UNIVERSITI PUTRA MALAYSIA

PHYSICAL AND SUPERCONDUCTING PROPERTIES OF CARBON NANOFIBERS AND CARBON NANOTUBES YBa•CuŽǑ– / SUPERCONDUCTOR

NURUL AUNI BINTI KHALID

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PHYSICAL AND SUPERCONDUCTING PROPERTIES OF CARBON NANOFIBERS AND CARBON NANOTUBES $YBa_2Cu_3O_{7-\delta}$ SUPERCONDUCTOR

By

NURUL AUNI BINTI KHALID

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

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

PHYSICAL AND SUPERCONDUCTING PROPERTIES OF CARBON NANOFIBERS AND CARBON NANOTUBES $YBa_2Cu_3O_{7-\delta}$ SUPERCONDUCTOR

By

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November 2016

Chairman: Mohd Mustafa bin Awang Kechik, PhD
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A co-precipitation (COP) process for the synthesis of ultrafine $YBa_2Cu_3O_{7-\delta}$ (Y-123) precursor has been developed with the addition of carbon nanofibers (CNFs) and carbon nanotubes (CNTs) nanoparticles with varying amounts of weight percentage (wt. %). Both nanoparticles acted as pinning center to improve the transport critical current density (J_c). The objective of this study was to investigate the flux pinning capability from carbon sources of CNFs and CNTs in $YBa_2Cu_3O_{7-\delta}$ superconductor. The effects of CNFs and CNTs addition on the superconducting properties (T_c and J_c), phase formation and structural properties, microstructure and surface morphology including the elemental distribution analysis were studied. A non-added Y-123 was used as a reference. The J_c for this sample is 11 A/cm^2. Introduction of CNFs and CNTs improved J_c significantly. Furthermore, the volume fraction of Y-123 phase was 96.2 % and EDX showed excellent atomic ratio. CNFs addition significantly enhanced the J_c of Y-123 bulk. The x = 0.4 wt. % sample showed the optimal amount as it exhibited the highest T_c and J_c of 88.0 K and 830 A/cm^2, respectively. CNTs addition showed J_c value of 477 A/cm^2 for the x = 0.2 wt. % sample which gave the optimal amount of CNTs addition. The largest grain size and high percentage of Y-123 phase was also observed in this sample together with good atomic % from EDX and high T_c value. The enhancement of J_c using CNFs and CNTs as artificial pinning centers from this work indicated that these two compounds are suitable for flux trapping. Both samples show increased values of T_c and J_c towards the optimum wt. % of addition and decreased after further addition of dopants.

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

FIZIKAL DAN CIRI-CIRI KESUPERKONDUKSIAN BAGI NANOFIBER KARBON DAN NANOTIUB KARBON SUPERKONDUKTOR $YBa_2Cu_3O_{7-\delta}$

Oleh

NURUL AUNI BINTI KHALID

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Proses sepemendakan untuk mensintesis serbuk halus pelopor $YBa_2Cu_3O_{7-\delta}$ (Y-123) telah disediakan dengan tambahan nanofiber karbon (CNFs) dan nanotiub karbon (CNTs) dengan jumlah peratusan berat (wt.%) yang berlainan. Kedua-dua nanozarah bertindak sebagai pengepin fluks untuk meningkatkan pengangkutan ketumpatan arus genting (J_c). Objektif kajian ini ialah untuk meningkatkan pengepinan fluks daripada sumber karbon CNFs dan CNTs dalam superkonduktor $YBa_2Cu_3O_{7-\delta}$. Kesan CNFs dan CNTs terhadap sifat-sifat superkonduktor (T_c dan J_c), fasa dan struktur, mikrostruktur dan morfologi termasuk analisis taburan unsur telah dikaji. Y-123 pukal tanpa tambah disediakan sebagai rujukan. J_c Y-123 tanpa penambahan ialah 11 A/cm^2. Penambahan CNFs dan CNTs telah meningkatkan J_c. Pecahan isipadu fasa Y-123 menunjukkan peratusan yang tinggi 90 > dengan peratus atom yang baik dari EDX. Penambahan CNFs menunjukkan peningkatan ketara nilai J_c. Sampel dengan tambahan bendasing x = 0.4 wt. % menunjukkan jumlah yang optimum kerana ia mempamerkan T_c dan J_c tertinggi dengan masing-masing bersamaan dengan 88.0 K dan 830 A/cm^2. Penambahan CNTs meningkatkan J_c kepada 477 A/cm^2 bagi sampel x = 0.2 wt. %. Saiz butiran terbesar dan peratusan fasa Y-123 yang tinggi juga dikesan melalui sampel ini dengan peratusan atom yang baik daripada EDX dan nilai T_c yang tinggi. Peningkatan J_c menggunakan CNFs dan CNTs sebagai pusat pengepin menunjukkan kedua-dua sebatian sesuai untuk memerangkap fluks. Kedua-dua sampel menunjukkan pertambahan dalam nilai T_c dan J_c ke arah nilai berat peratusan (wt. %) yang optimum dan penurunan berlaku apabila nilai kuantiti bendasing bertambah.

