UNIVERSITI PUTRA MALAYSIA

EFFECT OF GRAPHENE NANOPARTICLE ADDITION IN YBa_2Cu_3Oy USING THERMAL TREATMENT METHOD AND FABRICATION OF YBa_2Cu_3Oy WITH VARIOUS LIQUID PHASE RATIOS VIA INFILTRATION GROWTH

ALIAH NURSYAHIRAH BINTI KAMARUDIN

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By

ALIAH NURSYAHIRAH BINTI KAMARUDIN

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

May 2021

DEDICATION

I dedicate this work to my beloved parents who have been a great source of inspiration and encouraged me to go on every adventure especially in this master journey

KAMARUDIN BIN BUJANG
ZARIDAH BINTI MD SHAH

and to my siblings

Thank you for all your love and support

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

EFFECT OF GRAPHENE NANOPARTICLE ADDITION IN $YBa_2Cu_3O_y$ USING THERMAL TREATMENT METHOD AND FABRICATION OF $YBa_2Cu_3O_y$ WITH VARIOUS LIQUID PHASE RATIOS VIA INFILTRATION GROWTH

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In this study, the superconducting properties of $YBa_2Cu_3O_{7-\delta}$ (Y123) were studied using the thermal treatment method and the infiltration growth (IG) method. The synthesis of Y123 with graphene nanoparticles addition was prepared by the thermal treatment method using polyvinyl pyrrolidone (PVP) as a capping agent. Meanwhile, utilizing the liquid phase source of Y123 superconductors was studied by varying the Y123:$ErBa_2Cu_3O_y$ (Er123) ratios fabricated by the IG method. The microstructure and superconducting properties of Y123 were investigated by thermogravimetric analysis (TGA), X-ray diffraction (XRD), field-emission scanning electron microscope (FESEM), energy dispersive X-ray diffraction (EDX), alternating current susceptibility (ACS), and SQUID magnetometer. It was found that all samples were indexed with a P*mmm* orthorhombic crystal structure with Y123 as the major phase followed by a secondary phase of Y211. The increasing weight percentage up to 97.1 % of Y123 major phase in sample x = 0.3 wt. % added with graphene nanoparticles showed that this impurity was capable of enhancing the Y123 phase purity. The addition of graphene nanoparticles promoted into closely packed and denser, thus giving rise to the better grain growth of Y123 with less porosity. From the AC susceptibility, the highest $T_{c\text{-onset}}$ obtained in sample x = 1.0 wt. %, followed by x = 0.5 wt. % with 92.64 K and 92.59 K, respectively. The improvement of the $J_{cm,}$ at sample x = 1.0 wt. %, 25.94 A/cm^2 was observed. It showed that graphene nanoparticles acted as pinning centers, improving the $J_{cm.}$ In the IG method, the formation of smaller Y211 particles in IG samples with uniform distributions was also found to act as an effective pinning center. Hence, the critical current density, J_c of the sample Y_1Er_0 at 77 K with H//*c*-axis had the highest J_c 54.15 kA/cm^2 and 11.45 kA/cm^2 at self-field and 2 T, respectively. In conclusion, the existence of graphene nanoparticles and uniform distribution of the Y211 secondary phase plays an important role as an optimum flux pinning center to obtain good bulk of Y123 superconductors.

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

KESAN-KESAN PENAMBAHAN NANOPARTIKEL GRAPHENE DALAM $YBa_2Cu_3O_y$ MENGGUNAKAN KAEDAH TERMAL DAN FABRIKASI $YBa_2Cu_3O_y$ DENGAN PELBAGAI NISBAH FASA CECAIR MENGGUNAKAN KAEDAH PERTUMBUHAN PENYUSUPAN

