VIETNAM NATIONAL UNIVERSITY
UNIVERSITY OF SCIENCE

Pham Hoang Ha

ENHANCEMENTS OF CRITICAL CURRENT DENSITY OF MgB_2 BULK AND FILM SAMPLES USING ION IRRADIATION TECHNIQUE

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ABSTRACT OF Ph.D. DISSERTATION IN PHYSICS

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Supervisors:
1. Assoc. Prof. Tran Hai Duc
2. Dr. Nguyen Khac Man

Reviewer: Prof. Tran Hoai Nam
Phenikaa Institute for Advanced Study
- Phenikaa University

Reviewer: Assoc. Prof. Ho Khac Hieu
Duy Tan University

Reviewer: Assoc. Prof. Tran Dang Thanh
Institute of Materials Science - Vietnam Academy of Science and Technology

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ABSTRACT

Magnesium boron dioxide (MgB_2) superconductor, discovered in 2001, offers benefits such as a high critical temperature, high current density, simple crystal structure, low cost, and additional properties like weak-link free behavior, greater coherence length, and reduced electronic anisotropy. However, its practical applications are limited due to its rapid decline in magnetic field. This dissertation focuses on improving J_c of MgB_2 bulk and film samples, addressing weak link behavior in high temperature superconducting materials, requiring complex techniques.

First of all, the study aimed to fabricate MgB_2 bulk samples with the strongest superconductivity performance. *Ex-situ* MgB_2 powders were mixed with Mg and B, sintered at different times and temperatures. The optimal condition for fabricating MgB_2 bulk samples with the highest possible J_c value was found to Pure *ex-situ* MgB_2+ 0.5 Mg sintered at 1000°C for 1 hour.

Secondly, the study investigated the enhancement of J_c in MgB_2 bulk sample by adding impurities, with the greatest enhancement achieved with co-additions of B_4C and Dy_2O_3. The greatest J_c enhancement was obtained for the sample with the co-additions of 5 wt.% B_4C + 2.0 wt.% Dy_2O_3.

Lastly, the study investigated the deposition of high-quality MgB_2 thin films using hybrid physical-chemical vapor deposition (HPCVD). It was found that optimal irradiation of non-magnetic ions into MgB_2 films with a dose of 5×10^{13} ion/cm^2 was shown to be optimal for providing the highest J_c enhancement.

Chapter 1: INTRODUCTION

1.1. Superconductivity

1.1.1. History and crucial milestones

Superconductivity, a phenomenon where materials lose resistance below a critical temperature, was first observed in 1911 by Heike Kamerlingh Onnes. It led to the discovery of superconductors in various metals by the 1930s, with significant turning points including the Meissner effect, London equations, Ginzburg-Landau theory, and Josephson effect.

1.1.2. Classification

Superconductors are categorized into type-I and type-II groups based on their behavior in a magnetic field. Type-I superconductors, composed of about thirty pure metals, exhibit the full Meissner effect, allowing field penetration even at low fields. Type-II superconductors react differently, with coherence length determining vortex radius.

1.2. Vortex dynamics in superconductors

1.2.1. Flux pinning

The Lorentz force is caused by flux vortices in type-II superconductors, causing power dissipation. To prevent dissipation, a force opposing the Lorentz force is needed. Defects in superconductors provide vortex pinning sites, and the pinning force density (F_p) represents the total force applied to flux lines. Improving the density of defects can improve J_c.

1.2.2. Bean's critical state model

Bean's critical state model is utilized to calculate the J_c of superconductors from magnetization hysteresis loops. Inner filaments are protected by outer filament currents, which flow until the flux reaches the sample's core. The relationship between magnetization and J_c is represented by $J_c = a\Delta M/d$.

1.2.3. Dew-Hughes model

Dew-Hughes has created a model for describing flux-pinning in type-II superconductors, based on pinning center geometry and flux line interaction. The model calculates the pinning force per unit volume, ΔW and η, and the efficiency factor η. The flux lattice stiffness determines pinning force strength. The pinning process is defined by a curve plotting normalized volume pinning force against reduced magnetic field.

1.3. Collective pinning theory

Collective pinning theory is a study on the interaction between pierced vortices in type-II superconductors, focusing on field dependence of J_c and flux pinning mechanism in HTS samples. It was proposed in the 1980s, explaining how vortices form collective systems.

1.3.1. Collective pinning theory for bulk superconductors

The magnetic field induction in single vortex pinning is field independent when B is less than the crossover field, while small bundle pinning occurs when B > B_{sb}. Collective pinning theory studies type-II superconductors, vortice behavior, and thermal fluctuations.

