y-123, BFO and HMO added samples showed spectra consisting of two peaks. The g-factor for the composite samples changed in different manner from the Y-123 sample, while it decreased at lower concentrations ($x \leq 0.0075$) and increased at higher concentrations ($x \geq 0.01$) in BFO added samples, it increased at lower concentrations ($x \leq 0.01$) and increased at higher concentrations ($x \geq 0.03$) in HMO added samples.

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

KESAN PENAMBAHAN NANOZARAH MULTIFEROIK BFO DAN HMO KEATAS SIFAT STRUKTUR, ELEKTRIK DAN KESUPERKONDUKSIAN $YBa_2Cu_3O_{7-\delta}$

Oleh

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Mei 2019

Pengerusi : Profesor Abdul Halim Shaari, PhD
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Superkonduktor $YBa_2Cu_3O_7$ (Y-123) adalah salah satu daripada bahan-bahan yang telah dianggap berpotensi untuk memberi kesan yang besar kepada teknologi, khususnya mengenai isu ketumpatan arus kritikal. Permasalahan utama yang menghadkan applikasi bahan pukal Y-123 ialah konduktiviti sempadan butiran yang rendah disebabkan tautan dan pengepinan fluks yang lemah, menyebabkan J_c yang rendah dengan kehadiran medan magnet. Dalam kajian ini dua bahan multiferoik terkenal, iaitu $BiFeO_3$ dan $HoMnO_3$ bersaiz nano telah dimasukkan ke dalam Y-123 untuk membentuk komposit dengan komposisi berikut; $(YBa_2Cu_3O_{7-\delta})_{1-x}(BiFeO_3)_x$ atau Y-123/BFO dan $(YBa_2Cu_3O_{7-\delta})_{1-x}(HoMnO_3)_x$ atau Y-123 / HMO dengan x = 0.0025, 0.005, 0.0075, 0.01, 0.03 dan 0.05, 0.07, 0.10. Kajian untuk bahan tersebut tidak pernah dilaporkan. Komposit ini telah difabrikasi dengan menggunakan nanozarah $BiFeO_3$ disintesis secara sol-gel atau nano zarah $HMnO_3$ disintesis secara pemendakan bersama semasa proses tindak balas keadaan pepejal Y-123 superkonduktor. Semua sampel telah dicirikan dengan penggunaan pembelauan sinar-X (XRD), mikroskopi imbasan elektron pancaran medan (FESEM) dengan faciliti EDX, pengukuran rintangan empat titik, susceptometer AC, resonans spin elektron (ESR) dan penganalisis impedans.

Daripada keputusan XRD, satu fasa Y-123 mempunyai struktur ortorombik bersimetri *Pmmm* diperhatikan. Sebaliknya, BFO menunjukkan satu fasa $BiFeO_3$ dengan struktur heksagon bersimetri *R3c*, manakala HMO menunjukkan dua fasa campuran $HoMnO_3$ dalam kira-kira 85% daripada jumlah fasa dengan struktur heksagon bersimetri *P63cm* dan $HoMn_2O_5$ dalam kira-kira 15% dengan struktur ortorombik bersimetri *Pbam*.