Oleh

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Dalam kajian ini, sifat-sifat superkonduktor $YBa_2Cu_3O_y$ (Y123) telah disediakan dengan menggunakan kaedah rawatan termal dan kaedah pertumbuhan penyusupan (IG). Sampel Y123 dengan penambahan nanopartikel graphene disediakan menggunakan kaedah rawatan termal menggunakan *polyvinyl pyrrolidone* (PVP) sebagai agen pembatas. Sementara itu, penggunaan sumber fasa cecair superkonduktor Y123 disediakan mengikut nisbah Y123: $ErBa_2Cu_3O_y$ (Er123) disediakan menggunakan kaedah IG. Mikrostuktur dan sifat-sifat superkonduktor Y123 dikaji menggunakan analisis termogravimetrik (TGA), pembelauan sinar-X (XRD), mikroskop elektron pengimbas (FESEM), spektroskopi tenaga sinar-X (EDX), kerentanan arus ulang-alik (ACS), dan magnetometer SQUID. Semua sampel diindekskan dengan struktur hablur ortorombik P*mmm* dengan Y123 sebagai fasa utama diikuti oleh Y211 sebagai fasa sekunder. Sampel yang disediakan menggunakan kaedah termal menunjukkan penambahbaikkan peratus sehingga 97.1 % bagi fasa Y123 untuk sampel x = 0.3 wt. % menunjukkan bahawa dapat meningkatakan fasa penulenan Y123. Penambahan nanopartikel graphene menunjukkaan sampel mempunyai pertumbuhan yang rapat dan padat bagi menggalakan pertumbuhan butiran Y123 yang lebih baik dengan keliangan yang lebih sedikit. Pengukuran ACS menunjukkan T_c tertinggi diperoleh bgai sampel x = 1.0 wt. %, diikuti sampel x = 0.5 wt. % dengan 92.64 K dan 92.59 K, masing-masing. Peningkatan J_{cm} pada sampel x = 1.0 wt. %, 25.94 A/cm^2 menunjukkan bahawa nanopartikel graphene bertindak sebagai pusat penyematan dan meningkatkan J_{cm}. Dalam kaedah IG, pembentukan butiran Y211 yang lebih kecil dan seragam bertindak sebagai pusat pengepinan yang berkesan. Oleh itu, ia menyebakan nilai J_c bagi sampel Y_1Er_0 pada 77 K dengan paksi H//c mempunyai J_c tertinggi pada 54.15 kA/cm^2 dan 11.45 kA/cm^2 pada medan kendiri dan 2 T, masing-masing. Kesimpulannya, kewujudan nanopartikel graphene dan penyebaran seragam fasa sekunder Y211 memainkan peranan penting sebagai pusat pengepinan fluks yang optimum untuk mendapatkan Y123 superkonduktor pukal yang baik.

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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 SYMBOLS AND ABBREVIATIONS

A	Ampere
Å	Angstrom
a, *b*, *c*	Lattice parameters
ACS	Alternating current susceptibility
Al_2O_3	Aluminium oxide
B_a	Applied magnetic field
B_c	Critical field
Ba	Barium
BaO	Barium oxide
$BaCO_3$	Barium carbonate
$BaCuO_2$	Barium copper (II) peroxide
BCS	Bardeen-Cooper-Schrieffer Theory
BSCCO	Bismuth strontium calcium copper oxide
CNT	Carbon nanotube
COP	Co-precipitation
CuO	Copper (II) oxide
CuO_2	Copper (II) Peroxide
DTA	Derivative thermal analysis
DTG	Derivative thermogravimetric analysis
Er123	$ErBa_2Cu_3O_y$
EDX	Energy dispersive X-ray spectroscopy
FESEM	Field emission scanning electron microscope
FWHM	Full width half maximum
GPa	Gigapascal

HTS	High temperature superconductors
h	Hours
H_{c1}	Lower critical field
H_{c2}	Upper critical field
I_o	Critical Josephson current
ICSD	Inorganic crystal structure database
J_c	Critical current density
J_{cm}	Intergranular critical current density
K	Kelvin
M	Magnetisation
MgO	Magnesium oxide
min	Minutes
Nd123	$NdBa_2Cu_3O_y$
Oe	Oersted (unit of magnetic field)
Pt	Platinum
PVP	Polyvinyl Pyrrolidone
REBCO	Rare earth barium copper oxide
RE_2O_3	Rare earth oxide
RE211	$REBa_2CuO_5$
RE123	$REBa_2Cu_3O_y$
rpm	Rotation per minute
R-T	Resistance-temperature measurement
Sm123	$SmBa_2Cu_3O_{7\sim x}$
SQUID	Superconducting quantum interference device
T	Tesla (Standard unit of magnetic flux density)
T_c	Critical temperature

