INFLUENCE OF CNT, TiO_2 AND SiC NANOSTRUCTURE ADDITIONS ON MICROSTRUCTURAL AND SUPERCONDUCTING PROPERTIES OF $YBa_2Cu_3O_{7-\delta}$ SYNTHESIZED USING THERMAL TREATMENT

By

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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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October 2023

Chair : Associate Professor Mohd Mustafa Awang Kechik, PhD
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Yttrium Barium Copper Oxide $YBa_2Cu_3O_{7-\delta}$ (Y123) has emerged as one of the most promising high-temperature superconductors due to its potential applications in various fields. However, in high magnetic fields, Y123 applications are limited by weak links at grain boundaries, leading to low J_c. In this research, different weight percentages, x = (0.0, 0.2, 0.4, 0.6, 0.8, and 1.0 wt.%) of CNT (2 nm), TiO_2 (6 - 15 nm) and SiC (15 nm) nanostructures were added to Y123 bulk prepared using a thermal treatment method. The primary objective is to investigate how the incorporation of these nanostructures influences the structure and superconducting properties of Y123. The samples were characterised using various analytical techniques, including thermogravimetric analysis (TGA), X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) along with EDX, alternating current susceptibility (ACS), four-point probe technique (4PP) and electron-spin resonance (ESR). The XRD analysis revealed that the samples predominantly consisted of the Y123 phase, which exhibited an orthorhombic crystal structure. Additionally, the Y211 phase was detected in all additional samples as a minor phase. FESEM observations showed that the incorporation of CNT, TiO_2 and SiC nanostructures yielded more homogeneous specimens and improved inter-grain connectivity. EDX spectra performed for all Y123 samples showed the presence of Y, Ba, Cu and O elements with a precise match for the standard peak position for these elements. From ACS measurement, the presence of CNT enhanced onset critical temperature ($T_{c\text{-onset}}$) at x = 0.4 wt.% CNT. Samples with the addition of TiO_2 and SiC showed a slight decrease in $T_{c\text{-onset}}$, while T_{cj} greatly increased except for the higher contents of SiC. The temperature-dependent resistivity measurements, ρ-T confirmed the occurrence of superconductivity in all samples. However, samples at x = 0.8 and 1.0 wt.% SiC showed semiconducting like behaviour. $T_{c\text{-onset}}$ was observed at 95.6 K for a pure sample, and slightly decreased with the addition of nanostructures due to oxygen modification. The value of J_c as measured by current-voltage measurement was 2.08 A/cm^2 for the pure sample and increased to 8.1 A/cm^2 for the sample with x = 0.4 wt.% CNT, indicating enhanced grain connectivity. The ESR spectra for all samples displayed a symmetrical signal behaviour. Based on the results obtained, it can be concluded that

the incorporation of CNT, TiO_2 and SiC nanostructures into Y123 superconductors offers a viable route to enhance the superconducting properties with the best result achieved at x = 0.4 wt.%.CNT.in term of $T_{c\text{-}onset}$, and J_c.

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

PENGARUH PENAMBAHAN NANOSTRUKTUR CNT, TiO_2 DAN SiC TERHADAP SIFAT MIKROSTRUKTUR DAN MENSUPERKONDUKSI $YBa_2Cu_3O_{7-\delta}$ YANG DISINTESIS MENGGUNAKAN RAWATAN TERMA

