ENHANCING SUPERCONDUCTING PROPERTIES AND GRAIN CONNECTIVITY OF MAGNESIUM DIBORIDE VIA DOPANT ADDITIONS

By

NURHIDAYAH BINTI MOHD HAPIPI

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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DEDICATION

This work is dedicated to my beloved father and mother

MOHD HAPIPI BIN HANAFI
ASPALILA BINTI JUSOH

and to my sibling

NORHIWANI BINTI MOHD HAPIPI
MOHD IQRAM BIN MOHD HAPIPI
NURHIDAYATI BINTI MOHD HAPIPI

Thank you for everything!

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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In this work, both *ex-situ* and *in-situ* methods were used to synthesise MgB_2 samples. Pure *ex-situ* MgB_2 sample (Series 1) was sintered at various temperatures (600-900 °C) and times (1-7 h). Several dopants such as excess Mg (Series 2), (1.5 Mg + 2 B) (Series 3), nano-Si (Series 4), nano-Si + LaB_6 (Series 5), and Dy_2O_3 + La_2O_3 (Series 6) were added into MgB_2. For series 1, increasing the sintering temperature to 900 °C increased the J_c value (0 T, 20 K) to 4.2 × 10^3 A/cm^2, suggesting an enhancement in sample grain coupling. A prolonged sintering time of 3 h increased the J_c value to 3.2 × 10^3 A/cm^2 before decreasing to 0.5 × 10^3 A/cm^2 when the sintering time was prolonged to 7 h. Meanwhile, the addition of excess Mg into *ex-situ* MgB_2 (Series 2) successfully inhibits MgB_2 decomposition where no MgB_4 peaks were observed in the Mg-added sample, in contrast to pure *ex-situ* MgB_2 which exhibited MgB_4 peaks at higher sintering temperatures. When the sintering temperature increased, the addition of excess Mg reduced the average grain sizes and further strengthened the grain coupling of the samples, which subsequently increased the J_c value to 10^4 A/cm^2, which is more than 20 times. In Series 3, the addition of 0 to 50 wt.% of (1.5 Mg + 2 B) increased the J_c (0 T, 20 K) value from 3.0 × 10^3 A/cm^2 to 1.3 × 10^4 A/cm^2, respectively. The highest J_c (0 T, 20 K) value obtained for Series 3 was 2.1 × 10^4 A/cm^2 for the sample sintered at 1000 °C. XRD pattern for nano-Si added into *in-situ* MgB_2 samples (Series 4) shows the formation of Mg_2Si where excess Mg_2Si can obstruct the current pathway of the samples and lower the value of J_c. The addition of nano-Si from 0 to 10 wt.% decreased the value of J_c (0 T, 20 K) from 2.4 × 10^5 A/cm^2 to 1.7 × 10^5 A/cm^2, respectively. However, the J_c value at high field increased to 2.8 × 10^3 A/cm^2 with the addition of 5 wt.% of nano-Si. The co-addition of 0.03 mol LaB_6 and *x* wt.% nano-Si (Series 5) inhibited the grain growth of the samples as no significant changes in average grain size were observed. The addition of LaB_6 decreased J_c (0 T, 20 K) to 2.12 × 10^5 A/cm^2, and it further decreased to 1.7 × 10^5 A/cm^2 with co-addition of LaB_6 and 10 wt.% of nano-Si. Co-addition of Dy_2O_3 and La_2O_3 into MgB_2 (Series 6) enhanced the flux pinning of the samples and the J_c value, where the highest J_c (0 T, 20 K) value obtained was 4.3 × 10^5 A/cm^2 for 1.00 wt.% co-added samples.

