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BOLU ABANT İZZET BAYSAL UNIVERSITY
INSTITUTE OF GRADUATE STUDIES
Department of Physics



**PRODUCTION OF MgB_2 SUPERCONDUCTING WIRES AND
JOINTS BY MAGNESIUM DIFFUSION METHOD AND
THEIR CHARACTERIZATION**

DOCTOR OF PHILOSOPHY

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BOLU, DECEMBER 2023

APPROVAL OF THE THESIS

PRODUCTION OF MgB₂ SUPERCONDUCTING WIRES AND JOINTS BY MAGNEZYUM DIFFUSION METHOD AND THEIR CHARACTERIZATION submitted by Doğan AVCI and defended before the Examining Committee Members listed below in partial fulfillment of the requirements for the degree of **Doctor of Philosophy** in **Department of Physics, Institute of Graduate Studies of Bolu Abant İzzet Baysal University** in **28.12.2023** by

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ABSTRACT

PRODUCTION OF MgB₂ SUPERCONDUCTING WIRES AND JOINTS BY MAGNESIUM DIFFUSION METHOD AND THEIR CHARACTERIZATION

PHD THESIS

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BOLU, DECEMBER 2023

XVIII + 135

The aim of the thesis study is to design and implement innovative production methods to solve current problems encountered in IMD MgB₂ wires, such as unreacted boron clusters and layer, low engineering critical current density (J_{ce}), superconducting core non-uniformity along the wire, and joining difficulties caused by the thin MgB₂ ring layer. In the thesis study, designed wires were produced using non-stoichiometric Mg+B tablets (RD-IMD) and triple thin Mg rods (TH-IMD) in addition to the standard IMD (S-IMD) wire production in which Mg rod is centered in the boron powder. Using the RD-IMD method, MgB₂ wire was made with a porous structure instead of a large hole in the middle and a dense circular MgB₂ layer around this porous structure. In this way, it has been shown that a higher J_{ce} can be achieved compared to the S-IMD wire in self field. With another design called the TH-IMD method, it has been shown that the electrical properties are improved compared to the S-IMD wire by using three thin Mg rod sources in triangular geometry instead of using a central Mg rod. With this design, the length at which magnesium will diffuse into the boron during the heat treatment is reduced, ensuring that Mg spreads evenly throughout the boron layer. Finally, a special filling method designed to be half Mg and half B (H-EMD) was used to make the thin MgB₂ ring surface formed in the S-IMD MgB₂ wire more effective. It has been demonstrated that the effective MgB₂ surface obtained by the H-EMD method contributes to better contact between the superconducting joint bulk and the wire core and to reducing the critical current difference between the superconducting wires and the joint.

KEYWORDS: Superconductor, MgB₂, Wire, Joint, Magnesium Diffusion

ÖZET

MAGNEZYUM DİFÜZYON YÖNTEMİYLE MgB_2 SÜPERİLETKEN TEL VE EKLEMLERİN ÜRETİMİ VE

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BOLU, ARALIK - 2023

XVIII + 135

Tez çalışması kapsamında IMD MgB_2 tellerinde karşılaşılan reaksiyona girmemiş bor kümeleri ve bor katmanı, düşük mühendislik kritik akım yoğunluğu (J_{ce}), tel boyunca süperiletken öz düzgünsüzlüğü ve ince MgB_2 halka tabakasından kaynaklanan eklemleme zorlukları gibi güncel problemlerin çözümüne yönelik yenilikçi üretim yöntemlerinin tasarlanması ve uygulanması amaçlanmıştır. Tez çalışmasında önerilen yenilikçi yöntemler ile standart IMD (S-IMD) üretim yöntemine alternatif olarak merkezi Mg çubuk yerine stokiyometrik olmayan magnezyumca zengin Mg + B tabletlerin (RD-IMD), ve üç adet ince Mg çubuğun üçgen geometri (TH-IMD) düzeninde kullanıldığı tel üretimleri gerçekleştirilmiştir. Tasarlanmış iç Mg difüzyon işlemi adı verilen RD-IMD üretim yöntemi kullanılarak üretilen MgB_2 telinde S-IMD telinin merkezinde oluşan büyük boşluk yerine gözenekli bir MgB_2 yapısı ve bu yapıyı çevreleyen yoğun dairesel bir MgB_2 tabakası başarılı bir şekilde oluşturulmuştur. Bu sayede standart IMD teline kıyasla öz manyetik alan altında daha yüksek bir mühendislik kritik akım yoğunluğu elde edilebileceği gösterilmiştir. Bunun yanı sıra bir diğer yenilikçi tel üretim yöntemi olan TH-IMD yöntemi ile bor tozu merkezinde tek bir Mg kaynağı kullanmak yerine üçgen geometride yerleştirilmiş üç adet ince Mg çubuk kullanılmıştır. Mg'nin bor içerisinde difüze edeceği mesafe azaltılıp homojen bir şekilde yayılması sağlanarak telin elektriksel özelliklerinin geliştirilebileceği gösterilmiştir. Son olarak, S-IMD MgB_2 tellerde oluşan ince MgB_2 katman yüzeyinin daha etkin hale getirilmesi üzerine tasarlanan yarısı Mg diğer yarısı B olan özgün bir dolum yöntemi ile tel (H-EMD) ve süperiletken eklem üretimleri gerçekleştirilmiştir. H-EMD yöntemiyle elde edilen etkin MgB_2 yüzeyinin, süperiletken bağlantı ile tel çekirdeği arasındaki temasın daha iyi olmasına ve süperiletken teller ile bağlantı noktası arasındaki kritik akım farkının azaltılmasına katkıda bulunduğu kanıtlanmıştır.

ANAHTAR KELİMELER: Süperiletken, MgB_2 , Tel, Eklem, Magnezyum Difüzyonu

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LIST OF ABBREVIATIONS AND SYMBOLS

PIT	: Powder In Tube
PeIT	: Pellet In Tube
HIP	: Hot Isostatic Pressure
IMD	: Internal Magnesium Diffusion
LIMD	: Localized Internal Magnesium Diffusion
RLI	: Reactive Liquid Infiltration
EMD	: External Magnesium Diffusion
CHPD	: Cold High Pressure Densification
CTFF	: Continuous Tube Filling and Forming
MRI	: Magnetic Resonance Imaging
FCL	: Fault Current Limiter
SMES	: Superconducting Magnetic Energy Storage
LTSs	: Low Temperature Superconductors
HTSCs	: High Temperature Superconductors
ZFC	: Zero Field Cooling
NS	: Normal-Superconductor
GL	: Ginzburg-Landau
BCS	: Bardeen-Cooper-Schrieffer
DOS	: Density of State
EDS	: Electron Dispersive X-ray Spectroscopy
SEM	: Scanning Electron Microscope
XRD	: X-ray Diffraction
PVZ	: Pavezyum
PC	: Persistent Current
RRR	: Residual Resistance Ratio
EDAX	: Energy Dispersive X-ray Analysis
CCR	: Critical Current Ratio
OD	: Outer Diameter
ID	: Inner Diameter
SS	: Stainless-Steel
2D	: Two-Dimension
J_c	: Critical Current Density

J_{ce}	: Engineering Critical Current Density
J_e	: Engineering Current Density
T_c	: Critical Temperature
B_{c1}	: Lower Critical Field
B_{c2}	: Upper Critical Field
B_{irr}	: Irreversibility Field
I_c	: Critical Current
μ_0	: Permeability of Free Space
α	: Isotope Effect Coefficient
Φ	: Flux
κ	: Ginzburg-Landau Parameter
ξ	: Coherence Length
λ	: Penetration Depth
ψ	: Wave Function
h	: Planck Constant
M	: Magnetization
χ	: Magnetic Susceptibility
e	: Electron Charge
G	: Gibbs Free Energy
F_p	: Pinning Force
n	: Electron Density
γ	: Anisotropy Factor
A_f	: Active Cross-Sectional Area
ρ	: Resistivity
V	: Voltage
R	: Resistance
DC	: Direct Current
AC	: Alternative Current
I	: Current
K	: Kelvin
°C	: Degrees Celsius
A	: Ampere
T	: Tesla
cm	: Centimeter

Θ	: Bragg-angle
E_f	: Fermi Energy
μ	: Micro
mm	: Millimeter
Ω	: Ohm
$\parallel$	: Parallel
LHe	: Liquid-Helium
LH2	: Liquid-Hydrogen
GPa	: Giga Pascal
MPa	: Mega Pascal



ACKNOWLEDGEMENTS

I would like to thank my supervisor Prof. Dr. Hakan Yetiş for his advices and guidance throughout the research. He has always encouraged me to pursue innovative approaches during the experimental studies. He helped me greatly in the process of formation of the thesis.

I am also grateful to Assoc. Prof. Dr. Fırat Karaboğa for his contributions and suggestions to my thesis. He always helped me through the experimental study for my thesis. I would also like to thank Prof. Dr. İbrahim Belenli and Assoc. Prof. Dr. Canan Aksoy for their suggestions, comments and support during the thesis process. I would also like to thank Prof. Dr. Mustafa Akdoğan for contribution to construction of pulse measurement setup. I am grateful to Dr. Daniel Gajda, Dr. Michal Babij, and Dr. Lan Maria Tran due to their contribution to the electrical characterization measurements of wires and joints. I extend my heartfelt thanks to Mehmet Örgeç for standing by me through the challenges I encountered in the final phase of my thesis studies. Additionally, I am grateful to Kaan Yüksel Oyulmaz, Özgün Karadeniz, Umut Keskin, Sinan Öztel, and Murat Erdoğan for their invaluable support throughout the thesis research and writing journey.

I express my sincere gratitude to my mother, Bedriye Avcı, my sister, Ecem Avcı Aksöz, my brother, Umut Aksöz and my father, Erdoğan Avcı, for their unwavering tolerance, affection, encouragement, and assistance during the duration of my thesis research.

I express my gratitude to the Council of Higher Education for their support through the 100/2000 Doctoral Scholarship Program.

This study was supported by the TÜBİTAK, Grant No: 119M288

1. INTRODUCTION

Superconductors are playing an important role in the production of advanced technology devices day by day. Superconducting wires and tapes are used in many high-tech applications such as small volume high magnetic field magnets, wind turbine generators, magnetic energy storage (SMES), energy transfer lines, MRI, and electric motors, due to their high current carrying capacities. Today, the most common commercial application of superconductivity is in large coils that produce uniform, stable, and high-intensity magnetic field required for MRI machine. For commercial MRI coils, NbTi superconductor which is a member of low-temperature superconductors is used because high-temperature superconductors are not ready for cost-effective production of high-field magnets. However, the fact that NbTi has a critical temperature as low as 9.3 K is the biggest disadvantage of the NbTi conductor, which requires operation at liquid helium temperature. In recent years, there has been a notable increase in the demand for liquid helium, mostly driven by technological advancements and the use of liquid helium in various research studies. Unfortunately, due to its non-renewable nature, the availability of helium is limited, and there will inevitably come a time when the existing supply will not be able to meet increasing demand. Work is underway on an MRI system that does not require liquid helium, but these systems are not yet commercially available. In this respect, MgB₂ coils, which can be cooled to around 20 K with a cryo-cooler are of great importance in terms of cost and ease of use. It is generally accepted that the use of superconducting MgB₂ materials with a high $T_c = 39$ K in the superconducting coils of MRI systems for temperature regions of 10-20 K will bring significant technical and financial advantages.

For the construction of a large MgB₂ coil, it is necessary to produce kilometers of superconducting wire. MgB₂ wires may be fabricated using the PIT technique, which involves filling a metallic tube with a mixture of magnesium and boron powders (Mg + 2B). This process enables the production of MgB₂ wires at a kilometer scale. Nonetheless, the utilization of the IMD method has notable advantages over the PIT approach in terms of achieving a substantially higher critical current density under high magnetic fields. Consequently, the IMD method proves to be highly beneficial for the replacement of NbTi superconductors with MgB₂ in MRI superconducting coils. However, the heat treatment process in IMD

wires results in the formation of a significant central hole which is caused by the diffusion of the central magnesium (Mg) into the surrounding boron layer. Therefore, the engineering critical current density in IMD wires is lower than in PIT wires. As a result, production and joining of MgB₂ wire with a long but uniform core structure by IMD process and increasing the J_{ce} value are very important for large-scale superconducting coil applications.

The thesis covers the production of standard/designed MgB₂ IMD wires, superconducting joints and electrical/structural characterization measurements of the produced wires and joints. The results of experiments which reveal the improvement of the superconducting core region of MgB₂ wires and the joint of IMD wires are presented. The transport, electromechanical, and structural properties of single-filament MgB₂ wire produced using a redesigned IMD (RD-IMD) method based on the use of non-stoichiometric Mg+2B tablets containing excess Mg were examined. Structural analyses revealed that a porous MgB₂ structure was successfully formed in the center of this RD-IMD wire and a dense circular layer of MgB₂ surrounding this structure after reaction. The RD-IMD method allowed increasing the engineering critical current density of IMD wires in self-field. The mono filament MgB₂ wires with outer diameter of 0.81 mm were also produced by using three-hole IMD (TH-IMD) method. In this method, three thin Mg rods arranged in triangular geometry were used instead of using one Mg rod in the center of the wire. The purpose of this method is to reduce unreacted boron regions, and the production of TH-IMD wire designed for this purpose has been achieved. It has also been shown that the critical current of IMD wires is improved under an external high magnetic field with the TH-IMD method. The bending strain tolerance values of all wires were determined by many transport measurements on different wire. Using large numbers of wires for electromechanical experiments is challenging but reliable.

Superconducting joint is required to operate MgB₂ magnets in persistent current mode, as high-resolution MR images require an extremely stable magnetic field with $< 0.1 \text{ ppm.h}^{-1}$ decay rate. An MRI coil wound with a superconducting MgB₂ wire can be realized by combining individual coils in series. However, a very low joint resistance is needed in terms of current losses and heat generation. However, reliable superconducting joints, especially for the reacting MgB₂ wires, have not yet been fully optimized. The joints of S-IMD/H-EMD MgB₂ wires,

critical current measurements of joints and joined wires, resistance measurement and structural characterization of the joints were performed. In order to reduce the joint resistance, wire production was completed by forming Mg/B semi-cylindrical structure along the entire length of wire. With this new production method, an effective superconducting MgB₂ surface was formed in comparison to the circular thin MgB₂ layer in IMD MgB₂ wires. It was observed that the bulk type MgB₂ core reduced of I_c difference between MgB₂ superconducting wire and the joint.

In the second chapter of the thesis, a summary of the literature on MgB₂ material is given. In the third chapter, the production details for the wires and joints are presented. In the fourth chapter, some details about the experimental studies of the thesis are given. In the fifth chapter, structural, electrical, and electromechanical characterization measurement results for the wires are presented. Joint resistance measurements and structural features of the joints are also included in this chapter. In the last chapter, there are discussions on the results obtained and their important outputs.

1.1 Background of superconductivity

Heike Kamerlingh Onnes., a Dutch physicist, made the first discovery of superconductivity in 1911. Superconductivity is the ability of some materials to conduct an electric current without resistance. He used liquid helium to reach temperatures close to absolute zero (0 K), at which the electrical resistance of mercury completely disappeared. This breakthrough led to the study of many other materials with similar properties. The phenomena of superconductivity challenge the accepted theory of electrical resistance found in typical conductors. The superconducting state, characterised by zero electrical resistance and the repulsion of magnetic fields (the Meissner effect), is a state that superconducting materials have at low temperatures. Since then, many fields, including quantum computing, medical imaging and energy transfer, have found applications for this unusual behaviour.