Bagi sampel komposit, pola pembelauan XRD menunjukkan bahawa semua sampel untuk kedua-dua sistem Y-123 / BFO dan Y-123 / HMO boleh diindeks kepada kumpulan ruang (Pmmm) dengan struktur kristal ortorombik, manakala Y-211 dikesan sebagai fasa kecil dalam kedua-dua sistem. Selain itu, YBa_2BiO_6 dan YFe_2O_4 dikesan dalam sampel dengan penambahan BFO dan fasa-fasa $YBaMn_2O_5$, $YBaMn_2O_6$ dan $HoBa_2Cu_4O_8$ dikesan dalam sampel dengan penambahan HMO. Imej FESEM menunjukkan bahawa saiz butiran sebahagian besar Y-123, BFO dan HMO adalah masing-masing 11.0, 0.065 dan 0.085 nm. Saiz butiran menurun apabila penambahan zarah nano BFO dan HMO meningkat. Analisis EDX menunjukkan padanan tepat kedudukan puncak piawai untuk Y, Ba, Cu dan oksigen bagi Y-123 tulen. Spektrum juga menunjukkan kehadiran zarah teragglomerasi berkaitan dengan zarah nano Fe dan Bi tertanam di antara bijiran Y-123 dalam sampel yang ditambah dengan BFO. Begitu juga zarah teragglomerasi berkaitan dengan fasa Mn dan Ho telah dikesan terhuni di antara bijiranY-123. Y-123 tulen dipamerkan berkelakuan logam dalam keadaan biasa dan peralihan satu langkah kepada keadaan superkonduktor. Y-123 menunjukkan $T_{C(R\,=\,0)}$ sebanyak 92 K dan bermulanya $T_{C(onset)}$ suhu peralihan (permulaan) daripada 97 K. Kelakuan superkonduktor bagi sampel Y-123 / BFO menunjukkan penurunan suhu kritikal $T_{C(R\,=\,0)}$ apabila penambahan meningkat. Komposit dengan x = 0.01 menunjukkan suhu peralihan yang lebih tinggi, $T_{C(R=0)}$ = 90 K dan menurun kepada 44 K untuk x = 0.10. Apabila ditambah dengan HMO kelakuan superkonduktor ini meningkat dengan ketara untuk x = 0.0025 dengan $T_{C(R=0)}$ pada 91 K. Tiada penurunan utama diperhatikan dalam $T_{C(R=0)}$ apabila penambahan meningkat sehingga x = 0.03 ($T_{C(R=0)}$) = 88 K), kemudian beransur-ansur terus berkurangan kepada 83 K apabila x = 0.1. Secara umumnya sampel dengan penambahan HMO menunjukkan peralihan (ΔT) yang lebih sempit daripada sampel dengan penambahan BFO, menyarankan bahawa morfologi, mikrostruktur dan penghabluran yang lebih baik.

Pengukuran kerentanan AC untuk Y-123 tulen menunjukkan keluk peralihan diamagnet yang tajam di bahagian sahih (χ') yang dipamerkan oleh dua tangga peralihan berkaitan dengan intrabutiran dan gandingan interbutiran. Bahagian khayalan (χ'') menunjukkan dua puncak dikaitkan dengan perlakuan intrinsik intrabutiran kegandingan interbutiran. Ketumpatan arus kritikal interbutiran, $J_C(T_P)$, dan arus Josephson, I_0, untuk Y-123 tulen adalah masing-masing 16.54 Acm^{-2} dan 40.59 μm. Lengkung kerentanan AC di bahagian nyata (χ') bagi sampel BFO ditambah dimanifestasikan peralihan cenderung untuk semua sampel, manakala sampel dengan kurang kandungan BFO x = 0.0025 dan 0.005 menunjukkan gandingan interbutiran kuat. Manakala lengkung kerentanan AC bagi sampel dengan kandungan HMO menunjukkan peralihan tajam untuk sampel dengan x = 0.0025, 0.005 dan 0.0075. Dalam kedua-dua sistem, arus Josephson, I_0, dan ketumpatan arus kritikal $J_C(T_P)$ sampel komposit adalah lebih tinggi daripada Y-123 tulen. I_0 maksimum dan $J_C\ (T_P)$ telah diperhatikan untuk sampel dengan x = 0.0025 dalam kedua-dua sistem komposit. $J_C\ (T_P)$ maksimum diperhatikan untuk sampel BFO dan HMO masing-masing bernilai 18.07 Acm^{-2} dan 21.96 Acm^{-2}. Secara umumnya nilai I_0 adalah lebih tinggi dalam sampel dengan BFO manakala $J_C(T_P)$ nilai-nilai adalah lebih tinggi dalam sampel dengan tambahan HMO.

Parameter dielektrik ε_r 'dan ε_r" menurun apabila frekuensi bertambah untuk semua sample kekerapan yang meningkat bagi semua sampel Y-123 tulen juga bagi kedua-dua komposit. Pengukuran parameter ε_r terhadap perubahan frekuensi menunjukkan peningkatan dalam nilai ε_r' dan ε_r'' untuk semua sampel komposit berbanding dengan sampel Y-123. Nilai tertinggi untuk ε_r' dan ε_r'' diperolehi bagi sampel x = 0.1 dengan kehilangan tertinggi pada frekuensi yang lebih rendah. Nilai tertinggi ε_r' dan ε_r'' untuk (Y-123), (Y-123 / BFO) dan (Y-123 / HMO) adalah (2.05×102, 1.56×103), (3.67×103, 22.56×103) dan (5.15×103, 24.66×103) masing-masing. Plot Nyquist untuk impedans kompleks (Z'-Z") telah dianalisis di mana dua lengkung separa bulatan mewakili butiran dan kesan sempadan butiran telah dihasilkan dalam kedua-dua sistem.