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

MENINGKATKAN SIFAT MENSUPERKONDUKSI DAN SAMBUNGAN BUTIRAN MAGNESIUM DIBORIDE MELALUI TAMBAHAN BAHAN DOP

Oleh

NURHIDAYAH BINTI MOHD HAPIPI

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Di dalam kerja ini, kedua-dua kaedah *ex-situ* dan *in-situ* telah digunakan untuk mensintesis sampel MgB_2. Sampel MgB_2 *ex-situ* tulen (Siri 1) telah disinter pada pelbagai suhu (600-900 °C) dan masa (1-7 jam). Beberapa dopan seperti Mg berlebihan (Siri 2), (1.5 Mg + 2 B) (Siri 3), nano-Si (Siri 4), nano-Si + LaB_6 (Siri 5), dan Dy_2O_3 + La_2O_3 (Siri 6) telah ditambah ke dalam MgB_2. Untuk siri 1, peningkatan suhu pensinteran kepada 900 °C telah meningkatkan nilai J_c (0 T, 20 K) kepada 4.2 × 10^3 A/cm^2, mencadangkan peningkatan dalam gandingan butiran sampel. Masa pensinteran yang berpanjangan selama 3 jam telah meningkatkan nilai J_c kepada 3.2 × 10^3 A/cm^2 sebelum berkurangan kepada 0.5 × 10^3 A/cm^2 apabila masa pensinteran dipanjangkan kepada 7 jam. Sementara itu, penambahan Mg berlebihan ke dalam MgB_2 *ex-situ* (Siri 2) berjaya menghalang penguraian MgB_2 di mana tiada puncak MgB_4 diperhatikan dalam sampel tambah Mg, berbeza dengan MgB_2 *ex-situ* tulen yang mempamerkan puncak MgB_4 pada suhu pensinteran yang lebih tinggi. Apabila suhu pensinteran meningkat, penambahan Mg berlebihan telah mengurangkan saiz butiran purata dan mengukuhkan lagi gandingan butiran sampel, yang seterusnya meningkatkan nilai J_c kepada 10^4 A/cm^2 iaitu lebih daripada 20 kali ganda. Dalam Siri 3, penambahan 0 hingga 50 wt.% daripada (1.5 Mg + 2 B) meningkatkan nilai J_c (0 T, 20 K) daripada 3.0 × 10^3 A/cm^2 kepada 1.3 × 10^4 A/cm^2, masing-masing. Nilai J_c (0 T, 20 K) tertinggi yang diperolehi untuk Siri 3 ialah 2.1 × 10^4 A/cm^2 untuk sampel yang disinter pada 1000 °C. Corak XRD untuk nano-Si ditambah ke dalam sampel MgB_2 *in-situ* (Siri 4) menunjukkan pembentukan Mg_2Si di mana Mg_2Si berlebihan boleh menghalang laluan arus sampel dan menurunkan nilai J_c. Penambahan nano-Si daripada 0 hingga 10 wt.% telah menurunkan nilai J_c (0 T, 20 K) daripada 2.4 × 10^5 A/cm^2 kepada 1.7 × 10^5 A/cm^2, masing-masing. Walau bagaimanapun, nilai J_c pada medan tinggi telah meningkat kepada 2.8 × 10^3 A/cm^2 dengan penambahan 5 wt.% nano-Si. Penambahan bersama 0.03 mol LaB_6 dan *x* wt.% nano-Si (Siri 5) menghalang pertumbuhan butiran sampel kerana tiada perubahan ketara dalam saiz purata butiran dapat diperhatikan. Penambahan LaB_6 mengurangkan J_c (0 T, 20 K) kepada 2.12 × 10^5 A/cm^2, dan ia terus menurun kepada 1.7 × 10^5 A/cm^2 dengan penambahan bersama LaB_6 dan 10 wt.% nano-Si. Penambahan bersama Dy_2O_3 dan

La_2O_3 ke dalam MgB_2 (Siri 6) telah meningkatkan penyematan fluks sampel dan nilai J_c, di mana nilai J_c (0 T, 20 K) tertinggi yang diperolehi ialah 4.3 × 10^5 A/cm^2 untuk 1.00 wt.% sampel ditambah bersama.

