UNIVERSITI PUTRA MALAYSIA

INFLUENCE OF NbO_2 AND SnO_2 ADDITIONS ON THE PROPERTIES OF $YBa_2Cu_3O_{7-\delta}$ BULK SUPERCONDUCTOR SYNTHESISED VIA THERMAL TREATMENT METHOD

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FS 2018 53

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By

NUR NABILAH MOHD YUSUF

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

April 2018

DEDICATION

For my parents whose love for me beyond anything the world can measure

For my brothers who are always there to keep me 'sane'

For the true friends who have really supported me through thick and thin

For those people who have never gave up on me

For my fellow researchers in the pursuit of the same dream

And for my supervisor, the very backbone of my thesis writing.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Master of Science

INFLUENCE OF NbO_2 AND SnO_2 ADDITIONS ON THE PROPERTIES OF $YBa_2Cu_3O_{7-\delta}$ BULK SUPERCONDUCTOR SYNTHESISED VIA THERMAL TREATMENT METHOD

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Chairman : Mohd Mustafa Awang Kechik, PhD
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$YBa_2Cu_3O_{7-\delta}$ bulk sample had been synthesized using thermal treatment method. The microstructure and superconducting properties of the sample with the addition of NbO_2 and SnO_2 were studied. Both additions improve the resistance-temperature, *R-T* properties with smaller superconducting transition, ΔT compared to pure sample. While alternating current susceptibility, ACS measurement showed degradation of intergranular coupling with enhanced intragranular pinning strength in NbO_2 added sample, enhanced coupling is observed in SnO_2 added sample. Among the added sample, maximum Josephson current, I_o is found to be the highest in 0.8 wt% NbO_2 (131.44 μA) and 0.4 wt% SnO_2 (664.42 μA) compared to pure sample (146.05 μA). X-ray diffraction analysis revealed that NbO_2 added sample leads to the formation of YBa_2NbO_6 phase while SnO_2 addition reduces the Y211 secondary phase in the Y123 sample. Y123 phase is found to enhanced with SnO_2 addition up to 95.8 % (0.4 wt%) compared to 91.5% in pure sample. The scanning electron microscope, SEM images showed an increase in grain size as both addition increases. Sponge-like grain structures is observed in NbO_2 and is randomly distributed across the sample pertained to YBa_2NbO_6 phase while SnO_2 addition causes Sn to precipitate on the surface sample and agglomerates at the grain boundaries. Here, SnO_2 addition seems to have the upper hand as the overall current transport in high T_c is governed by the intergranular coupling where weak links is associated.

