SYNTHESIS AND CHARACTERIZATION OF $YBa_2Cu_3O_{7-\delta}$ (YBCO) ADDED BLACK PHOSPHORUS VIA SOLID-STATE REACTION

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons)

By

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Sesi Pengajian: **2021/2022 Semester 2**

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfillment of requirements for the degree of Bachelor of Manufacturing Engineering (Hons.)

The members of supervisory committee are as follow:

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(Dr. Mohd Shahadan Bin Mohd Suan)

ABSTRAK

Superkonduktor ialah bahan yang mencapai superkonduktiviti iaitu keadaan jirim yang tiada rintangan elektrik dan tiada medan magnet boleh melaluinya. Dalam kebanyakan kes, superkonduktiviti hanya boleh dicapai pada suhu yang sangat rendah. Superkonduktor suhu tinggi $YBa_2Cu_3O_{7-\delta}$ dengan Fosforus Hitam ditambah telah disintesis melalui tindak balas keadaan pepejal. Kebaharuan kerja penyelidikan ini ialah kaedah tindak balas keadaan pepejal menggunakan kurang tenaga berbanding kaedah sintesis lain untuk memproses superkonduktor $YBa_2Cu_3O_{7-\delta}$ dan menghasilkan pengedaran telaga nanozarah Fosforus Hitam. Tindak balas automatik-pembakaran mengubah gel sitrat-nitrat prekursor yang dirumus menjadi produk abu halus. Ia menghasilkan fasa $YBa_2Cu_3O_{7-\delta}$ selepas proses pengkalsinan yang selanjutnya dirawat haba untuk mencapai superkonduktiviti. Kesan komposisi berbeza Fosforus Hitam ditambah $YBa_2Cu_3O_{7-\delta}$ pada struktur dan sifat superkonduktor $YBa_2Cu_3O_{7-\delta}$ telah disiasat dan dinilai. Kerja ini menunjukkan bahawa tindak balas keadaan pepejal adalah kaedah yang berkesan untuk memperkenalkan Fosforus Hitam sebagai zarah nano yang diedarkan secara homogen dalam superkonduktor $YBa_2Cu_3O_{7-\delta}$

ABSTRACT

A superconductor is a substance that achieves superconductivity which is a state of matter that is no electrical resistance and no magnetic fields may pass through it. In most cases, superconductivity can only be attained at extremely low temperatures. In this study, the high temperature superconductor $YBa_2Cu_3O_{7-\delta}$ with added Black Phosphorus was synthesized via solid-state reaction. The novelty of this research work is the solid-state reaction method consumed less energy compared with other synthesis methods for processing $YBa_2Cu_3O_{7-\delta}$ superconductor and produced well distribution of Black Phosphorus nanoparticles. The auto-combustion reaction transformed the formulated precursor citrate-nitrate gel into fines ashes product. It yielded $YBa_2Cu_3O_{7-\delta}$ phases after calcination process which was further heat treated to achieve superconductivity. The effects of different compositions of Black Phosphorus added $YBa_2Cu_3O_{7-\delta}$ on the structural and superconducting properties of $YBa_2Cu_3O_{7-\delta}$ were investigated and appraised. This work shows that the solid-state reaction is an effective method to introduce Black Phosphorus as nanoparticles homogeneously distributed in the $YBa_2Cu_3O_{7-\delta}$ superconductor.

DEDICATION

Only

My beloved father, Goh Wai Keong

My appreciated mother, Leow Mei Pin

My adored brother, Goh Chun Kit

For giving me moral support, money, cooperation, encouragement and also understandings

Thank you so much & Love You All Always.

