



**UNIVERSITI PUTRA MALAYSIA**

***STRUCTURAL, MAGNETIC AND ELECTRICAL PROPERTIES OF YBCO-123 SUPERCONDUCTOR REACTED WITH  $Y_2O_3$  AND  $BaZrO_3$  NANOPARTICLES***

**NURHIDAYAH MOHD HAPIPI**

**FS 2018 19**



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By

**NURHIDAYAH BINTI MOHD HAPIPI**

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

**December 2017**

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## **DEDICATION**

This work is dedicated to my beloved father and mother

**MOHD HAPIPI BIN HANAFI**

**ASPALILA BINTI JUSOH**

and to my sibling

**NORHIWANI BINTI MOHD HAPIPI**

**MOHD IQRAM BIN MOHD HAPIPI**

**NURHIDAYATI BINTI MOHD HAPIPI**

Thank you for everything

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

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By

**NURHIDAYAH BINTI MOHD HAPIPI**

**December 2017**

**Chairman: Assoc. Prof. Chen Soo Kien, PhD**

**Faculty: Science**

In this work, superconducting properties of YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7- $\delta$</sub>  (Y-123) reacted with 5.0 mol.% of Y<sub>2</sub>O<sub>3</sub> and different molar percentages (mol.%) of BaZrO<sub>3</sub> (BZO) nanoparticles were studied. Series 1 samples are Y-123 reacted with  $x$  mol.% BZO nanoparticles and Series 2 samples are Y-123 reacted with 5.0 mol.% of Y<sub>2</sub>O<sub>3</sub> and  $x$  mol.% BZO nanoparticles ( $x = 0.0 - 7.0$  mol.%). The samples were prepared using co-precipitation (COP) method. The phase formation, microstructure, magnetic and electrical properties of the samples were investigated using thermogravimetric analysis (TGA), X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), temperature dependence of resistance measurement, and alternating current susceptibility (ACS). XRD patterns indicated that all of the samples could be indexed to space group ( $Pmmm$ ) with orthorhombic crystal structure. Y-123 is the major phase while Y-211 was detected as minor secondary phase in all the samples. Besides, BZO peaks started to appear when its addition level was increased from 1.0 mol.%. The SEM images showed that all the samples have irregular shaped grains and they are randomly distributed. The average grains size for both series increased in the range of 0.30  $\mu\text{m}$  to 0.50  $\mu\text{m}$  with increasing amount of BZO nanoparticles addition. Yet, the grain size for Series 2 samples is slightly higher than that in Series 1 indicating that Y<sub>2</sub>O<sub>3</sub> may promote grain growth. The temperature dependence of resistance measurements showed a metallic behaviour at normal state and a superconducting transition to zero resistance for all the samples. The value of  $T_c$  for the pure sample is 91.6 K and it decreased to 81.5 K and 87.6 K for Series 1 and Series 2 samples, respectively for 7.0 mol.% BZO addition. The  $T_c$  values and the transition width,  $\Delta T_c$  for the Series 2 are slightly higher than Series 1. The higher value of transition width,  $\Delta T_c$  shows the degradation of homogeneity within the samples. The ACS measurement showed the decreasing of  $T_{c\text{-onset}}$ ,  $T_{c_j}$  and  $T_p$  with the increase of BZO nanoparticles addition for all the samples. The decrease of  $T_{c_j}$  and  $T_p$  is

due to the weakening of the intergranular coupling and the decrease of pinning forces. The obtained  $I_0$  and  $J_{cm}$  for Series 2 are higher than that for Series 1 indicating that the grain coupling of the former is stronger. As a conclusion, the results shows that the co-addition of  $Y_2O_3$  and BZO (Series 2) can maximize the  $J_{cm}$  value and improve a flux pinning if compared to the addition of BZO only (Series 1). However, the optimum value for co-addition of BZO is only up to 2.0 mol.% since further addition will degrade the superconductivity of the samples. The highest  $J_{cm}$  is 2.096 A/ cm<sup>2</sup> for co-addition of 2.0 mol.% BZO and  $Y_2O_3$ .



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

**SIFAT-SIFAT STRUKTURAL, MAGNETIK DAN ELEKTRIK DALAM  
SUPERKONDUKTOR YBCO-123 YANG BERTINDAK BALAS DENGAN  $Y_2O_3$   
DAN  $BaZrO_3$  NANOPARTIKEL**