Superconductors are rare materials having zero electrical resistance under a specific critical temperature as well as exceptional diamagnetic characteristics. Every superconducting material has a specific critical temperature and critical magnetic field value below which superconductivity can persist. Above these

critical temperature and field, the material loses its superconducting properties. The material will have a magnetic field approximately equal to the applied magnetic field because it is not in the superconducting state when a magnetic field is applied externally to a superconducting material at a temperature above its critical temperature. The magnetic field lines will be forced out of the superconductor surface and the magnetic field inside the superconductor will be zero if the temperature is decreased below the critical temperature. The superconductor exhibits outstanding diamagnetic properties. The Meissner effect (Schmidt, 1997) describes how the magnetic field is excluded from a superconductor's interior. The difference between superconductors and perfect conductors emerges when they are cooled to their critical temperatures by exposure to an external magnetic field. An ideal conductor does not maintain a zero internal magnetic flux value when the external field is removed after cooling under the field, whereas the magnetic field is always zero within the superconductor.

In 1957, John Bardeen, Leon Cooper, and Robert Schrieffer presented the theoretical justification for superconductivity. Their important research often referred to as the BCS theory explained how interactions with the lattice vibrations (phonons) cause electrons in a superconducting material to form pairs (Cooper pairs), which are then able to conduct electricity. As a result of the attractive forces acting on these pairs, they are able to pass across the lattice without dispersing and meet no electrical resistance. As superconductivity research continued to advance, new types of superconductors were discovered. These can be broadly divided into two categories: conventional (Type I) superconductors, which require very low temperatures, and high-temperature (Type II) superconductors, which exhibit superconductivity at temperatures higher than nitrogen's boiling point (77.355 K). By making it easier to cool materials to extremely low temperatures, Bednorz and Müller's discovery of high-temperature superconductivity in the late 1980s opened up new opportunities for practical applications of superconductors.

Type I superconductors prevent the penetration of magnetic flux into them up to a certain critical magnetic field (B_c) when an external magnetic field (B) is applied in the region $T < T_c$. The magnetic field value inside the superconductor is zero and the superconductor is in the Meissner state. However, when B is higher than the B_c value of the superconductor, the magnetic field completely penetrates the sample and destroys the superconducting state. In contrast to type I

superconductors, type II superconductors have two critical magnetic field values, B_{c1} and B_{c2} . If the external magnetic field intensity is less than B_{c1} , which is called the lower critical magnetic field, the magnetic flux in the sample is completely excluded. Type II superconductors up to B_{c1} exhibit magnetic properties similar to type I superconductors. The magnetic properties of type II superconductors are completely different in applied magnetic fields between B_{c1} and B_{c2} . Unlike type I superconductors, above B_{c1} the magnetic field partially penetrates the sample rather than completely. A mixed state, also known as the vortex state, occurs where normal and superconducting regions coexist. The magnetic flux enters the superconductor in a unique form known as a vortex in the $B_{c1} < B < B_{c2}$ range. A vortex has a normal core that is encircled by supercurrents. The magnetic field at the vortex center equals to external field due to the super-currents move around the core. The magnetic flux quantum for each vortex is $\Phi_0 = h/2e$. Here h is Planck's constant and e is the electron charge. Above the upper critical field value of B_{c2} , a phase transition from the superconducting state to the normal state occurs since normal cores of the vortices overlap.

Superconductivity is being used, prototype or real, in wind turbine generator, SQUID, MRI, particle accelerator and maglev train to provide clean energy, precise measurements and high-performance solutions. Superconducting qubits are at the forefront of quantum computing research and promise unheard of computing power. Our power grids can become more energy efficient and secure with the use of superconductors, which have the potential to revolutionize energy transmission and storage systems.

1.2 Critical parameters of superconductivity

The basic parameters related to superconductivity cover different properties that describe the state or behavior of a material during its transition to the superconducting state. The parameters covered in this study consist of the critical temperature (T_c), which expresses the temperature at which a material exhibits superconductivity. When the temperature of the material drops below the critical temperature, a phase transition occurs, causing the material to change from behaving as a normal conductor to becoming a superconductor. The critical magnetic field (B_c) refers to the upper limit of the external magnetic field that a

superconductor can withstand without compromising its superconducting characteristics. Once the magnetic field strength above a certain threshold, superconductivity ceases to exist and the material returns to its conventional conductive state. The critical current density (J_c) is a parameter that characterizes the maximum current density at which a superconducting material may carry electrical current without losing superconductivity. This parameter specifies the upper limit of the electric current that the material can carry when it is in the superconducting phase.

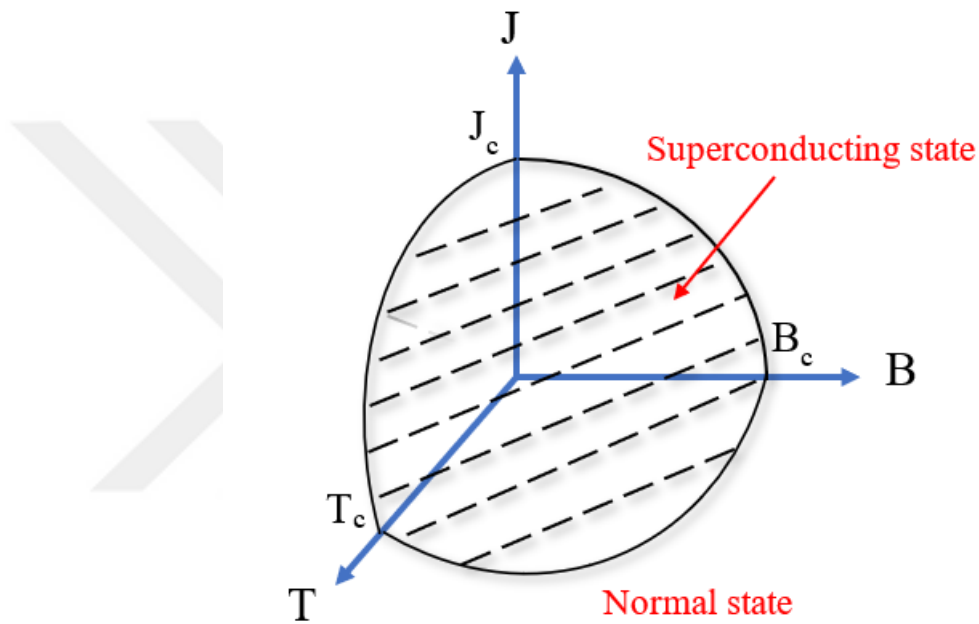


Figure 1.1. The phase diagram of superconductivity.

1.3 Overview of diffusion

Diffusion refers to the displacement of particles (comprising atoms, molecules, or ions) from regions characterized by higher concentrations to those exhibiting lower concentrations, thus facilitating the achievement of a homogenized distribution within a given medium. This phenomenon manifests itself in various media such as gases, liquids, and solids, which is of great importance in scientific fields such as physics, chemistry, biology, and materials science. Rate and extent of diffusion; It depends on a variety of influencers, including temperature, pressure differences, concentration gradients, and inherent characteristics of the medium.

The origin of solid-state diffusion dates back to the 19th century, based on Robert Brown's (1773 - 1858) seminal observations of Brownian motion, in which atoms or molecules move randomly. Diffusion, albeit slowly, occurs in solids through this movement, it exhibits a diffusion distance of approximately one micrometer per second near its melting point and only one nanometer per second at half that temperature. The theoretical framework of diffusion in continuous media finds its pioneering in the specific efforts of Adolf Eugen Fick (1829 - 1901) (A. E. Fick, 1855), these studies were catalyzed by Thomas Graham's (1805 - 1869) pioneering work to explain salt diffusion in water (T. Graham, 1833). Albert Einstein (1879 - 1955) facilitated the understanding of Brownian motion by proposing a relationship between mean square displacements and diffusion coefficient (A. Einstein, 1905). William Chandler Roberts-Austen (1843-1902) initiated the study of solid diffusion through meticulous studies of gold diffusion in solid lead, supported by contemporary gold radioisotope-derived diffusion coefficients (J. Groh *et al*, 1921).

The inception of solid-state physics, attributed to Max von Laue (1879-1960), were based on his discovery on the periodic arrangement of atoms in solids through X-ray diffraction on crystals. However, perfect crystals deviate from reality, prompting Jakov Il'ich Frenkel's (1891-1952) proposition of atom migration from lattice to interstitial positions due to thermal agitation, leading to lattice vacancies (J. I. Frenkel, 1926) Wagner and Schottky (C. Wagner, W. Schottky, 1930) later investigate the disorder in binary compounds and elucidated the existence of vacancies, interstitials, and antisite defects. The accepted role of atomic imperfections as facilitators of core and solution diffusion in solids is a prevailing concept (A. E. Fick, 1855). Ernest Kirkendall (1914 - 2005) contributed significantly to understanding interdiffusion in copper and brass diffusion pairs, emphasizing the Kirkendall effect in which the interface between these materials exhibits movement, indicative of different diffusion rates (A. D. Smigelskas, E.O. Kirkendall, 1947). This observation provided convincing evidence for mechanisms such as direct exchange or ring mechanisms governing atomic diffusion.

1.3.1 Fick's law

Diffusion operates as a pivotal mechanism governing the mobility of atoms within solid substrates. Its significance within the realm of metals and alloys is

immense and it mainly contributes to the establishment of intrinsic material properties and microstructural qualities. Adolf Fick's groundbreaking contributions during the 19th century became the cornerstone of understanding diffusion mechanisms in solids. Fick's first law depicts the significant influence of species concentration gradients on diffusion rates, while his second law describes the temporal evolution of concentration profiles. These basic principles are rigorously expressed in precise mathematical representations. The Fick's laws expressed as follows:

$$\text{First Fick's Law, } J = D \frac{dc}{dx}$$

$$\text{Second Fick's Law, } \frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2}$$

In this context, the symbols represent fundamental parameters: J denotes the diffusion flux, D signifies the diffusion coefficient, c stands for concentration, x represents position, and t signifies time. These principles serve as the cornerstone for quantitatively assessing and forecasting diffusion phenomena within metals and alloys.

1.3.2 Diffusion mechanism

Diffusion within metals and alloys manifests itself through a variety of mechanisms, particularly including vacancy diffusion and interstitial diffusion. While vacancy diffusion involves atom migration through lattice vacancies, interstitial diffusion entails atoms occupying interstitial positions within the crystal lattice. The specific diffusion mechanism observed depends on factors such as temperature, alloy composition, and crystalline structural configuration.

1.3.2.1 Vacancy mechanism

The phenomenon of diffusion is explained by the vacancy mechanism, wherein an atom positioned in a standard lattice site migrates to an adjacent, vacant site within the lattice. The migration of atoms between sites requires energy, resulting from the thermal energy inherent in atomic vibrations. In the context of

the vacancy exchange mechanism, atoms require a vacancy in the adjacent region in both the lattice and sublattice, as shown in Figure 1.2.

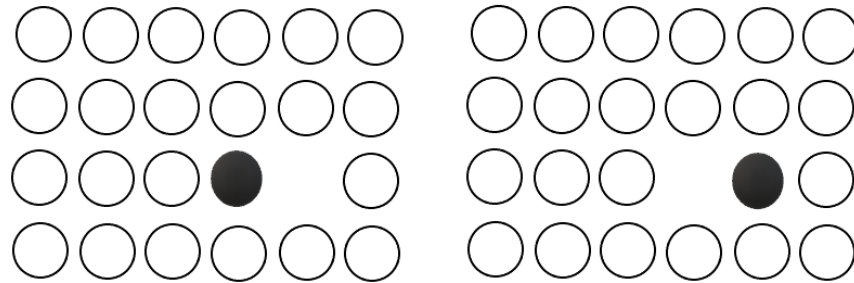


Figure 1.2. Vacancy mechanism of diffusion.

1.3.2.2 Interstitial mechanism

The diffusion process is described through the so-called indirect interstitial mechanism, in which atoms are predominantly found in interstitial positions within a larger atom host crystal. The movement of interstitial atoms causes substantial distortions in the lattice structure. This mechanism can be associated with natural point defects but does not concern the formation of pairs containing lattice atoms and defects. When the interstitial atom is smaller than the atoms occupying the normal lattice positions, this mechanism can occur, as depicted in Figure 1.3.

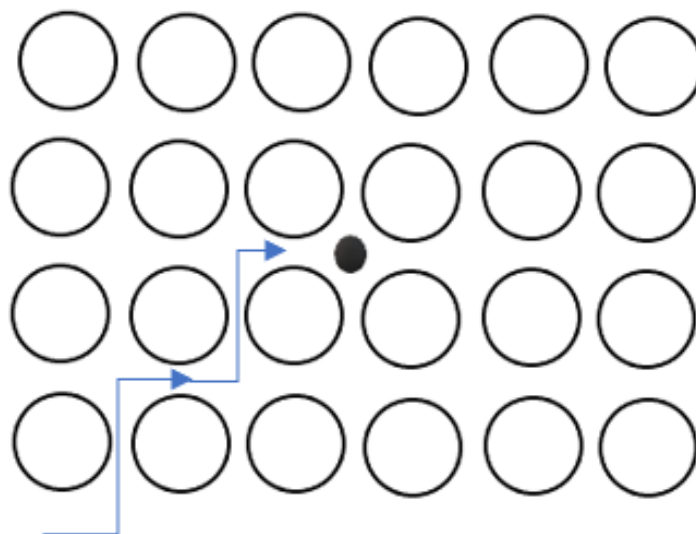


Figure 1.3. Interstitial mechanism of diffusion.

1.3.3 Diffusion in MgB₂ superconducting wire

The MgB₂ wires produced using the powder-in-tube process often exhibit significant porosity and little intergranular connection (J. M. Rowell, 2003 and E. W. Collings *et al*, 2008). Consequently, attaining the necessary critical current density for systems operating at temperatures of 20 K and high magnetic fields is a significant challenge. The diffusion technique was invented by Giunchi *et al*, utilizing a Mg rod and B powder as the initial ingredients. A magnesium rod was introduced into the central region of a steel outer sheath. Following the placement of B powder into the gap between the Mg rod and the Nb barrier, a wire was subsequently produced. During the process of heat treatment, Mg diffused into the B layer, resulting in the formation of a layer composed of MgB₂. This process also led to the creation of voids in the positions formerly occupied by the magnesium atoms. The procedure was denoted as the reactive liquid infiltration (RLI) process, while the wires were designated as hollow MgB₂ wires (Giunchi *et al*, 2003). Recent studies have provided evidence that the process of infiltration is successful in generating compact and interconnected MgB₂ material, exhibiting J_c above 1.0×10^5 A.cm⁻² at a temperature of 4.2 K and under 10 T (K. Togano *et al*, 2010 and S. J. Ye *et al*, 2013). These J_c values are notably greater compared to those typically observed in MgB₂ conductors produced using the PIT method. During infiltration, Mg undergoes a phase change from solid to liquid state, thereby penetrates through B region. The gaps present in the initial B region are completely filled by melted Mg as shown in Fig. 1.4. The infiltration of Mg also continues for the gaps formed due to the volume contraction during the $\text{Mg} + 2\text{B} \rightarrow \text{MgB}_2$ reaction. As a result, the initial B region completely transforms into a dense MgB₂ structure. The formation of dense MgB₂ layer ceases shortly after the initiation of the heat-treatment process. Consequently, an unreacted B layer remains, leading to a decrease in engineering critical current density (G. Z. Li *et al*, 2015 and H. Kumakura, 2012).