$T_{c\text{-onset}}$	Starting temperature for superconductivity to occur
$T_{c\text{-zero}}$	Temperature at zero resistance
T_p	Intergranular temperature
T_{pm}	Intragranular temperature
T_{cj}	Phase lock-in temperature
T_{max}	Maximum temperature
ΔT_c	Transition of critical temperature
TGA	Thermogravimetric Analysis
wt. %	Weight percentage
x	Parameter for weight percentage
XRD	X-ray Diffraction
Y	Yttrium
Y_2O3	Yttrium oxide
Y035	$Ba_3Cu_5O_y$
Y123	$YBa_2Cu_3O_{7-\delta}$ (YBCO)
Y124	$YBa_2Cu_4O_8$
Y211	Y_2BaCuO_5
Y247	$Y_2Ba_4Cu_7O_{15}$
Y358	$Y_3Ba_5Cu_8O_{18}$
Y257	$Y_2Ba_5Cu_7O_{15}$
δ	Oxygen deficiency
ξ	Coherence length
κ	Ginzburg-Landau constant
λ	London penetration depth
θ	Angle of diffraction

Ω	Ohm
Δφ	Phase difference
χ'	Real part of ACS
χ”	Imaginary part of ACS

CHAPTER 1

INTRODUCTION

1.1 Introduction of superconductivity

Over 110 years ago, the development of superconductor's materials was empirically discovered. The discovery of yttrium barium copper oxide, $YBa_2Cu_3O_y$ (YBCO) as high-temperature superconductor (HTS) materials by Paul Chu in 1987 has received significant attention since it was declared as the first to show the superconductivity above the boiling point of liquid nitrogen, 77 K (Hor et al., 1987). Extensive research effort has been dedicated to the research and development of YBCO superconductors to improve their superconducting properties (Hapipi et al., 2018; Yusuf et al., 2019; Arlina et al., 2015; Abd-Shukor et al., 2008; Mikheenko et al., 2010; Baqiah et al., 2016; Ramli et al., 2016; Arlina et al., 2016). Many superconductors materials were discovered for their powerful potential in different fields and ease human life and activities. Various applications had been widely used, especially in technical applications, scientific and industrial applications such as in levitation platforms, microwave filters, magnetic levitation devices and electronic power transmission cables (Yang et al., 2006; Hazelton and Selvamanickam, 2009; Long et al., 2010; Aghabagheri et al., 2018).

Recently, HTS materials have been widely studied in magnetic levitation transportation systems and transmission cables (Yu et al., 2020; Buchholz et al., 2021). One of the modern developments that have given great attention nowadays is the Magnetic levitation (Maglev) train. Strong development of maglev train happens in Japan, China, Germany and the United States due to its promising approach to be used as ideal transportation in inner and intensity city areas (Chen et al., 2021; Zhao et al., 2004; Ohsaki, 2008; Bae et al., 2007). On top of that, the development of HTS electric cables has been used as alternative ways due to its higher current-carrying capacity in electric power transmission (Yang et al., 2021; Choi et al., 2019). This development can provide high electric power distribution that had been in high demand, especially in populated urban areas (Da Costa Branco and Dente, 2012; Deng et al., 2012). Bismuth Strontium Calcium Copper Oxide (BSCCO) is a first-generation for potential HTS cables and wires (Masuda et al., 2005). However, the second generation wire, YBCO, has even better potential applications to be less expensive of construction and operation, including its brilliant properties to reduce AC loss and has higher current carrying capability than BSCCO (Mukoyama et al., 2007; Ekicibil et al., 2011). The availability of higher quality and cheaper HTS are welcome to improve superconductor applications.