Oleh

**NURHIDAYAH BINTI MOHD HAPIPI**

**Disember 2017**

**Pengerusi: Prof. Madya Chen Soo Kien, PhD**  
**Fakulti: Sains**

Dalam kajian ini, sifat-sifat superkonduktor  $YBa_2Cu_3O_{7-x}$  (Y-123) yang bertindak balas dengan 5.0 mol.%  $Y_2O_3$  dan nanopartikel BZO pada peratusan molar yang berbeza (mol.%) telah dikaji. Sampel Siri 1 adalah Y-123 yang bertindak balas dengan  $x$  mol.% nanopartikel BZO dan sampel Siri 2 adalah Y-123 yang bertindak balas dengan 5.0 mol.%  $Y_2O_3$  dan  $x$  mol.% nanopartikel BZO ( $x = 0.0 - 7.0$  mol.%). Sampel Y-123 telah disediakan menggunakan kaedah se-pemendakan. Kesan penambahan nanopartikel pada pembentukan fasa, mikrostruktur, sifat-sifat magnetik dan elektrik ke atas sampel telah dikaji menggunakan analisis termogravimetrik (TGA), pembelauan sinar-X (XRD), mikroskop elektron pengimbas (SEM), spektroskopi serakan tenaga sinar-X (EDX), pergantungan suhu pada pengukuran kerintangan, dan kerentanan arus ulang-alik (KAU). Corak XRD menunjukkan bahawa semua sampel boleh diindekskan kepada kumpulan ruang ( $Pmmm$ ) dengan struktur hablur ortorombik. Y-123 adalah fasa utama manakala Y-211 dikesan sebagai fasa minor kedua di dalam semua sampel. Selain itu, puncak BZO mula kelihatan apabila aras penambahan meningkat daripada 1.0 mol.%. Imej SEM menunjukkan bahawa semua sampel mempunyai bentuk tak menentu dengan taburan rawak. Purata saiz bijirin bagi kedua-dua siri bertambah dalam kadar 0.30  $\mu m$  hingga 0.50  $\mu m$  dengan peningkatan jumlah penambahan nanopartikel BZO. Walau bagaimanapun, saiz bijirin bagi sampel Siri 2 adalah tinggi sedikit berbanding Siri 1 menunjukkan  $Y_2O_3$  menggalakan pertumbuhan bijirin. Semua sampel dalam Siri 1 dan Siri 2 menunjukkan sifat logam pada keadaan normal dan peralihan mensuperkonduksi ke rintangan sifar seperti yang ditunjukkan oleh pergantungan suhu pada pengukuran rintangan. Nilai  $T_c$  bagi sampel tulen adalah 91.6 K dan nilai ini berkurang kepada 81.5 K dan 87.6 K bagi sampel Siri 1 dan Siri 2, masing-masing dengan penambahan 7.0 mol.% nanopartikel BZO. Nilai  $T_c$  dan lebar peralihan,  $\Delta T_c$  bagi Siri 2 adalah tinggi sedikit berbanding Siri 1. Ketinggian nilai lebar peralihan,  $\Delta T_c$  menunjukkan

ketakhomogenan di dalam sampel. Pengukuran bagi KAU menunjukkan penurunan bagi  $T_{c-onset}$ ,  $T_{cj}$  dan  $T_p$  dengan peningkatan penambahan nanopartikel BZO untuk semua sampel. Penurunan  $T_{cj}$  dan  $T_p$  adalah disebabkan gandingan antara butiran yang melemah dan penurunan daya pengepinaan. Keputusan arus Josephson,  $I_0$  dan ketumpatan arus genting antara butiran,  $J_{cm}$  bagi Siri 2 adalah lebih tinggi berbanding Siri 1 menunjukkan bahawa gandingan butiran bagi Siri 2 adalah lebih kuat. Sebagai konklusi, keputusan menunjukkan bahawa penambahan bersama  $Y_2O_3$  dan BZO (Siri 2) boleh memaksimumkan nilai  $J_{cm}$  dan menambahbaik pengepinaan fluks jika dibandingkan dengan penambahan BZO sahaja (Siri 1). Walaubagaimanapun, nilai optimum bagi penambahan bersama BZO adalah sehingga 2.0 mol.% memandangkan penambahan selanjutnya akan merosotkan kesuperkonduksian sampel.  $J_{cm}$  yang tertinggi adalah 2.096 A/cm<sup>2</sup> bagi penambahan bersama 2.0 mol.% BZO dan  $Y_2O_3$ .





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I certify that a Thesis Examination Committee has met on 7 December 2017 to conduct the final examination of Nurhidayah binti Mohd Hapipi on her thesis entitled "Structural, Magnetic and Electrical Properties of YBCO-123 Superconductor Reacted with  $Y_2O_3$  and  $BaZrO_3$  Nanoparticles" 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.

Members of the Thesis Examination Committee were as follows:

**Nurul Huda binti Osman, PhD**

Senior Lecturer  
Faculty of Science  
Universiti Putra Malaysia  
(Chairman)

**Yap Wing Fen, PhD**

Senior Lecturer  
Faculty of Science  
Universiti Putra Malaysia  
(Internal Examiner)

**Kong Wei, PhD**

Associate Professor  
Kuala Lumpur Infrastructure University College  
Malaysia  
(External Examiner)



---

**NOR AINI AB. SHUKOR, PhD**

Professor and Deputy Dean  
School of Graduate Studies  
Universiti Putra Malaysia

Date: 27 February 2018

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:

**Chen Soo Kien, PhD**

Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Chairman)

**Mohd Mustafa Awang Kechik, PhD**

Senior Lecturer  
Faculty of Science  
Universiti Putra Malaysia  
(Member)

**Tan Kar Ban, PhD**

Associate Professor  
Faculty of Science  
Universiti Putra Malaysia  
(Member)