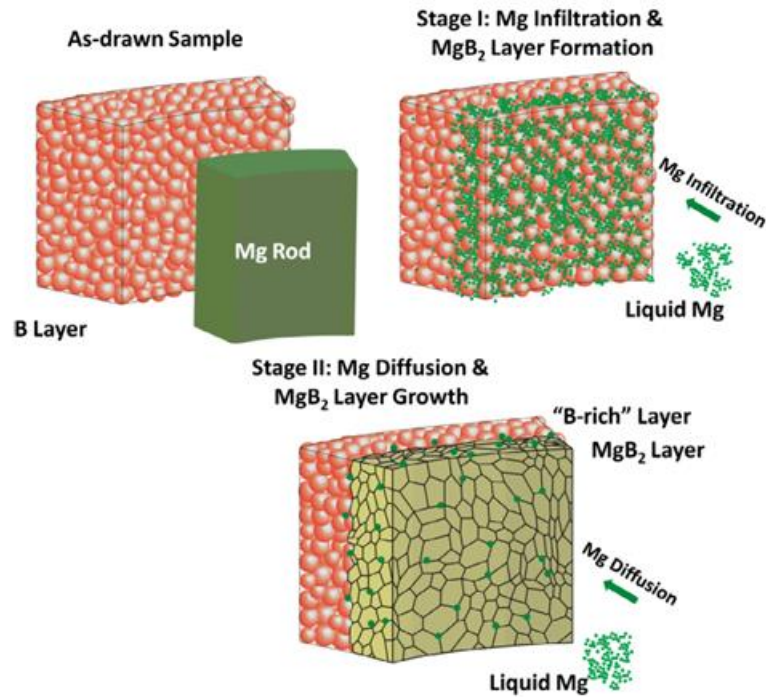


Figure 1.4. The pictures represent Mg infiltration to the B layer. (G. Z. Li *et al*, 2015)

G. Z. Li *et al*, studied on limiting layer thickness has led to improvements in wire configuration, precursor material selection, and heat treatment parameters. They achieved a complete reaction within an infiltration-based composite, resulting in an elevated critical current density and expanded MgB₂ cross-sectional area. This wire design, called advanced internal Mg infiltration, was proposed in 2012 (G. Z. Li *et al*, 2012).

Yong Liu *et al*. studied the kinetic mechanism of the reaction between Mg and B, forming the MgB₂ layer in normal and Cu-coated IMD wires. They found that the formation of the MgB₂ layer is controlled by various mechanisms, with the slow Mg diffusion-limited mechanism becoming dominant as the reaction progresses. Cu coating can accelerate the formation of the MgB₂ phase (Yong chang Liu *et al*, 2017).

2. OVERVIEW OF MgB₂ SUPERCONDUCTOR

Synthesis and structural confirmation of MgB₂ were first achieved in 1954 by Jones and Marsh (Jones and Marsh, 1954). Superconductivity in MgB₂, an intermetallic compound, was first observed by Professor Jun Akimitsu in 2001. Numerous efforts have been devoted on this intermetallic superconductor, focusing on its high critical temperature, significant critical current density, and cost-effectiveness. MgB₂ has been synthesized in various forms, including bulk samples (Takano *et al*, 2001), single crystals (Chen *et al*, 2011), thin films (Petaccia *et al*, 2006), wires (Yetiş H. *et al*, 2022), and tapes (Kumakura H. *et al*, 2012).

2.1 Crystal structure of MgB₂ superconducting material

The structure of MgB₂ is classified as a simple hexagonal AlB₂-type structure, which is distinguished by the presence of graphite-like B layers arranged in a honeycomb pattern. These boron layers are located between the hexagonal close-packed (hcp) Mg layers as depicted in the schematic picture of MgB₂ in Figure 2.1 (Larbalestier *et al*, 2001). The contribution of the metallic B layers is of great importance in the superconductivity of MgB₂, as emphasized by Korkus *et al*. (2001). It is worth mentioning that MgB₂ exhibits a notable electronic anisotropy in the lengths of B–B bonds, as reported by Nagamatsu *et al*. (2001). Specifically, the distance between boron planes along the *c*-axis is considerably larger than the B–B distance inside the plane along the *a*-axis. The anisotropy, which impacts the superconducting characteristics, was illustrated by Kim *et al*. (Kim *et al*, 2002) in both the crystal structure of MgB₂ grains and single crystals.

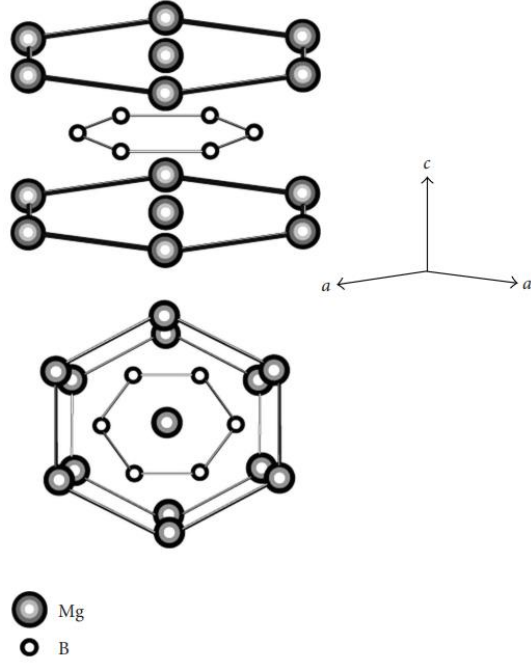


Figure 2.1. The schematic representation of crystal structure of MgB_2 superconductor with a , b , and c plane (Jean-Baptiste Laloe *et al*, 2011).

The interatomic distances inside the same layer for B-B and Mg-Mg are 0.1780 nm and 0.3084 nm, respectively. On the other hand, the interatomic distances between different layers are measured as B-B 0.17 nm, Mg-B 0.25 nm, and Mg-Mg 0.3524 nm (Jones and Marsh 1954, 1953; Vinod *et al*, 2007). According to Buzea and Yamashita (2001), the lattice parameters of hexagonal MgB_2 are reported as follows: $a = b = 0.3088$ nm and $c = 0.3522$ nm. Laughlin and Hono classified MgB_2 within the $P6/mmm$ space group (Laughlin and Hono, 2014). They determined that the Mg atoms occupy positions in (0,0,0) sites, while B atoms are located in $(1/3, 2/3, 1/2)$ and $(2/3, 1/3, 1/2)$ sites. In the context of crystalline structures, it is seen that each Mg atom donates one electron to the B ring.

2.2 Overview for MgB_2 wire developments

With the discovery of the superconductivity of MgB_2 at 39 K in 2001 (Nagamatsu *et al*, 2001), many scientific studies have been carried out on MgB_2 both at the basic research level and on the use of this material in practical applications. MgB_2 superconducting material has structural and superconducting properties that provide significant advantages in terms of applications compared to

other superconductors, high temperature superconductors (such as YBCO, BSCCO) and low temperature superconductors (such as NbTi, Nb₃Sn). The most important of these properties is that it has the highest T_c value among binary metallic compounds. MgB₂, which can be cooled with a cryocooler around 20 K instead of low-temperature superconductors that work only in liquid helium, is of great importance in terms of cost and ease of use. A high T_c value of 39 K allows MgB₂ to still exhibit promising superconducting properties around the liquid hydrogen temperature (20.2 K) (Shirai *et al*, 2016). The main parameters that determine superconducting properties are J_c , B_{c2} and the irreversible magnetic field values, respectively. Since MgB₂ does not have grain orientation and weak links that limit the current, it can be formed into a wire simpler than other high temperature oxide superconductors. In terms of ease of use and production cost, this material has the advantages of abundant raw materials (magnesium and boron) and low density, meaning a lighter product. When all these advantages are considered, MgB₂ superconductor is an ideal candidate for large-scale applications involving electrical power and magnetic field generation.

MgB₂ superconducting wire is mainly manufactured using the powder-in-tube method (Glowacki B. A., 2001); the starting chemical powder is filled into a metal tube and the resulting composite is formed into wire by cold groove rolling or cold drawing processes. Depending on the powders used, the PIT process can be divided into two groups: internal (*in-situ*) PIT (using a mixture of Mg and B powders) and external (*ex-situ*) PIT (using ready-made MgB₂ powder). In the *in-situ* PIT method, porosity is a problem due to the volume shrinkage caused by the chemical reaction between Mg and B (because MgB₂ has a higher mass density than Mg and B), while in the *ex-situ* PIT method, low grain connectivity occurs since MgB₂ is a hard ceramic. Therefore, both internal and external PIT lead to the production of MgB₂ wires with low critical current densities. Porosity is the main reason for the low J_c of *in-situ* PIT MgB₂ wires. The most important step towards solving the porosity problem in this method was taken by Giunchi *et al*. (Giunchi *et al*, 2003). In this method, which was developed based on the *in-situ* PIT method, an Mg rod is placed in the center of a steel outer sheath with an Nb internal barrier and after filling the B powder into the space between the Mg rod and the Nb barrier, the wire is produced as shown in the schematic diagram in Figure 2.2. During the heat treatment, Mg diffuses into the boron powder, leaving behind a large hole,

resulting in the formation of a dense, non-porous and thin MgB_2 layer on the inner walls of the Nb barrier (Figure 2.3). This method was called “reactive liquid infiltration” (RLI) and the wires produced were called hollow MgB_2 wires. The first measured J_c values were about $3 \times 10^2 \text{ A.cm}^{-2}$ at $T = 4.2 \text{ K}$ and $B = 10 \text{ T}$, but this value was increased to $2.5 \times 10^3 \text{ A.cm}^{-2}$ at 4.2 K and 12 T by carbon doping (Giunchi G., 2007). In 2008, they obtained a high J_c value of $4.1 \times 10^4 \text{ A.cm}^{-2}$ at 4.2 K and 10 T for an Fe-sheathed wire using a fine B powder with nanosized SiC addition (Hur *et al.* 2008). This value was the highest J_c value reported for MgB_2 wire at that time, and the method was named internal Mg diffusion (IMD). The highest J_c value reported so far was $1.5 \times 10^5 \text{ A.cm}^{-2}$ at 4.2 K and 10 T for single filament wires, increasing the carbon content to 3 mol %. This method was later named “advanced internal Mg infiltration” (AIMI) (Li *et al.* 2013). Studies show that the preferred method to produce MgB_2 wire should be *in-situ* technique. At this point, the preferred filling method is to use IMD instead of PIT because, as mentioned above, porosity leads to low J_c values, that is, current paths are prevented by pores (Dongliang *et al.* 2017). The manufacturing parameters of second-generation IMD wires have not yet been optimized and further research is needed (Ye *et al.* 2016).

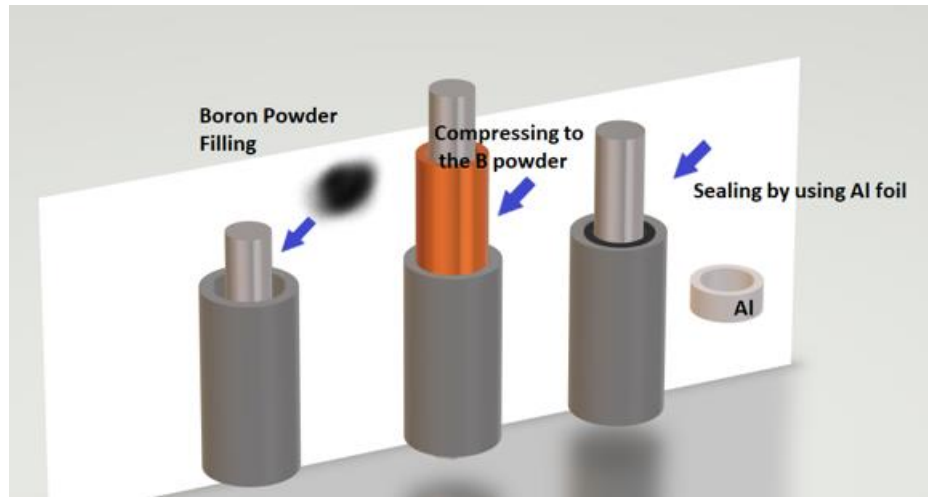


Figure 2.2. Schematic representation of internal magnesium diffusion (IMD) method.

The construction of a large MgB₂ coil requires the production of kilometers of superconducting wire. MgB₂ wires produced by the *in-situ* PIT method can now be produced in lengths on the order of km (Baig *et al*, 2017; Tanaka *et al*, 2020). The IMD method, which provides higher J_c under higher magnetic fields than the PIT method, is more advantageous in terms of using MgB₂ instead of NbTi in the MRI magnet (Takahashi *et al*, 2006; Ye *et al*, 2013; Ye *et al*, 2014). However, the J_{ce} value of wires produced by the internal Mg diffusion method is lower than that of PIT wires because a hole is formed in the center of the IMD wire due to the diffusion of the central Mg rod into the surrounding boron layer during heat treatment (Giunchi *et al*, 2003; Hur *et al*, 2008). Furthermore, variations in the packing density of boron powder lead to variations in the superconducting properties along the IMD wire after heat treatment and make it difficult to achieve self-uniformity in long IMD wires. For these reasons, improving the longitudinal self-uniformity and J_{ce} in IMD wires are essential for large-scale applications (Ye *et al*, 2016; Kovac *et al*, 2021).

Superconducting coils are produced using two different techniques, called wind & react and react & wind methods. While the former has the advantage of high current carrying capacity, it can impose high additional costs in manufacturing due to the need for very large furnaces for heat treatment and the requirement for heat-resistant insulation material. The react & wind method seems to be more suitable to produce large superconducting coils (Young *et al*, 2015). However, the reacted MgB₂ wire has a brittle structure, so the bending strain tolerance should be improved to reduce the deformations caused by the winding process in the superconducting core (Tanaka *et al*, 2018). The bending strain tolerance for MgB₂ wire is closely related to the core structure, filament number, sheath material and heat treatment conditions (Nishijima *et al*, 2012; Kovac *et al*, 2016; Tanaka *et al*, 2019; Yoo *et al*, 2019). It has been suggested that MgB₂ wire produced by the IMD process has a better strain tolerance than MgB₂ wire produced by the PIT method under various bending processes (Nishijima *et al*, 2012). The good mechanical properties obtained for IMD wire are attributed to both the low annealing temperature, which does not adversely affect the mechanical strength of the sheath, and the strong grain connectivity (Kovac *et al*, 2020). However, it has also been reported that the degradation in J_c is only tolerated up to a bending strain of up to 0.5 % in advanced IMD multifilament wires, while PIT wire is independent of

bending strain within this limit (Yang *et al*, 2015). Sheath material is an important factor in determining the appropriate bending diameter for reacted MgB_2 wires (Katagiri *et al*, 2005; Avronsart *et al*, 2018). Measurements on strain bending tolerance for reacted MgB_2 wires and tapes have shown that the use of Fe sheathing increases the strain limit by up to 0.7 % and allows a low bending diameter for reacted MgB_2/Fe multifilament tape (Avronsart *et al*, 2018). Yoo *et al*, suggested that the sheath layer thickness plays an important role in determining the appropriate bending diameter (Yoo *et al*, 2019). They confirmed that the minimum bend diameter should be 110 mm for single filament PIT wires and 90 mm for multi-filament PIT MgB_2 wires.