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LIST OF ABBREVIATIONS

θ	Angle of diffraction
Å	Angstrom (Unit of length equal to 10^{-10} m)
H_{ac}	Applied magnetic field
a.u.	Arbitrary unit
ξ	Coherence length
J_c	Critical current density
T_c	Critical temperature
Dy_2O_3	Dysprosium (III) oxide
FESEM	Field emission scanning electron microscopy
FWHM	Full width at half maximum
κ	Ginzburg-Landau constant
HTS	High-temperature superconductor
ICSD	Inorganic Crystal Structure Database
H_{irr}	Irreversibility field
K	Kelvin (Standard unit of temperature)
LaB_6	Lanthanum hexaboride
La_2O_3	Lanthanum (III) oxide
a-, *b*-, *c*-axis	Lattice parameter
LTS	Low-temperature superconductor
F_L	Lorentz force
Mg	Magnesium
MgB_2	Magnesium diboride
MgO	Magnesium oxide
Φ_0	Magnetic flux

mol.%	Molar percentage
$T_{c\text{-onset}}$	Onset critical temperature
$T_{c\text{-offset}}$	Offset critical temperature
F_p	Pinning force
Si	Silicone
SQUID	Superconducting Quantum Interference Devices
T	Tesla (Standard unit of magnetic flux density)
TGA	Thermogravimetry Analysis
ΔT_c	Transition temperature width
wt.%	Weight percentage
XRD	X-Ray Diffractometer
λ	X-ray wavelength

CHAPTER 1

INTRODUCTION

1.1 Background Study

Superconductivity is a physical phenomenon that occurs when the electrical resistance of a material is zero and magnetic flux is expelled from the material. Any material that exhibits these properties is known as a superconductor (Rose-Innes and Rhoderick, 1978). Superconductors can be divided into low-temperature superconductors, LTS (niobium-based alloy), and high-temperature superconductors, HTS (copper-oxide-based). If the critical temperature, T_c, of a superconductor is above 30 K, it is classified as HTS. In the meantime, a T_c value below 30 K is referred to as LTS. Many researchers are more interested in studying HTS compared to LTS due to its higher T_c and J_c values.

However, following the discovery of superconductivity in MgB_2, researchers became interested in studying MgB_2. This is because MgB_2 has a simple crystal structure and high critical temperature ($T_c \approx 39$ K), making it a most promising candidate for cryogen-free operation to replace conventional NbTi and Nb_3Sn-based technology. Two common methods to prepare MgB_2 bulk samples are *in-situ* (Arvapalli et al., 2019; Gao et al., 2010; Kim et al., 2007; Muralidhar et al., 2017) and *ex-situ* method (Malagoli et al., 2010; Mizutani et al., 2014; Tanaka et al., 2012). *In-situ* is a method of synthesising MgB_2 by simply mixing Mg and B powders at the appropriate ratio of Mg: B = 1: 2. During heat treatment, Mg grains melt and diffuse into B grains through a liquid-solid reaction. Meanwhile, the *ex-situ* method involves heat treatment of the pre-reacted MgB_2 powders. Although commercial MgB_2 powders are readily available as it has been synthesized since the early 1950s, the quality of MgB_2 powder may not be as good as desired (Buzea and Yamashita, 2001).

MgB_2 also possesses a high critical current density, J_c (> 10^5 A/cm^2 at 20 K, 0 T), strong grain coupling due to its large coherence lengths (ξ_{ab} ~ 6.5 nm at 0 K), and weak-link-free behaviour at grain boundaries. MgB_2 is a lightweight and inexpensive material that is ideal for commercial and industrial applications (Buzea and Yamashita, 2001; Larbalestier et al., 2001; Xu et al., 2001; Yamamoto et al., 2004; Zhao et al., 2001). However, further improvement of J_c and flux pinning is crucial for the material to be used in high magnetic field applications. Prior studies show the addition of dopants such as Mg (Zeng et al., 2008; Zhang et al., 2015), La (Kimishima et al., 2004; Shekhar et al., 2005), La_2O_3 (Gao et al., 2010), Dy_2O_3 (Chen et al., 2006), Si (Tan et al., 2015; Zhang et al., 2010) and carbon (Ohmichi et al., 2004; Tan et al., 2015; Yeoh et al., 2004) into MgB_2 can act as flux pinning that enhanced the value of J_c, resulting in high-quality MgB_2 samples.