Oleh

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Yttrium Barium Copper Oxide $YBa_2Cu_3O_{7-\delta}$ (Y123) telah muncul sebagai salah satu superkonduktor suhu tinggi yang paling menjanjikan kerana aplikasinya yang berpotensi dalam pelbagai bidang. Walau bagaimanapun, dalam medan magnet yang tinggi, aplikasi Y123 dihadkan oleh pautan lemah pada sempadan butiran, yang membawa kepada J_c yang rendah. Dalam penyelidikan ini, peratusan berat yang berbeza, x = (0.0, 0.2, 0.4, 0.6, 0.8, dan 1.0 wt.%) CNT (2 nm), TiO_2 (6 - 15 nm) dan SiC (15 nm) telah ditambah kepada Y123 pukal yang disediakan menggunakan kaedah rawatan terma. Objektif utama adalah untuk menyiasat bagaimana penggabungan struktur nano ini mempengaruhi struktur dan sifat mensuperkonduksi Y123. Sampel dicirikan menggunakan pelbagai teknik analisis, termasuk analisis termogravimetri (TGA), belauan sinar-X (XRD), mikroskop elektron pengimbasan pelepasan medan (FESEM) bersama-sama dengan EDX, kerentanan arus ulang-alik (ACS), teknik prob empat titik (4PP) dan resonans putaran elektron (ESR). Analisis XRD mendedahkan bahawa sampel kebanyakannya terdiri daripada fasa Y123, yang mempamerkan struktur kristal ortorombik. Selain itu, fasa Y211 dikesan dalam semua sampel tambahan sebagai fasa minor. Pemerhatian FESEM menunjukkan bahawa penggabungan struktur nano CNT, TiO_2 dan SiC menghasilkan lebih banyak spesimen homogen dan meningkatkan ketersambungan antara butiran. Spektrum EDX yang dilakukan untuk semua sampel Y123 menunjukkan kehadiran unsur Y, Ba, Cu dan O dengan padanan yang tepat bagi kedudukan puncak piawai untuk unsur-unsur ini. Daripada pengukuran ACS, kehadiran CNT telah meningkatkan suhu kritikal permulaan CNT ($T_{c\text{-}onset}$) pada x = 0.4 wt.% CNT. Sampel dengan penambahan TiO_2 dan SiC menunjukkan sedikit penurunan dalam $T_{c\text{-}onset}$, manakala T_{cj} meningkat dengan banyak kecuali bagi kandungan SiC yang lebih tinggi. Pengukuran kerintangan kebergantungan suhu, ρ-T mengesahkan berlakunya superkonduktiviti dalam semua sampel. Walau bagaimanapun, sampel pada x = 0.8 dan 1.0 wt.% SiC menunjukkan tingkah laku seperti semikonduktor. $T_{c\text{-}onset}$ diperhatikan pada 95.6 K untuk sampel tulen, dan sedikit berkurangan dengan penambahan struktur nano akibat pengubahsuaian oksigen. Nilai J_c seperti yang diukur dengan pengukuran voltan semasa ialah 2.08 A/cm^2 untuk sampel tulen dan meningkat kepada 8.1 A/cm^2 untuk

sampel dengan x = 0.4 wt.% CNT, menunjukkan peningkatan ketersambungan butiran. Spektrum ESR untuk semua sampel menunjukkan tingkah laku isyarat simetri. Berdasarkan keputusan yang diperoleh, dapat disimpulkan bahawa penggabungan struktur nano CNT, TiO_2 dan SiC ke dalam superkonduktor Y123 menawarkan laluan yang berdaya maju untuk meningkatkan sifat superkonduktor dengan hasil terbaik dicapai pada x = 0.4 wt.% CNT dalam istilah $T_{c\text{-onset}}$, dan J_c.

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LIST OF ABBREVIATIONS

A	Ampere
Å	Angstrom
AC	Alternating current
ACS	Alternating Current Susceptibility
a, *b, c* axis	Lattice parameters
B	Magnetic field
BCS	(Bardeen-Cooper-Schrieffer) Theory
BSCCO	Bismuth lead strontium calcium copper oxide
°C	Degree Celsius
CNT	Carbon nanotube
DC	Direct current
d_p	Crystallite size
DTG	Derivative Thermogravimetric Analysis
EDX	Energy dispersive x-ray Analysis
ESR	Electron-spin resonance
FESEM	Field emission scanning electron microscopy
F_L	Lorentz force
f_s	Free energy in the normal state
FWHM	Full width half maximum
G-L	Ginzburg-Landau theory
h	Hour
H_{ac}	Applied field
H_{c1}	Lower critical field
H_{c2}	Upper critical field
HTSC	High temperature superconductor
Hz	Hertz
I	Current