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LIST OF SYMBOLS AND ABBREVIATIONS

Jc	-	current density
Tc	-	critical temperature
XRD	-	X-ray diffraction
SEM	-	Scanning electron microscope
K	-	Kelvin
E_g	-	minimum energy
k_B	-	Boltzmann’s constant
B_c	-	critical magnetic field
B_{c1}	-	lower critical magnetic field
B_{c2}	-	upper critical magnetic field
E	-	electric field
m	-	mass
n_s	-	number density
e	-	charges of electrons
A	-	vector potential
u_0	-	permeability of the vacuum
λ_L	-	London penetration
F_s	-	free energy density
ξ	-	coherence length
h	-	Planck constants
F_L	-	Lorentz force
F_p	-	pinning force density
N_p	-	number of density of pinning centre
f_p	-	elementary pinning force
d	-	spacing between diffracting planes

θ	-	incident angle
λ	-	beam wavelength

CHAPTER 1

INTRODUCTION

This chapter provide background of study, problem statement, objectives, scope and significant of study and organization of the report.

1.1 Background of Study

Superconductor is discovered by famous Dutch scientist, Kamerlingh-Onnes soon after he found how to liquefy helium in the early 1900s. He began research into the electrical resistance of very pure metals at cryogenic temperatures. Kamerlingh-Onnes was testing electrical resistance of pure mercury at low temperature in 1911. He found out below 4K, the resistance of mercury decreased to zero. He discovered that mercury enter a new condition below 4K which is called the "superconductivity". Superconductors are metals, ceramics, and organic materials. Superconductivity occurs when an electrical current is passed through a material that has no resistance. This indicates that superconductor can carry electrons without releasing heat or generating energy. When a superconductor is subjected to a magnetic field, its magnetic lines are expelled out. This effect is known as Meissner effect. High- temperature superconductor was characterized by their complex chemical reaction. Yttrium Barium Copper Oxide (YBCO) is a most widely superconductor that displays high-temperature superconductivity. In 1986, J.G. Bednorz and K.A. Mueller found high-temperature superconductivity in $LaBaCuOx$ and were given the Nobel Prize in Physics shortly after. This material exhibited a critical temperature, Tc of 35 K.The groups of M.K. Wu and C.W. Chu discovered superconductivity in the $Y - Ba - Cu - O$ system a year later with $YBa_2Cu_3O_{7-\delta}$ also known as "YBCO-123" having a Tc onset of 92 K.

$YBa_2Cu_3O_{7-\delta}$ is the first material discovered to become superconducting above the boiling point of liquid nitrogen (77K).

There are two type of superconductor. Type I superconductors and Type II superconductors are two different types of superconductors. The majority of Type I superconductors are pure metals with conductivity at normal temperature. Type I superconductors were the first to be found and they require a lower temperature to conduct. Type I superconductors include aluminium, mercury, and lead. They were referred to as "soft superconductors." Metal alloys or complicated oxide ceramics are commonly used in Type II superconductors. Type II superconductors commonly known as high-temperature superconductors (HTS) and "hard superconductors". Type II superconductors include YBCO, BSCCO, TBCCO, and HBCCO.

The Yttrium barium copper oxide (YBCO) superconductor is now being studied because of its ease of synthesis, reliability, and low cost. The $YBa_2Cu_3O_{7-\delta}$ superconductor is highly recommended due to its diverse applications. As a consequence, the research for new type of superconductor has never ended since the discovery of superconductivity in $YBa_2Cu_3O_{7-\delta}$ at 92 K.

1.2 Problem Statement

$YBa_2Cu_3O_{7-\delta}$ superconductor is an attractive material for potential applications such as energy storage systems, current limiters, magnetic bearings and so forth due to their comparatively high critical temperature. These applications required a high critical density even with high applied magnetic field. For maintaining high superconducting properties in $YBa_2Cu_3O_{7-\delta}$, it has important limiting factors such as low grain boundary, weak links and poor flux pinning. In the presence of magnetic field, the $YBa_2Cu_3O_{7-\delta}$ results in low current density, Jc and critical temperature, Tc. Current density can be enhanced by preventing the vortex from moving which can be accomplished by pinning flux lines using suitable impurities which is known as pinning centres materials. Pinning centre materials can be generated by chemical doping or addition of nanoparticles in $YBa_2Cu_3O_{7-\delta}$ superconductor.