Dari ukuran resonans elektron semua sampel Y-123, BFO dan HMO tulen mempamirkan spektrum terdiri daripada dua puncak. Faktor-*g* bagi sampel komposit berubah dalam cara yang berbeza dari sampel Y-123, semasa ia menurun pada kepekatan yang lebih rendah ($x \leq$ 0,0075) dan peningkatan pada kepekatan yang lebih tinggi ($x \geq$ 0.01) dalam sampel BFO; ia meningkat pada kepekatan ($x \leq$ 0.01) yang lebih rendah dan meningkat pada kepekatan yang lebih tinggi ($x \geq$ 0.03) dalam sampel komposit HMO.

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LIST OF ABBREVIATIONS AND SYMBOLS

BFO	Bismuth Ferrite (multiferroic material)
HMO	Holmium Manganite (multiferroic material)
Bi	Bismuth
Fe	Iron
Ho	Holmium
Mn	Manganese
LTSC	Low Temperature Superconductor
HTSC	High Temperature Superconductor
YBCO	Yttrium Barium Copper Oxide
Y-123	Family member in $YBa_2Cu_3O_{7-\delta}$
Y-124	Family member in $YBa_2Cu_4O_{7-\delta}$
Ca	Calcium
Ba	Barium
REBCO	Rear-earth Barium Cooper Oxide
RE	Rare-earth
T_C	Critical Temperature (K)
$T_{c\ (R=zero)}$	Zero resistance temperature
$T_{c\ (onset)}$	Onset of superconducting transition
Tc max	Maximum critical temperature
ΔTc	Delta critical temperature
K	Kelvin
T	Temperature in Kelvin scale
J_C	Critical Current Density (A/m2)
J	Current Density (A/m2)
SQUID	Superconducting quantum interference devices

XRD	X-ray diffraction
FESEM	Field emission scanning electron microscope
EDX	Energy Dispersion X-ray
AC	Alternating current
ρ	Resistivity
A	Ampere
I	Current
R	Resistance
f	frequency
Z′	Real part of impedance
Z″	Imaginary part of impedance
AC	Alternating current
DC	Direct Current
V	Voltage
ZFC	Zero Field Cooled
X	Magnification
BSCCO	Bismuth strontium calcium copper oxide is a family of superconductor
B	Magnetic induction
H	Magnetic Field
He	Applied magnetic field
Hc	Critical field
M	Magnetization
Hc2	Upper critical magnetic field
Hc1	Lower critical magnetic field
V	Voltage
ε_r'	Relative dielectric constant.

ε_r''	Relative dielectric loss.
ε_0	Permittivity of vacuum (8.854 x 10-12 F/m).
μ_0	Vacuum permeability (4 x π x 10-7 H/m)
ρ	Resistivity (Ω.m).
τ	Time constant (s)
a, b, c	Lattice parameters
V	Unit cell volume
P	Hole concentration

CHAPTER 1

INTRODUCTION

1.1 Introduction

The compounds that had been found, developed and used over the history serve as a perfect indicator of progress in technology and civilization. The earliest materials are those that were simply available in nature, like wood, stone, clay and metals. Therefore, the technology involved was aimed to physically restructuring these materials to be fitted to specific purposes such as therapeutics, weaponry, utensils and the devices for storing energy. The discovery of procedures for the extraction of metals and fabrication of alloys was a major revolution in the history of materials. It was a situation of complete transformation of physical, chemical and electrical properties of a material. Equally remarkable was the discovery of superconductors; a process in which the magnetic and electrical properties of the normal ionic metals were totally modified. Presently, superconductors are part and parcel of electronic industry, due to the properties they offer for effective, economy energy source and other potential applications.

1.2 History of high temperature superconductors (HTS)

High temperature superconductors (HTS) are materials that superconduct at temperatures above 30 K and its superconducting state can be reached by cooling it using liquid nitrogen (77 K). In late 1986, a first HTS was discovered when Alex Muller and Georg Bednorz produced a brittle ceramic compound (La-Ba-Ca-O compounds) that showed superconductivity above 30 K. Since then, researchers commenced to study every composition of ceramics to acquire a higher T_c. Further, in January 1987, a team from University of Alabama-Huntsville obtained a T_C, of 92 K, when they substituted lanthanum by yttrium in yttrium barium copper oxide ($YBa_2Cu_3O_{7-x}$) which is the first superconductor with T_C, above the boiling point of liquid nitrogen (Cyrot & Pavuna, 1992; Wu et al., 1987). Following, in 1988, Maeda and co-workers at the National Research Institute, Japan presented the Bi-cuprate oxides ($Bi_2Sr_2Ca_2Cu_3O_{10}$) with T_c = 110 K (Maeda et al., 1988). The Tl-cuprate oxides ($Tl_2Sr_2Ca_2Cu_3O_{10}$) was found by Sheng and Hermann with T_c = value at 125 K (Sheng and Hermann, 1988).