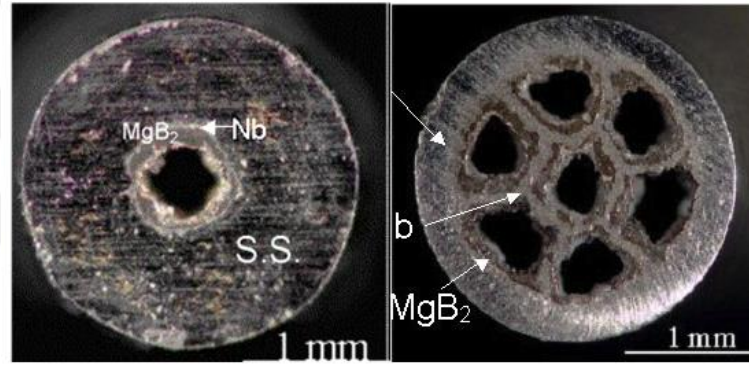


Figure 2.3. Scanning electron microscope images of Nb/SS sheathed single and 7 filament MgB_2 wires produced by IMD method (Giunchi *et al*, 2003). The black areas inside the wire are the hollow parts left behind by the magnesium rod after heat treatment.

In terms of practical applications, the J_c value of the second generation IMD wire at ~ 20 K needs to be brought to a level that can compete with the J_c value of commercially used NbTi wire at 4.2 K (Ballarino *et al*, 2017). In addition to the J_c value of the MgB_2 wire produced by IMD, the J_{ce} value should also be high. Studies show that there is no obstacle to the use of IMD wires in large-scale applications in terms of mechanical properties (Saglietti *et al*, 2010), AC losses (Kovac *et al*, 2015) and other properties. IMD MgB_2 wires are known to have much higher J_c and J_{ce} values compared to PIT MgB_2 wires, but J_c and J_{ce} values at 20 - 30 K are still not sufficient for large-scale applications. On the other hand, it is not enough to target J_c and J_{ce} values for a specific practical application, as the requirements may vary

depending on the application area. Therefore, the fact that J_c and J_{ce} are not high enough at temperatures above the temperature of liquid helium remains the main obstacle to the large-scale application of IMD MgB_2 wires. As can be seen in Figure 2.3, the large hole in the central region of the IMD wire due to magnesium diffusion limits the J_{ce} values of these wires. There are many factors that affect the J_c and J_{ce} values of IMD MgB_2 wires more or less. These include the properties of boron powder and carbon addition, effective MgB_2 density, grain size, magnetic flux pinning properties, intergranular coupling, maximum Mg infiltration distance, B layer thickness, wire diameter, heat treatment conditions and external pressure. For example, carbon doping is often used to increase J_c and J_{ce} values under magnetic field.

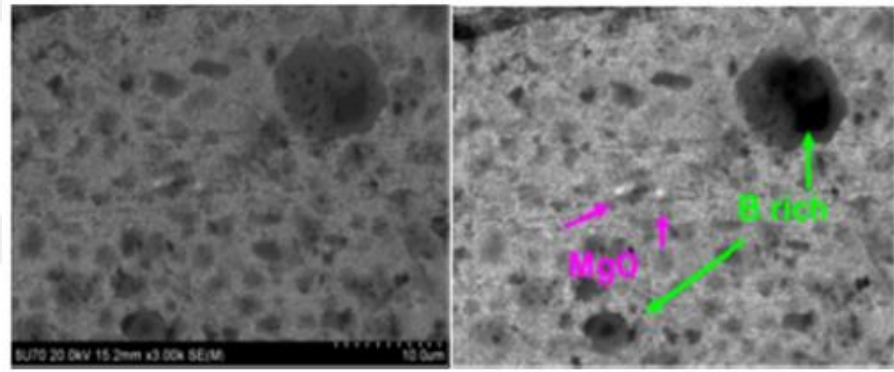


Figure 2.4. Scanning electron microscopy (SEM) images of MgO, pores and unreacted boron clusters observed in IMD MgB_2 wires (Ye *et al.*, 2013).

In the MgB_2 crystal lattice, electron scattering increases when the boron atom is replaced by a carbon atom, leading to a decrease in the average free electron path and an increase in the upper critical magnetic field value. The increase in B_{c2} leads to an improvement in the critical current properties of MgB_2 wires in strong magnetic fields. Particle size, amorphous and impurity content (especially oxide content) of boron powder are the most important factors affecting the J_c value. The choice of boron with small particle size (<250 nm) is important to reduce the amount of unreacted boron formed in the MgB_2 layer of IMD wires. The distance that Mg diffuse from the centered Mg rod to the interior of B layer is long, which prevents the Mg from diffusing to the distant regions of the B layer, resulting in the

formation of unreacted boron clusters. Therefore, if smaller B particles are used, the amount of unreacted B will decrease in volume. In studies on this subject, the J_{ce} value was obtained as approximately $1.0 \times 10^4 \text{ A.cm}^{-2}$ at 4.2 K and 10 T. The J_c value obtained at 10 K for the same wire was reported to be comparable to the J_c value measured for NbTi at 4.2 K (Ye *et al*, 2013). To date, the highest J_c and J_{ce} values reported for MgB₂ wires under 5 T and at 20 K reached $7.6 \times 10^4 \text{ A.cm}^{-2}$ and $5.3 \times 10^3 \text{ A.cm}^{-2}$, respectively. The high J_c and J_{ce} values obtained for IMD MgB₂ superconducting wires are mainly due to the use of nano-sized B powder to reduce the amount of unreacted B and the small wire diameter to reduce the required Mg infiltration distance. However, very small unreacted B particles are still present in the results observed from TEM images. To increase the J_c value of IMD MgB₂ wires, it is necessary to remove unreacted B particles, small voids and MgO amounts at the same time. As can be seen in Figure 2.4, the use of nano-sized boron is not sufficient to eliminate the excess boron regions because the use of nano boron in the production of *in-situ* MgB₂ wires brings about agglomeration and oxidation effects. These are the factors that limit the production of wires at industrial scale and need to be resolved.

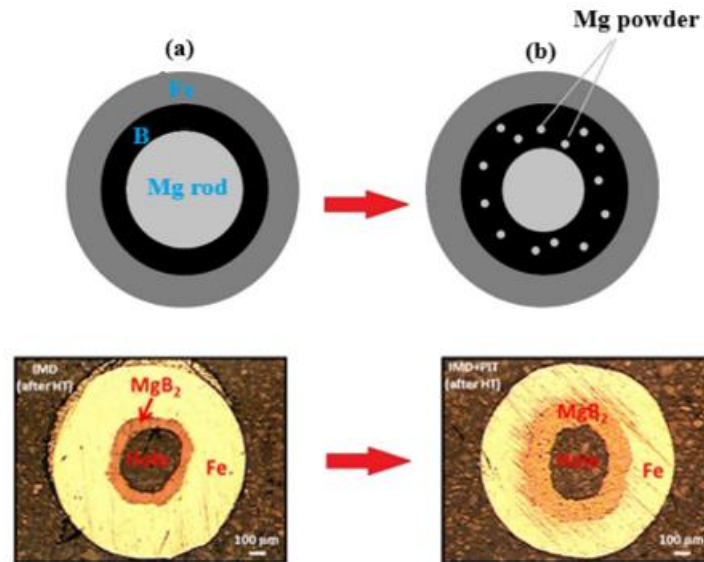


Figure 2.5. a) Schematic representations of thick Mg rod and (b) wires produced by PIT+IMD hybrid method before heat treatment (top) and optical microscope images after heat treatment (bottom) (Ye *et al*, 2014).

The maximum Mg infiltration distance is mainly influenced by B compaction density, B particle size, reaction temperature and time between Mg and B, and impurities in B powders. In fact, the content ratio of Mg to B also affects the maximum Mg diffusion distance. In Figure 2.5, two different excessive Mg utilization schemes were used to reduce the boron excess regions remaining in the MgB₂ layer in IMD wires. As seen in Figure 2.5(a), the choice of a thicker Mg rod in the center of the wire causes a larger central hole after heat treatment and a decrease in the J_{ce} value (Ye *et al*, 2016). On the other hand, combining PIT and IMD methods causes porosity to form in the MgB₂ layer after heat treatment. As shown in Figure 2.5(b), in order to reduce the amount of unreacted B particles, a small amount of Mg was added to the boron powder in the *in-situ* IMD MgB₂ wires (hybrid IMD wire), but this resulted in the formation of a porous structure (Ye *et al*, 2014). In another later study, the IMD process was applied in reverse and called the "modified internal magnesium diffusion" method. The difference of this method from the standard IMD production is that Mg tube is used instead of Mg rod and the boron powder is compressed into Mg tube. Since the wall thickness of the Mg tube is thick, a gap and unreacted Mg remain between the MgB₂ core and the sheath material after the reaction (Brunner *et al*, 2017). It is difficult to decide the initial ideal Mg and B content in IMD wire production because some Mg and B remain unreacted. In this respect, adjusting the maximum Mg infiltration distance and B layer thickness is very important to achieve high J_c and J_{ce} for IMD MgB₂ wires.

Studies have shown that various sheath and barrier materials used for IMD MgB₂ wires have little effect on J_c . In the wires produced so far, Nb barrier and Monel outer sheath, Ta barrier and Cu-Ni sheath or only an Fe sheath are generally used. The barrier or sheath material should be selected from materials that will not react with Mg / B, are resistant to cold deformation processes, and have good thermal and electrical conductivities. Iron as a sheath material eliminates the use of chemical barrier materials (such as Nb, Ta) that increase production cost and labor, and provides many of the above-mentioned properties (Li *et al*, 2012; Ye *et al*, 2013).

The manufacturing of long wires with a uniform structure is essential for practical applications of superconductors. Therefore, in IMD wires, the Mg rod must be exactly in the center of the boron powder filled around it. If linearity is lost in some localized regions, these regions will be enlarged during wire extension and

will limit the wire's maximum length or uniformity. Today, IMD wires can be extended up to 100 m (Wang *et al*, 2016). Although multi-filament superconducting wires reduce alternating current losses, it is more difficult to produce multi-filament wires using the IMD process. Currently, there are efforts to produce 7-, 19- and 37-filaments wires, but the filament shapes are still generally irregular (Figure 2.6).

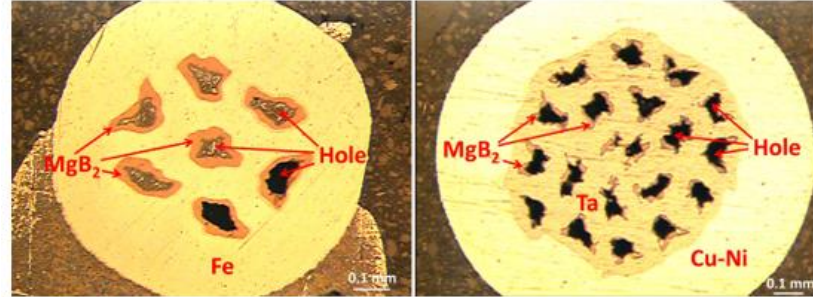


Figure 2.6. Optical microscope images of 7- and 19-core wires produced by IMD method (Ye *et al*, 2013).

3. PRODUCTION METHODS FOR MgB₂ WIRES AND JOINTS

3.1 Fabrication techniques for wires

Superconducting MgB₂ wires are fabricated by powder-in-tube or continuous tube filling and forming (CTFF) methods (Glowacki *et al*, 2001; Tomsic *et al*, 2007; Ma *et al*, 2008). PIT involves sealing one end of a clean metallic tube, filling it with the prepared powder, and sealing the other end before forming it into wire form. CTFF aims to increase powder density by pouring precursor powder onto a metal strip, folding it into a tube shape, and stabilizing it within another metal tube, usually copper.

The PIT method involves two different situations: filling the previously prepared MgB₂ powder (*ex-situ*) or the Mg+2B powder mixture (*in-situ*) into a metal tube. *In-situ* PIT allows wire production at low heat treatment temperatures around 650 °C, and suitable additives such as carbon can be included in the mixture before the reaction. However, *in situ* PIT-processed MgB₂ wires suffer from high porosity due to the volume contraction after reaction of Mg with B and limited intergranular connections. Typical core density is about 50 % of the theoretical density of MgB₂ (2.62 g/cm³).

Methods such as heat treatment under high isostatic pressure (Gajda *et al*, 2018) and cold high-pressure densification (CHPD) (Hossain *et al*, 2014) are used to increase the core density. Since the HIP process is performed under gas pressure, it can only be applied to short wire samples of 1-2 cm in length. The CHPD method can be applied to long wires. With this method, a denser core is obtained, but it is not as effective as the HIP method in terms of reducing porosity.

The pellet-in-tube technique, applied as an alternative to the PIT method, increases the initial filling density up to 60 % of the theoretical mass density of MgB₂ and significantly increases the core density despite the remaining porosity after reaction (Akdoğan *et al*, 2015). In PeIT method, filling the Mg+2B mixture as a tablet into the metal tube allows a uniform powder density along the metal tube. This is an important parameter in the production of long wires. (Karaboga *et al*, 2018).

Another approach is the internal magnesium diffusion (IMD) technique, in which a magnesium rod is placed in the center of the metallic tube, and the space between the rod and the metal tube is filled with boron powder (Giunchi et al, 2003). A central hole is formed in the middle of the IMD wire after the reaction. IMD wire provides a dense MgB_2 ring without creating porosity in the core region, but the gap in the middle reduces the J_{ce} value. The production of IMD MgB_2 wires with high J_c values by carbon doping is promising (Hur *et al.*, 2008). Although short IMD wires have high J_c values, achieving the same J_c level in long IMD wires remains a challenge and encourages ongoing efforts to achieve this goal (Wang *et al*, 2017).

3.2 Fabrication techniques for joints

Joints are typically composed of three fundamental configurations: Butt joints, lap joints, and hybrid joints. Butt joints involve end-to-end wire connection, ideal for coil windings where magnetic field homogeneity is crucial (G. D. Brittles *et al*, 2015). Lap joints arrange conductors in an overlapping manner, particularly in monofilamentary tapes. Hybrid joints are a combination of both types using scarf and step joint techniques. Swenson and Markiewicz presented a review article on the available joint fabrication methods for NMR magnet (Swenson C. A. and Markiewicz W. D., 1999). Kim and Joo used the lap joint, which has the stepped surfaces to increase contact area and improve current transport efficiency (Kim J. H. and Joo J., 2002).

Indirect joint can be established using superconductive substances such as solders (Thornton R. F., 1986), powders/ slurries (Li X. H. *et al*, 2008), or tape bridge components (Ryan D. T., 1997). However, intermediate materials often have weak superconducting properties. The wires can be joined at the ends with a "termination" joint, ensuring proper alignment. If the joint needs to extend or replace the conductor, a "continuation" connection is needed. The relative orientation of joined wires within the joint can be arranged or joints near the coils are typically placed within the reduced stray field region.

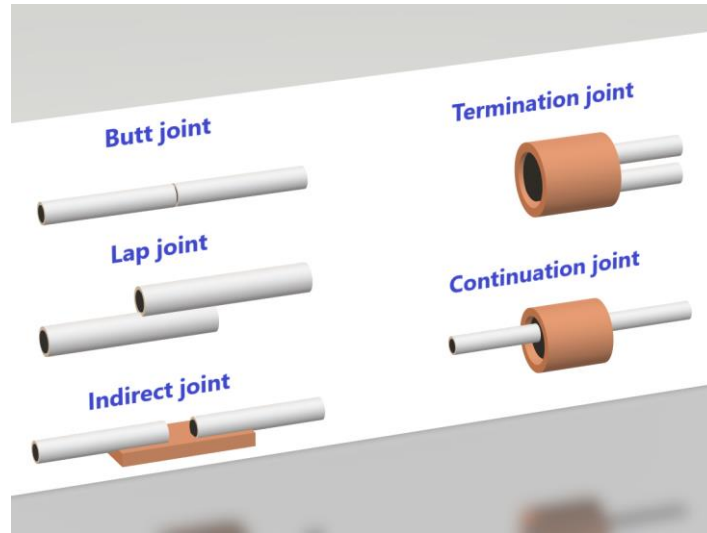


Figure 3.1. Typical joint types.

