UNIVERSITI PUTRA MALAYSIA

MICROSTRUCTURE AND SUPERCONDUCTING PROPERTIES OF Y-Ba(Ca/K)-Cu-O (Y-123 AND Y-358) SYSTEMS SYNTHESIZED USING THERMAL TREATMENT METHOD

MUSTAFA MOUSA ALI DIHOM

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

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May 2018

Chairman : Professor Abdul Halim Shaari, PhD
Faculty : Science

There is a lack of reports on superconducting behavior of $Y_3Ba_5Cu_8O_{18-\delta}$ (Y-358) superconductor which belong to Y-Ba-Cu-O family. In addition, the role of alkali metals, Ca and K, substitution on the Ba site on the microstructural property of Y-Ba-Cu-O superconductors have not been well understood. In this work, bulk Y-358 and $YBa_2Cu_3O_{7-\delta}$ (Y-132) superconductors were synthesized by a new technique based on thermal treatment method using PVP as capping agent. The samples were sintered in flowing O_2 at 980°C for 24 hour. In addition, the effect of alkali metals (M = Ca and K) substitutions in Ba site of Y-123 and Y-358 on the microstructure and superconducting properties were systematically investigated using X-rays diffraction (XRD), field emission scanning electron microscope (FESEM), energy-dispersive X-ray spectroscopy (EDX), resistivity behaviour (ρ–T), temperature dependence of resistance measurement, and alternating current susceptibility (AC) and electron spin resonance (ESR) techniques.

From XRD results, the Y-123 and Y-358 showed orthorhombic crystal structure besides small amount of secondary phased such as Y-211. In the case of Y-123, the orthorhombicity and crystallite size changed differently with Ca and K substitutions. For Ca substituted Y-358, the orthorhombicity and crystallites size increases up to $x = 0.01$ and 0.02 respectively and then decreases. The intensity of XRD peaks changed unsystematically with K substitution in Y-358, however it shows improvement at $x = 0.03$ and 0.14.

From Field Emission Scanning Electron Microscope (FESEM) micrographs, the grain size of pure Y-358 is larger and more compact compared to Y-123. The grain size was found to be larger when Ba is substituted with either Ca or K than the pure samples in both Y-123 and Y-358. For both Y-123 and Y-358 samples, the grains become much finer, almost with different shape and well-connected as K contents increases.

Both Y-123 and Y-358 samples exhibited good metallic behaviour in the normal state and one step transition. The Y-123 and Y-358 showed critical temperature $T_{c(R=zero)}$ at 87 and 92 K and onset of superconducting transition $T_{c\ (onset)}$ at 93 K and 98 K, respectively. The $T_{c(R=zero)}$ for Ca substituted Y-123 and Y-358 was decreased. The changing of lattice parameters in Y-123 and Y-358 structure due to Ca substitution may disturb the oxygen content and hence affect T_c. The $T_{c(R=zero)}$ was increased as K substitution in Y-123 increased. In general Y-123 and Y-358 samples with initial Ca and K substitution show sharper superconducting transition (ΔT_c) than pure, which could be due to good microstructural morphology and better crystallinity.

The AC susceptibility measurement show that for the Ca substituted Y-123 and Y-358 samples a decrease in diamagnetism onset temperature $T_{c\text{-onset}}$, was observed from real part (χ') which exhibited two-step transitions related to the superconducting intra and intergrain coupling. The $T_{c\text{-onset}}$ decreased in the case of Ca substituted samples and increased in the case of K substituted samples. This decrease is mainly due to the decrease and increase of hole concentration respectively. The intergranular critical current density, J_{cm}, of pure Y-123 and Y-358, 34.4 A cm^{-2} and 34.7 A cm^{-2} respectively, increased to 35 Acm^{-2} at (x=0.08) and 36.3 Acm^{-2} at (x=0.2) for Ca substituted Y-123 and Y-358, respectively and K to 35 Acm^{-2} at (x=0.1) and 38.8 Acm^{-2} at (x=0.12) for Ca substituted Y-123 and Y-358, respectively, which could be due the improvement of the grain boundary and the hence the grains' coupling. On the other hand, Josephson current, I_o, and Josephson energy, E_j decreased and increased with the Ca and K concentration respectively due to degrading and coupling between the grain connectivity.