---

**ROBIAH BINTI YUNUS, PhD**

Professor and Dean  
School of Graduate Studies,  
Universiti Putra Malaysia

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Signature: \_\_\_\_\_

Name of Chairman  
of Supervisory

Committee: Chen Soo Kien, PhD

Signature: \_\_\_\_\_

Name of Member  
of Supervisory

Committee: Mohd Mustafa Awang Kechik, PhD

Signature: \_\_\_\_\_

Name of Member  
of Supervisory

Committee: Tan Kar Ban, PhD

## TABLE OF CONTENTS

		Page
	<b>ABSTRACT</b>	i
	<b>ABSTRAK</b>	iii
	<b>ACKNOWLEDGEMENT</b>	v
	<b>APPROVAL</b>	vi
	<b>DECLARATION</b>	viii
	<b>LIST OF TABLES</b>	xiii
	<b>LIST OF FIGURES</b>	xv
	<b>LIST OF SYMBOLS AND ABBREVIATIONS</b>	xx
<b>CHAPTER</b>		
<b>1</b>	<b>INTRODUCTION</b>	
	1.1 Background	1
	1.2 History of High Temperature Superconductor (HTS)	1
	1.3 Application of HTS	2
	1.3.1 Maglev Train	2
	1.3.2 SQUIDs	3
	1.4 Problem Statement and Research Objectives	3
	1.5 Thesis Overview	4
<b>2</b>	<b>LITERATURE REVIEW</b>	
	2.1 Method of Synthesizing YBCO Bulk	5
	2.1.1 Solid-State Method	5
	2.1.2 Co-Precipitation Method	6
	2.2 Addition of Nanoparticles into YBCO	7
	2.2.1 Addition of BaZrO <sub>3</sub> Nanoparticles into YBCO	8
	2.2.1.1 Effect on Crystal Structure, Phase Formation, and Microstructure	8
	2.2.1.2 Effect on Superconducting Transition Temperature, $T_c$	9
	2.2.1.3 Effect on Critical Current Density, $J_c$	9
	2.2.2 Co-addition of BZO nanoparticles and Y <sub>2</sub> O <sub>3</sub> into YBCO Superconductor	10
	2.2.2.1 Effect on Crystal Structure, Phase Formation, and Microstructure	11
	2.2.2.2 Effect on Superconducting Transition Temperature, $T_c$	12
	2.2.2.3 Effect on Critical Current Density, $J_c$	12
<b>3</b>	<b>BASIC PROPERTIES AND THEORY</b>	
	3.1 Brief History of Superconductivity	15
	3.2 Properties of Superconductor	16
	3.2.1 Zero Electrical Resistance	16
	3.2.2 Ideal Diamagnetism (Meissner Effect)	17

3.3	Classification of Superconductor	22
3.3.1	Type-I Superconductor	23
3.3.2	Type-II Superconductor	24
3.4	Critical Current Density, $J_c$	26
3.5	Vortex States and Flux Pinning in the Superconductor	26
3.6	Formation of Cooper pairs and BCS Theory	29
3.7	YBCO System	31
<b>4</b>	<b>MATERIALS AND METHOD</b>	
4.1	Introduction	34
4.2	Analysis of Chemical Reaction Equation and Calculation of Raw Materials	34
4.3	Overview of YBCO Process via COP Method	36
4.4	Synthesis of Y-123 with Addition of BZO and Co-Addition of BZO and $Y_2O_3$	37
4.4.1	Co-Precipitation (COP) of Y-123	37
4.4.2	Addition of BZO Nanoparticles	41
4.4.3	Co-Addition of BZO Nanoparticles and $Y_2O_3$	41
4.5	Heat Treatment	42
4.5.1	Calcination Process	42
4.5.2	Sintering and Annealing Process	43
4.6	Sample Characterization	44
4.6.1	Thermogravimetric Analysis (TGA)	44
4.6.2	X-ray Diffraction (XRD)	47
4.6.2.1	Crystallite Size and Lattice Strain	48
4.6.3	Scanning Electron Microscope (SEM) and Energy-Dispersive X-ray Spectroscopy (EDX) Analysis	48
4.6.4	Temperature Dependence of Electrical Resistance Measurement	50
4.6.5	Alternating Current Susceptibility (ACS)	52
<b>5</b>	<b>RESULTS AND DISCUSSION</b>	
5.1	Thermogravimetry Analysis (TGA)	55
5.2	Addition of BZO nanoparticle into Y-123 (Series 1)	58
5.2.1	X-ray Diffraction (XRD) Analysis	58
5.2.2	Microstructure and Elemental Analysis	65
5.2.2.1	Scanning Electron Microscopy (SEM)	65
5.2.2.2	Energy Dispersive X-Ray (EDX) Analysis	69
5.2.3	$T_c$ Deduced from Temperature Dependence of Electrical Resistance Measurement	72
5.2.4	Alternating Current Susceptibility (ACS) Measurement	76
5.3	Co-addition of BZO Nanoparticle and $Y_2O_3$ into Y-123 (Series 2)	81
5.3.1	X-ray Diffraction (XRD) Analysis	81
5.3.2	Microstructure and Elemental Analysis	88

5.3.2.1	Scanning Electron Microscopy (SEM)	88
5.3.2.2	Energy Dispersive X-Ray (EDX)	92
5.3.3	$T_c$ Deduced from Temperature Dependence of Electrical Resistance Measurement	96
5.3.4	Alternating Current Susceptibility (ACS) Measurement	99
<b>6</b>	<b>CONCLUSION</b>	
6.1	Conclusion	105
6.2	Recommendation/ Future Work	107
	<b>BIBLIOGRAPHY</b>	108
	<b>APPENDICES</b>	117
	<b>BIODATA OF STUDENT</b>	129
	<b>LIST OF PUBLICATIONS</b>	130