1.3.2. Collective pinning theory for thin film superconductors

Larkin and Ovchinnikov introduced collective pinning in 1979, a method where multiple defects can significantly alter vortex dynamics. This method assumes that individual pinning forces on the vortex line add up arbitrarily, with variations in defects' density and force determining the total force for a vortex segment. The strength of samples can be assessed using the exponent β.

1.4. MgB_2 Superconductor and Subject to be discuss

1.4.1. Basic parameters

In 2001, Akimitsu's research group discovered superconductivity in magnesium diboride (MgB_2) with a 39 K T_c, a breakthrough in superconductivity history. MgB_2 has a hexagonal AlB_2 type crystal structure with a p6/mmm space group, containing boron and magnesium layers, and a graphite honeycomb network.

1.4.2. Superconducting mechanism in MgB_2

MgB_2 is a phonon-mediated BCS type superconductor with a layered structure with two superconducting energy gaps. Its electronic state at the Fermi level is characterized by boron atom bonding through covalent connections and metallic linkages. The two energy gaps are caused by electron coupling in 2D bands and weak coupling in 3D.

1.4.3. Properties of MgB_2 Superconducting Materials

The study explores the characteristics of MgB_2 superconductors, focusing on their high coherence lengths and low-temperature penetration depth, but also examining their structure due to oxygen, carbon, hydrogen, and uneven boron distribution, and their preparation methods including carbon, carbon-containing compounds, silicon carbide, titanium, tantalum, and zirconium.

1.4.4. Raman spectroscopy in MgB_2 superconductors

MgB_2's superconductivity is due to electron-phonon interactions, with the E_{2g} phonon mode being a single active mode. This Raman active mode, related to band electrical transport, can be studied using Raman scattering, providing data on crystal symmetry, particle size, and contaminants.

1.4.5. Potential application of MgB_2 superconductors

High temperature superconductor (HTS) and MgB_2 superconductors are popular for shields, DC magnetic field generation, and small/medium power motors. They trap magnetic fields, are lightweight, and can be fabricated using techniques like hot pressing and spark plasma sintering.

Figures 1.11–1.13 show equipment for bulk MgB2 production using high-pressing, hot-pressing, and spark plasma sintering techniques, producing large, stable blocks suitable for practical applications.

1.4.5.1. Trapped Magnetic Field (Quasi-Permanent Magnets)

Magnetized MgB_2 and HTS bulks offer quasi-permanent magnets with high magnetic fields, making them suitable for flywheel energy storage systems. MT-YBCO bulks trap 17.24 T magnetic fields but can be destroyed by Lorentz force. MgB_2 bulks are simpler, less expensive, and time-efficient, suitable for various applications.

1.4.5.2. Fault Current Limiters

Fast-operating nonlinear fault current limiters (FCLs) can limit high fault currents in power systems by increasing impedance. Superconducting materials have perfect conductivity and a quick phase transition, making them ideal for power systems. Inductive

SFCLs can protect high-voltage direct-current systems, and their circuit current and voltage drop remain unaffected by synthesis settings and ring sizes.

1.4.5.3. Electrical Machines

Superconductors replace metal wires in electrical machines, with advancements in electromagnetic characteristics enabling bulk-superconducting rotor elements. MgB_2 bulk superconductor-based motors are less expensive and more efficient, with potential applications in liquid hydrogen systems.

1.4.5.4. Magnetic Field Shields

Bulk MgB_2 superconductors exhibit excellent magnetic shielding properties, making them useful for passively protecting devices and orbital stations from cosmic radiation. Their basic ingredients are widely accessible and free of noble, poisonous, or rare earth elements. The shielding factors, which are based on the Hall probe position, reach their highest value around 105 at the cup's bottom.

1.4.6. Enhancements of critical current density of MgB_2 bulk superconductors

MgB_2 is a promising material for commercial and industrial applications due to its high coherence lengths, weak-link-free behavior, and simple hexagonal crystal structure. Researchers have developed methods to produce factitious pinning centers for high magnetic field applications. *Ex-situ* MgB_2 bulks were sintered at different temperatures and times, with additives like Mg and B increasing critical current density. Recent studies show self-feld J_c of MgB_2 bulk samples in the range of $2 - 4 \times 10^4$ A/cm^2.

1.4.7. Enhancements of critical current density of MgB_2 thin-film superconductors

1.4.7.1. Enhancements of critical current density of MgB_2 thin-film superconductors by using buffer layers

The study investigates flux pinning of MgB_2 films on buffered hastelloy tapes with different SiC layer thicknesses. Results show improved field dependence of J_c with increasing SiC layer thickness. Lattice mismatch between MgB_2 film, ZnO buffer layer, and Hastelloy substrate significantly influences phonon behavior.