The highest temperature superconductor was found in the three-layer system. It was discovered in $HgBa_2Ca_2Cu_3O_{8+}$ system, (Hg-1223) with T_C = 133.5 K. In January 2008, a research group in Japan led by Hideo Hosono found a new class of high-temperature superconductors (non-Cu-based superconductors) in layered iron arsenic compounds with T_C = 26 K. It was discovered that the parent compound, LaOFeAs became superconducting when some of the oxygen are replaced with fluorine (Kamihara et al., 2008). In 2015, the highest T_C of 203 K was obtained in hydrogen sulphide (H_2S) under extremely high pressure around 150 GPa (Sheng & Hermann, 1988). The Y-123 is more favourable for superconducting applications advantages which is ascribed to its physical robustness and superior superconducting behaviour in a higher magnetic field (Maple, 1998). A great tendency in superconductivity field is encouraged by the promising uses of it in the subsequent terms for electronic power transmission without losses and in the construction of quantum high power generators. Moreover, superconductivity is engaged to build up quantum computers (Tsai, 2010).

1.3 Problem of statement and research objectives

Researchers had widely studied the high temperature superconductor (HTS). The diamagnetism and zero resistance properties of HTS have made them of great benefits in the world industry. Yet, there are certain limitations of HTS applications due to the critical transition temperatures which are well below room temperature. Accordingly, lots of work has been done to improve the critical transition temperature T_C and the critical transition current density, J_C (Barnes et al., 2009; Horvath et al., 2008; Klie et al., 2005). The superconducting behaviors of Y-123 is defined by three main parameters; the critical temperature,T_c, the critical current density, J_c and the critical magnetic field H_{c2}. However, two main issues were arised and restricted technology applications of bulk Y-123, namely, low grain boundary conductivity, that is weak links and poor flux pinning, causing low J_c in the presence of magnetic field (Maple, 1998). Moreover the T_c is sensitive to oxygen content in the system (Skakle, 1998). Introducing artificial pinning centers and fabrication of good quality bulk Y-123 are among strategies to improve current capability with stablized T_c (Azzouz et al., 2007; Dihom et al., 2017; Kung et al., 1993; Mele et al., 2008; Rejith et al., 2014; Zhao et al., 2005). Remarkable studies have been focused on the impacts of magnetic impurities added into bulk and thin film Y-123. Most magnetic impurities involved were in the form of oxides such γ-Fe_2O_3, Fe_3O_4, $NiFe_2O_4$ and ZnO or of metal ions such as Fe, Co, Ni, Mn and Zn. whereas bulk Y-123 added with multiferroic nanoparticles is rarely investigated. However the physical phemenomen associated with interface between layered structure of manganites such $La_{0.7}Ca_{0.3}MnO_3$(LCMO), $La_{0.67}Sr_{0.33}MnO_3$(LSMO) and $Pr_{0.68}Ca_{0.32}MnO_3$ (PCMO) with Y-123 has been the subject of intensive research. For instance it was found that the fundamental length scale of the interface between Y-123 and LCMO is in the range of subnanometre (Kourkoutis et al., 2013). Moreover, the charge density and T_c has been modulated in $BiFeO_3$ and Y-123 hetrostrucure (Crassous et al., 2011) and it was realized that the Y-123/BFO hetrostucture can be used to produce Y-123 Josephson junctions (Yang et al., 2013). Composition of BFO on Y-123 bilayers has been published as well (Springer et al., 2016). Nano-BFO doped Y-123 single-grain superconductor has been fabricated indicating an improved in superconducting properties (Li et al., 2017). The hybridization of superconductors with mulfiferroics may lead to the development of new superconducting devices (Liu et al., 2015). To the present date there were less publications reported on the effects of the addition of bismuth ferrite $BiFeO_3$ (BFO) nanoparticles or holmium manganite $HoMnO_3$ (HMO) nanoparticles to the bulk Y-123 superconductor. The BFO and HMO might act such as advantage pinning centers in Y-123. Moreover, nano addition of magnetic impurities to basic superconductors may be not only affect the pinning centers, but also brings the concept of Josephson like system that can be employed as adapting mechanism to the Josephson critical current junctions (Jung et al., 2000; Van Look et al., 1999; Wördenweber et al., 2000). This leads to decreasing the noise in SQUID devices and improves the carried current density in superconducting wires and cables (Dantsker et al., 1997; Vélez et al., 2008; Wördenweber et al., 2000). In the present work the structural, superconducting and dielectric properties of both multiferroics added to Y-123 have been studied.