## LIST OF TABLES

Table		Page
2.1	Summary of the studies related to Y-123 with the addition of BZO and $Y_2O_3$	13
3.1	The critical temperature, $T_c$ for selected type-I and type-II superconducting materials	23
4.1	The mass of chemical materials used to prepare 20 g of pure Y-123	35
5.1	Chemical reaction involved in the formation of Y-123	57
5.2	Different weight percentages of Y-123, Y-211 and BZO phases formed for Y-123 + $x$ mol.% of BZO nanoparticles	61
5.3	Lattice parameter of $a$ -, $b$ -, and $c$ -axis and unit cell volume for Y-123 + $x$ mol.% of BZO samples	63
5.4	Crystallite size and lattice strain for Y-123 + $x$ mol.% of BZO samples. Crystallite size was calculated using the Scherrer and Williamson-Hall methods while lattice strain was calculated using the Williamson-Hall method	64
5.5	Average grain size of Y-123 + $x$ mol.% of BZO samples	68
5.6	The atomic percentages and atomic ratios obtained from EDX spectra for Y, Ba, Cu and O elements for each sample	72
5.7	Superconducting properties and hole concentration for the Y-123 + $x$ mol.% of BZO samples	75
5.8	Coupling peak temperature, $T_p$ , onset critical temperature, $T_{c-onset}$ , phase lock-in temperature, $T_{cj}$ , and Josephson current, $I_0$ for Y-123 + $x$ mol.% of BZO samples in AC field of 0.5 Oe	78
5.9	The cross-section area of sample, peak temperature, $T_p$ and the intergranular critical current density, $J_{cm}$ for Y-123 + $x$ mol.% of BZO nanoparticles in AC field of 0.5 Oe	80
5.10	Weight percentages of Y-123, Y-211 and BZO phases formed for Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ + $x$ mol.% of BZO nanoparticles	84
5.11	Lattice parameter and unit cell volume for Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ + $x$ mol.% of BZO samples	86

5.12	Crystallite size and lattice strain for Y-123 and co-addition of 5.0 mol.% $Y_2O_3 + x$ mol.% of BZO samples. Crystallite size was calculated using the Scherrer and Williamson-Hall methods while lattice strain was calculated using the Williamson-Hall method	87
5.13	Average grain size of Y-123 samples with co-addition of 5.0 mol.% $Y_2O_3 + x$ mol.% BZO samples	91
5.14	The atomic percentages and atomic ratios obtained from EDX spectra for Y, Ba, Cu and O elements for each sample	95
5.15	Superconducting properties and hole concentration for the Y-123 with co-addition of 5.0 mol.% $Y_2O_3 + x$ mol.% BZO samples	98
5.16	Coupling peak temperature, $T_p$ , onset critical temperature, $T_{c-onset}$ , phase lock-in temperature, $T_{c_j}$ , and Josephson current, $I_0$ for Y-123 and co-addition of 5.0 mol.% $Y_2O_3 + x$ mol.% of BZO samples in AC field of 0.5 Oe	102
5.17	The cross-section area of sample, peak temperature, $T_p$ and the intergranular critical current density, $J_{cm}$ for Y-123 with co-addition of 5.0 mol.% $Y_2O_3 + x$ mol.% of BZO nanoparticles in AC field of 0.5 Oe	104

## LIST OF FIGURES

Figure	Page
3.1 The relation between resistances with temperature at the superconducting state	17
3.2 Magnetic levitation phenomenon on superconducting bulk material in liquid nitrogen bath (Meissner effect)	18
3.3 The sequence events of magnetic behavior of a “perfect” conductor (a)-(b) specimens becomes resistanceless in the absence of field (c) magnetic field applied to resistanceless specimen (e)-(f) specimen becomes resistanceless in applied magnetic field (d)-(g) applied magnetic field removed	20
3.4 The sequence events of magnetic behavior of a superconductor (a)-(b) specimens becomes resistanceless in the absence of field (c) magnetic field applied to superconducting specimen (e)-(f) specimen becomes superconducting in applied magnetic field (d)-(g) applied magnetic field removed	21
3.5 Schematic diagram of magnetic behavior for type-I superconductor (a) a plot of internal field versus applied magnetic field (b) a plot of magnetization versus applied magnetic field	24
3.6 Schematic diagram of magnetic behavior for type-II superconductor (a) a plot of internal field versus applied magnetic field (b) a plot of magnetization versus applied magnetic field	25
3.7 Schematic diagram of the mixed state. The dark regions are vortices in type-II superconductor	27
3.8 Schematic diagrams of vortex in type-II superconductor carrying current through mixed state. The Lorentz force, $F_L$ on a magnetic flux is perpendicular both to the axes of the tubes and to the transport current, $J$	28
3.9 The formation of Cooper pair	30
3.10 The movement of electrons in a one-dimensional lattice	31
3.11 The crystal structure of (a) $\text{YBaCu}_3\text{O}_6$ (b) $\text{YBaCu}_3\text{O}_{6+\delta}$	32
3.12 Schematic structure of $\text{YBaCu}_3\text{O}_{6+\delta}$	33
4.1 The flow diagram of the COP method for sample preparation	36