From electron spin resonance (ESR), the Ca and K substituted in Y-123 and Y-358 showed ESR spectra consisted of two peaks. The g-factors increased with increment of Ca and K content in both Y-123 and Y-358 samples, which could be due to changes in the oxygen ordering in Y-123 and Y-358.

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

MIKROSTRUKTUR DAN SIFAT SUPERKONDUKTOR SISTEM Y-BA (Ca / K) -Cu-O (Y-123 DAN Y-358) YANG DISINTESIS DENGAN MENGGUNAKAN KAEDAH RAWATAN HABA

Oleh

MUSTAFA MOUSA ALI DIHOM

Mei 2018

Pengerusi : Profesor Abdul Halim Shaari, PhD
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Terdapat kekurangan laporan mengenai kelakuan superkonduktor $Y3Ba5Cu8O18-\delta$ (Y-358) superkonduktor yang dimiliki oleh keluarga Y-Ba-Cu-O. Di samping itu, peranan logam alkali, Ca dan K, penggantian pada tapak Ba terhadap sifat mikrostruktur superkonduktor Y-Ba-Cu-O tidak difahami dengan baik. Dalam kajian ini, bahan pukal Y-358 dan $Y2Ba3Cu8O18-\delta$ (Y-132) superkonduktor telah disintesis oleh teknik baru berdasarkan kaedah rawatan haba menggunakan PVP sebagai ejen pengehadapan. Sampel-sampel telah disinter dengan mengalirkan O2 pada 980 ° C selama 24 jam. Selain itu, kesan penggantian logam alkali (M = Ca dan K) di tapak Ba Y-123 dan Y-358 terhadap sifat-sifat mikrostruktur dan superkonduktor disiasat secara sistematik dengan menggunakan spektrum pembelaun sinar-X (XRD), spektroskopi penyebaran tenaga sinar-X (EDX), mikroskop elektron pengimbasan pelepasan medan (FESEM), kelakuan resistiviti (ρ-T), kebergantungan suhu pengukuran rintangan, dan teknik resonans spektrum elektron (AC) dan resonan spin elektron (ESR).

Dari hasil XRD, Y-123 dan Y-358 memperlihatkan struktur kristal ortorombik selain sejumlah kecil fasa menengah seperti Y-211. Dalam Y-123, saiz ortorombik dan kristalit berubah secara berbeza dengan penggantian Ca dan K. Bagi Ca digantikan Y-358, saiz ortorombik dan kristalit meningkat sehingga x = 0.01 dan 0.02 masing-masing dan kemudian berkurangan. Keamatan puncak XRD berubah secara sistematik dengan penggantian K dalam Y-358, namun ia menunjukkan peningkatan pada x = 0.03 dan 0.14.

Dari mikrograf pengimbasan pelepasan elektron mikroskop (FESEM), saiz bijirin tulen Y-358 lebih besar dan lebih kompak berbanding dengan Y-123. Saiz bijian didapati lebih besar

Apabila Ba digantikan dengan Ca atau K daripada sampel tulen di kedua-dua Y-123 dan Y-358. Untuk kedua-dua sampel Y-123 dan Y-358, biji-bijian menjadi lebih halus, hampir dengan bentuk yang berbeza dan terhubung dengan baik apabila kandungan K meningkat.