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I certify that a Thesis Examination Committee has met on (01 November 2016) to conduct the final examination of (Nurul Auni binti Khalid) on her thesis entitled "Physical and Superconducting Properties of Carbon Nanofibers and Carbon Nanotubes $YBa_2Cu_3O_{7-\delta}$ Superconductor" 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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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:

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LIST OF ABBREVIATIONS

A	Ampere
Å	Angstrom
BCS Theory	Bardeen-Cooper-Schrieffer Theory
$BaCO_3$	Barium carbonate
cm^2	Centimetre square
CNFs	Carbon nanofibers
CNTs	Carbon nanotubes
COP	Co-precipitation
EDX	Energy Dispersive X-ray Analysis
FESEM	Field Emission Scanning Electron Microscope
H_{c1}	Lower critical field
H_{c2}	Upper critical field
HTS	High Temperature Superconductor
ICDD	International Centre for Diffraction Data
J_c	Critical current density
K	Kelvin
nm	Nanometre
SEM	Scanning Electron Microscope
T_c	Critical temperature
TGA	Thermogravimetric analysis
wt. %	Weight percentage
XRD	X-ray Diffraction

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Y-123	$YBa_2Cu_3O_{7-\delta}$
Y-124	$YBa_2Cu_4O_8$
δ	Oxygen deficiency
ξ	Coherence length
λ	Wavelength, Penetration depth

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CHAPTER 1

INTRODUCTION

1.1 Overview

Superconductors are of completely different orders, such strange and mysterious materials as it becomes a perfect conductor of electricity after being chilled to unimaginably cold temperatures. In the year of 1911, a physicist unexpectedly noticed that in certain materials, an electric current could flow without removing any heat and it is resistance-less. Mercury was the first superconducting element which exhibited a dramatically drop in resistivity (Dahal, 2011;Kirkpatrick, 2010). Following the year of the first discovery, a number of superconducting materials have been discovered such as lead, aluminium, titanium and tin which superconduct when they were cooled below a critical temperature, T_c (Abd-Shukor, 2004). Those materials are classified as conventional superconductors which are made up of metals and alloys in which the T_c is normally less than 24 Kelvin, until the discovery of MgB_2 in 2001 with the highest T_c of 39 K.

Paul Chu is not a conventional thinker and he wasn't interested in doing the conventional things. By the year of 1986, Paul Chu and his research team discovered the exotic superconducting mixture of yttrium, barium, copper and oxygen (YBCO), with a superconducting transition temperature of 93 K (Hazen, 1989;Blundell, 2009). It was a revolutionary discovery of YBCO which was named as Y-123 with the chemical formula of $YBa_2Cu_3O_{7-\delta}$, as it was the first ever material with T_c above the boiling point of liquid nitrogen (77 K). This simply means that superconductors would no longer require a complex cooling system for their applications, unlike other materials that need liquid helium in their operation which is rather costly. Y-123 superconductor gave huge impacts to the market penetration for power applications (Hawsey and Christen, 2006) due to its high T_c. However, the weak flux pinning and granularity of Y-123 especially at temperatures above 20-30 K seems to have small critical current density, J_c (Dadras, 2009).

In recent years, various synthetic strategies have been employed concerning the pinning center properties in superconducting system (Berdan Özkurt, 2013). A technique of doping and addition of impurities including metal and non-metal elements, nanoparticles, carbon sources and other compounds have shown the dependent on superconducting properties. The influence of the type and amount of chemical compound and impurities can be used to improve both inter-granular and intra-granular critical current density, J_c in different high temperature superconductors (Dadras, 2009). Moreover, the requirement of high performance YBCO bulks in current science and technological applications indicates that doping of YBCO samples with another suitable compound with a right proportions would be a reliable method in order to

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achieve highly qualified complex structures (Tepe *et al*., 2014)by improving its grain boundary superconducting properties (Jian Yong Xiang, 2008).