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

PENGARUH NbO_2 DAN SnO_2 KE ATAS CIRI-CIRI KESUPERKONDUKSIAN BAGI SUPERKONDUKTOR $YBa_2Cu_3O_{7-\delta}$ PUKAL

Oleh

NUR NABILAH MOHD YUSUF

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Sampel pukal $YBa_2Cu_3O_{7-\delta}$ telah disintesis melalui kaedah rawatan termal. Perubahan sifat elektrik dan mikrostruktur sampel tersebut dengan penambahan NbO_2 dan SnO_2 telah dikaji. Kedua-dua jenis penambahan telah menunjukkan kemajuan dalam sifat suhu-rintangan (*R-T*) dengan pengecilan julat suhu peralihan kesuperkonduksian, ΔT berbanding sampel asal. Bagi sampel berpenambahan NbO_2, kerentanan semasa alternative, ACS telah menunjukkan kemerosotan dalam pengepinan antara butiran dan menguatkan pengepinan dalam butiran. Penambahan SnO_2 pula menguatkan lagi pengepinan antara butiran. Nilai arus kritikal Josephson, I_o dalam kalangan sampel berpenambahan memberikan nilai tertinggi pada sampel 0.8 wt% NbO_2 (131.44 μA) dan 0.4 wt% SnO_2 (664.42 μA) berbanding sampel asal, (146.05 μA). Analisis difraksi X-ray mendedahkan bahawa penambahan NbO_2 membentuk fasa baru, YBa_2NbO_6 manakala penambahan SnO_2 dapat mengurangkan fasa sekunder Y211 dalam sampel. Fasa major Y123 pula didapati meningkat dengan adanya penambahan SnO_2 sehingga 95.8 % (0.4 wt%) berbanding 91.5% dalam sampel asal. Imej mikroskop pengimbasan elektron, SEM mempamerkan pembesaran saiz butiran seiring pertambahan berat peratusan NbO_2 dan SnO_2. Penambahan NbO_2 membawa kepada pertumbuhan berbentuk spong dek fasa YBa_2NbO_6. Pertambahan berat peratusan SnO_2 menyebabkan penepuan Sn berlaku lalu membentuk aglomerasi Sn di sempadan butiran pada permukaan sampel. Memandangkan arus superkonduktor lebih dipengaruhi oleh sifat ikatan yang lemah dan pengepinan di antara butiran berbanding dalam butiran, maka SnO_2 dilihat mempunyai kelebihan berbanding NbO_2.

ACKNOWLEDGEMENTS

Alhamdulillah, praise be to Allah, the Most Beneficent and the Most Merciful for all the courage, patience, and strength given to me throughout the completion of this thesis. My sincerest gratitude for Dr. Mohd Mustafa Awang Kechik, Associate Prof. Chen Soo Kien, Professor Dr. Abdul Halim Shaari and Associate Prof. Dr. Lim Kean Pah for their endless guidance throughout my master studies.

Special thanks to Dato' Prof. Dr. Roslan Abdul Shukor from UKM, for allowing the characterisation process performed in his laboratory, not forgetting his student, Raihan, Akasyah, Aizat and Ilham for all the help given during the intensive days. My gratitude is also extended to my research group members and seniors for their contributions towards the completion of this research study.

Federal scholarship from *Jabatan Perkhidmatan Awam* (JPA) under *Program Mahasiswa Cemerlang* and Grant Research Fellowship, UPM are also greatly acknowledged.

I certify that a Thesis Examination Committee has met on 2 April 2018 to conduct the final examination of Nur Nabilah binti Mohd Yusuf on her thesis entitled "Influence of NbO_2 and SnO_2 Additions on the Properties of $YBa_2Cu_3O_{7-\delta}$ Bulk Superconductor Synthesised via 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 Master of Science.

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LIST OF ABBREVIATIONS

ΔT_c	Transition of critical temperature
A	Ampere
Å	Angstrom
a,b,c	Lattice parameters
AC	Alternating current
Ba	Barium
$BaCO_3$	Barium carbonate
B_c	Critical field
B_{c1}	Lower critical field
B_{c2}	Upper critical field
BCS	Bardeen-Cooper-Schrieffer Theory
CO_2	Carbon dioxide
COP	Co-precipitation
Cu	Copper
DSC	Derivative Scanning Calorimetric
DTA	Derivative Thermal Analysis
DTG	Derivative Thermogravimetric Analysis
E	Electric field
EDX	Energy Dispersive X-ray Analysis
F	Force
FWHM	Full width half maximum
H_{c1}	Lower critical field
H_{c2}	Upper critical field
hrs	Hours
HTS	High Temperature Superconductor

I	Current
I_o	Critical Josephson current
ICDD	International Centre for Diffraction Data
J	Critical current
J_c	Critical current density
J_{cm}	Critical current density calculated by means of magnetization
K	Kelvin
M	Magnetisation
min	Minutes
Nb	Niobium
NbO_2	Niobium oxide
NO_2	Nitrogen oxide
O	Oxygen
PAAc	Polyacrylic Acid
PVP	Polyvinyl Pyrrolidone
R	Resistance
rpm	Rotation per minute
R-T	Resistance-temperature measurement
SEM	Scanning electron microscope
Sn	Tin
SnO_2	Tin Oxide
T'	Peak temperature for secondary phase
T_c	Critical temperature
T_{cj}	Phase lock-in temperature
$T_{c\text{-offset}}$	Temperature at zero resistance
$T_{c\text{-onset}}$	Starting temperature for superconductivity to occur
TGA	Thermogravimetric Analysis