1.4.7.2. Enhancements of critical current density of MgB_2 thin-film superconductors by using ion irrradiation

MgB_2 films offer advantages like high T_c, large coherence length, simple crystal structure, low cost, and high $J_c(0)$. However, their practical use is limited by the quick J_c(B) fall under a magnetic field. Ion irradiation has been studied to create artificial pinning sites in super conducting materials, improving in-field J_c and B_{c2} in MgB_2, iron-based superconductors, and high-T_c cuprates.

1.4.8. Subject to be discuss

MgB_2 superconductor offers low cost, two energy gaps, simple crystal structure, and high self-field J_c, but its practical applications are limited due to quick J_c decline in magnetic field. The thesis aims to enhance J_c of MgB_2 bulks and thin films. The overall structure of this dissertation is as follows:

- Fabrication conditions for pure MgB_2 bulk superconductors showing high T_c and J_c. Adding 0.5 mol Mg and sintering at 1000 ◦C for 1 hour, sample S5 showed the lowest value of T_c, whereas its J_c enhancement achieved the highest value.
- Enhancing J_c of MgB_2 bulk by co-addition of B_4C and Dy_2O_3. The coaddition of B_4C and Dy_2O_3 likely improved the grain connectivity, which enhance B_{c2} and J_c
- Highest J_c enhancement of MgB_2-thin films by ion irradiation. Sn ion irradiation showed an improvement in J_c at 5 and 20 K; the maximum J_c was reached for the 5×10^{13} atoms/cm^2 sample.

Chapter 2: EXPERIMENTAL METHODS

2.1. MgB_2 bulk sample fabrications

2.1.1. Pure MgB_2 bulk sample fabrications

MgB_2 bulk samples are prepared using *in-situ* or *ex-situ* methods. *In-situ* reactions involve melting Mg grains and diffusing them into B grains, resulting in high J_c and strong intergrain coupling. *Ex-situ* MgB_2 has a higher packing factor but weaker intergrain coupling. Heat treatment improves grain-to-grain current channel and maximizes MgB_2 phase production.

The study involved mixing *ex-situ* MgB2 with 0.5 mol of Mg to create Mg-added samples. In bulk MgB_2 samples, 1.5 mol of Mg and 2 mol of B were added. Sintering was performed at 600 and 700 ◦C for 1 hour, 3 hours at 700 ◦C, and 1 hour at 1000 ◦C.

2.1.2. MgB_2 samples co-added B_4C and Dy_2O_3

Magnesium diboride (MgB_2) powder was obtained from Alfa Aesar and mixed with 0.5 mol of Mg. Different weight percentages of B_4C were added to the MgB_2 samples, resulting in different samples. The mixture was ground, compressed into circular pellets, and heat-treated at 900 ◦C for 1 hour. The powder handling process was conducted in air. The samples are listed in Table 2.2. The process involved grinding, compression, and heat-treatment.

2.2. MgB_2 thin film sample fabrications

2.2.1. Hybrid Physical-Chemical Vapor deposition (HPCVD) system

The hybrid physical-chemical vapor deposition (HPCVD) method was used to create MgB_2 thin films, maintaining thermodynamic stability at high temperatures. The process includes a load-lock chamber, heater, reactor, gas inlet, flow, pressure, temperature, and exhaust systems, using diborane as boron precursor gas and magnesium pieces as sources.

2.2.2. Growth of MgB_2 thin films on Al_2O_3 substrates

The MgB2 thin-film growing process involves purging a chamber with Ar gas, filling it with a susceptor, substrate, and magnesium chips. Under ambient hydrogen gas, the substrate and chips are heated inductively, forming thin coatings. Single-crystal Al_2O_3 is used due to its hexagonal structure. The Volmer-Weber growth mode forms columnar structures, requiring superconducting samples for disorder impacts investigation.

2.2.3. Ion irradiation of MgB_2 thin film

A 412-nm-thick MgB_2 was created on c-cut Al_2O_3 substrates using a hybrid physical-chemical vapor deposition technique. High-purity magnesium particles were heated to 700 ◦C, and

B_2H_6 gas was supplied. The HUS-5SDH-2 tandem pelletron accelerator system was used for ion irradiation. Low-energy irradiation enhanced in-field J_c and $\mu_0 H_{c2}$, while rapid heavy-ion irradiation enhanced J_c in iron-based superconductors and high-T_c cuprates.

2.3. Measurement methods

2.3.1. X-ray diffraction (XRD)

X-ray diffraction (XRD) is a method used to study material crystal structures. The Bruker D8 Advance model was used to determine MgB_2 bulk and thin films. The study used Bragg's Law and XRD data for background determination, profile fitting, refinement, phase identification, and calculation of lattice constants.