4.2	Yttrium (III) acetate tetrahydrate was being heated for 30 minutes at 80 °C	38
4.3	A mixture of metal acetate and acetic acid was being heated for 2 hours at 80 °C (Solution (A))	38
4.4	A mixture of oxalic acid and distilled water: 2-propanol (1: 1.5) (Solution (B))	38
4.5	Both solutions were cooled in the ice bath until the temperature dropped to below 10 °C	39
4.6	The mixture of solution (A) and solution (B) after being cooled in the ice bath	39
4.7	The mixture solution underwent the filtration process	40
4.8	The residue was left for 12 hours at 100 °C	40
4.9	The blue precursor powder after drying (12 hours)	41
4.10	Heating and cooling profile for 24 hours calcination process	43
4.11	Heating and cooling profile for 15 hours of sintering (red circle) and 8 hours of annealing (blue circle) processes under oxygen flow	44
4.12	A schematic diagram of the thermogravimetric instrument	46
4.13	Scattered X-rays by crystal lattice	47
4.14	The schematic diagram of a scanning electron microscope (SEM)	49
4.15	Schematic drawing of the four-point probe	50
4.16	(a) Graph of normalized resistance against temperature (b) Graph of derivative ( $dR/dT$ ) against temperature	51
4.17	Schematic diagram of an AC Susceptometer (ACS)	52
4.18	Temperature dependence of the ACS	53
5.1	TGA thermograms of Y-123 blue precursors prepared by the co-precipitation method	56
5.2	X-ray diffraction patterns of Y-123 + $x$ mol.% of BZO nanoparticles samples	59
5.3	The XRD patterns of the peak corresponding to the (0 1 3) and (1 0 3) planes of $YBa_2Cu_3O_{7-\delta}$ in the Y-123 + $x$ mol.% of BZO samples	60

5.4	The XRD patterns of the peak corresponding to the (0 1 1) plane of BaZrO <sub>3</sub> in the Y-123 + <i>x</i> mol.% of BZO samples	60
5.5	Lattice parameter <i>a</i> -, <i>b</i> - and <i>c</i> -axis and unit cell volume for Y-123 samples with addition of (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% (F) 7.0 mol.% BZO nanoparticle. Solid lines are given as guides to the eye only	62
5.6	Crystallite size (Scherrer and Williamson-Hall) and lattice strain variations for Y-123 samples with addition of (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% (F) 7.0 mol.% BZO nanoparticles. Solid lines are given as guides to the eye only	64
5.7	SEM images of Y-123 fractured surfaces samples added with (a) 0.0 mol.% (b) 1.0 mol.% (c) 2.0 mol.% (d) 3.0 mol.% (e) 5.0 mol.% (f) 7.0 mol.% of BZO nanoparticle at 5000× magnification. The distribution of grain size is shown on the right hand side of the images	66
5.8	Average grain size distribution of Y-123 samples with addition of (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% and (F) 7.0 mol.% BZO nanoparticles	68
5.9	The difference spectrum taken from the SEM images (left hand side) and the EDX spectra obtained from the spectrum (right hand side) for (a) 0.0 mol.% (b) 1.0 mol.% (c) 2.0 mol.% (d) 3.0 mol.% (e) 5.0 mol.% and (f) 7.0 mol.% BZO nanoparticles	70
5.10	Normalized resistance versus temperature of Y-123 + <i>x</i> mol.% of BZO samples	73
5.11	Derivative of resistance $\delta R / \delta T$ against temperature of the Y-123 + <i>x</i> mol.% of BZO samples	73
5.12	Comparison of $T_{c-onset}$ and $T_{c-zero}$ for Y-123 samples with addition of (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% (F) 7.0 mol.% BZO nanoparticles. Solid lines are given as guides to the eye only	75
5.13	Hole concentration versus $T_{c-onset}$ for Y-123 samples with addition of (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% (F) 7.0 mol.% BZO nanoparticles	76
5.14	Temperature dependence of the ACS for the Y123 + <i>x</i> mol.% of BZO samples in AC field of 0.5 Oe	77

5.15	Variation of the onset temperature, $T_{c-onset}$ , phase lock-in temperature, $T_{cj}$ , and Josephson current, $I_0$ for Y-123 samples with (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% (F) 7.0 mol.% BZO nanoparticles addition. Solid lines are given as guides to the eye only	79
5.16	The variation of peak temperature, $T_p$ and intergranular critical current density, $J_{cm}$ for Y-123 samples with (A) 0.0 mol.% (B) 1.0 mol.% (C) 2.0 mol.% (D) 3.0 mol.% (E) 5.0 mol.% (F) 7.0 mol.% BZO nanoparticle in AC field of 0.5 Oe. Solid lines are given as guides to the eye only	80
5.17	XRD patterns of the pure Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ and + x mol.% of BZO samples	82
5.18	The XRD patterns of the peak corresponding to the (0 1 3) and (1 0 3) planes of $YBa_2Cu_3O_{7-\delta}$ in the Y-123 samples with co-addition of 5.0 mol.% $Y_2O_3$ + x mol.% of BZO samples	83
5.19	The XRD patterns of the peak corresponding to the (0 1 1) plane of $BaZrO_3$ in the Y-123 samples with co-addition of 5.0 mol.% $Y_2O_3$ + x mol.% of BZO samples	83
5.20	Lattice parameter $a$ -, $b$ - and $c$ -axis and unit cell volume for samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ and (B) 0.0 mol.% BZO (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% (G) 7.0 mol.% BZO nanoparticles. Solid lines are given as guides to the eye only	85
5.21	Crystallite size (Scherrer and Williamson-Hall) and lattice strain variations for samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ and (B) 0.0 mol.% (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% and (G) 7.0 mol.% BZO nanoparticle. Solid lines are given as guides to the eye only	87
5.22	SEM images for fractured surfaces of samples (a) Y-123; Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ and (b) 0.0 mol.% (c) 1.0 mol.% (d) 2.0 mol.% (e) 3.0 mol.% (f) 5.0 mol.% and (g) 7.0 mol.% of BZO nanoparticle at 5000 $\times$ magnification. The distribution of grain size is shown on the right hand side of the image	89
5.23	Average grain size distribution of samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% $Y_2O_3$ and (B) 0.0 mol.% (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% and (G) 7.0 mol.% BZO nanoparticles	92