I_0	Critical Josephson current
ICDD	International Centre for Diffraction Data
J_c	Critical current density
J_{cm}	Intergranular critical current density
K	Kelvin
La-Ba-Cu-O	lanthanum barium copper oxides
LTS	low temperature superconductor
MgB_2	Magnesium diboride
min	Minute
mm	Millimetre
nm	Nanometre
Oe	Oersted
P	Hole concentration
PVP	Polyvinyl pyrrolidone
R	Resistance
rpm	Rotation per minute
sec	Second
SiC	Silicon carbide
SQUID	Superconducting quantum interference device
T^*	Pseudogap temperature
TBCCO	Thallium barium calcium copper oxide
T_c	Critical temperature
T_{cj}	Phase lock-in temperature
$T_{c\text{-onset}}$	Onset critical temperature
$T_{c\text{-zero}}$	Transition temperature at zero resistivity
TGA	Thermogravimetric Analysis
TiO_2	Titanium Oxide
T_p	Coupling peak temperature
T_{pm}	Intergranular peak temperature
T_{pg}	Intragranular peak temperature

V	Voltage
wt.%	Weight percentage
XRD	X-ray diffraction
$YBa_2Cu_3O_{7-\delta}$ (YBCO, Y123)	Yttrium barium copper oxide
Y211	Y_2BaCuO_5
Y124	$YBa_2Cu_4O_8$
Y247	$Y_2Ba_4Cu_7O_{15}$
ΔT_c	Transition width
ρ	Electrical resistivity
ρ_0	Residual resistivity
ρ-T	Resistivity-temperature measurement
χ'	Real part of ACS
χ''	Imaginary Part of ACS
ψ	Order parameter
Ƙ	Ginzburg-Landau constant
ϕ	Phase of the order parameter
Φ_0	Quantum of flux
λ	Penetration depth
ξ	Coherence length
δ	Oxygen deficiency
θ	Angle of the diffracted
Ω	Ohm
μm	Micrometre

CHAPTER 1

INTRODUCTION

1.1 Superconducting Phenomenon: its Discovery and Evolution

The phenomenon of superconductivity was first discovered in 1911 by H. Kamerlingh Onnes and Gilles Holst, who found that at a temperature below 4.2 K, the dc resistivity of mercury abruptly drops to zero. At this point, the transition temperature, T_c, between the normal and superconducting states is the first quantity used to characterise a superconducting material. This temperature is also known as the critical temperature. Walther Meissner and Robert Ochsenfeld discovered in 1933 that a magnetic field is expelled from the inside of a material cooled down below its critical temperature when superconductors are exposed to an external magnetic field (Forrest, 1983). Thus, no applied magnetic field is permitted inside when metal becomes superconducting. This phenomenon is one of the most fundamental properties of superconductors and is referred to as the Meissner effect.

In 1934, Dutch scientists C. J. Gorter and H. B. G. Casimir proposed a phenomenological explanation of superconductivity based on the idea that the conducting electron "fluid" has two components: normal and superconducting in the superconducting state. Electrons with normal properties are identical to the electron system in a normal metal, whereas superconducting electrons are the cause of the anomalous features. In 1935, following the discovery of the Meissner effect, the brothers F. and H. London introduced two equations to explain the diamagnetism of superconductors in a weak external field. The London equations defined the first characteristic length of superconductivity, which is now referred to as the London penetration depth L. The London equations, along with the Maxwell equations, explained the behaviour of superconducting electrons within the framework of the two-fluid model. In contrast, the Maxwell equations only described the behaviour of normal electrons.