2.3.2. Physical property measurement system (PPMS)

2.3.2.1. DC susceptibility

Susceptibility measurements were conducted using a direct current field on a surface using the Quantum Design PPMS EverCool II systems' vibrating sample magnetometer. The critical temperature (Tc) was measured between 10 and 50 K using zerofeld cooled conditions.

2.3.2.2. Resistivity vs. temperature measurement

The transport characteristics of MgB2 bulk and thin films were measured using a physical property measurement system (PPMS) with a magnetic field of up to ±9 Tesla and temperature change between 1.9 K and 400 K. The system's resistivity function was used to measure electro-transport.

2.3.2.3. Magnetization vs. field measurement

The Physical Property Measurement System (PPMS) was utilized for magnetization of MgB_2 bulk and thin films, with a resolution of less than 10-8 emu. The system, cryogen-free, used a cryostat, superconducting magnet, and measurement probes. Bean's critical state model was used to compute Jc from the magnetization hysteresis loop.

2.3.3. Raman spectrocopy

The WITec Micro-Raman Spectrometer System was used to measure Raman spectra of MgB_2's superconducting capabilities, revealing that the E_{2g} mode's active frequency affects T_c fluctuation, confirming the BCS theory.

2.3.4. Magnectic force measurement

The study aims to determine the absolute value of the pinning force for single Abrikosov vortices and explore the spatially resolved absolute value of the London penetration depth using a special He-4 MFM with vector magnet capabilities.

Chapter 3: FABRICATIONS OF PURE MgB_2 BULK SUPERCONDUCTORS

This chapter investigates the superconducting properties of MgB_2 bulk samples under various conditions. *Ex-situ* samples were mixed with Mg and B, sintered at different times and temperatures, and Raman spectroscopy was used to investigate electron-phonon coupling (EPC). The lowest EPC value was found to be 1.094 for the MgB_2 sample added with 0.5 mol Mg and sintered at 1000°C for one hour. The collective pinning theory revealed that δT_c pinning is the predominant flux pinning mechanism across all MgB_2 bulk samples.

This study investigates the effects of adding Mg and B to *ex-situ* MgB_2 bulks, which were sintered at different temperatures and times, on the temperature-induced displacement (T_c), joint conduction (J_c), and volume pinning force (F_p). Raman spectroscopy was used to analyze the effect of individual phonon properties on T_c. Results show that the displacement induced by Mg and B, along with sintering temperature, significantly influences T_c, J_c, and F_p mechanisms.

3.1. Effect of sample fabrication conditions on T_c of MgB_2 bulk superconductors

The superconductivity of samples is determined by T_c, which stabilizes at 38.5 K and drops to 38.1 K for sample S5. Additions like Mg and increasing sintering temperature can reduce transition temperature, but S5 samples have the lowest T_c.

The modification of strain-induced local structural deformity and disorder significantly impacted the T_c variation of MgB_2, possibly due to incomplete reaction between Mg and B during heat treatment. *Ex-situ* MgB_2 particles may deaccelerate phase formation. Raman spectra were used to understand T_c change in MgB_2 samples.

3.2. Explaining the possible reason for the changes in values of T_c of MgB_2 bulk superconductors

MgB_2 superconductors' T_c variations are caused by lattice distortions and electron-phonon interactions. Raman spectrum measurements show an increase in E_{2g} peak intensity and a decrease in half-width due to the addition of Mg and B. Lattice distortions, intense coupling, and phonon-phonon interaction may account for this shift. The addition of Mg and B reduces T_c and EPC value, leading to elevated impurity rates and significantly affecting the Raman spectrum.

3.3. Effect of sample preparation conditions on J_c of MgB_2 bulk superconductors

The study reveals that the addition of Mg and B to high-temperature superconductors enhances the flux pinning mechanism, resulting in increased J_c under various fabrication conditions. The results show that J_c is enhanced in samples S3, S4, S5, S6, and S7 at all temperatures and investigated fields. Sample S5 showed the highest improvement, while J_c descended slowly under the applied field. The study also found that adding Mg and B broadened the regime of small and large bundles, improving J_c at high external magnetic fields.

The J_c value of MgB_2 bulk samples increased significantly when sintered at 1000°C for one hour. The J_c value reached 1.2×10^5 A/cm^2, 5 times higher than previous research samples. The J_c value was 3.2 times higher at 0.5 T, 2.3 times higher at 1 T, 1.3 times higher at 1.5 T, and 1.2 times higher at 2 T.