Addition of nanoparticles is most currently in research due to its easy process. Nanoparticles such as $BaZrO_3, ZrO_2, ZnO, SnO_2$ and Al_2O_3 can react with superconductor materials like $YBa_2Cu_3O_{7-\delta}$ and some of them have been shown to have a considerable effect on superconductor critical parameters, notably increasing Jc . The addition of nanoparticles to $YBa_2Cu_3O_{7-\delta}$ system enhance current carrying capability which prevent magnetic flux mobility therefore prevent Jc suppression. Furthermore, the addition of nanoparticles to $YBa_2Cu_3O_{7-\delta}$ system improve current carrying capability due to size of nanoparticles is almost identical to magnetic flux diameter of superconductor. Thus, the addition of nanoparticles is likely to change other superconducting properties of $YBa_2Cu_3O_{7-\delta}$ by locally altering the crystalline structure and generating defects such as twin, tweed, and inhomogeneous micro-defects. In this study, black phosphorus nanoparticles was selected to be pinning centre material in $YBa_2Cu_3O_{7-\delta}$.

Black phosphorus act as pinning centre material is still rare, less study and research was conducted. Since then, black phosphorus was the most stable allotrope of the phosphorus element. Black phosphorene is one of the rare 2D materials having exceptional mechanical, electrical, and optical characteristics for device applications. Black phosphorus has been gaining popularity ever since the fabrication of field-effect transistors. Recent research indicates that 2D Black Phosphorus and its bulk phase have a high charge carrier mobility, in-plane anisotropic structure, and a tunable direct bandgap. Moreover, according to its wider layer spacing and folded structure, 2D Black Phosphorus has a theoretical specific capacity that is 432.8 mAh g^{-1}, which is significantly higher than graphite (372 mAh g^{-1}). Due to the armchair phosphorene nanoribbon structure in Black Phosphorus, it have excellent energy storage capability in both theoretically and experimentally. Therefore, Black phosphorus has emerged as a potential pinning centre material for superconductors.

There are several methods used to synthesized $YBa_2Cu_3O_{7-\delta}$ such as coprecipitation , combustion method and solid-state reaction. Coprecipitation is method where metal oxalate, carbonate or hydroxide precipitates with cation mole ratio of $\mathrm{Y:Ba:Cu} = 1:2:3$ were formed from aqueous solution of metal nitrates. The resulting precursors are acquired in the form of fine powder using this procedure. The difficulty in controls the final composition of the precipitates is an issue in this procedure. The citrate-nitrate auto-combustion reaction was also used to make superconductor ceramic material results in nanostructured homogenous materials with high yield. Solid state reaction was the most extensively used method which allows common compounds to be calcined into superconducting materials. This method has been used to fabricate all the superconductors described in the present work. In 2009, A.Aliabadi used solid state reaction at 840°C for 12 hours to synthesize superconductor $Y_3Ba_5Cu_8O_{18-\delta}$ (Y38), the last member of YBCO family. With the exception of CuO chains and CuO_2 planes, the sample had Tc around 102 K and crystalline structure of this phase is remarkably similar to $YBa_2Cu_3O_{7-\delta}$. In this study, sample with different composition of Black Phosphorus added into $YBa_2Cu_3O_{7-\delta}$ will prepared using solid state method. Then, the structural and superconductivity properties of sample will be investigated and discussed in this study.