The joining process plays a crucial role in determining the suitability of a superconductor for use in industrial-scale applications. The process of joining MgB_2 wires is less challenging in comparison to that of high-temperature superconductors (HTS) because superconducting properties of HTSs are quite dependent on microstructure, especially for REBCO superconductors. However, the critical current ratio (CCR) values of the joints produced between the reacted MgB_2 wires are still significantly lower than the joints between the unreacted MgB_2 conductors, and the joint resistances are quite high at 20 K (G. D. Brittles *et al*, 2015). The difficulty in joining IMD MgB_2 wires, particularly for joining multifilament wire, arises from the limited availability of filler surface area caused by the significantly thinner MgB_2 surface compared to PIT MgB_2 wires. Currently, there are limited research studies on superconducting joint of IMD MgB_2 wires. However, more research is needed to improve the joint to ensure widespread use of the IMD MgB_2 wire (Ye *et al*, 2016; Ballarino *et al*, 2017; Yoo *et al*, 2018; Matsumoto *et al*, 2018). An ultra-stable magnetic field is required to obtain high-resolution MRI images. The use of a superconducting MgB_2 wire in the construction of an MRI coil can be designed by sequential connection of individual coils. This requires the use of superconducting joints to maintain persistent current in the MRI magnets. However, the joint resistance is expected to be very low to reduce heat generation (Patel *et al*, 2016, 2019, 2021). A recent study (Matsumoto *et al*, 2018) has shown that joining unreacted multifilament MgB_2 wires has a joint

resistance as low as $10^{-15} \Omega$. The optimization of superconducting joints is not yet completed, because it is necessary to maintain both the high CCR and the low resistance value (Li *et al*, 2008; Ling *et al*, 2017; Luo *et al*, 2019; Kim *et al*, 2021). It has been determined that the J_c value of the joint for IMD monofilament MgB_2 wires is around 60 % of the J_c value of the wire. However, it is important to note that this ratio is obtained by joining unreacted IMD wires. It may be preferable to use reacted wires to eliminate the need to heat treat a large coil (Ye *et al*, 2016; Park *et al*, 2018; Patel *et al*, 2020; and Xi *et al*, 2021).



4. MATERIALS AND METHODS

4.1 Production of wires

This section contains information about the manufacture of MgB_2 mono filament wires. Three different wire preparation methods were proposed as an alternative to standard internal magnesium diffusion (S-IMD) method. These are redesigned internal magnesium diffusion (RD-IMD), half external magnesium diffusion (H-EMD), and three-hole internal magnesium diffusion (TH-IMD) methods. The iron (Fe) tubes with 12 mm outer / 9 mm inner diameters were used as a sheath material for each method. The filled composite tubes were formed into thin wires with a diameter of 0.81 mm in three steps: In the 1st step, the tube diameter was reduced from 12.0 mm to 6.0 mm using an octagonal groove rolling machine (Figure 4.1(a)), in the 2nd step from 6.0 mm to 3.0 mm using a cylindrical groove rolling machine (Figure 4.1(b)), and in the 3rd step from 3.00 mm to 0.81 mm linear & cylindrical cold drawing machines (Figures 4.2(a) and 4.2(b)). Intermediate heat treatments below the reaction temperature of MgB_2 were performed at certain reduction rates in order to lessen the mechanical stress build-up on the surface and deep structure of the sheath material during the cold deformation processes. Intermediate heat treatments were carried out under argon gas flow in a long tube furnace shown in Figure 4.3.

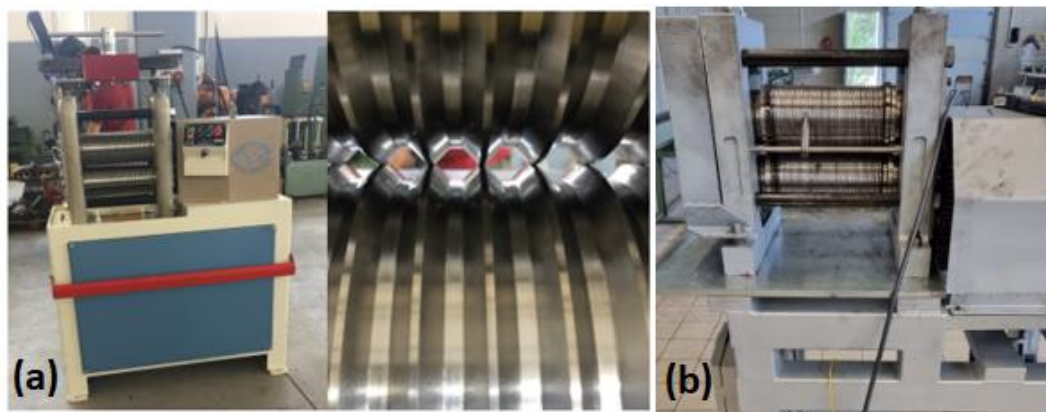


Figure 4.1. The photos show (a) octagonal, and (b) cylindrical groove rolling machines.

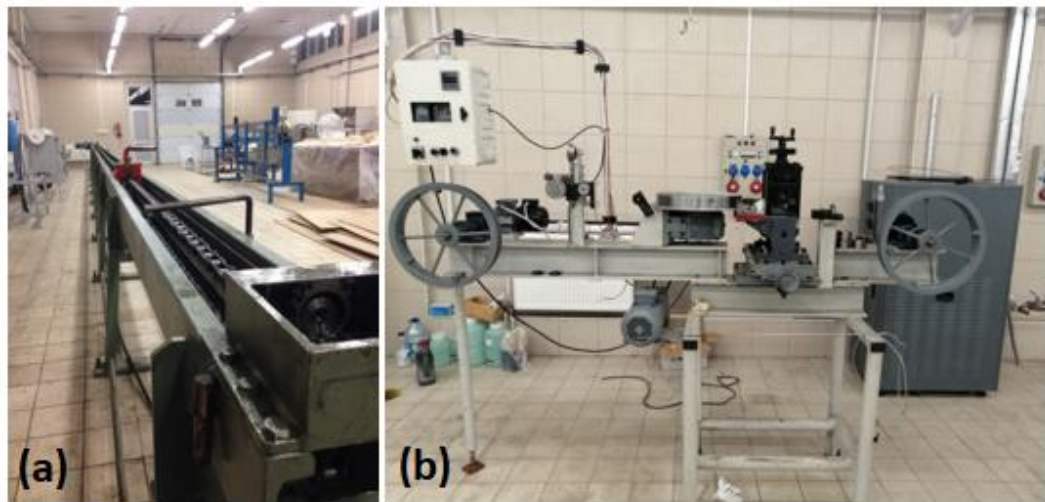


Figure 4.2. The photos of (a) line and (b) cylindrical cold drawing machines.



Figure 4.3. The photos of long tube furnace used for the intermediate heat treatment.

4.1.1 Designed internal magnesium diffusion method

Wire production was realized using a large number of uniform and designed tablets in this method. A press mold with an inner diameter of 6.4 mm was used for tablet production. The dimensions of the mold were determined to ensure Mg+2B powder stoichiometry in an iron tube with a diameter of 9 mm (inner), which was used as an outer sheath material in the filling process. Preparation of magnesium-rich Mg+2B powder mixture; magnesium and boron powders (percentage weight ratios of 75 % to 25 %) were filled into a glass jar with ceramic balls of different sizes and mixed for three hours. The prepared powder mixtures were filled into molds and placed in a hydraulic press with a load capacity of 25 tons as shown in Figure 4.4 and the tablets were produced under a load of 3 tons.

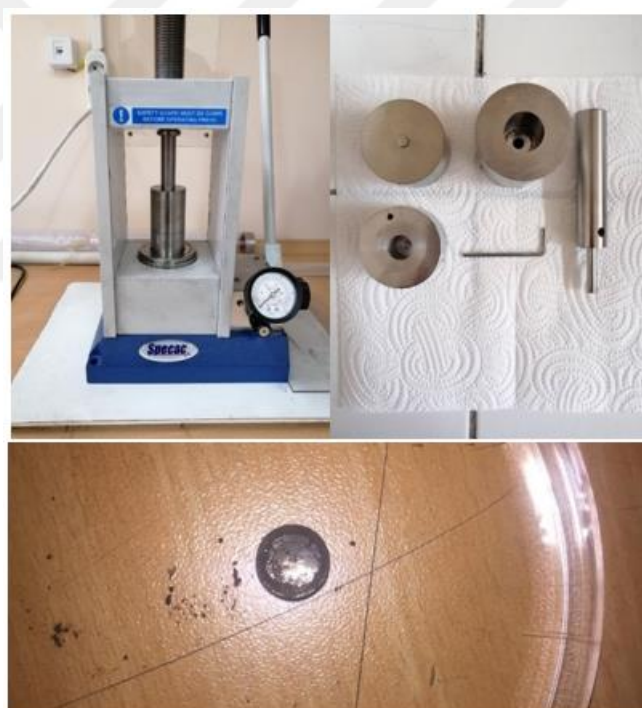


Figure 4.4. The pictures show the hydraulic press, the cylindrical press mold, and a produced tablet.

Boron powder (PVZ 98.5 % purity, <250 nm particle size) was filled into an iron tube with an outer diameter of 12 mm and an inner diameter of 9 mm and compressed to form a cylindrical layer with a wall thickness of 1.2 mm. Magnesium-rich Mg + 2B tablets were then placed one by one into the hole at the center as shown in Figures 4.5 and 4.6. Finally, the open end of the iron tube was

sealed with an aluminum tablet. Thus, the filling process was completed in accordance with the $\text{Mg}+2\text{B}$ stoichiometry.

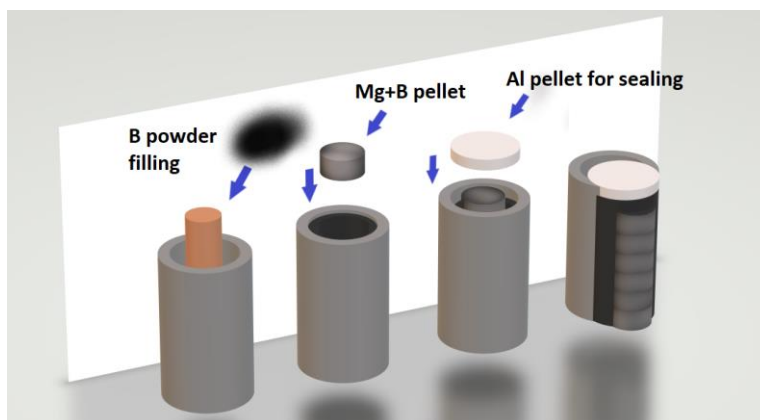


Figure 4.5. A schematic representation for filling process of the redesigned IMD method.

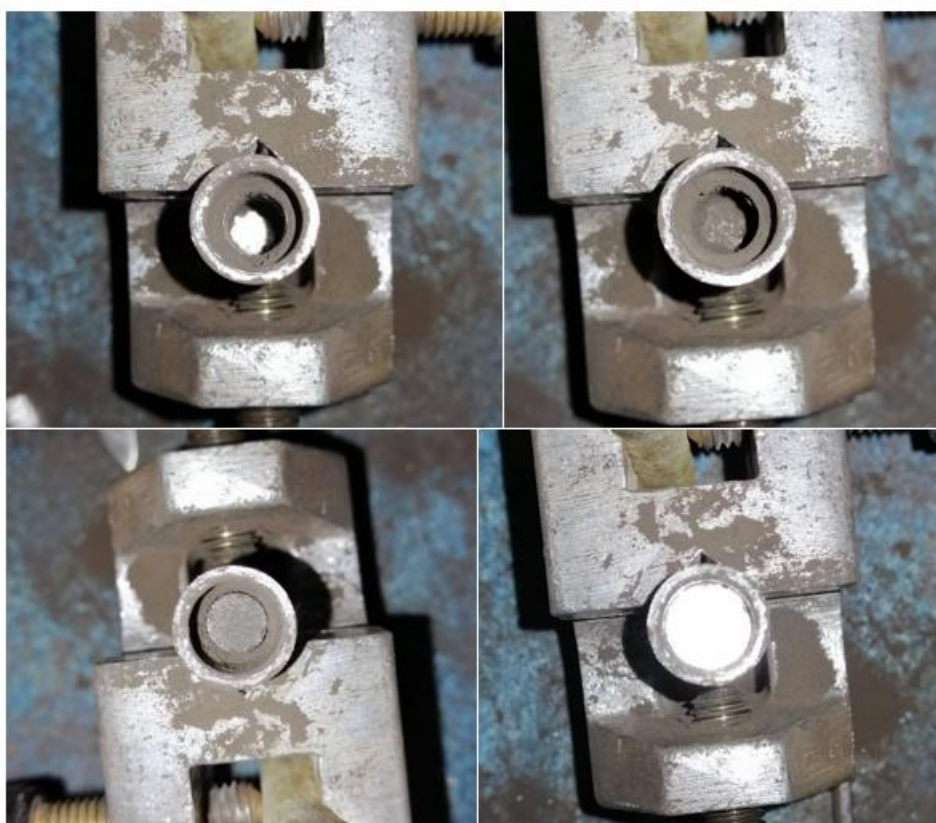


Figure 4.6. The photos show each step during the filling process of the RD-IMD method.

4.1.2 Three hole internal magnesium diffusion method

An innovative design is proposed to improve superconducting core of MgB_2 wire produced by internal magnesium diffusion method. This method is called Three-Hole IMD (TH-IMD) and is based on the insertion of three thin Mg rods into boron powder in a triangular geometry instead of a single central Mg rod used in the IMD method. As shown in Figure 4.7, three Mg rods with a diameter of 2.65 mm are inserted into holes drilled to form a triangle on a pellet cut from an Al rod. These Mg rods are then placed into the iron tube and remaining gap is filled with amorphous nano boron powder (<250 nm). Finally, the filled boron powder is compacted by using Al mold with three holes. All steps for the filling process of the wire sample such as inserting the Mg rods, filling the boron powder, and compacting process are shown in Figure 4.8.

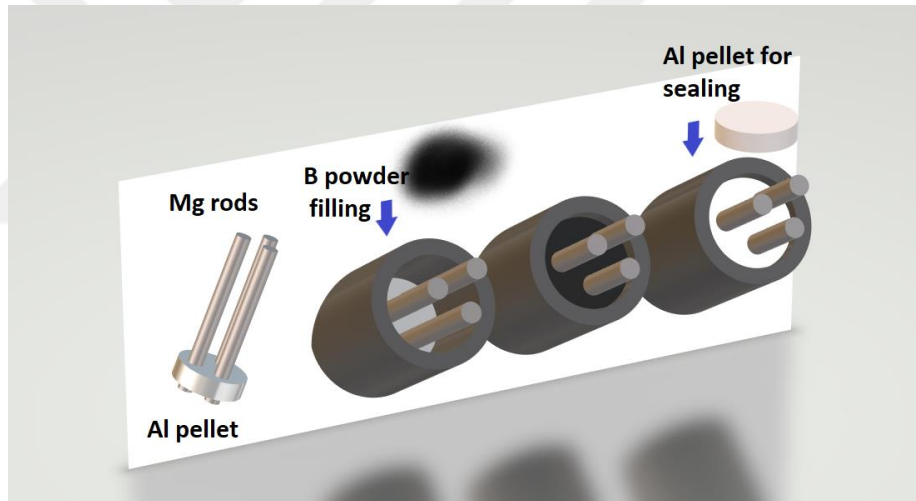


Figure 4.7. A representative drawing which shows the filling process for the three-hole IMD wire.