5.24	The difference spectrum taken from the SEM images (left hand side) and the EDX spectra obtained from the spectrum (right hand side) for (a) Y-123; Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> and (b) 0.0 mol.% (c) 1.0 mol.% (d) 2.0 mol.% (e) 3.0 mol.% (f) 5.0 mol.% and (g) 7.0 mol.% BZO nanoparticles	93
5.25	Normalized resistance versus temperature of Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> and + x mol.% of BZO samples	96
5.26	Derivative of resistance $\delta R / \delta T$ against temperature of the Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> + x mol.% of BZO samples	97
5.27	Comparison of $T_{c-onset}$ and $T_{c-zero}$ for samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> and (B) 0.0 mol.% (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% (G) 7.0 mol.% BZO nanoparticles. Solid lines are given as guides to the eye only	98
5.28	Hole concentration versus $T_{c-onset}$ of samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> and (B) 0.0 mol.% (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% (G) 7.0 mol.% BZO nanoparticles	99
5.29	Temperature dependence of the ACS for the Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> + x mol.% of BZO samples in AC field of 0.5 Oe	100
5.30	Variation of the onset temperature, $T_{c-onset}$ , phase lock-in temperature, $T_{c,l}$ , and Josephson current, $I_o$ for samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> and (B) 0.0 mol.% (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% (G) 7.0 mol.% BZO nanoparticle addition. Solid lines are given as guides to the eye only	103
5.31	The variation of the peak temperature, $T_p$ and intergranular critical current density, $J_{cm}$ for samples (A) Y-123; Y-123 with co-addition of 5.0 mol.% Y <sub>2</sub> O <sub>3</sub> and (B) 0.0 mol.% (C) 1.0 mol.% (D) 2.0 mol.% (E) 3.0 mol.% (F) 5.0 mol.% (G) 7.0 mol.% BZO nanoparticle in AC field of 0.5 Oe. Solid lines are given as guides to the eye only	104

## LIST OF SYMBOLS AND ABBREVIATIONS

ACS	Alternating current susceptibility
$\theta$	Angle of diffraction
Å	Angstrom (Unit of length equal to $10^{-10}$ m)
$H_{ac}$	Applied magnetic field
a.u.	Arbitrary unit
BaZrO <sub>3</sub> (BZO)	Barium Zirconate
BaCO <sub>3</sub>	Barium Carbonate
BaO	Barium Oxide
BSCCO	Bismuth Strontium Calcium Copper Oxide
$\xi$	Coherence length
CuO	Copper (II) Oxide
CuO <sub>2</sub>	Copper (II) Peroxide
COP	Co-precipitation
$T_p$	Coupling peak temperature
$J_c$	Critical current density
$T_c$	Critical temperature
DSC	Differential scanning calorimetry
EDX	Energy-dispersive X-ray spectroscopy
Bi <sub>2</sub> Sr <sub>2</sub> Ca <sub>2</sub> Cu <sub>3</sub> O <sub>10</sub> (Bi-2223)	Family member in BSCCO
YBa <sub>2</sub> Cu <sub>3</sub> O <sub>7-<math>\delta</math></sub> (Y-123)	Family member in YBCO
Y <sub>2</sub> BaCuO <sub>5</sub> (Y-211)	Family member in YBCO
FWHM	Full width at half maximum
$\kappa$	Ginzburg-Landau constant
HTS	High temperature superconductor
$p$	Hole concentration
$d$	Interatomic spacing
$J_{cm}$	Intergranular critical current density
ICSD	Inorganic Crystal Structure Database
$\chi'$	In-phase (real) susceptibility
$I_0$	Josephson's current
K	Kelvin (Standard unit of temperature)
$a$ -, $b$ -, $c$ -axis	Lattice parameter
LTS	Low temperature superconductor
$F_L$	Lorentz force
$\Phi_0$	Magnetic flux
$T_c^{\max}$	Maximum critical temperature
mol.%	Molar percentage
Oe	Oersted (Unit of magnetic field)
rpm	Rotation per minute
$T_{c-onset}$	Onset critical temperature
$\chi''$	Out-of-phase (imaginary) susceptibility
$T_{cj}$	Phase lock-in temperature
$F_p$	Pinning force
rpm	Revolutions per minute
SEM	Scanning electron microscope
SQUIDS	Superconducting Quantum Interference Devices
T	Tesla (Standard unit of magnetic flux density)