Up to now, YBCO, $YBa_2Cu_3O_{7\sim\delta}$ (Y123) superconductors still well known as an essential role in HTS system as it has critical temperature, T_c above the boiling point of nitrogen at 77 K as it is much cheaper and easier to handle than liquid helium that is very costly and difficult to handle (Hor et al., 1987). Enormous research towards the YBCO compound proved that the optimum superconducting properties of the YBCO system, especially for its high T_c and critical current density, J_c (Chen et al., 1989; Nakazato et al., 2014; Muralidhar et al., 2016; Ramli et al., 2016; Radušovská et al., 2016; Dihom et al., 2017; Khalid et al., 2018). Therefore, the discovery of YBCO superconducting properties did not stop as more compounds of YBCO such as Y_2BaCuO_5 (Y211) (Sandiumenge et al., 1997), $YBa_2Cu_4O_8$ (Y124) (Matsu et al., 1997), $Y_3Ba_5Cu_8O_{18}$ (Y358) (Aliabadi et al., 2009), $Y_2Ba_4Cu_7O_{15}$ (Y247) (Sato et al., 2010) and $Y_2Ba_5Cu_7O_{15}$ (Y257) (Kruaehong., 2014) were supremely studied and will be discussed more in the next chapter.

Many efforts from devoted researchers have been made to improve the superconducting properties of Y123 for technological applications. Various research has been focused on processing parameters to control and optimize the growth conditions of Y123 bulk superconductors (Hapipi et al., 2019; Slimani et al., 2018; Malik et al., 2017; Dihom et al., 2017; Abd-Shukor et al., 2008). It was recently found that a very high J_c at 77 K and self-field of around 224 kAcm^{-2} was reported to be achieved in the literature by controlling the processing conditions by infiltration growth technique (Muralidhar et al., 2016). This process involves melting the liquid phase ($BaCuO_2$ + CuO) into the (Rare Earth = RE) RE211 to form RE123 superconductors. This technique attracted much attention from researchers and industries due to its best performance that offers near net-shaped for the final bulk with precisely dispersed Y211 particles. This technique also improved the pinning centers of YBCO bulk superconductors resulting in high critical current density, J_c values (Chen et al., 1994; Iida et al., 2001; Sushma, 2020). Further details about this technique will be discussed more in Chapter 3.

Over the past years, Y123 is well known as one of the most exciting HTS materials due to its high performance, especially in superconducting properties T_c and J_c. However, it is challenging to raise their properties to the next level due to some limitations. Generally, the high superconducting performance of Y123 can be achieved by enhancing flux pinning in the Y123 system by ensuring good condition during the processing of Y123 bulks to obtain good quality and ultimate performances YBCO materials (Wang et al., 2015; Muralidhar et al., 2016; Pinmangkorn et al., 2021). Depending on the applied magnetic field strength for the practical applications, the HTS material may classify into two groups which are small-scale applications and large-scale applications. A wide range and large scale of industrial applications such as flywheel energy storage systems, HTS DC cables, and superconducting levitation train systems required superior superconducting properties, including high magnetic fields (Werfel et al., 2013). The small-scale applications are relevant and have been used in electronic applications, such as HTS devices and detectors (Torino et al., 2001). However, poor superconducting properties at high magnetic fields has disrupted the YBCO material performance in several practical applications (Hannachi et al., 2020). Thus, the preparation of these YBCO superconductors materials could be realized by altering the process conditions and using different synthesis methods to produce desired YBCO materials.

1.2 Problem statement

The applications of Y123 with high J_c is limited to non-transporting fields due to the weak-link nature of its grain's boundary, which is attributed to the lower $J_{c.}$ When the vortices of the Y123 move under the influence of the transport current, it will turn the materials into no longer remain in a zero-resistance state and lead to energy dissipation.