The presence of two critical magnetic fields for type-II superconductors and the mixed state was discovered in 1937 by L. V. Shubnikov and co-workers. Actually, they discovered vortices in superconductors. The isotope effect in superconductors was found by E. Maxwell and C. A. Reynolds in 1950. This finding was essential to establishing the correct theory of superconductivity. The phenomenological theory of superconductivity that uses the second-order phase transition theory was proposed by Ginzburg and Landau in the same year, 1950. The Ginzburg-Landau theory was able to explain how superconductors, both conventional and unconventional, react when subjected to intense magnetic fields. The Ginzburg-Landau theory offered an expression for the coherence length, which is the second characteristic length of superconductivity.

Cooper, (1956) showed that if two conducting experience an attractive interaction on the Fermi surface, they will form a stable paired state. A pair like these is known as a Cooper

pair. Based on these findings, J. Bardeen, L. Cooper, and R. Schrieffer (Abrikosov, 1957) developed the first microscopic theory in 1957, which is now widely known as the BCS theory. Following BCS, B. D. Josephson predicted research on tunnelling effects between two bulk superconductors separated by an insulating barrier (a few nanometres thick) (Josephson, 1962). Within a year, the predictions of Josephson were confirmed, and they became known as the Josephson effects.

1.2 High-Temperature Superconductivity (HTSC): A history

The history of high-temperature superconductivity started in late 1986, when George Bednorz and Karl Müller observed evidence for superconductivity in lanthanum barium copper oxides (La-Ba-Cu-O) ceramics at temperatures up to 38 K (Bednorz et al., 1986). In 1987, scientists at the Universities of Alabama and Houston, led by Wu and Chu, jointly announced the finding of the 93 K superconductor in yttrium barium copper oxide (Y-Ba-Cu-O) (Wu et al., 1987). The focus then turned to copper oxides, and shortly after, the compounds BSCCO with T_c = 105 K and TBCCO with T_c = 125 K were discovered in 1988 (Maeda et al., 1988; Sheng et al., 1988). In 1993, the highest critical temperature, T_c = 135 K for Hg-based cuprates ($HgBa_2Ca_2Cu_3O_x$) was observed (Chu et al., 1993). The T_c for the mercury compounds can reach 164 K at high pressure (Sheng et al., 1988).

Figure 1.1 shows the evolution of the transition temperature, T_c, of different cuprate superconductors. The figure includes the metallic compound MgB_2 and the iron group. Meanwhile, the MgB_2 compound was discovered in 2000 with a transition temperature of 39 K (Nagamatsu et al., 2001). The physics community was highly active as a result of this discovery, and significant characteristics of this substance were determined over the next two years. Comparable to the "classical" metallic superconductors, MgB_2 exhibits similar characteristics. In 2008, an iron pnictide with transition temperatures as high as 56 K was discovered (Takahashi et al., 2008).

The mechanism by which high-temperature superconductors achieve their properties is still the topic of discussion. The data collected in the cuprates within a year of their superconducting discovery showed that the features of high-T_c superconductors varied from those predicted by the BCS theory. The BCS isotope effect, for example, is almost non-existent in optimally doped cuprates. This has resulted in the development of the mechanisms of non-phonon electronic coupling. Within the past several years, bulk HTSC that have large critical currents, high trapped fields, and powerful levitation forces have been developed. This development in bulk HTSC growth was particularly noticeable for the $YBa_2Cu_3O_{7-\delta}$ or (Y123) compound, which plays a major role in applications of superconducting materials. The Y123 is one of the most promising high-T_c superconductors for applications at 77 K due to its relatively small anisotropy, which enables large critical current densities even when applied magnetic fields are present. The highest T_c value for $YBa_2Cu_3O_{7-\delta}$ is 93 K at $\delta = 0.1$. It has been found that if Y is substituted by another rare-earth element, such as Nd, Eu, Gd, Er, Sm, Dy, Tm, or Ho, the resulting T_c is essentially unaffected.