T_p	Intergranular temperature
T_{pm}	Intragranular temperature
V	Voltage
wt %	Weight percentage
x	Parameter for weight percentage
XRD	X-ray Diffraction
Y	Yttrium
Y123	$YBa_2Cu_3O_{7-\delta}$
Y124	$YBa_2Cu_4O_{\delta}$
δ	Oxygen deficiency
κ	Ginzburg-Landau constant
λ_l	London penetration depth
μ_0	Permeability
ξ	Coherence length
χ’	Real part of ACS
χ”	Imaginary Part of ACS

CHAPTER 1

INTRODUCTION

1.1 Introduction to superconductor and its potentials

In order to fight the enormous challenge of climate change and annual depletion of fossil fuels, renewable energy is our alternative solution. Unlike oil and gas, renewable energies are generated by natural sources such as sunlight and wind which are constantly replenish. However, reducing our dependence on oil and gas is only part of the solution. We need to conserve energy and improve the energy efficiency of our electrical system.

Despite the growing demand for electrical energy, the electrical system we have today is incredibly inefficient especially during the transport of electricity from one source to another. Although copper are good conductors for electrical wiring, approximately 60 to 80 percent of the energy that goes into the system is lost and wasted mostly to heat and sound during the transportation. Thus, further investment, research and development are required to find better material that has better efficiency to meet the ever increasing energy demand (Kaygusuz, 2012).

This is where superconductor comes in handy. Unlike conventional conductors, superconductor has the ability to conduct electricity with zero resistance. As such, there will be no loses in the form of energy. This phenomenon of superconductivity however can only be achieved upon cooling the material below its transition temperature, T_c in which the material loses its resistance (Khare, 2003). In other words, superconducting materials act like perfect conductors but with better striking properties such as perfect diamagnetism (Tinkham, 1996).

The absence of resistance in the superconductor allows the current to flow without measurable decay with time hence, perfect conductivity. Perfect conductivity successfully explains the exclusion of external magnetic field from entering the superconductor (Tinkham, 1996). However, later it appears that not only the external field but, the field which is originally inside a superconductor itself is expelled too when cooled below its T_c. This is known as perfect diamagnetism (Ginzburg & Andryushin, 2004). These properties are the prerequisite for most potential electrical applications, such as high-current transmission lines or high-field magnets (Sarrao et al., 2006).

In electrical power devices, the ability of superconductors to sustain higher current densities and lower losses enables it to have higher specific power and efficiency compared to conventional conductors. Superconductors are also able to generate large magnetic flux density with values that are constant in time. They can trap magnetic flux and have strong diamagnetic response to changes in magnetic flux with nonlinear relationship between current density and electric field near the superconducting transition (Sarrao et al., 2006).

1.2 Material aspects of superconductor

While it is desirable to replace the wires in conventional electrical power devices or electromagnets with superconducting wires, the high cost of cooling makes it unrealistic for widespread use. This is due to the fact that only at extremely cold temperature would the material possess superconductivity. However, there is an alternative to superconducting wires that provides the same benefits. It is 'bulk superconductors', a generic class of material that includes large single-grain blocks and sintered structures (Hull & Murakami, 2004).

To this day, many researches have been done to find materials that can sustain superconductivity at room temperature. One of the promising materials with excellent ability to carry high superconducting critical current in high magnetic field despite having a lower T_c of 92 K is $YB_2C_3O_{7-\delta}$ (Kechik, 2010). To date, the feasibility of YBCO to be manufactured in a large scale makes it one of the most studied high temperature superconductor (HTS) in the industry. A more detailed account regarding the properties of this material is given in section 2.3.