Figure 4.8. The photos show each step during the filling process of the TH-IMD method.

Diffusivity of magnesium for the S-IMD and TH-IMD wires is represented in Figure 4.9. It is seen that the use of three Mg rods are beneficial in terms of diffusion of Mg inside the wire core. The main idea behind the TH-IMD method is to shorten diffusion path of magnesium by distributing the Mg source in a triangular geometry and obtain a uniform MgB_2 core without leaving any unreacted boron layer. Mg diffusion is provided almost throughout the core of TH-IMD wire as seen in Figure 4.9.

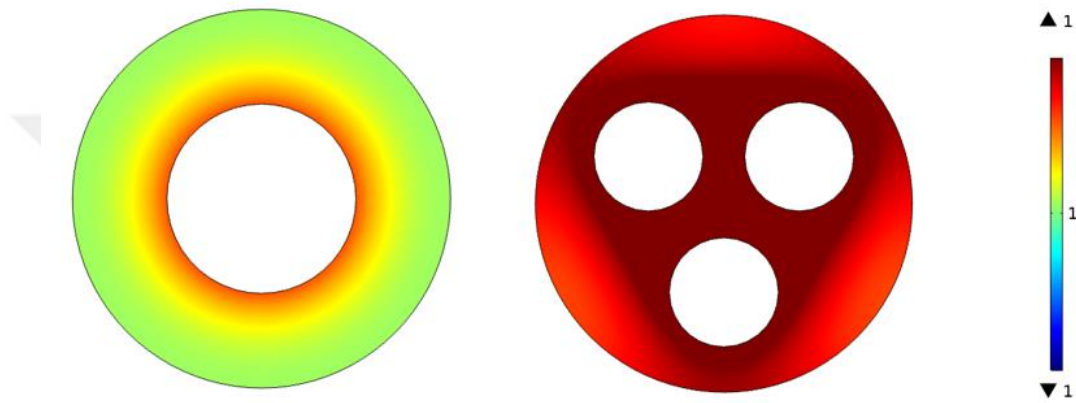


Figure 4.9. Mg diffusivity at the cross-section of S-IMD (left) and TH-IMD (right) wires.

4.1.3 Half external magnesium diffusion method

In this method, another novel core design was carried out with an innovative approach, especially for a superconducting joint fabrication. A half external magnesium method (H-EMD) was developed to fabricate an MgB_2 wire with a semicircular MgB_2 core structure. The H-EMD wire initially contains semicircular magnesium and boron columns before heat treatment process. With the semicircular identical steel bars shown in Figures 4.10(a) and (b), a complete circle with a diameter of 8.8 mm was formed in an iron tube with an inner diameter of 9 mm. As can be seen in Figure 4.11, one of the semicircular steel bars was placed inside the iron tube and boron powder was filled in the remaining space (Figure 4.11(b)). The filled B powder was then compressed with the other steel bar by hand as shown in Figure 4.11(a). Finally, after the boron compression process was completed, the steel bar was removed from the iron tube and the remaining empty space was filled

with magnesium powder as shown in Figures 4.11(c) – (d) and the H-EMD core structure was obtained.

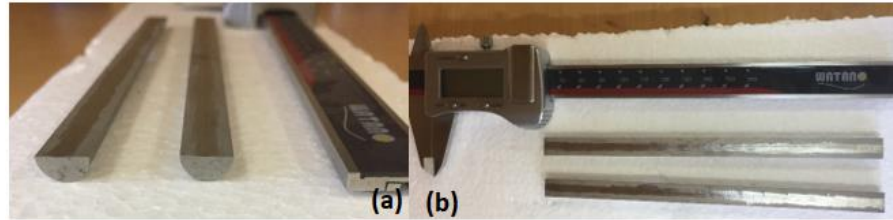


Figure 4.10. Semicircular steel bars with a total diameter of 8.8 mm and a length of 15 cm.

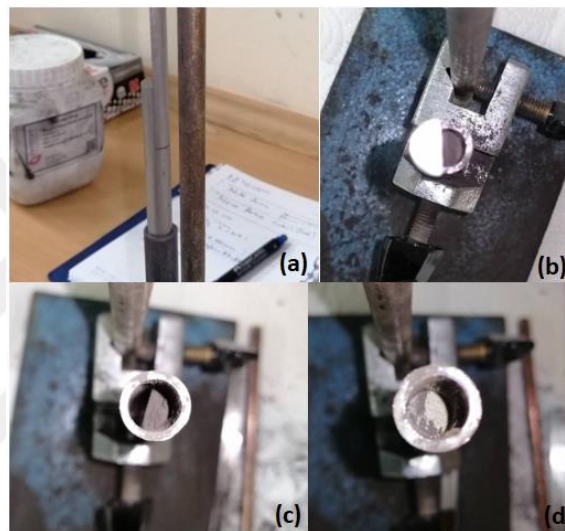


Figure 4.11. Photos show each step during the filling process of H-EMD method.

The diffusion of magnesium into the boron layer in the H-EMD method is shown representatively in Figure 4.12. The main advantage of the H-EMD method is that the self-contact surface of the H-EMD wire is more effective than the standard IMD wire because the MgB_2 structure in the H-EMD wire is formed in the form of approximately semicircular bulk, not in the form of a thin ring layer. The H-EMD method allows obtaining more current paths between the wire - joint - wire region in terms of joining MgB_2 wires.

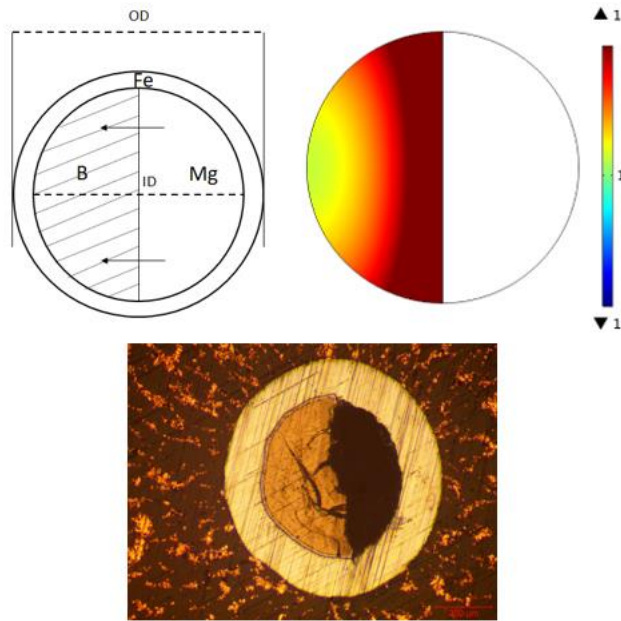


Figure 4.12. A representative drawing for Mg diffusion on the cross-section of H-EMD wire. OD: outer diameter of sheath, ID: inner diameter of sheath.

4.1.4 Standard internal magnesium diffusion method

An Mg rod with a diameter of 4.6 mm was placed in the center of a closed end iron tube (OD: 12 mm, ID: 9 mm). Amorphous nano-sized boron powder was then filled into the space between the Mg rod and the iron tube. As a final step, boron powder compressed and the open end of the Fe tube was sealed with Al foil as shown in Figure 4.13(a). Figure 4.13(b) shows a representative cross-sectional view of the S-IMD wire after wire fabrication is complete.

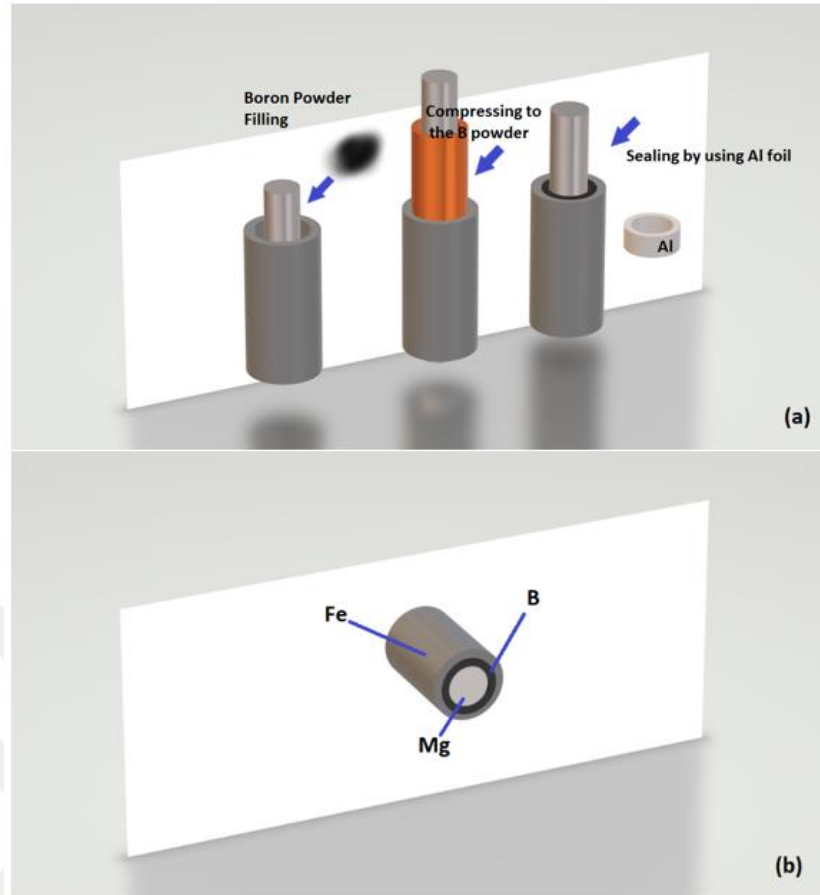


Figure 4.13. (a) Filling process steps for S-IMD wire. (b) Cross-sectional view of S-IMD wire.

4.1.5 Powder internal magnesium diffusion method

In this method, a steel rod with a 6.0 mm diameter was placed in the center of the Fe tube (OD: 12mm, ID: 9mm) and the amorphous nano B powder was filled into the gap between steel rod and Fe tube. The boron powder compressed and the steel rod was removed from the Fe tube. Mg powder was then filled into hollow space which remains after removing steel rod from Fe tube. As a final process, Fe tube was sealed by using Al foil as seen in Fig. 4.14(a), and the fabrication of Powder-IMD (P-IMD) sample, the transactional view was shown in Fig. 14(b).

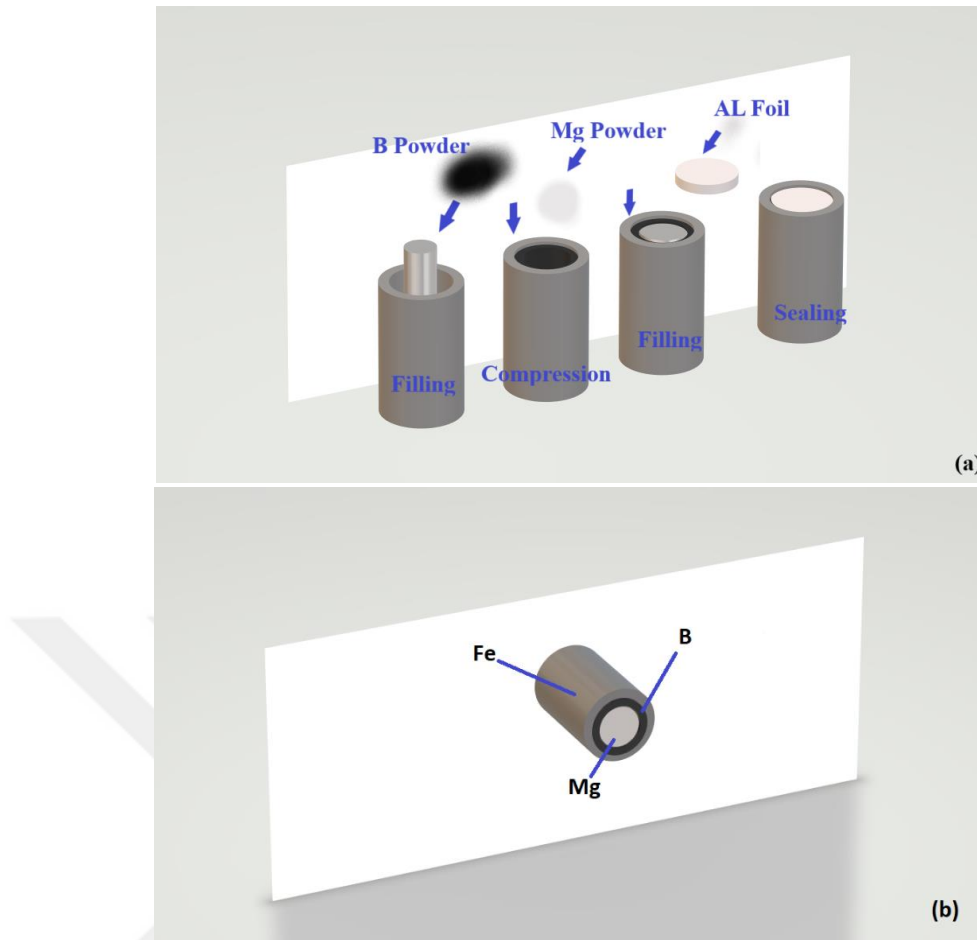


Figure 4.14. (a) The filling steps of P-IMD method. (b) Representative view of P-IMD wire.

4.2 Joint production

Different joint types have been used to join MgB_2 wires. The reacted or unreacted MgB_2 wires were jointed using continuation-type or termination-type connections. In joint production, an angle is usually formed for the wire ends to be inserted into the joint mold. The purpose of angling the ends of the wires by cutting or polishing them is to increase the contact surface between the wire core and the joint core. For this reason, an angle of 15° was formed at the ends of the wires as shown in Figure 4.15. A thick Cu/Nb/Mg+B composite wire with a diameter of 5 mm was used for the joint mold. The wire-type joint mold was prepared by means of H-EMD method as shown in Figure 4.16.



Figure 4.15. Picture of unreacted MgB_2 wires with 15° angled ends.



Figure 4.16. Schematic drawings for the wire type mold used produced by H-EMD method.

In wire-type joining; two holes, 4 mm long and 0.8 mm in diameter, were drilled on one (for termination type) or both (for continuation type) sides of the boron layer, and the 15° angled ends of the wires were placed in these holes. After the wires were placed in the joint mold, the molds were compressed in three stages by using the square grooved rolling machine shown in Figure 4.17 or by applying unidirectional force with the hydraulic press shown in Figure 4.4. The images of the joints obtained after compression are shown in Figure 4.18.



Figure 4.17. Square-grooved rolling machine.