1.4 Aim and objectives of the study

This study has been planned to determine the effects nano-BFO and nano-HMO addition synthesized via wet chemical routes on structural, magnetic, electrical and dielectric properties of (Y-123) synthesized via standard solid state reactions based on the problems stated in section 1.3 and on the hypotheses that the critical transition temperature T_C and the critical transition current density, J_C, could be improved by introducing nano-metric scale addition of magnetic impurities to basic superconductors that may not only affect the pinning centers, but also brings the concept of Josephson like system that can be employed as adapting mechanism to the Josephson critical current junctions. The specific research objectives of the study are as follows;

1. To fabricate a high purity bulk Y-123 superconducting material with micro size grain particles using solid state reaction method and to determine its superconducting and dielectric characterizations.
2. To synthesize the nano-powders of BFO multiferroic materials using Sol-Gel chemical method.
3. To prepare the nano-powders of HMO multiferroic materials using coprecipitation chemical methods.
4. To investigate the influence of nanoparticles BFO and HMO multiferroics addition on the structural, magnetic, superconducting and dielectric properties of Y-123.

1.5 Significance of the study

At the end of this study, the effects of the synthesized nanoparticle multiferroics, BFO which is belonging to so called ferrites and the HMO that belongs to manganites on the properties of the superconductor system Y-123 will be realized. The output of this work will add useful information for the knowledge and comprehension of the subject and should reveal more insight into academia and innovative issues which will help in the improvement of new YBCO materials for broad scope of applications.

1.6 Scope and limitation of the research study

This research work investigates the effect of the addition of both the multiferroics bismuth ferrite BFO and holmium manganite HMO synthesized via sol-gel method and coprecipitation method respectively on the bulk Y-123 superconducting material prepared via the standard solid state reaction method. Starting with the synthesis of the three pure samples of Y-123 with micro-size particles, nanoparticle powder of BFO and nanoparticle powder HMO, following that the preparation of nine samples according to the formulas of each $(Y\text{-}123)_{1-x}(BFO)_x$ and $(Y\text{-}123)_{1-x}(HMO)_x$ (x = 0.00, 0.0025, 0.005, 0.0075, 0.01, 0.03, 0.05, 0.07 and 0.1). Prepared powders and sintered samples will be characterised by X-Ray Diffraction (XRD), four point probe, Field Emission Scanning Electron Microscope FESEM/Dispersive X-Ray Analysis EDX, Ac susceptibility, Electron Spin Resonance (ESR) and Impedance Analyser. The XRD was used to examine the crystalline phase and the structural parameters. The critical transition temperature is carried out using the four point probe technique. The surface morphology and microstructure of the samples are examined using FESEM/EDX. AC susceptibility measurements are used to examine the magnetic properties of the sintered samples at a temperature below Tc with the resistivity. The spin properties are studied at room temperature using ESR technique. Finally, the dielectric properties are carried out at room temperature using the complex Impedance Analyser.

1.7 Outline of the thesis

This thesis is reported into six chapters. First chapter gives a general introduction of presented research work and a review of the historical developments of superconductivity. This chapter also gives the background of the study, statement of the problem, objectives, significance, scope of the study, and outline of the research work. Chapter 2 deals with detailed literature review. It contains background information to assist in understanding the aims and results of this investigation, and also reported previous studies published by other researchers with which these results can be compared. Chapter 3 covers the fundamentals and describes some aspects of the theory of superconductivity, structural, phase transformation, compounds properties including electrical, magnetic, dielectric and electronic spin resonance. Chapter 4 describes the materials and methodology of sample preparation and the different characterization techniques involved related to this research work. Chapter 5 presents the results and discussion, while chapter 6 concludes and summaries all the results and contributions derived from the research.

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