1.2 Applications of Superconductor

Superconductors have brought great success and technological changes, especially in energy storage, transportation, electronics, and medical sector. Some of the applications are discussed as follows:

1.2.1 Superconducting Wire

Superconductor wire is an electrical wire made of superconductor material that exhibits no electrical resistance when cooled below T_c. Figure 1.1 shows that, although bismuth-based superconductor wire has a smaller cross-sectional area than copper wire, it can carry the same current magnitude. It means that superconductor wire can carry approximately 200 times more electric current than copper wire with the same cross-sectional area (Hayashi, 2020).

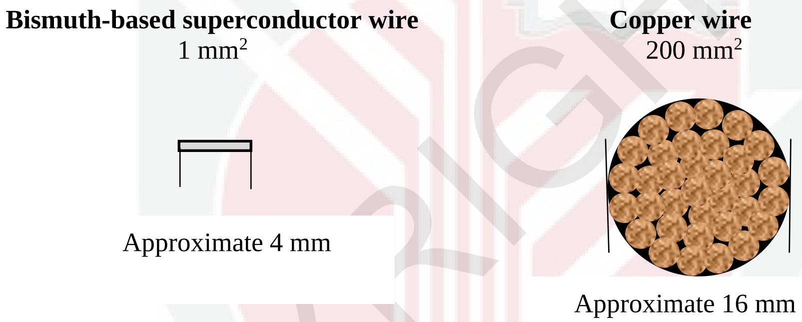

Figure 1.1: Comparison of the cross-sectional area for bismuth-based superconductor wire and copper wire carrying the same current magnitude

Commonly, conventional superconductors such as niobium-titanium (NbTi) and niobium-tin (Nb_3Sn) are used as the superconducting wire. However, these conventional superconductors are made from low-temperature superconductors that require very expensive liquid helium for cooling. Besides, Nb_3Sn is brittle and difficult to fabricate. High-temperature superconductor (HTS) wires were later introduced to the market. HTS wire can operate in liquid nitrogen, which is cheaper than liquid helium. HTS wires are also more tolerant of AC loss and have better thermal stability than low-temperature superconducting (LTS) wires. There are two types of high-temperature superconductor wires: first-generation (1G) wire, bismuth strontium calcium copper oxide, BSCCO, and second-generation (2G) wire, rare-earth barium copper oxide, ReBCO. 1G HTS superconductor wires are widely used on various HTS power devices such as transmission cables, transformers, motors, and generators. The first company to produce long bismuth-based superconductor wire (DI-BSCCO) is Sumitomo Electric Japan. The transition from 1G HTS wire to 2G HTS wires promises that 2G HTS wires will be cheaper than existing 1G wires. This 2G HTS wire provides cost benefits and excellent performance benefits (Hayashi, 2020).

1.2.2 High-Temperature Superconductor (HTS) Cable

High-temperature superconductor (HTS) power cable is made of a group of sheath-coated wires. It can carry two to five times the electrical current more than conventional cables such as XLPE (Cross Link Poly Etheline) of the same size. Furthermore, HTS cable can operate at high current levels with minimal heat and electricity loss, which aids in energy conservation. HTS cables are also smaller than conventional cables, allowing them to occupy less space. Therefore, it does not require extensive construction work for installation, potentially lowering construction costs. In Japan, the Railway Technical Research Institute (RTRI) has developed a superconducting feeder cable system. This superconducting feeder cable connects the Hino Civil Engineering Testing Station to the regular feeding circuit of the Chuo line (408 m cable), where it needs to be cooled with cryogenic or liquid nitrogen. The test confirmed that currents up to 2200 A or larger could flow from the substation to the test train as the train is accelerated. Most importantly, the shut-off test confirmed that the train could keep running, powered by the regular feeding circuit, even after the superconducting system is shut off (*Superconducting Feeder Cable System*, 2019).