Kedua-dua sampel Y-123 dan Y-358 mempamerkan sifat logam yang baik dalam keadaan normal dan peralihan satu langkah. Y-123 dan Y-358 menunjukkan suhu kritikal Tc(R=zero) pada 87 K dan 92 K dan permulaan superconducting transition Tc (onset) pada 93 K dan 98 K, masing-masing. Tc(R=zero) untuk Ca digantikan Y-123 dan Y-358 telah menurun. Perubahan parameter kekisi dalam struktur Y-123 dan Y-358 disebabkan oleh penggantian Ca boleh mengganggu kandungan oksigen dan seterusnya menjejaskan Tc. Tc(R=zero meningkat apabila penggantian K dalam Y-123 meningkat. Secara umum sampel Y-123 dan Y-358 dengan pengubahsuaian Ca dan K menunjukkan peralihan mensuperkonduksi lebih tajam (□Tc) daripada tulen, yang boleh disebabkan oleh morfologi mikrostruktur yang baik dan kristalografi yang lebih baik.

Pengukuran kerentanan AC menunjukkan bahawa untuk Ca menggantikan sampel Y-123 dan Y-358, penurunan pada suhu awal diamagnetisme Tc (onset), diperhatikan dari bahagian sebenar (χ') yang memperlihatkan peralihan dua langkah yang berkaitan dengan intra dan inter butiran superkonduktor gandingan. Tc (onset) berkurangan dalam kes sampel Ca yang digantikan dan meningkat dalam kes sampel K yang digantikan. Penurunan ini adalah disebabkan oleh penurunan dan peningkatan penumpuan lubang masing-masing. Ketumpatan arus kritikal inter butiran, Jcm, tulen Y-123 dan Y-358, 34.4 Acm-2 dan 34.7 Acm-2 masing-masing, meningkat kepada 35 Acm-2 pada (x = 0.08) dan 36.3 Acm-2 pada (x = 0.2) untuk Ca digantikan masing-masing Y-123 dan Y-358 dan K hingga 35 Acm-2 pada (x = 0.1) dan 38.8 Acm-2 pada (x = 0.12) untuk Ca digantikan Y-123 dan Y-358 masing-masing, yang mungkin disebabkan oleh peningkatan sempadan butiran dan gabungan butiran. Sebaliknya, tenaga Arus Josephson, Io, dan Josephson, Ej menurun dan meningkat dengan penumpuan Ca dan K masing-masing kerana direndahkan dan gandingan antara gabungan butiran.

Dari resonans spin elektron (ESR), Ca dan K yang digantikan dalam Y-123 dan Y-358 menunjukkan spektrum ESR terdiri daripada dua puncak. Faktor g meningkat dengan peningkatan kandungan Ca dan K dalam kedua-dua sampel Y-123 dan Y-358, yang mungkin disebabkan oleh perubahan dalam susunan oksigen di dalam Y-123 dan Y-358.

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In the name of Allah, Most Gracious, Most Merciful. All praise be to Allah, the Supreme Creator, Nourisher and Sustainer of the universe. We beseech the Almighty to bestow His choicest blessings on our illustrious Master, Nabi Muhammad (peace be upon Him), on His pure family, His noble companions and all believers in every era.

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I certify that a Thesis Examination Committee has met on 15 May 2018 to conduct the final examination of Mustafa Mousa Dihom on his thesis entitled "Microstructure and Superconducting Properties of Y-Ba (Ca/K)-Cu-O (Y-123 and Y-358) Systems Synthesized Using Thermal Treatment Method" 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 Doctor of Philosophy.

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LIST OF ABBREVIATIONS AND SYMBOLS

HTSC	High Temperature Superconductor
LTSC	Low Temperature Superconductor
YBCO	Yttrium Barium Copper Oxide
Y-123	Family member in $YBa_2Cu_3O_{7-\delta}$
Y-124	Family member in $YBa_2Cu_4O_{7-\delta}$
Y-358	Family member in $Y_3Ba_5Cu_8O_{18-\delta}$
Ca	Calcium
Ba	Barium
K	Potassium
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
^{o}C	Degrees Celsius
T_p	Coupling peak temperature
K	Kelvin
T	Temperature in Kelvin scale
PG	Pseudogap
T^*	Pseudogap temperature
J_C	Critical Current Density (Acm^{-2})
J	Current Density (Acm^{-2})
J_m	inter-grain current density
J_g	intra-grain currents with density
R &D	Research and Development
SQUID	Superconducting quantum interference devices
XRD	X-ray diffraction
FESEM	Field emission scanning electron microscope
EDX	Energy Dispersion X-ray