3.4. Effect of sample preparation conditions on flux pinning mechanism of MgB_2 bulk superconductors

The study explores the flux pinning mechanism in MgB_2 bulk superconductor by adding Mg and B, focusing on improvements in J_c. The core interaction is the most efficient vortex-pinning center interaction in type-II superconductors. Two primary pinning mechanisms exist: δl pinning and δT_c pinning. The results confirmed the supremacy of the δT_c pinning mechanism in all MgB_2 bulks at different temperatures.

3.5. Effect of sample preparation conditions on geometry of pinning centers in MgB_2 bulk superconductors

The study explores the impact of adding Mg and B to MgB_2 bulk samples on flux pinning, focusing on the magnetic field dependence of F_p. The Dew-Hughes model was used to study supplementary flux pinning centers. The study found that the addition of Mg and B, along with temperature and sintering time, enhanced grain coupling and solid-state self-sintering, strengthening core surface pinning in MgB_2 bulk superconductors.

Conclusion of Chapter 3

The study investigates the local structure and superconducting properties of ex-situ MgB_2 bulk samples under different conditions. The electron-phonon (e-ph) and superconducting characteristics of MgB_2 bulk samples, such as T_c, J_c, and F_p, were examined. The results show that T_c remains constant for samples S1–S3, declines in samples S5, and increases in samples S6–S7. The EPC constant, determined by Raman spectroscopy, significantly impacts superconducting transition behavior. J_c increasing the highest in sample S5, Pure *ex-situ* MgB_2 + 0.5 Mg (1000 °C – 1 hour). The addition of Mg and B increased J_c(B) and sintering conditions, revealing growth of small and big bundle modes. The Dew–Hughes model was used to understand the role of Mg and B addition in the flux pinning process.

Chapter 4: ENHANCEMENTS OF CRITICAL CURRENT DENSITY OF MgB_2 BULK WITH B_4C AND Dy_2O_3 ADDITIONS

The study focuses on improving the grain coupling (J_c) of MgB_2 ceramic by adding rare earth oxides (REO) and carbon (C) to create efficient flux pinning centers. Boron carbide (B_4C) can be used as a precursor to form C-doped MgB_2 with high field-dependent J_c. ReO materials like Dy_2O_3 can also increase J_c. The study plans to use both B_4C and Dy_2O_3 to increase J_c of *ex-situ* MgB_2.

This chapter synthesizes MgB_2 bulk superconductors with B_4C and Dy_2O_3, confirming MgB_2 as the predominant phase. The properties are characterized by resistivity and magnetization measurements, with optimal additions enhancing B_{c2} and J_c. δl pinning is identified as the dominant mechanism.

This study investigated the effects of adding Mg, Dy_2O_3, and B_4C to MgB_2 ceramics, focusing on the crystal structure and superconducting properties. The pinning mechanism was analyzed using collective pinning theory, with core interactions driving random spatial variations in transition temperature (δT_c pinning) and fluctuations in the charge-carrier mean free path (δl pinning) in high-temperature superconductors. The dominant pinning mechanism was found to be coexistence of δl and δT_c pinning. The addition of B_4C and Dy_2O_3 likely improved grain connectivity and enhanced B_{c2} and J_c.

4.1. Crystal structure of MgB_2 bulk superconductors with co-additions of B_4C and Dy_2O_3

The XRD patterns of MgB_2 samples with 0.5 mol of added Mg and different concentrations of Dy_2O_3 and B_4C reveal hexagonal crystal structures, possibly due to oxygen incorporation during sample preparation. B_4C decreases MgB_2 and MgO peaks, while B_4C and Dy_2O_3 increase them.

4.2. Effect of co-additions of B_4C and Dy_2O_3 on T_c of MgB_2 bulk superconductors

The crystal structure of MgB_2 bulk with co-additions of B_4C and Dy_2O_3 is examined, revealing variations in superconducting transition and T_c values. The critical temperature (T_c) decreases with increasing coaddition content, and the transition width broadens with increasing coaddition content. Sample S5-1 has a T_c of 38.57 K, while sample S5-4 has a T_c of 34.70 K.

4.3. Effect of co-additions of B_4C and Dy_2O_3 on $\mu_0 H_{c2}$ of MgB_2 bulk superconductors

The study examined the effect of B_4C and Dy_2O_3 on the upper critical field of MgB_2 samples. It found that the superconducting transition width increased with magnetic field strength. The optimal dopant was 5 wt.% B_4C. The temperature dependence of B_{c2} showed a positive curvature, attributed to MgB_2's two-band superconductivity.

Previous studies showed that MgB_2 bulk samples doped with nanosized SiC, Carbon nanotube, B_4C, and Dy_2O_3 had B_{c2} values above 15 T. The MgB_2 + 0.5 mol Mg + 5wt.% B_4C (S5-2) sample has a higher B_{c2} value.