TGA	Thermogravimetry Analysis
$\Delta T_c$	Transition temperature width
TEM	Transmission Electron Microscopy
$J$	Transport current
wt. %	Weight percentage
XRD	X-Ray Diffractometer
$\lambda$	X-ray wavelength
YBCO	Yttrium Barium Copper Oxide
$Y_2O_3$	Yttrium Oxide
$T_{c-zero}$	Zero critical temperature



# CHAPTER 1

## INTRODUCTION

### 1.1 Background

A superconductor is a material that has a zero resistance when cooled below its critical temperature,  $T_c$ . The critical temperature or the transition temperature is the temperature at which a superconductor loses its resistance. Resistance causes the energy flows through the material to lose. All metals and alloy materials have a resistance and the resistance decreases at lower temperature due to the decrease in thermal vibrations of the atoms resulting in less scattering of conduction electrons. However, superconducting materials would suddenly lose all trace of electrical resistance once they are cooled into the superconducting state below the  $T_c$  (Rose-Innes and Rhoderick, 1978).

They are two distinctive properties to consider a material as a superconductor. First, no resistance is observed in a superconductor below its  $T_c$  value and superconductivity will disappear if the current passed is higher than the critical current density,  $J_c$ . Secondly, no magnetic induction,  $B = 0$  inside the superconductor in weak external magnetic field when cooled below its  $T_c$  value (Cyrot and Pavuna, 1992).

The phenomenon of superconductivity was observed for the first time in 1911 when Heike Kamerlingh Onnes cooled mercury to the temperature of liquid helium, 4 K and he found that the resistance of mercury disappeared (Eck, 1999). Over the years, it was believed that superconductivity could only occur when the materials were held at very low temperature (low temperature superconductor, LTS). Nevertheless, in 1986, the discovery of cuprates marked the beginning of superconductivity at higher temperatures (high temperature superconductor, HTS) hence provides a new prospective in the superconducting area (Khare, 2003).

### 1.2 History of High Temperature Superconductor (HTS)

High-temperature superconductors (HTS) are materials that superconduct at higher temperatures (above 30 K) and can be cooled to superconducting state using liquid nitrogen (77 K). The first HTS was discovered in late 1986, when Alex Müller and Georg Bednorz synthesized a brittle ceramic compound (La-Ba-Cu-O compounds) that was shown to be superconducting at the temperature above 30 K. However, the critical temperature of La-Ba-Cu-O compound was increased above 40 K when a pressure was

applied (Chu et al., 1987). One interesting part was that scientists had never thought of ceramic could be HTS candidates because ceramic is normally insulating and cannot conduct electricity (Eck, 1999).

Since then, researchers began to study every combination of ceramics to get higher  $T_c$ . However, in January of 1987, a research team from University of Alabama-Huntsville achieved 92 K of  $T_c$ , when they substituted yttrium for lanthanum 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 (Wu et al., 1987; Cyrot and Pavuna, 1992). Later, in 1988, Maeda and co-workers at the National Research Institute, Japan discovered Bi-cuprate oxides ( $Bi_2Sr_2Ca_2Cu_3O_{10}$ ) with  $T_c$  value at 110 K (Maeda et al, 1988) and Tl-cuprate oxides ( $Tl_2Sr_2Ca_2Cu_3O_{10}$ ) was discovered by Sheng and Hermann with  $T_c$  value at 125 K (Sheng and Hermann, 1988).

The search for new HTS materials with higher  $T_c$  reached the climax when the mercury based HTS compounds were discovered. The highest temperature superconductor was found in the three-layer system of  $HgBa_2Ca_2Cu_3O_{8+\delta}$  (Hg-1223) with  $T_c$  value at 133.5 K. In January 2008, a research group led by Hideo Hosono in Japan discovered a new class of high-temperature superconductors (non-Cu-based superconductors) in layered iron arsenic compounds with  $T_c$  of 26 K. It was found that the parent compound,  $LaOFeAs$  became a superconducting upon replacing some of the oxygen with fluorine and this is known as “pnictides” (compounds of the nitrogen group) (Kamihara et al., 2008). In 2015, the highest  $T_c$  value at 203 K was found in hydrogen sulfide ( $H_2S$ ) under extremely high pressure around 150 GPa (Drozdov et al., 2015).

### 1.3 Application of HTS

Superconductors have made a significant breakthrough in technological applications, especially in transportation, electronic, medical and superconducting energy storage system. A few of these applications are discussed as follow:

#### 1.3.1 Maglev Train

In 1934, the train based on superconductivity was proposed and known as the Maglev train. Maglev stood for magnetic levitation. Maglev train is moved when the magnetic fields created by electrified coils within the track walls that propel the train. These strong superconducting magnets eliminate the friction between the trains and its tracks. Due to the frictionless properties, it allows the train to travel over long distances at speeds of hundreds of miles per hour (Galiano, 2011; Liu et al., 2015). The principle used for maglev trains to levitate is “Meissner effect” that will be explained later.

### 1.3.2 SQUID

Superconducting quantum interference device or SQUID magnetometer have been used to measure and recording small magnetic signals from the brain, heart, stomach and other organs. Besides, magnetic field sensors based on SQUIDs also has been widely used in geophysics for measuring the magnetic field oscillations of the earth and in some other fields (Wiksw, 1995).