AC	Susceptibility measurement
ESR	Electron Spin Resonance
EPR	Electron paramagnetic Resonance
AAS	Atomic absorption spectrometer
GL	Ginzburg-Landau theory
ρ	Resistivity
A	Ampere
I	Current
R	Resistance
AC	Alternating current
DC	Direct Current, in opposition to alternative current
V	Voltage
BCS	Bardeen, Cooper and Schrieffer theory
ZFC	Zero Field Cool
X	Magnification
BSCCO	One of the three forms of the superconductor above.
B	Magnetic Field
H	Magnetic Field
He	Magnetic field applied
Hc	Critical field
H_{ac}	Applied field
M	Magnetization
$Hc2$	Upper critical magnetic field
$Hc1$	Lower critical magnetic field
U	Voltage (V)
wt.	Weight
nm	Nanometer
mm	Millimeter
µm	Micrometer
h	Houre
min	Minute
sec	Second
Hz	Hertz

g	Gram
w	Width of a superconduting tape (mm).
ε	Permittivity of medium (F/m).
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).
ψ	Order parameter
ϕ	Phase of the order parameter
τ	Time constant (s)
a,*b*,*c*- axis	Lattice parameter
V	Unit cell volume
P	Hole concentration
x	Amount of weight percent added
θ	Angle of diffraction
ξ	Coherence lelength
δ	Delta
$=$	Equal
$\leq$	Less than or equal to
$\geq$	Greater than or equal to
$>$	Bigger than
Å	Angstrom (Unit of length equal to 10^{-10} m)
ICSD	Inorganic Crystal Structure Database
κ	Ginzburg-Landau constant
ac	Alternate current
dc	direct current

CHAPTER 1

INTRODUCTION

1.1 Background of the study

High temperature superconductivity (HTSC), based on cuprate system, was discovered by Bednorz and Muller in 1986 which introduces a new era for superconductivity research area of interest (Keimer et al., 2015; Maple, 1998). Since that time many efforts have been devoted to understanding nature of superconductivity, find new materials and to make these materials ready for the uses in technology application (Chu et al., 2015). Currently, more than 200 members of copper-based oxides based superconductors were found by self-doping or doping into the parent materials of some copper based oxides (Chu et al., 2015). $BiSr_2Ca_2Cu_3O_{10}$ and $YBa_2Cu_3O_7$ (Y-123) systems appear to be the most candidate for superconductor's technology. The Y-123 is more favourable for superconducting application advantage this is attributed to its physical robustness and superior superconducting behaviour in a higher magnetic field (Maple, 1998). The great trend in superconductivity field is motivated by the promise possible uses of it in the near future for electric power transmission without losses and also in the construction of quantum high-power generators. In addition, superconductivity phenomenon is involved to develop quantum computers (Tsai, 2010).

1.2 Y-Ba-Cu-O (YBCO) family

The Yttrium barium copper oxide (Y-Ba-Cu-O) was the first material showing superconductivity above 77 K (boiling point of nitrogen) (Bednorz & Müller, 1986). Several stoichiometry formula were synthesized where superconducting properties depend on the number of CuO chains and CuO_2 planes (Hackl, 2011). Currently, Y-Ba-Cu-O family include $YBa_2Cu_3O_{7-\delta}$ (Y-123), $YBa_2Cu_4O_8$ (Y-124), $Y_2Ba_4Cu_7O_{15}$ (Y-247), and $Y_3Ba_5Cu_8O_x$ (Y-358). Y-123 has one CuO chain and two CuO_2 planes with 91 K critical temperature T_c. Y-124 has one double CuO chain and two CuO_2 planes with T_c = 80 K while Y-247 has a mixture of single and double CuO chains with a mix of two CuO_2 planes and exhibiting T_c (30 – 90) K depending on its oxygen content (Slimani et al., 2014). The phases mentioned above were previously identified as secondary phases in Y-123 system, hence it is difficult to obtain it in a single phase (Chu et al., 2015). Recently, Y-358 compound was firstly prepared by Aliabadi et al. with T_c = 98 K which was confirmed to be attributed to the strong correlation interaction within layered copper oxides (Bednorz & Müller, 1988; Lee et al., 2006). Y-358 is similar to that of Y-123, but with two additional CuO chains and three additional CuO_2 planes (Mazaheri et al., 2015). There is a discrepancy in the literature regarding structure properties of Y-358 phase. While some group have claimed that it has same lattice parameters like Y-123 (Topal et al., 2011; Udomsamuthirun et al., 2010), other reported that the lattice parameters *a* and *b* are same to Y-123 while *c* lattice parameter three time that of in Y-123 (Aliabadi et al., 2009). Due to the similarity of the two phases, Y-123 and Y-358, further characterization is needed to shed light on the physical and superconducting behavior of Y-358 phase.