The fact that the properties of YBCO or any other superconducting material can be enhanced by introducing defects or altering the parameter during the synthesis technique makes the study about the subject matter still on going. Never the less, the researches' aim is always to get higher T_c superconductor with enhanced critical current density, J_c for application wise.

1.3 Applications of superconductors

Due to its ability to have higher power and efficiency in conducting current, more and more instruments as well as electrical devices today have started to employ superconductor in its system. Magnetic Resonance Imaging (MRI) is one of the commonly used machines in hospitals that employs superconductor in its system.

A superconducting MRI magnet is an electromagnet made of niobium-titanium superconducting wire. Once current is caused to flow in the coil, it will continue to flow as long as the coil is kept immersed in liquid helium during the MRI scan. Although some losses do occur over time due to infinitely small resistance in the coil, these losses are usually only a few parts per million of the main magnetic field per year (Coyne, 2017)

Trains that employ superconductor have also been faster and more efficient. Magnetic Levitation Train, MAGLEV, has been widely used in the Europe and even China with the idea of levitating the train on top of the railway so that it moves without friction. The speed and direction can be fully controlled by the superconducting magnets which are made by coils of superconducting wires immersed in liquid nitrogen.

1.4 Today's challenge in superconductor research

Though many efforts have been made to further understand the phenomenon of a superconductor, there are still more questions than answers regarding this remarkable behaviour. Through the experience of other researches, it is rather known that in most cases, critical temperature, T_c of a superconductor does not have a linear relationship with its critical current density, J_c. Most of them showed that while trying to increase T_c, J_c would be slowly diminished and vice versa. While the aim is to find superconducting materials with high T_c to operate at higher temperature, having larger J_c is also crucial for practical industrial use. Thus, until today, endless researches have been made to find any possible way to elevate T_c or improve J_c of superconductors if not both.

Although we know that introducing defects by adding impurities in a superconductor can act as flux pinning centre, there is not quite a handbook of which element would or would not work on improvising the properties of a superconductor. Different result could also be obtained simply by altering the temperatures and the holding time during the heat treatment process, what more using different synthesis methods.

To date, thermal treatment method has been widely used to produce nano magnetic material, but it is yet to be implemented in the synthesis of a superconductor. Although this method is expected to yield nano materials, in terms of $YBa_2Cu_3O_{7-\delta}$, micro-size grains is expected due to its high sintering temperature (Dihom et al., 2017). Compared to other wet methods, thermal treatment uses less material and has less by-product effluents. Hence, for a more environmental friendly route, it is one of the aim of this study to implement this method in the synthesis of $YBa_2Cu_3O_{7-\delta}$.

Various elements and compounds such as Ca, K, Zr, Al_2O_3, $BaZrO_3$, Nd_2O_3 and CNT have been introduced in the $YBa_2Cu_3O_{7-\delta}$ matrix and had successfully enhance either J_c or T_c of the material (Dihom et al., 2017; Guner et al., 2012; Jha & Khare, 2010; Khalid et al., 2018; Ramli et al., 2016; Yeoh et al., 2009). However, less study has been made for the addition of SnO_2 and NbO_2 in $YBa_2Cu_3O_{7-\delta}$. Up to now, the effects of NbO_2 and SnO_2 addition in $YBa_2Cu_3O_{7-\delta}$ compound synthesised via thermal treatment method has not yet been reported.

1.5 Objectives

In this study, $YBa_2Cu_3O_{7-\delta}$ or Y123 is prepared via new method called thermal treatment method which will be further discussed in Chapter 3 with additions of NbO_2 and SnO_2 powder in between the heat treatments. The objectives are as stated below:

1) To synthesise Y123 bulk superconductor via thermal treatment method.

2) To study the influence of NbO_2 and SnO_2 addition towards the morphology of Y123 bulk sample.

3) To investigate the effects of NbO_2 and SnO_2 addition towards the superconducting properties of Y123.

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