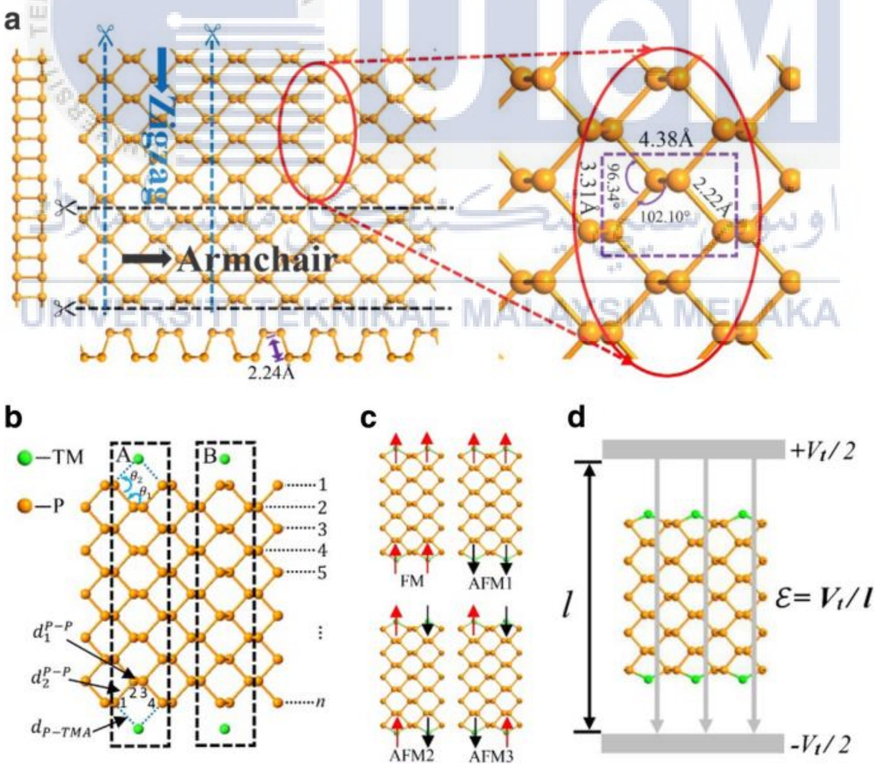

Figure 1.1 Armchair structure of 2D Black Phosphorus

1.2 Objectives

I. To formulate the Black Phosphorus added $YBa_2Cu_3O_{7-\delta}$ superconducting nanoparticle by using solid-state reaction.

II. To elucidate the structural properties of Black Phosphorus added $YBa_2Cu_3O_{7-\delta}$ by using X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM) analysis.

III. To correlate the structural properties of Black Phosphorus with the electrical conductivity of $YBa_2Cu_3O_{7-\delta}$ superconductor by using cryogenic Four-Point Probe.

1.3 Scope of Work

The scopes of this work are synthesizing and characterizing $YBa_2Cu_3O_{7-\delta}$ added black phosphorus nanoparticles produced by solid-state reaction. This study seeks to systematically study the effect of black phosphorus with different weight percentage added into $YBa_2Cu_3O_{7-\delta}$ superconductor target on structural properties and superconducting properties. This study will entail structural analysis utilizing X-ray diffraction (XRD), Scanning electron microscope (SEM) and Resistance-Temperature $(R - T_c)$ measurements in sample for superconducting properties will be measure through Four- Point Probe. To summarize, this study will introduce black phosphorus as pinning centre material to increase the superconducting properties with minimum effects toward Tc of $YBa_2Cu_3O_{7-\delta}$ superconductor.

1.4 Significance of Study

This research present a method of synthesizing superconductors composite. Black phosphorus nanoparticles was introduced as pinning centre materials and distributes into $YBa_2Cu_3O_{7-\delta}$ superconductor. The black phosphorus shows great potential as a candidate of pinning centre materials and has attracted tremendous interest in scientific community due to its advantageous structural properties and higher carrier mobility. The method used in this study is solid state reaction which has higher productivity and lower cost. The reaction efficiency in this technique is great and provide better distribution in high temperature superconductor. As a results, the structural and superconducting properties of $YBa_2Cu_3O_{7-\delta}$ are expected to be increased.