1.3 Role of chemical substitution

Chemical substitution strongly affects the structure, physical and superconducting properties of cuprate superconductors. These oxides, as in the example of Y-123, consists of active conducting block [(CuO_2) Y (CuO_2)] separated by [(BaO) (CuO) (BaO)] that can be considered as charge reservoir that controls the carrier into the conducting layer. Doping can be done by partially replacing one ion by another with different valence or by changing oxygen content and self-doping. Superconductivity was induced in antiferromagnetic insulating La_2CuO_4 by increasing charge carriers in CuO_2 layer as a result of doping of La site with Ba. In Self-doping, increasing the oxygen content (δ), the holes are brought into the CuO_2 planes and change the structure from tetragonal $YBa_2Cu_3O_6$ (antiferromagnetic phase) to orthorhombic $YBa_2Cu_3O_{6+\delta}$ (Y-123 superconducting phase). On the other hand, doping of Y, Ba, or Cu by univalent or divalent elements led to changes in its structure and electronic properties. Various research works on substitutions in oxide superconducting materials were of immense importance, as variations in critical transition temperature were usually observed (M. Sahoo & Behera, 2014; Talantsev et al., 2014). Thus, the doping can basically vary the concentration of the hole in a controlled manner and influence the materials' superconducting properties. Many research activities were mainly devoted to explore substitution effects at Y site (Awana et al., 1994; Fisher et al., 1993; Guan et al., 1996; Tallon & Flower, 1993). The concentration of charge carrier and effects of charge transfer from Cu-O chains to the conducting layers CuO_2 in Y-123 take place by Ca^{2+} partial substitution for Y^{3+}. An enhanced T_c is observed as the Ca^{2+} ion replaces the trivalent Y^{3+} ion (Delorme et al., 2002). The Ca ionic radius is slightly greater than that of Y, so a hole is created in the CuO_2 plane by the substitution, and increases the concentration of electron hole per unit Cu–O (Haibin & Welch, 2005). In general, superconductivity is induced by Oxygen in Ca-free Y-123 and Ca does likewise in oxygen-deficient Y-123. $\delta = 6.92$ is found to be the optimal doping for oxygen in Y-123 is, with a T_c of about 90 K. The superconducting T_c can be suppressed by alkaline earth metals substitution at Y site (Feng et al., 2001; Nishizaki et al., 2005). Specifically, the temperature range of 90 to 80 K has been a subject of vast importance and is reported to raise the critical current density, J_c, of the material, which is an important parameter for applications Krabbes et al. (2006), and is dependant on the number of pinning centers per unit volume in the superconducting phase (Campbell, 1996). The current carrying capability of Y-123 across its grain boundaries is improved by 35 percent calcium substitution. Doping lowers its 91 K critical temperature T_c to a level uncomfortably close to that of the liquid nitrogen coolant, 77 K. Up to now, two variants of YBCO systems that can accommodate calcium without T_c loss were discovered namely,$Y_2CaBa_4Cu_7O_{16-\delta}$ (T_c ~ 96 K) and $Y_3CaBa_4Cu_8O_{18-\delta}$ (T_c ~ 99 K). Like other types of HTSC, these two types of the Y-

123 superconducting system have their problems of poor mechanical properties and brittle nature as well (Mellekh et al., 2006).