4.4. Effect of co-additions of B_4C and Dy_2O_3 on the improvement of J_c of MgB_2 bulk superconductors

Figure 4.3 displays M-H hysteresis loops of samples at different temperatures, with wider loops indicating increased J_c under different manufacturing conditions.

The field dependence of magnetization of samples was measured at 10 K using modified Bean's model. The self-field J_c of each sample was determined from magnetization hysteresis loops. The pure MgB_2 sample had a self-field J_c of 19 kA/cm², while the addition of Mg (sample S5-1) increased it. Samples S5-3, S5-4, and S5-6 showed improved J_c values under magnetic fields.

The J_c value of a sample with a 5 wt.% B_4C + 2.0 wt.% Dy_2O_3 coaddition improves by 1.5 – 3.2 times when subjected to a 0.1 to 2 T field at 10 K, as shown in Table 4.2.

The collective pinning theory explains vortex behavior in high-temperature superconductors by varying the influence of the magnetic field on J_c. Sample S5-5 showed increased B_{sb} and B_{lb} values, indicating the effectiveness of additional pinning centers.

4.5. Effect of co-additions of B_4C and Dy_2O_3 on flux pinning mechanism of MgB_2 bulk superconductors

Core pinning is classified into two dominant types: δl and δT_c. δl is attributed to variations in charge carrier mean free path near defect sites, while δT_c is related to fluctuations in the Ginzburg–Landau parameter. The dependence of J_{sv} on reduced temperature t differs from δl to δT_c pinning. The relationship between B_{sb} and J_{sv} is B_{sb}–$j_{sv}B_{c2}$. The coexistence of δl and δT_c pinning mechanisms was observed in samples, similar to impurity-doped bulk MgB_2. The role of δT_c pinning is more pronounced in higher-temperature regions.

4.6. Effect of co-additions of B_4C and Dy_2O_3 on geometry of pinning centers in MgB_2 bulk superconductors

The study examined the impact of adding B_4C and Dy_2O_3 to MgB_2 bulk samples, focusing on their pinning properties. The Dew-Hughes model predicted normal core-surface and core point pinning, with core-correlated pinning being dominant. The addition of B_4C and Dy_2O_3 may enhance connectivity between MgB_2 grains.

Conclusion of Chapter 4

The study investigates the improved flux pinning properties of MgB_2 bulk samples doped with B_4C and Dy_2O_3, revealing small lattice distortions. The greatest increase in J_c was observed in the sample with co-addition of 5 wt.% B_4C and 2.0 wt.% Dy_2O_3. The greatest increase in B_{c2} was observed in the sample with 5 wt.% B_4C. The dominant pinning mechanism was δl pinning, with two-dimensional pinning centers associated with grain boundaries. These findings can be used to enhance manufacturing parameters for MgB_2 bulk samples.

Chapter 5: ENHANCEMENTS OF CRITICAL CURRENT DENSITY OF MgB_2 THIN FILM WITH Sn^{2+} ION IRRADIATED

Ion irradiation significantly impacts in-field J_c and B_{c2} of MgB_2, with high-T_c cuprate superconductors often improving by columnar failures along ion tracks. Defects in type-II superconductors disrupt vortex movement, increasing essential superconducting characteristics like in-field J_c and B_{c2}. Understanding faults' impact on superconductivity is crucial for scientific development and technological adoption.

This chapter discusses the effects of 2 MeV Sn^{2+} irradiation on MgB_2 films grown on an Al_2O_3 substrate. The study used a hybrid physical-chemical vapor deposition technique and the HUS-5SDH-2 tandem pelletron accelerator system to expose 2 MeV energetic Sn ions to different doses, determining their mean projected ranges and damage events.

Raman spectroscopy was used to investigate changes in T_c and B_{c2} in MgB_2 films, a superconductor facilitated by phonons. The study found that ion irradiation displacement is essential for modifying T_c, in-field J_c, and B_c. The study also examined the relationship between EPC from Raman analysis and superconductivity properties, revealing the relationship between B_{c2} and thermodynamic critical fields.

5.1. Effect of Sn^{2+} ion irradiation on T_c and RRR of MgB_2 thin films

The study examined the temperature dependence of samples' resistivity to determine their superconductivity. Results showed that all samples exhibited metallic behavior in high temperatures, with a superconducting transition occurring as temperature steadily decreased. The irradiated samples showed a decrease in resistivity (T_c) due to lattice distortions in MgB_2 films. Ion irradiation altered T_c by altering local texture and strain-induced structural modification, such as deformity and clutter of the crystal lattice.