1.2.3 Maglev Train

In the 21st century, few countries such as Japan, South Korea, and China have developed powerful electromagnetic high-speed trains called maglev trains. Maglev is derived from "magnetic" and "levitation". Maglev trains operate on magnetic repulsion principles between a train and a track, where magnetic levitation can be achieved using an electrodynamic suspension system (EDS).

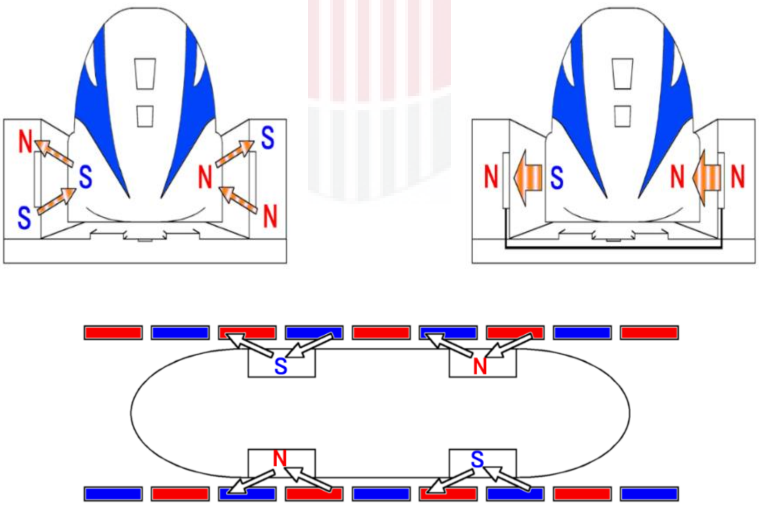

Figure 1.2: The illustration of the superconductor Maglev levitation and propulsion system

Figure 1.2 shows an illustration of the Maglev superconductor levitation and propulsion system. The Maglev train railway consists of two sets of cross-connected metal coils wound into a "figure eight" pattern along both the guideway walls to form electromagnets. These coils are also cross connected underneath the rails to accelerate the cars and guide and stabilise them. Due to the magnetic field induction effect, the magnetic field of the superconducting magnets induces a current into these coils when the train accelerates.

The train's movement starts when it moves forward slowly on the wheels, allowing the magnets beneath the train to interact with the guideway. Once the train reaches 150 kilometres per h, the magnetic force is strong enough to lift the train 4 inches off the ground. The magnetic force will then eliminate friction between the car and the guideway, allowing for faster speeds. This magnetic force also makes the train move forward and continue to be centred within the guideway. If the train is centred with the coils, the electrical potential will be balanced, and no currents will be induced. However, when the train runs on rubber wheels at a lower speed, a magnetic field positioned below the coils' centre will cause an unbalanced electrical potential. When stopped, the train will rest on rubber wheels.

The advantage of the Maglev train is that it can float on the rails, which means there is no rail friction. It allows trains to travel at speeds of hundreds of miles per h. Since the trains rarely touch the track, there is not much noise and vibration compared to regular trains. As a result, the Maglev train produces minor mechanical damage. The Central Japan Railway Company and the Railway Technical Research Institute developed the first Maglev superconductor trains in the 1970s. In April 2015, Japan Railway maglev trains recorded 603 km/h, far faster than Maglev trains operating in Shanghai, China (431 km/h to 500 km/h) and in South Korea (109 km/h). In the latest related development, Chuo Shinkansen maglev line is planned to connect Tokyo and Nagoya by 2027. The Chuo Shinkansen maglev line is expected to cover the 178-mile distance (Tokyo to Nagoya) at 500 km/h, slashing the travel time to just forty minutes. It means that the Chuo Shinkansen maglev line can reduce travel time by around 50% compared to the current Tokaido Shinkansen line.