Among the cationic substitutions in Y-123, potassium substitution for barium has gain more interest (Felner & Barbara, 1988). The potassium ionic radius (1.33 Å) is close to that of Ba (1.35 Å), which makes it an ideal substituent for Ba in Y-123. The potassium substitution for Ba in Y-123 has been performed by some groups. While some groups reported that T_c decreases with increasing potassium doping (Mukherjee et al., 1992; Wu et al., 2000), some other groups have claimed it increases up to 124 K using KOH (Beales et al., 1992) or 135 K using K_2CO_3 as the source of potassium (Khan, 1988). Thus the effect of potassium addition on superconductivity in YBCO system is still controversial.

The compound substitution on Y-358 phase is still not common till recently. It was observed that doping with Ca into Y358 phase reduced T_c significantly without affecting the orthorhombic structure (Ayas et al., 2011). The studies of superconducting properties of the Ca and K-doped in Y-358, especially in its polycrystalline state, are limited or have not been studied and reported yet. Therefore, in this research, our aims are to investigate the effect of Ca and K substituted at the Ba-site for the Y-123 and Y-358 phases, according to structural, superconducting and electrical properties. Thermal treatment technique was carried out to prepare the materials. The findings are discussed based on XRD, FESEM, electrical measurements, magnetic and ESR spectra.

1.4 Synthesis of YBCO

Various synthesis methods have been employed to prepare Y-123 with the aim of improving the morphology and superconducting properties. Frequently, these ceramics were produced by the solid-state reaction and wet chemical methods (Yeoh & Abd-Shukor, 2008). These methods have many advantages and disadvantages as will be extensively discussed in the following chapter. In the route of the solid state synthesis, the reaction of the reactants takes place in the solid state method to obtain the end product. Of all the techniques, the solid-state technique is the most widely used when it comes to the synthesis of a vast number of oxide, ceramic materials, boride, carbide, and nitride. However, this method has some drawbacks such as difficulty to obtain homogenous oxide and the need of several steps of grinding and annealing process. Wet chemical based methods have been extensively used to fabricate superconductors in order to obtain the good quality product (Yeoh & Abd-Shukor, 2008). These methods yield an excellent homogeneous oxide and smaller size precursors in comparison with traditional solid state reaction method. Recently, thermal treatment has emerged as a new method to synthesize fine particles oxides. This method is based on an aqueous solution of metals nitrates and polymer such as polyvinyl pyrrolidone (PVP) that acts as a capping agent without the addition of other chemicals. Several oxides nanoparticles have been successfully prepared using this method (Al-Hada et al., 2016; Al-Hada et al., 2014; Naseri et al., 2011). However, there is a lack in the literature on superconductor synthesis using the thermal treatment method. It is expected that by using this method, good quality Y-Ba-Cu-O based materials could be obtained.

1.5 Statement of the problem

YBCO family has wider range potential application compared to other materials HTSC and thus, they become a major research field (Maple, 1998). However, J_c value are limited basically by their insufficient flux pinning properties (Vinu et al., 2009). This is caused by their poor flux pinning properties and grain boundaries (Larbalestier et al., 1991; Mannhart et al., 2000). Although the J_c for single YBCO crystals is very high, the value is very low for polycrystals; it only allows a small current to pass while maintaining superconductivity. Also, another problem with Y-123 compound is that the content of oxygen is not stable (Choudhary et al., 2004). The above mentioned issues can be overcome by elemental doping or substitution in YBCO materials synthesized by new method (Anjela et al., 2000; Berenov et al., 2002; Celebi et al., 2000; Daniels et al., 2000; Mohan et al., 2007; A Veneva et al., 1996). So far, synthesis of Y-123 and Y-358 using thermal treatment method has not yet been performed. While the role of alkali metals (Ca, K) substituting in Ba-site on the morphology, structural superconducting behaviour of Y-123 and Y-358 systems is not well known yet. Therefore the aim of this work is to study the effect of Ca and K substitution on barium site in both Y-123 and Y-358 systems prepared using thermal treatment method. The ultimate hope is to shed more light on the critical temperature, grain boundary, critical current density, critical magnetic field, AC susceptibility, resistivity, by using X-ray Diffraction (XRD), FESEM analysis, four point probe method, AC susceptibility and ESR spectra.