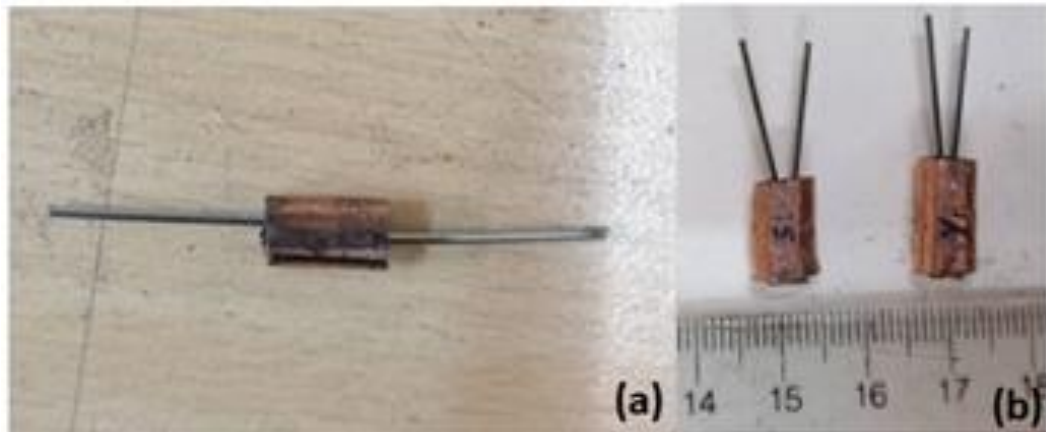


Figure 4.18. Joints fabricated using wire type joint mold. (a) Continuation type and (b) termination type.

Single turn MgB_2 coils with a superconducting MgB_2 joint were fabricated to determine the electrical resistance of the joint. A short MgB_2 wire with 6 - 8 cm in length was first angled 15° from its ends by cutting. The wire was then bent circularly and inserted into two blind holes with a diameter of 0.8 mm and a length of 5 mm pre-drilled into the boron layer of the mold with their angled surfaces facing each other, as shown in Figure 4.19. Finally, the joint mold was slightly compressed by applying a force of 3.24 kN perpendicular to the lateral surface of the joint mold with a hydraulic press.

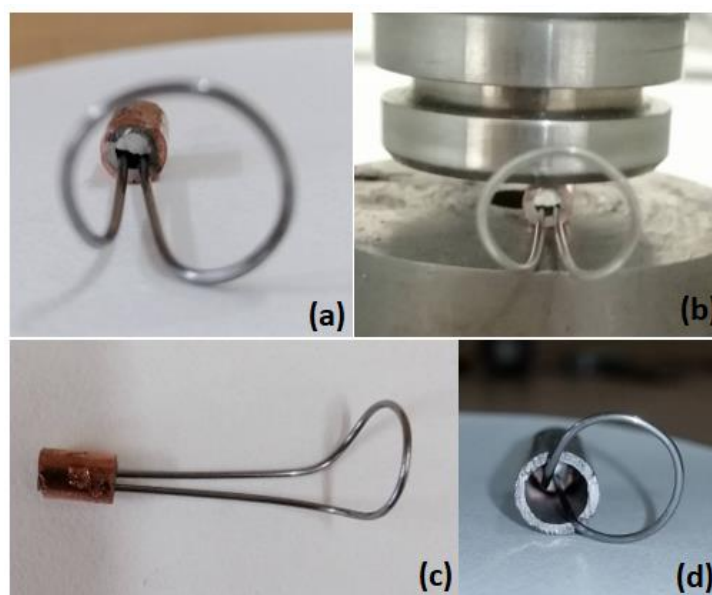


Figure 4.19. A single turn MgB₂ coil with a joint.

4.3 Heat treatment process

Heat treatments for short MgB₂ wires and joints were carried out at different annealing temperatures and times. The heat treatments were carried out under 5 bar argon atmospheres using a programmable three-zone furnace (Protherm) shown in Figure 4.20. The samples were heat treated in a stainless-steel tube placed inside the furnace, taking into account homogeneous temperature zone of 50 cm, as shown in Figure 4.21. Before the samples were annealed, the closed steel tube was cleaned three times, that is, 2 bar argon gas was sent to the steel tube and the argon gas was evacuated using a rotary vacuum pump. After the cleaning process was accomplished, 5 bar argon gas was filled into the steel tube to prevent oxidation on the samples during annealing process. The furnace temperature was increased to annealing temperature with 5 °C/min rate for all samples.



Figure 4.20. Three-zone temperature-controlled programmable furnace.

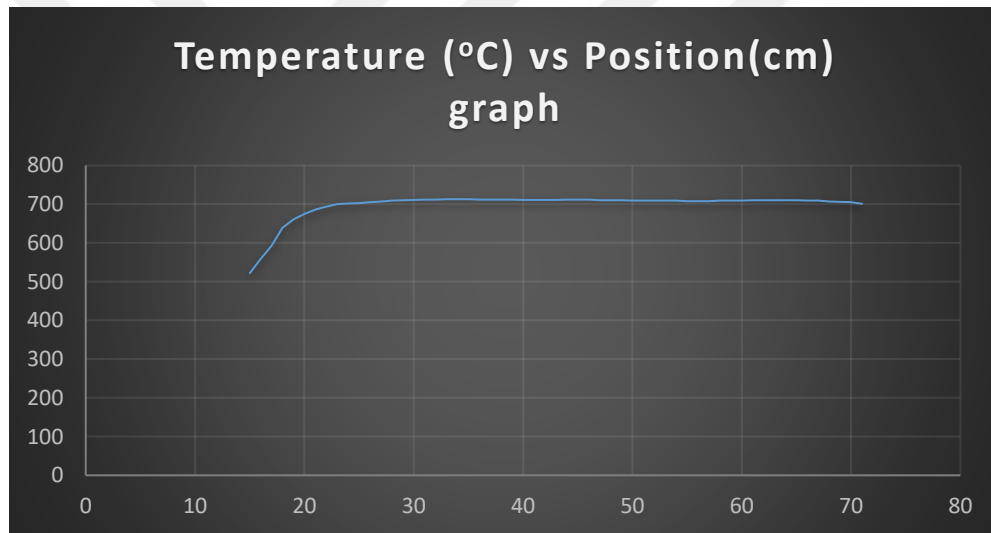


Figure 4.21. Temperature vs. position plot for a three-zone heat treatment furnace. The data was acquired using a K-type thermocouple with a drift rate of 1 cm/min.

4.4 Electrical characterization and electromechanical measurements

The electrical properties of all MgB_2/Fe wires were investigated by resistance-temperature ($R - T$) and current-voltage ($I - V$) characterization measurements. These measurements were performed in a closed cycle gas contact helium cryostat system shown in Figure 4.22. $I - V$ measurements were made for a maximum dc transport current of 1 A, and a maximum magnetic field of 7 T. The magnetic field direction was perpendicular to current direction and the cryostat

system was cooled under zero magnetic field (ZFC). Resistance-temperature measurements were done by applying 50 mA dc current to the sample between 20 K and 45 K and standard four-point probe method was used. Critical current density (J_c) values were determined from current-voltage curves using the 1 $\mu\text{V}/\text{cm}$ criterion. The other closed cycle helium gas contact cryostat system shown in Figure 4.23 has a maximum magnetic field of 4.5 T. A system capable of delivering 24 A sequential pulse current for $I - V$ measurements is adapted into the cryostat system. Figure 4.25 shows I_c values derived from $I - V$ measurements at 20 - 25 K using pulse current and DC current. I_c values obtained in different measurement systems for the same wire are consistent. Critical current (I_c) measurements for the wires and joints were performed using helium vapor cooling system (Oxford Instruments Susceptometer, INTIBS-PAN, Poland) or in a liquid helium environment with a maximum 150 A current and up to an external magnetic field of 14 T. The fast $I - V$ measurements were performed in a conduction cooling system. Current linear ramps of 1 s were used to avoid causing heating of the wire during measurements.

For joint resistance measurement, the joint was located next to the temperature sensor (Cernox) on the sample holder and the Hall Effect sensor (LakeShore, HGCA-3020, InAs) was mounted in the center of the joint loop as shown in Figure 4.24. The joint resistance was determined by means of magnetic field decay measurement for ZFC case. A joint was first cooled to 20 K without an external magnetic field, and then the external magnetic field was increased from 0 to 1 T at 20 K. Finally, the magnetic field was reduced from 1 T to 0 T in order to trap magnetic field within the superconducting loop. The coil constant for the superconducting source magnet is 0.1 T/A. I_c measurements for the joints were performed in three ways: ZFC case by applying an increasing magnetic field from 0 T to 4 T at a fixed temperature, ZFC case by applying a decreasing magnetic field from 4 T to 0 T at a fixed temperature, field cooling (FC) case in which the temperature is decreased below T_c under the external magnetic field.



Figure 4.22. Closed cycle cryostat system with 7 T superconducting magnet used for transport measurements.



Figure 4.23. Closed cycle cryostat system with 4.5 T superconducting magnet used for transport and magnetic field decay measurements.

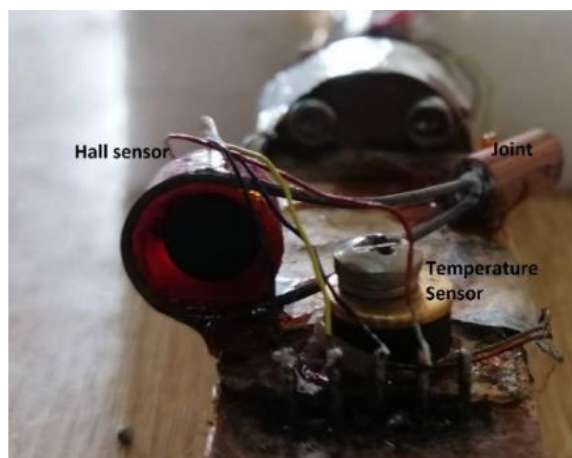


Figure 4.24. Single turn coil with a termination type joint sample placed on the sample holder for a magnetic field decay measurement.

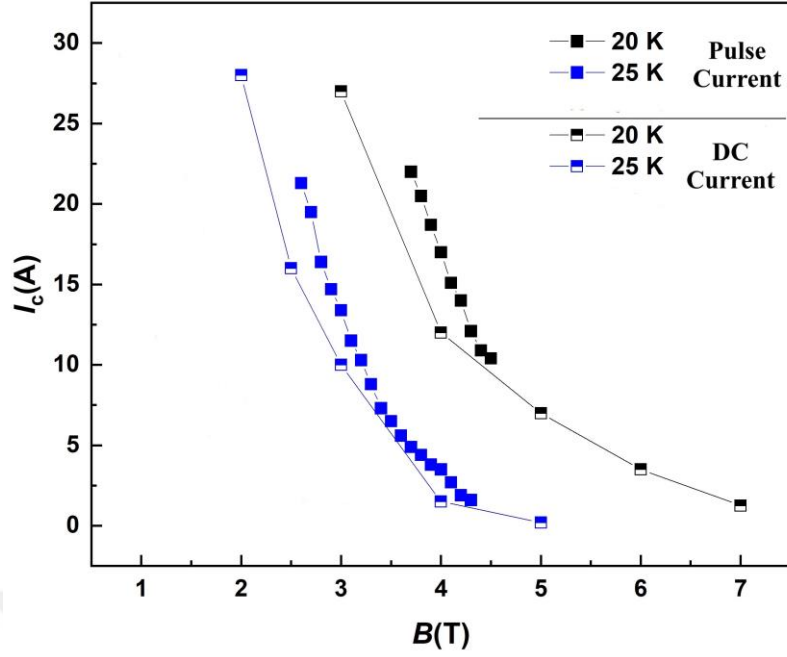


Figure 4.25. $I_c(B)$ values obtained using pulse and DC current sources.

Transport measurements were also performed on reacted MgB_2 wires bent to different bending diameters. The bending apparatus, which has many discs with bending diameters ranging from 100 mm to 350 mm, is shown in Figure 4.26. The reacted wires were first bent at room temperature, and then the bent samples were placed in the cryostat system. Measurements were made to determine bending tolerance values of the wires. Each bending process was performed on different wire pieces cut from the same wire. The straight wire pieces were selected to be close to each other according to their critical current values.



Figure 4.26. Circular apparatus used to bend MgB_2 wire pieces. Each disc has a different diameter.

5. RESULTS AND DISCUSSIONS

5.1 Experimental results for IMD MgB₂ wires

5.1.1 Current - voltage and resistivity - temperature measurements

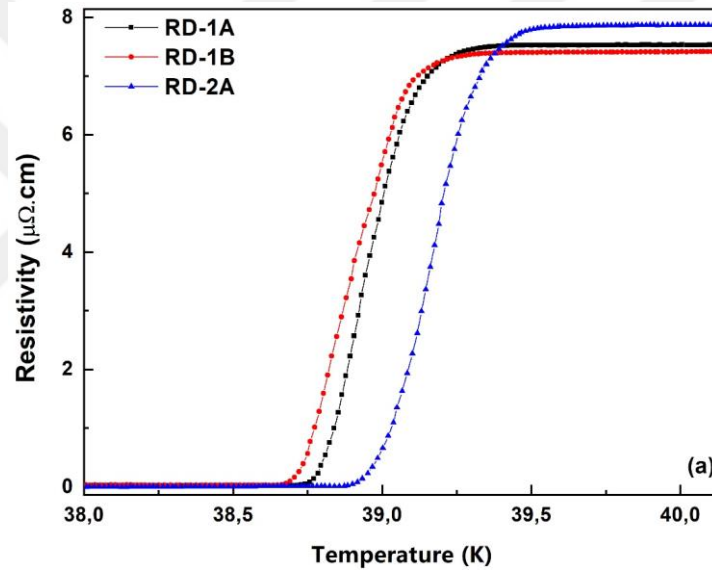
In Table 5.1, some properties, production parameters and codes of the IMD wires produced by using Mg rich Mg + B tablets, Mg powder, and Mg rod are given.

Table 5.1. The letters A and B in the wire codes indicate the annealing times of 60 and 240 minutes, respectively. “RD” stands for design IMD MgB₂/Fe wires, “P” stands for powder IMD MgB₂/Fe wires, and “S” stands for standard IMD MgB₂/Fe wires. The wire samples heat treated at 650 °C and 700 °C are coded as 1 and 2, respectively. The wire diameter for all samples is 0.81 mm.

WIRE CODE	TEMPERATURE (T _A) (°C)	TIME (T _S) (MIN.)	T _C , OFFSET (K)	ΔT _C (K)	P (40 K)
RD-1A	650	60	38.70	0.73	7.52
RD-1B	650	240	38.65	0.72	7.41
RD-2A	700	60	38.85	0.73	7.86
S-1A	650	60	37.40	1.06	8.02
S-1B	650	240	38.74	0.75	8.89
S-2A	700	60	37.65	1.22	6.39
P-1B	650	240	37.29	0.90	3.53

Figure 5.1 shows the resistivity-temperature (ρ - T) curves of design and standard IMD wire samples annealed at different annealing times and temperatures. The normal state resistivity values of RD-1A, RD-1B, and RD-2A wires, produced by the tablet method, are close to each other and are below 8 $\mu\Omega$.cm for all of them. A superconducting phase transition was observed in all (RD) IMD wires and the phase transition width (ΔT_c) from the normal state to the superconducting state for these wires is below 1 K as shown in Table 5.1(a). The wire with the highest $T_{c \text{ offset}}$ value is RD-2A sample. The fact that the (RD) IMD wires have a very sharp phase transition indicates that good MgB₂ phase formation is achieved, the impurities are low in the structure, or the very few impurities present do not produce a resistive effect (P. C. Canfield *et al*, 2001). It was observed that (RD) IMD wires have very sharp phase transitions and high $T_{c \text{ offset}}$ values even at shorter annealing times

compared to standard IMD wire samples produced under similar conditions. For instance, in the study of Kovac *et al.*, IMD wires annealed for 40 minutes and 90-minutes at 650 °C, a phase transition width of 2-3 K (P. Kovac *et al*, 2016). Figure 5.1(b) shows that the normal state resistivity and $T_{c \text{ offset}}$ values in (S) IMD wires change depending on the heat treatment temperature and time, revealing that (S) IMD wires are more affected by heat treatment temperature and time compared to (RD) IMD wires. The superconducting phase transition temperatures at zero resistivity ($T_{c \text{ offset}}$) for (S) IMD wires are also given in Table 5.1. Depending on the annealing temperature and time, the transition width was observed to be more than 1 K for the S-1A and S-2A wires and less than 1 K for S1-B wire.