### 1.4 Problem Statement and Research Objectives

Over the years, scientists had studied high temperature superconductor (HTS) extensively. HTS is very useful for industries because of their special properties which are zero resistance and perfect diamagnetism. However, there are some limitations of HTS application. For example, it only can behave as a superconductor below the room temperature which is above the  $T_c$  value. Therefore, a lot of studies had been done to improve the critical temperature,  $T_c$  and the critical current density,  $J_c$  (Klie et al., 2005; Horvath et al., 2008; Barnes et al., 2009). With this improvement, it can save lots of cost and energy since the current can be transported without any loss of energy. But, it is challenging to improve both the  $T_c$  and  $J_c$  value.

In this work, the addition of BaZrO<sub>3</sub> (BZO) nanoparticles and the co-addition of BZO nanoparticles and Y<sub>2</sub>O<sub>3</sub> into Y-123 were studied. It is believed that both the addition and co-addition into Y-123 samples will improve the  $J_c$  without affecting much the  $T_c$  value. Previous study of bulk Y-123 composite with BZO by applying hot isostatic pressing (HIP) for sintering claimed that the nanoparticles size of BZO (30 - 50 nm) gave a good dispersion of pinning centres in the matrix and the introducing of BZO enhanced the  $J_c$  value up to 20000 A/ cm<sup>2</sup> (Awano et al., 2000). It was supported by Jin et al. (2015), when a smooth surface and a good texture have been observed in the YBCO + 5.0 mol.% BZO film, which exhibited the best  $J_c$  value (4.9 MA/ cm<sup>2</sup>) for the YBCO + 5.0 mol.% BZO film at 77 K and 0 T. Although the  $J_c$  value was increased, it did not affect much the  $T_c$  values of these films that can be obtained around ~ 90 K. Ding et al. (2012), claimed that the co-addition of 7.0 mol.% BZO and 7.0 mol.% Y<sub>2</sub>O<sub>3</sub> into Y-123 films showed a higher self-field  $J_c$  value of 6.5 mA/ cm<sup>2</sup> compared to the pure Y-123 film as 3.2 mA/cm<sup>2</sup>. Besides, the  $T_c$  value of Y-123 film with co-addition of 7.0 mol.% BZO and 7.0 mol.% of Y<sub>2</sub>O<sub>3</sub> was ~ 90 K, a value which is closed to that of the pure Y-123.

The method used was co-precipitation (COP) which is being used widely in the industry compared to solid-state method (Ochsenkuhn-Petropoulou et al., 2002; Cardwell and Ginley, 2003). The samples from COP method can be produced at large scale for various applications such as fly wheel motor, transmission power cable, wires, tapes and magnets. Until now a few conventional materials, like NbTi or Nb<sub>3</sub>Sn have been commercialized (Wang, 2013). The wires made of HTS oxides such as YBCO and BSCCO are still under development. Since the previous studies had focused more on the

thin films (Haugan et al., 2007; Ciontea et al., 2008), therefore this work focused more on bulk superconductor.

Hence, the objectives of this work are:

- i. To prepare high purity of Y-123 precursor by using co-precipitation method.
- ii. To study the influence of nanoparticles addition, BZO on structural, magnetic and electrical properties of Y-123 bulk superconductor.
- iii. To investigate the effects of co-addition of BZO nanoparticles and  $Y_2O_3$  on structural, magnetic and electrical properties of Y-123 bulk superconductor.

## 1.5 Thesis Overview

Basically, this thesis is consist of six chapters. The first chapter is about introduction of superconductor and the history of high temperature superconductor. Then, the applications of superconductor, the problem statement and the objectives of this work are given here too. Chapter 2 reviews some previous studies of Y-123 superconductor especially the study involved the co-precipitation method, the addition of BZO nanoparticles and the co-addition of BZO and  $Y_2O_3$ . Chapter 3 explains the theory and fundamental of superconductivity. The phenomena of superconductivity, classification of superconductors, properties of superconductors, vortex state, and the Cooper pair's formation are discussed here. Then, chapter 4 discusses the materials and method used for this work. Discussion in this chapter also includes sample characterization such as TGA, XRD, SEM, temperature dependence of electrical resistance measurement and AC Susceptibility. In chapter 5, the results obtained from all the samples are analyzed and discussed. Lastly, chapter 6 concludes the results for this work and the recommendation for future research is given.

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## LIST OF PUBLICATIONS

### Publication in Index Journal

Mohd Hapipi, N., Shaari, A. H., Awang Kechik, M. M., Tan, K. B., Abd-Shukor, R., Mohd Suib, N. R., and Chen, S. K. (2017). Effect of heat treatment condition on the phase formation of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-8}$  superconductor. *Solid State Phenomena*, 268, 305–310.

### Journal and Abstracts in conference proceedings

Nurhidayah Mohd Hapipi, Nursyafiqah Ismail, Cheong Choon Min, Abdul Halim Shaari, Mohd Mustafa Awang Kechik, Lim Kean Pah and Chen Soo Kien. "Comparative study of phase formation of  $\text{YBa}_2\text{Cu}_4\text{O}_8$  prepared via solid state and co-precipitation method", Programme and Abstract Book for 5<sup>th</sup> International Conference on Solid State Science and Technology 2015, 13-15 December 2015.



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