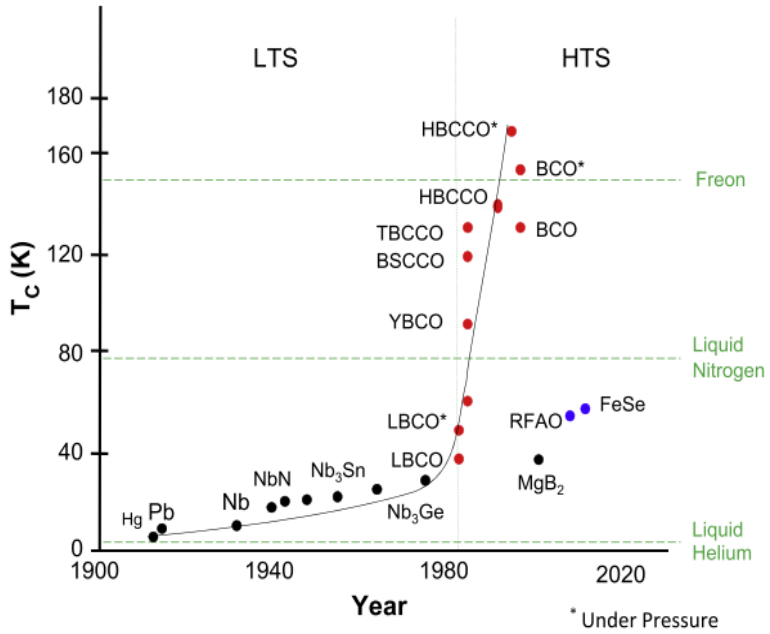

Figure 1.1: Evolution of the superconducting transition temperature as a function of the year of discovery (Chu et al., 2015)

1.3 Applications of High-T_c Superconductors

As soon as high-T_c superconductors were discovered, it was realised that significant economic savings would be possible using liquid nitrogen rather than liquid helium (low temperature superconductor LTS such as NbTi and Nb_3Sn). HTSC will contribute significantly to energy savings and greenhouse gas reductions when novel designs of power components and systems become available. The commercial applications of HTSC became increasingly fascinating after their discovery. Indeed, superconductor wire's electrical efficiency and great power density result in powerful and highly compact devices that are more dependable, efficient, and environmentally friendly. Hence, their applications include various areas such as medical, transportation, electrical grids, and industry. The most advanced HTSC used in power systems are power transmission cables. In fact, 2001 has proven to be a pivotal year for HTSC cable prototypes, with important pre-commercial demonstrations taking place on many continents, some of which are supplying electricity for commercial and residential usage (Hassenzahl, 2001; Kelley et al., 2001).

The diamagnetic property of bulk HTSC superconductors has been employed to produce levitators and bearings (Hull, 2000). Because bulk HTSC can effectively trap magnetic fields, they are worth seriously considering for usage in power generation. Bulk HTSC have been used to generate a trapped field of 17 T at 29 K, with the value being restricted by the strength of the energising magnet (Tomita & Murakami, 2003). The development

of HTSC motors started soon after they were discovered (Driscoll et al., 2000). This effort led to the creation of experimental devices with ever-growing sizes. Bulk HTSC motors have lately been studied as analogues to reluctance (Oswald et al., 1999) and hysteresis (Kovalev et al., 1999) permanent-magnet motors. For generator applications, it is expected that HTSC generators will make it possible to compete at 100 MVA and higher because of the greater temperature cooling related to HTSC (Rabinowitz, 2000). Superconducting bulk YBCO has been employed as a bearing system in a flywheel device to store power with a capacity of about 1 kW/h (Hull, 1997). Another application for bulk YBCO is water cleaning by magnetic separation technology (Hayashi et al., 2003). The high critical temperature and high current density of YBCO thin films make it ideal for use in small superconducting devices.

1.4 Problem Statement

The promise of superconductivity to revolutionise power applications has been there since its discovery. The interest in using superconductors in power technology was renewed by the discovery of a material that could superconduct at temperatures higher than the boiling point of liquid nitrogen. Materials with a high critical current density, J_c, and minimal power losses are crucial for bulk applications.