1.5 Organization of Report

There are five chapters included in this project. Chapter 1 describes background study, problem statement, objectives, scope of work and significance of study are covered in order to complete this project. Chapter 2 describes about the literature review based on the project. This chapter provide basic theory about superconductors and characteristics of type II superconductor. Chapter 3 describes the details about preparation of sample and characterization techniques used in this project. For chapter 4, which is results and discussion will discuss about the results obtain from this study. Lastly, chapter 5 describes about conclusion of this project and recommendation in this study.

1.6 Summary

This chapter has successfully described the background of this study and a brief introduction of this study.

CHAPTER 2

LITERATURE REVIEW

This chapter is mainly describe the theory and research which have been defined and done by various researcher years ago. Related information of previous studies are extracted as references and discussion based on their research about YBCO superconductor, structure, mechanical and physical properties.

2.1 A Brief History of Superconductivity

Heike Kamerlingh Onnes discovered superconductivity in 1911 while studying the characteristics of metals at low temperatures. He became the first one to liquefy helium which has a boiling point of 4.2 K at atmospheric pressure. This had opened up a new range of temperature for experimental study. During his research on the conductivity of metals at low temperatures, he discovered that just at the boiling temperature of liquid helium the resistance of mercury sample decreased to an unmeasurable low value. Figure 2.1 shows the initial measurement. This unexpected phenomenon was called "superconductivity" by Karmerlingh Onnes. The critical temperature (Tc) is known as the temperature below which the mercury becomes superconducting.

Over the next 75 years, scientists made significant progress in their understanding of superconductors. During that period, various alloys that were superconductors at slightly higher temperatures were discovered. Unfortunately, none of these alloy superconductors was capable of operating at temperatures higher than 23 K. As a result, liquid helium remained the sole practical refrigerant for use with these superconductors. Many additional elements have been revealed to be superconductors after this discovery. In 1980, superconductivity had been discovered in numerous metals and alloys. Remarkably, traditional ferromagnets like Nickel (Ni) and Iron (Fe) did not show superconductivity. Superconductivity has only been documented in the non-magnetic state and under very high pressure. For example, in iron, Tc=2 K. Discovery of superconductors with high Tc has been a strong motivation since the beginning. However, until 1980 the A-15 compound, Nb_3Ge was the superconductor with highest Tc around 22 K. Thus, many alloys and impact of applying pressure were investigated an attempt to obtain higher Tc values.

Consequently, numerous possible applications were ruled out due to lack of commercial viability. Furthermore, most scientists consider superconductivity as a small field with limited potential for increasing critical temperatures. All this suddenly changed when K. A. Bednorz and J. G. Müller discovered high-temperature superconductivity. New superconductors from different material classes were discovered shortly after 1980. K. A. Bednorz and J. G. Müller published a paper in April 1986 on the possibility of superconductor in ceramic material called $\mathrm{La-Ba-Cu-O}$ with Tc around 35K. This was the first cuprate superconductor to be discovered. Müller and Bednorz said that the French work on $\mathrm{La-Ba-Cu-O}$ system inspired their research. The era of "High-Temperature Superconductivity" began when the researchers from the University of Tokyo validated Müller and Bednorz findings.

At the end of 1986 and the beginning of 1987, the discovery of $\mathrm{Y-Ba-Cu-O}$ (YBCO) superconductor with a Tc of 92 K was highlighted by the synthesis of rare-earth metal oxides with higher Tc. This is a huge breakthrough because the material was become superconducting above 77K, the boiling point of liquid nitrogen. Because of the simplicity in which YBCO could be synthesized, it was investigated by a several laboratories. In February 1988, researchers from Japan, China and the United States discovered superconductivity in copper-containing oxides without rare earth. Bismuth or thallium are included in these non-rare earth superconductors. These superconducting compounds have been reported with Tc around 127 K. In addition, unlike rare earth superconductors, these materials are more stable since they do not lose oxygen or react with water. Figure 2.3 show history of some of the discovered superconducting compounds.