The residual-resistance ratio (RRR) measures the purity and structure of a superconductor based on temperature dependence. The 7E13 sample had the lowest T_c value at 30.47 K, with a decrease in RRR values with increasing ion dose, possibly due to lattice disorder after irradiation.

5.3. Effect of Sn^{2+} ion irradiation on electron-phonon coupling properties of MgB_2 thin film superconductors

Raman scattering is used to study the E_{2g} phonon mode in MgB_2, a phonon-mediated superconductor. The Raman spectrum of Sn^{2+} irradiated films shows peaks at 750 cm^{-1} and 792 cm^{-1}, with weak influence on critical temperature. As irradiation dose increases, phonon density of states peaks increase. The relationship between Raman signals and superconducting properties is explained by the McMillan formula and Bardeen–Cooper–Schrieffer theory.

5.4. Effect of Sn^{2+} ion irradiation on the improvement of J_c of MgB_2 thin film superconductors

The study explores the enhancement of in-field J_c in MgB_2-thin films at 10 K. The pristine films had a substantial J_c at zero field, which disappeared quickly in magnetic fields. Sn^{2+} ion irradiation significantly improved the in-field J_c, matching the pristine sample's value at 1.33 T. Sample 5E13 showed the best J_c value at 10 K, with an irreversible field increasing more than three times from 2.23 to 6.49 T.

The J_c value of MgB_2 thin film samples was found to be 2.5×10^6 A/cm^2 when irradiated with Sn^{2+} ions at a dose of 5×10^{13} ions/cm^2 and 2 MeV, a 2.5 times higher value than previously reported values.

5.5. Effect of Sn^{2+} ion irradiation on B_{c2} of MgB_2 thin film superconductors

The study explores the effect of Sn^{2+} irradiation on the B_{c2} of MgB_2 samples in a magnetic field. It finds that Sample 5E13 is the optimal fluence for increasing B_{c2}, while pristine samples have a pristine $B_{c2}(0)$ of 5.889 T. Sn^{2+} irradiation enhances $B_{c2}(0)$ but suppresses T_c suppression. The study suggests that a superconducting layer enhances superconducting characteristics while reducing T_c.

Previous studies showed that MgB_2 films irradiated with He and Co ions had B_{c2} values of 8.9 and 9.5 T, respectively. Sn^{2+} ion irradiation, at a dose of 5×10^{13} ions/cm^2 and 2 MeV, had a B_{c2} value of 12.941 T.

5.6. Observations of magnectic vortex cores MgB_2 thin films before and after Sn^{2+} ion iradiation

The study used a homemade ^{4}He probe to measure magnetic force gradients in superconducting samples. Homogeneous vortices were observed in Nb and pristine MgB_2 samples, while sample 5E13 showed inhomogeneity. The Meissner response force gradient was used to screen the magnetic field. The pristine and 5E13 samples had larger penetration depths.

5.8. Comparision of doping effect on the J_c of MgB_2 bulk superconductor and ion irradiation effect on the J_c of MgB_2 thin films

The study systematically examined the impact of fabrication conditions on the J_c of MgB_2 bulk samples, revealing that pure ex-situ MgB_2 + 0.5 Mg sintered at 1000 °C for 1 hour increases J_c the most. The study also explored the effect of doping on J_c, with the greatest enhancement achieved with 5 wt.% B_4C + 2.0 wt.% Dy_2O_3. The research also focused on MgB_2 thin film superconductor, with the strongest increase observed when irradiated with Sn^{2+} ions.

Conclusion of Chapter 5

The study explains variations in electron-phonon and superconductivity properties in MgB_2-thin film samples by irradiation with Sn^{2+} ions. The study found a high correlation between the E_{2g} mode and T_c, suggesting the desired E_{2g} mode in MgB_2. The behavior of superconducting transitions is significantly influenced by the EPC constant, suggesting disorder strength influences the superconducting process of MgB_2. The value of T_c gradually decreased, whereas the values of J_c and B_{c2} increased and reached a maximum at 5E13. Moreover, the opposition between the high value of $\mu_0 H_{irr}$ and low value of β can improve J_c values. The study suggests MgB_2-thin films irradiated with Sn^{2+} ions are attractive for power applications.