To improve the superconducting properties, Y123 superconducting materials have been prepared by different techniques: the thermal treatment method and the IG technique with some artificial and strong pinning centers are required to achieve high $J_{c.}$ The existence of inhomogeneities such as impurities and defects can act as effective flux pinning centers to maintain the superconductivity of the Y123 superconductors (Dadras et al., 2017; Yang and Wang, 2013). The graphene nanoparticles and the Y211 particles were added as impurities to act as the flux pinning and pinned the vortex motion in the Y123 compounds, thus leading to high J_c under applied magnetic field. Graphene nanoparticles are known as carbon-based nanomaterials composed of a two-dimensional (2D) hexagonal lattice with special properties such as high surface area, good electrical conductivity and better thermal conductivity (Kogularasu et al., 2017; Sahoo et al., 2019). Thus, the addition of these impurities with good processing conditions methods will improve the J_c values that help act as good pinning centers of the Y123.

1.3 Objectives

The objectives of this research are:

1. To investigate the effect of superconducting properties of Y123 bulk superconductors with graphene nanoparticles additions on the structural properties, surface morphology, T_c and J_c by thermal treatment method.
2. To elucidate the influence of the Y123/Er123 ratios on the structural, surface morphology and superconducting properties, T_c and J_c of Y123 bulk superconductors by the infiltration growth process.

1.4 Scope of the research works and limitations

This study focuses on the Y123 bulk superconductors prepared by different techniques: the thermal treatment method (Part 1) and the IG method (Part 2). The preparation of the Y123 bulk by the thermal treatment method will be focused on the different weight percentages, x = 0.0, 0.1, 0.3, 0.5 and 1.0 wt. % graphene nanoparticles. For the IG method, Y123 will be fabricated by varying the ratios of (Y123: Er123 = 1:0, 0.75:0.25, 0.5:0.5, 0.25:0.75 and 0:1) in a liquid phase. The IG method has been conducted during the research exchange program at Superconductivity Research Laboratory, Shibaura Institute of Technology (SIT), Tokyo, Japan. The samples were characterized using thermogravimetric (TGA) analysis, X-ray diffraction (XRD), Fields emission scanning electron microscope (FESEM)/ energy dispersive X-ray (EDX) analysis. The

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LIST OF PUBLICATIONS

Publication in Index Journal

Kamarudin, A.N., Awang Kechik, M.M., Miryala, M., Pinmangkorn, S., Murakami, M., Chen, S.K., Baqiah, H., Ramli, A., Lim, K.P. and Halim, S.A. (2021). Microstructural, Phase Formation, and Superconducting Properties of Bulk $YBa_2Cu_3O_y$ Superconductors Grown by Infiltration Growth Process Utilizing the $YBa_2Cu_3O_y$ + $ErBa_2Cu_3O_y$ + $Ba_3Cu_5O_8$ as a Liquid Source. C*oatings* 11(4).

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Optimisation of The Liquid Source $YBa_2Cu_3O_y$ +$ErBa_2Cu_3O_y$ + $Ba_3Cu_5O_8$ in Bulk $YBa_2Cu_3O_y$ Superconductors Grown by Infiltration Growth Process on International Conference on Solid State Science and Technology (ICSSST 2021) at the 5th International Innovation, Design and Articulation (i-IDeA 2020).

Effect of Graphene Nanoparticles Addition on Superconductivity of $YBa_2Cu_3O_{7\sim\delta}$ (Y123) Synthesized via Thermal Treatment Method on International Fundamental Science Congress (IFSC) 2021 with collaboration between Universiti Putra Malaysia, Malaysia, Kasetsart University, Thailand and IPB University, Indonesia.

Effect on Structural and Superconducting Properties of $YBa_2Cu_3O_{7\sim x}$ (Y123) Superconductor Added with Graphene Nanoparticles via Thermal Treatment Method on International Symposium on Superconducting Magnetic & Energy Materials (ISSM) 2020 with collaboration between Universiti Putra Malaysia, Malaysia and Shibaura Institute of Technology, Japan.

Top-Seeded Infiltration Growth Process of YBCO Bulk Materials for Superconducting Cables Technology on Materials Technology Challenges (MTC) 2020, Universiti Putra Malaysia, Malaysia (Gold Medal Award).

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