Many successful groups claim that the improvement of J_c in high temperature superconductor can be obtained by the introduction of nanoparticle addition, which act as artificial pinning centers (Sushant Gupta, 2011;R. Mawassi, 2014;Abd-Ghani, 2012). The effective artificial pinning centers will further enhance a strong J_c at higher fields and higher temperature, which can be achieved when the flux lines are pinned and automatically, their movement will be prevented (Kechik, 2010). In high temperature superconductor (HTS), the effects on the vortex movements, determination of J_c, and other superconducting properties are depending on the engineering artificial pinning centers, over the pinning of flux line through crystal imperfections or so-called defects (Pathak *et al*., 2008) such as low-angle grain boundaries, twin boundaries, dislocations and non-superconducting phase (Dang, 2010;Zhou *et al*., 2002). The route of artificially introducing inhomogeneity or second-phase materials as flux pinning sites in the processing of bulk YBCO is a long standing issue (Hamrita *et al*., 2014).

Nano-phase particles in superconductor links can improve the intra-granular critical current density under high magnetic field (Dadras, 2009). On the other hand, due to the effects of intra-granular, the enhancement of the inter-granular electrical links can have an effective role in the increase of J_c, since the existence of electrically coupled grains can increase inter-granular current flow. Carbon precursor’s sources like carbon nanofibers, carbon nanotubes and graphene are particularly interesting due to their nanometer diameter that can serve as the effective pinning centers. In this project, carbon nanofibers (CNFs) and carbon nanotubes (CNTs) are chosen as the impurities to the addition of Y-123.

Carbon nanofibers consist of multi-graphene sheets rolled into concentric cylinders. There are hollow core nanofibers consisting of a single graphite layer or double graphite layers that are stacked parallel or at a certain angle from the fiber axis (Al-Saleh and Sundararaj, 2011). The stacked layers are nested with each other and have different structures including bamboo-like, parallel and cup-stacked. CNFs have excellent mechanical properties, high electrical conductivity and high thermal conductivity, which can be imparted into a wide range of matrices including ceramics and metals. It is very well known that CNFs have a higher tendency to form agglomerates because of their high surface area (1000 m^2/g). Carbon nanotubes (CNTs) which act as the carbon precursors usually result in some improvements in upper critical field (H_{c2}) and critical current density (J_c). In addition, CNTs also show some individuality for their high aspect ratio and nanometer width. CNTs and CNFs is a graphitic filament with diameters ranging from 0.4 to 500 nm and lengths in the range of several micrometers to millimetres (Dadras, 2009). Moreover, this kind of carbon does not decay, however it may turn into a part of the crystal matrix as a whole, where they are performed as one of the effective pinning centers (Shekhar *et al*., 2007).

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1.2 Problem Statement

Over the years, many researches have been carried out to investigate the superconducting properties of high temperature superconducting (HTS). High transition temperatures (T_c) and current density (J_c) value play important roles in the practical application of YBCO. However, there is insufficient and low J_c in YBCO samples because of the serious weak links and granularity. This research project proposes to prepare a good quality of pure YBCO-123 via co-precipitation method with view in having the possibility of controlled particle size and morphology. A nanoparticle of CNFs and CNTs are also added to YBCO. Up until now, the effects of CNFs and CNTs adding or doping on Y-123 compound via co-precipitation (COP) method have yet to be reported.

1.3 Objectives

The main objectives of this project are to synthesise and prepare $YBa_2Cu_3O_{7-\delta}$ (Y-123) by co-precipitation method. Other purpose is to study the effects of the addition of nano-phase particles from carbon sources into superconductor matrix, which act as artificial pinning center. So, the observation on every possible role played by CNFs and CNTs addition in improving the flux pinning properties of Y-123 compound is studied. The parameters in term of weight percentage (wt. %) is varied with pure sample (0 wt. %) and added samples (0.2 wt. %, 0.4 wt. %, 0.6 wt. % and 0.8 wt. %). Thus, the test and study is carried out through:-

i) The determination of their superconducting properties such as the trend of critical temperature, T_c and critical current density, J_c.

ii) The determination of their physical properties such as the phase formation and surface morphology.

1.4 Thesis Contents

Chapter 1 sees a brief introduction on overview of superconductors about YBCO, strategies regarding the pinning center properties, introduction of nanoparticles addition and carbon family (CNFs and CNTs), follows by problem statement and objectives. In Chapter 2, the discussion is focused on the literature reviews and theories which support the project. Some literatures about YBCO system with nanoparticle inclusions have also been reviewed. Meanwhile, the theory of fundamentals in superconductivity is studied based on the discovery, Type I and Type II superconductors as well as the flux pinning and the coherence length. In Chapter 3, the discussion will be on the advantages of the methodology with sample preparation, follows by the characterizations in superconducting and physical properties. In Chapter 4, the experimental data and results obtained as well as the discussions are presented. Finally in the last chapter, Chapter 5, overall explanations and discussions are concluded with the additional suggestions for future research.

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