CONCLUSION

The present dissertation delves into the investigation of the critical current density and flux pinning mechanism in MgB_2 superconductors. The main findings of this dissertation, which include advances in J_c in MgB_2 superconductors, can be summed up as follows:

Initially, the fabrication of MgB_2 bulk samples was optimized by using the conventional solid state reaction technique. By adjusting the additions of starting materials of Mg and B as well as the sintering temperature, critical temperature (T_c), critical current density (J_c), and the flux pinning force density (F_p) of the MgB_2 bulk were found to vary. The variation of T_c was found to link to the corresponding variation in electron-phonon coupling and the formation of phonon density of state of the samples probed by Raman spectra. The J_c enhancements were attributed to the improvements of grain connectivity, which was believed to effectively worked as 2D pinning centers by using the Dew-Hughes model. Therefore, the optimal sample fabrication condition to increase J_c was MgB_2 + 0.5 Mg sintered at 1000 °C for 1 hour.

To the further enhancements of J_c of MgB_2 bulk, the additions of proper impurity into solid-state reacted MgB_2 bulk were carried out. By applying the optimal fabrication conditions of MgB_2, the improved flux-pinning properties of MgB_2 bulk samples with co-additions of B_4C and Dy_2O_3 were investigated. The greatest J_c enhancement of ~ 1.5 – 3.2 times in the range of 0.1 to 2 T at 10 K was obtained for the sample with 5 wt.% B_4C + 2.0 wt.%

Dy_2O_3. More interestingly, the co-additions of B_4C and Dy_2O_3 were shown to provide the enhancements of B_{c2}, with the maximum B_{c2} was achieve for the 5 wt.% B_4C added MgB_2 bulk sample up to 1.6 – 1.7 times. The improvements of grain connectivity and the dominance of 2D pinning centers in B_4C and Dy_2O_3 added MgB_2 bulk were also revealed by using the Dew-Hughes model. The evidences were likely to be reasons for enhancements of J_c and B_{c2}.

To further satisfy the power related application of MgB_2, the nearly single crystalline MgB_2 thin films were fabricated by using HPCVD technique. The J_c of the MgB_2 films was found to be much higher than that of the MgB_2 bulk and the dominant pinning mechanism was still identified to be 2D pinning centers. The addition of different kind of pinning centers was performed by using the ion irradiation technique. The Sn^{2+} ions were irradiated into and expected to thoroughly pass the MgB_2 films. Evidence for the formation of point-like defects in form of ion track or crystal imperfections were indicated by the shifts in the XRD and Raman E_{2g} peaks. By varying the Sn^{2+} ion dose from 2×10^{12} to 7×10^{13} ion/cm^2, the obvious enhancements of J_c and B_{c2} were reached, those were ~ 2.5 – 1000 times in the range of 0.1 to 2 T at 10 K and 1.4 – 1.5 times, respectively. The pinning strength - obtained by using the collective pinning theory - of the Sn^{2+} irradiated MgB_2 films was found to be improved. The pinning mechanism was slightly shift from 2D to 1D pinning as suggested by the Dew Hughes model, which was possibly originated from the point-like defects. The researches on irradiation process to improve critical parameters of superconductors might be expanded with different kinds of ions.

DISSERTATION PUBLICATIONS

[1]. **Ha H. Pham,** An T. Pham, Phi Thi Huong, Nguyen Hoang Nam, Nurhidayah M. Hapipi, Soo Kien Chen, Muralidhar Miryala, Dzung T. Tran, Jungseek Hwang, Yu Seong Seo, Dang T. B. Hop, Nguyen Thanh Binh, and Duc H. Tran. (2023). Correlation between electron-phonon coupling and superconductivity of Sn2+ ion irradiated MgB2-thin films. *Ceramics International*, *49*(12), 20586–20593. https://doi.org/10.1016/J.CERAMINT.2023.03.188

[2]. **Ha H. Pham,** Tien Le, An T. Pham, Won Nam Kang, Nguyen H. Nam, Do T. K. Anh, Nguyen Hoang Anh, Hoo Keong Peh, Soo Kien Chen, Nguyen K. Man, Vuong Thi Anh Hong, Hanoh Lee, Tuson Park, Le Viet Cuong, Phan Hai, Nguyen Quang Huy, and Duc H. Tran (2024). Improved flux pinning properties of Mg exceed MgB2 ceramics with B4C and Dy2O3 additions. *Ceramics International*, *50*(23), 51509–51515. https://doi.org/10.1016/J.CERAMINT.2024.10.069

[3]. **Ha H. Pham,** Tien Le, The Nghia Nguyen, Nguyen Hoang Nam, Nhung T. Nguyen, Min Kyun Sohn, Dae Joon Kang, Tuson Park, Jinyoung Yun, Yeonkyu Lee, Jeehoon Kim, Duc H. Tran, and Won Nam Kang (2023). Role of electron–phonon coupling in the superconducting state of MgB2 polycrystals fabricated at different conditions. *Ceramics International*, *49*(21), 34053–34061. https://doi.org/10.1016/J.CERAMINT.2023.08.105