The major problem that has stood in the way of using Y123 bulk superconductors in technological applications is that these materials tend to have a granular consistency. This characteristic leads to weak flux pinning at the boundaries of the grains, which results in lowering J_c, causing the materials to lose its superconducting properties at high magnetic fields. (Bishop et al., 1992; Larbalestier, 1996; Tanaka, 2006). It is necessary to find a way to strength the weak flux pinning of Y123 materials to make it suitable for high magnetic field applications.

The beneficial way to enhance the weak flux pinning and superconducting properties with stabilising T_c is by introducing suitable impurities within the structure of the superconducting system. In addition, to ensure the effectiveness of the pinning centres, the size of the pinning should be on the order of the coherence length (distance over which the two electrons in the Cooper pair can bond) (Hari Babu et al., 2006; Yang & Wang, 2013). The presence of nano-sized impurity phases as defects in Y123 superconductor can act as artificial pinning centers to enhance its superconducting properties. (Lian et al., 1990; Ochsenkühn-Petropoulou et al., 2002; Rodriguez & Lazo, 2018; Wei et al., 2016; Xu et al., 2012; Xu et al., 2002). Hence, the aim of this research is to study the addition of CNT, TiO_2, and SiC nanostructures to Y123 via the thermal treatment method and investigate their potential to enhance weak flux pining and superconducting performance. While various studies have investigated Y123 superconductors' performance enhancement through different synthesis methods, none have applied the thermal treatment method to introduce CNT, TiO_2, and SiC into the Y123 matrix. The current challenge lies in the lack of reported studies employing this synthesis method to introduce these specific nanostructures into Y123. It is expected that the structure and related superconducting properties of the Y123 superconductor will be enhanced when an adequate amount of impurity is introduced to the Y123 system using thermal treatment method. The role of the incorporation of different amounts of CNT,

TiO_2, and SiC into the microstructure and superconducting properties of Y123 bulk was thoroughly investigated and discussed.

1.5 Research Objectives

The following is a summary of the objectives for this research:

1. To synthesis a good quality of Y123 bulk superconductor using the thermal treatment method with PVP as a capping agent and determine its structure and superconducting properties.

2. To study the effect of the distribution of different percentages of CNT, TiO_2 and SiC nano powders on phase formation, microstructural and morphology of Y123 bulk.

3. To investigate the influence of CNT, TiO_2 and SiC nano powders addition on the electrical, magnetic, and superconducting properties of Y123 bulk.

1.6 Outline of the Thesis

The thesis is organised into six chapters, as follows: In Chapter 1, significant historical events connected to the phenomenon of superconductivity are reviewed. This chapter also presents a historical overview of high temperature superconductivity and its applications. The problem statement, objectives of the research, and outline of the thesis are given in this chapter. Chapter 2 explains the basic theories of superconductivity and discusses the fundamental properties of superconductors. The following topics are covered in this chapter: The BCS, zero electrical resistivity, the Meissner-Ochsenfeld effect, the Josephson effects, quantisation of magnetic flux, critical magnetic field, critical current density, penetration depth, and coherence length, type-I and type-II superconductors, flux pining mechanism in type I1 superconductors, the mixed state, the vortex state, grain boundary problem, grain alignment, the pseudogap and variation of T_c with oxygen stoichiometry. Chapter 3 gives an overview of the literature review on the Y123 superconductor prepared by the thermal treatment method and the effect of the addition of CNT, TiO_2, and SiC nano powder on the Y123 system. The preparation method and sample characterisation techniques are presented in Chapter 4. The following chapter, Chapter 5, went over the results and discussed crystal structure, morphological, elemental distribution, AC susceptibility measurement, electrical resistivity and ESR spectra analysis. Finally, the outcomes that can be derived from this study are summarised in Chapter 6.

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