1.3 Problem Statement and Research Objective

The main problem in the process of synthesising MgB_2 is determining suitable and optimal sintering conditions. This is because the optimal sintering value can reduce the grain size of the sample which increased the flux pinning at the grain boundary and enhanced the grain connectivity of the samples (Kobayashi et al., 2015; Matthews et al., 2020; Tanaka et al., 2012; Yamamoto et al., 2012). Unlike previous studies that varied the sintering conditions at high temperatures (Shim et al., 2005) and for a long time (Tanaka et al., 2012), this study is more focused on shorter sintering time (1-7 h) and lower sintering temperature (600-1000 °C).

Furthermore, another major issue for MgB_2 is that Mg is highly volatile and oxidised, especially at elevated temperatures. Addition of excess Mg is expected to compensate for the loss of Mg, reduce the formation of the MgO phase, and increase the grain connectivity, which can increase the value of J_c, especially at low temperatures and magnetic fields (Zeng et al., 2008; Zhang et al., 2015). However, prior studies (Arvapalli et al., 2019; Zeng et al., 2008; Zhang et al., 2015) focused on the addition of excess Mg to *in-situ* MgB_2 instead of *ex-situ* MgB_2. Both *in-situ* and *ex-situ* methods have their shortcomings. Although *in-situ* sample has strong grain coupling and easily reaches a high J_c value, it has a low bulk density and low connectivity. Meanwhile, *ex-situ* sample has a higher bulk density, but the grain coupling for *ex-situ* MgB_2 sample is weaker than *in-situ* MgB_2 sample (Li et al., 2012; Yamamoto et al., 2012). Therefore, in this work, both methods are combined to compensate for each other's shortcomings.

It is necessary to further improve the flux pinning and J_c value of MgB_2 sample to make it suitable for high magnetic field applications. This problem can be overcome by introducing dopants such as Si and LaB_6 into MgB_2. The addition of Si into MgB_2 can increase the value of J_c at a higher field (Wang et al, 2003). It has also been claimed that the addition of La_2O_3 resulted in the formation of LaB_6, which acts as an effective pinning center that increased the intragrain J_c (Gao et al., 2010; Shekhar et al., 2005). Hence, this study continues to focus on the co-addition of Si and LaB_6 to improve the flux pinning center and increase the value of J_c at all magnetic fields.

Similar to LaB_6, the formation of DyB_4 (Chen et al., 2006) can act as an effective flux pinning and increase the value of J_c. Although it is important to increase the value of J_c, the addition of dopants should not affect the value of T_c. Studies have shown that the addition of Dy_2O_3 (Chen et al., 2006) or La_2O_3 (Zhao-Shun et al., 2010) did not result in a drastic reduction of T_c, most likely due to the insignificant substitution of the lattice structure of MgB_2. Therefore, the co-addition of Dy_2O_3 and La_2O_3 into MgB_2 is expected to form LaB_6 and DyB_4 which can act as flux pinning centers and further improve the J_c value.

Hence, the objectives of this work are:

i. To enhance grain coupling of *ex-situ* MgB_2 via optimisation of sintering temperature and time.

ii. To elucidate the influence of materials addition such as excess Mg and (1.5 mol Mg + 2.0 mol B) on structural and superconducting properties of the *ex-situ* MgB_2.

iii. To investigate the effects of LaB_6, nano Si and co-addition of LaB_6 and nano-Si on structural and superconducting properties of *in-situ* MgB_2.

iv. To investigate the effects of co-addition of Dy_2O_3 and La_2O_3 on structural and superconducting properties of *in-situ* MgB_2.

1.4 Thesis Overview

This thesis consists of six chapters. Chapter 1 introduces a brief history of superconductors and their applications. The problem statement and the objectives of this work are also discussed in Chapter 1. Chapter 2 reviews the previous works of MgB_2 superconductors focusing on the preparation methods, heat treatment conditions, and dopant additions used by previous researchers. The theory and fundamentals of superconductivity, especially MgB_2, will be explained in Chapter 3. Chapter 4 focuses on the materials and methods used for this work. Sample characterisation, such as XRD, FESEM, and SQUID measurement, is also discussed in detail. Chapter 5 discusses all the analysed results obtained from all the sample characterisations. Finally, in Chapter 6, the outcomes of this research will be summarised, followed by recommendations for future research.

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