1.6 Objectives of the study

Our research has been designed based on the following objectives:

1. To synthesize single phase Y-123 and Y-358 superconducting ceramics using thermal treatment method with PVP as capping agent.
2. To synthesize single phase of Y-123 and Y-358 substituted with Ca, K at Ba site via thermal treatment method with PVP as capping agent.
3. To determine the differences between the properties of bulk of Y-123 and bulk Y-358 superconducting ceramics.
4. To investigate the effect of (Ca, K) substitutions at Ba-site on the microstructural, electrical, magnetic and superconducting properties of Y-123 and Y-358 superconductors.

1.7 Significance of the study

Technological advances demand more complex portable devices with various functions. This includes efforts to fabricate HTSC materials, production of long superconducting wires and cables with higher J_c, superconducting magnets. Electric power networks which include transformers, generators, fault-current limiters, motors, and magnetic energy storage and superconducting quantum interference (SQUID) devices are among the significant issues. Due to the HTSC strong impact potentials on business and society, a new technology needs to be developed in Research and Development by scientists and engineers toward fabricating YBCO materials which should be less expensive and better than other systems. The outcome of this research will add to the knowledge and understanding of the subject and ought to shed more light on scientific and technological issues which will help in the development of synthesis of YBCO materials for wide range of applications.

1.8 Scope of the research work

The study will investigate the effect of doping of calcium Ca and potassium K in barium site, with an aim to find higher T_c value in the high temperature of bulk Y-123 and Y-358 superconducting materials that will be carried out using thermal treatment method only. By so doing, the findings of this study can be compared with previous results as an effort to complement the work of other researchers. The bulk Y-123 and Y-358 will be prepared based on composition with a stoichiometric formula $Y(Ba_{1-x}Ca_x)_2Cu_3O_{7-\delta}$, $Y(Ba_{1-x}K_x)_2Cu_3O_{7-\delta}$, $Y_3(Ba_{1-x}Cax)_5Cu_8O_{18-\delta}$ and $Y_3(Ba_{1-x}K_x)_5Cu_8O_{18-\delta}$ for x = (0.00, 0.01, 0.02, 0.03, 0.04, 0.05, 0.8, 0.1, 0.125), (0.00, 0.03, 0.05, 0.08, 0.1, 0.125, 0.15, 0.175, 0.2, 0.25), (0.00, 0.01, 0.02, 0.03, 0.05, 0.08, 0.1, 0.2) and (0.00, 0.03, 0.05, 0.08, 0.1, 0.12, 0.14, 0.16 0.20) respectively. The synthesized powder and the sintered samples will be characterized using X-ray diffraction (XRD), Field emission scanning electron microscope (FESEM)/dispersive X-ray analysis (EDX). The electric properties are studied by using four point probe measurements and the magnetic properties will also be studied by AC susceptibility measurement. The spin properties are studied by Spin Electron Resonance (ESR) signals.

1.9 Outline of the thesis

This work is reported in 6 chapters. The first chapter describes a short review of the historical developments of superconductivity. This chapter also gives the statement of the problem, background of the study, objectives, significance, and scope of the study, motivation, 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 present previous reports by other researchers with which these results can be compared. Chapter 3 describes the theory of superconductivity and the properties of HTSC. Then, chapter 4, the methodology of sample preparation and characterization techniques related to this research work are described. In Chapter 5 gives the results and discussion of the structure, morphological, AC susceptibility and superconducting properties of YBCO (123) and YBCO (358) substitution within Ca and K in the Ba site are presented. Lastly, Chapter 6 gives the conclusion derived from the research and suggestion for future work.

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