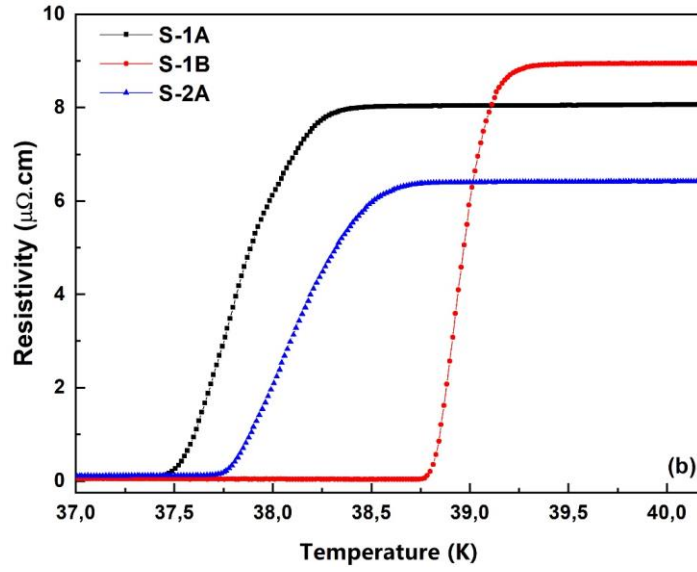


Figure 5.1. Temperature dependent resistivity curves for (a) (RD) and (b) (S) IMD MgB₂/Fe wires.

The $I - V$ measurements were also performed on (RD) and (S) IMD wires under external magnetic field of 3.5 T at 28.5 K. Figures 5.2 (a) and (b) show the E vs. J_e curves for these wires. The electric field (E) is obtained dividing the measured voltage to a distance between voltage contacts ($d_{(V+)-(V-)}$) and engineering current density (J_e) is calculated dividing the applied current to total cross-sectional area of the wires. RD-1A wire annealed at 650 °C for 1h has the lowest J_e value while RD-1B wire annealed at 650 °C for 4h has the highest J_e value as seen in Figure 5.2 (a). It seems that long annealing time improves the transport properties of (RD) IMD wires. The fact that the J_{ce} value of RD-2A is higher than that of RD-1A shows that the annealing temperature also plays a positive role on the transport properties of (RD) IMD wires. It is shown in Figure 5.2 (b) that when the annealing temperature or time is changed, the differences in J_e values of (S) IMD wires is more pronounced compared to (RD) IMD wires. The J_e values of the S-1A and S-2A wires are lower than that of (RD) IMD wires but the J_e value of S-1B wire is higher than the J_e value of RD-1B wire. These remarkable variations indicate that transport properties in (S) IMD wires are more dependent on annealing temperature and time than (RD) IMD wires.

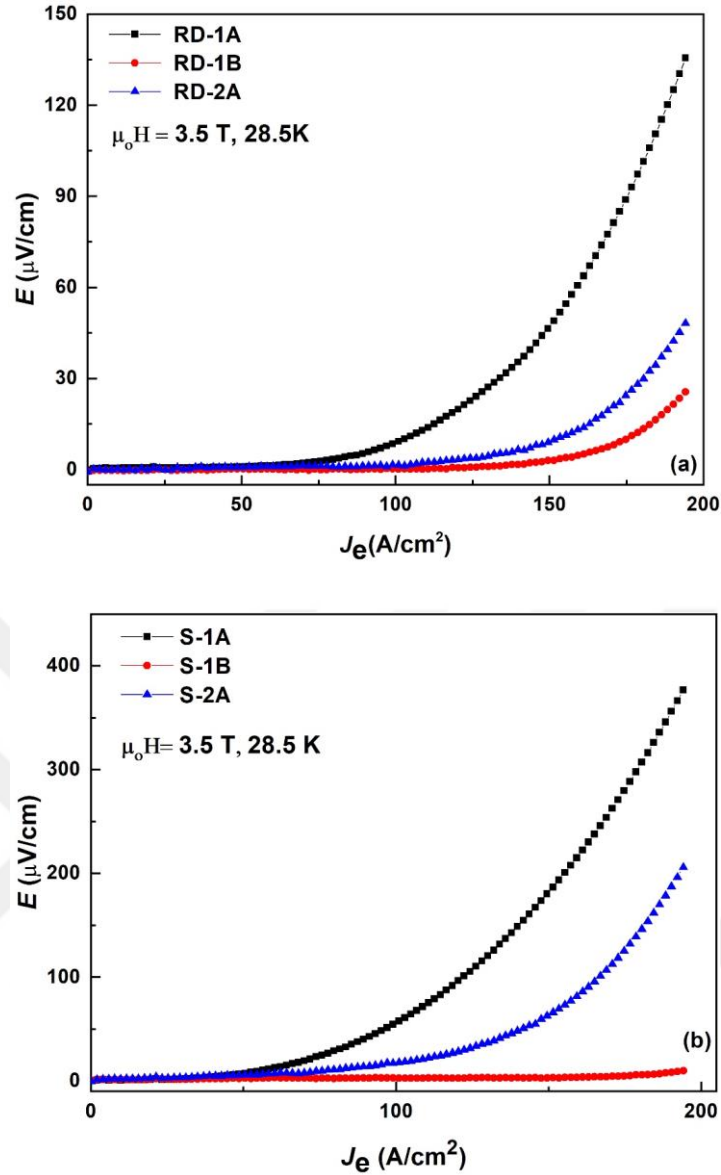


Figure 5.2. The current density versus electric field curves of (a) (RD) and (b) (S) IMD MgB₂/Fe wires.

The magnetic field-dependence of J_e values, obtained from the $E - J_e$ curves of the (RD) and (S) IMD wires, are shown in Figure 5.3 (a). It is seen that the highest engineering critical current density belongs to S-1B which is annealed at 650°C for 4h. When the $J_{ce}(B)$ curves are examined, annealing at 650°C for 1h or at 700°C for 1h worsens the transport properties of (S) IMD wire. It is seen that S-1B wire has a slightly higher J_{ce} value than RD-1B wire but the J_{ce} values of S-1A and S-2A wires are lower than RD-1A and RD-2A wires. When the J_{ce} values of the produced IMD wires are evaluated with reference to the J_c values of the IMD wires between 25 K and 30 K (Shujun Ye *et al*, 2016), it is understood that the transport

properties of (RD) and (S) IMD wires are quite good. The n -values are obtained by fitting the $E - J_e$ curves of the (RD) and (S) IMD wires according to the power law ($E \sim J^n$) and given in Figure 5.3(b). The n -value is important for the use of MgB_2 wire in coil applications (such as magnetic resonance (MRI) coils) and is expected to be greater than 30 at around 20 K. Here, it is seen in Figure 5.3(b) that S-1B and RD-1B wires have n values of 9 and 8.5 under the external magnetic field of 3.5 T at 28.5 K, respectively. The wires are capable to achieve required n -value for MgB_2 magnets to be used in permanent mode at 20 K (A. Motaman *et al*, 2014).

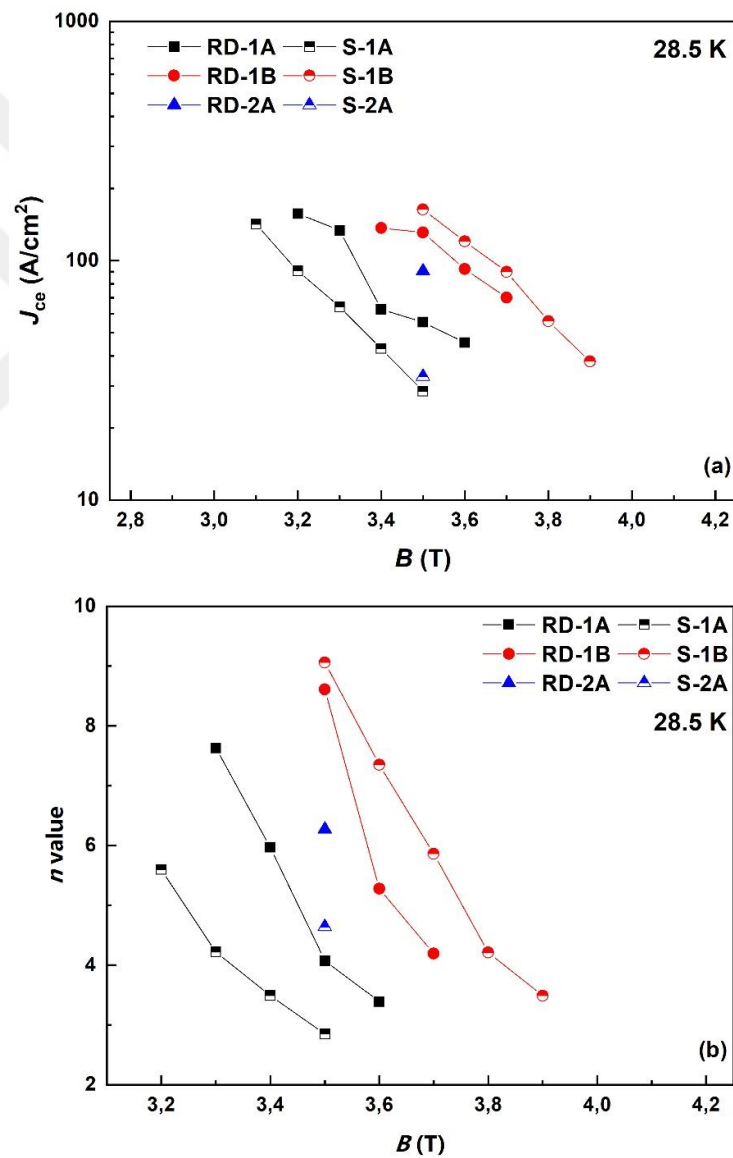


Figure 5.3. Magnetic field dependence of (a) J_{ce} and (b) n values obtained from $E - J_e$ curves of (RD) and (S) IMD wires.

5.1.2 Structural analysis

Figure 5.4 shows the images taken from the cross-sectional surface of (RD) and (S) IMD wires. The images were obtained after the wires were polished by placing them in a conductive bakelite mold. The images in Figures 5.4(a), (b), and (c) are of (RD) IMD wires. It is seen that the central porous superconducting MgB_2 structure and a dense MgB_2 ring surrounding it were successfully obtained. On the other hand, there is a central hole in the center of the (S) IMD wires produced by using a Mg rod as shown in Figures 5.4 (d), (e), and (f). It is seen in the images given in the right column of Figure 5.4 that this hole filled with conductive bakelite. When (RD) and (S) IMD wires fabricated under the same heat treatment conditions are examined in terms of their core structures, remarkable differences between (RD) and (S) IMD wires are seen depending on the annealing temperature and time. Especially, when the core structures for RD-2A in Figure 5.4(b) and for S-2A in Figure 5.4 (e) are analyzed, it is seen that MgB_2 phase formation is not complete for S-2A. Mg diffusion partially occurs and a sickle-shaped boron region remains (in gray color), where diffusion cannot reach to a large extent. This boron layer, in which the reaction is not completed, causes a resistance when the current is transferred to the superconducting core in the wire and cannot contribute to the critical current value. When RD-2A wire is examined, it is seen that MgB_2 formation is more complete than S-2A but there are also small boron regions. Another significant difference is between RD-1B and S-1B wires, that S-1B wire has a thicker superconducting MgB_2 ring structure. The superconducting cores of the RD wires shown in Figures 5.4 (a), (b), and (c) have a dark orange color under the optical microscope. This indicates that (RD) IMD wires have a denser MgB_2 core structures than (S) IMD wires because a dense MgB_2 is more reflective feature (S. Ye *et al*, 2016). The main reason for the formation of a thinner MgB_2 IMD layer in (RD) IMD wires than (S) IMD wires after the reaction is that the total stoichiometry between Mg and B can be achieved with a thin boron layer during the filling process for (RD) IMD wire production.

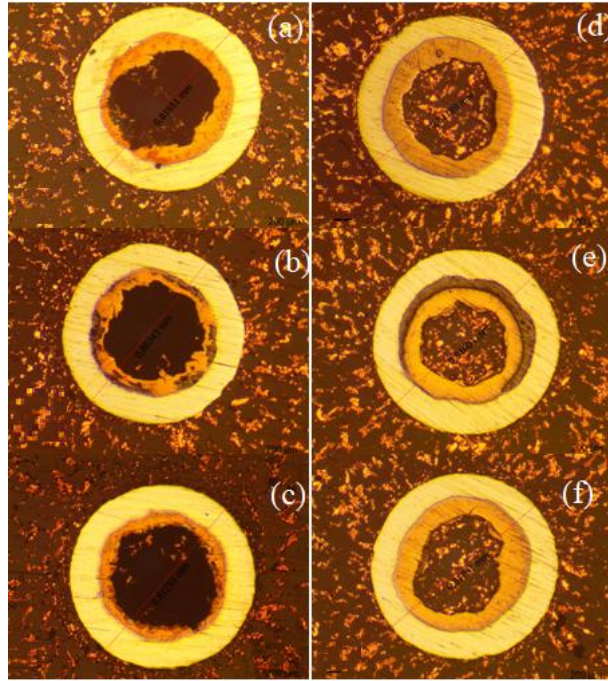


Figure 5.4. (a-b-c) show the polished cross-sectional optical microscope images (5x magnification) of RD-1A, RD-2A and RD-1B, and (d-e-f) of S-1A, S-2A and S-1B wires, respectively.

Scanning electron microscope images revealing the core structure in detail are shown in Figure 5.5. Similar to optical microscope images, SEM images given in Figures 5.5(a), (b), and (c) show that (S) IMD wires have a central hole and surrounding MgB_2 ring structure. On the other hand, the porous structure formed in the central region of (RD) IMD wires is revealed using SEM as shown in Figures 5.5(d), (e), and (f). SEM images show that (RD) IMD wires were successfully fabricated and a porous structure was created instead of a central hole as intended. In addition, it is understood from the SEM images that a circular dense MgB_2 structure was formed around this porous structure by diffusing the powder magnesium, which is in a mixture with boron powder in the wire center. It is possible to control the porosity in (RD) wires, but it may be necessary to dispense with the stoichiometry. It has been understood that MgB_2 phase formation is better at low temperatures and short-term annealing for (RD) IMD wires but the wires should be optimized structurally. The positive contribution of core design to Mg diffusion can be seen, considering the boron layer contained in the S-2B sample given in Figures 5.4(e) and 5.5 (b). It is seen that the dense MgB_2 layer formed internal region of S-2B IMD wires at annealing temperature of 700 °C prevents Mg

to diffuse further (S. Ye *et al*, 2016). To overcome this difficulty, IMD wires with very thin MgB_2 layer were produced, but IMD wire with a large hole has low engineering critical current density. It is also difficult to maintain the structural uniformity for an IMD wire with a thin MgB_2 layer throughout the wire (P. Kováč *et al*, 2021). The observed changes in the transport properties of S1-A and S1-B IMD wires can be considered as a result of longitudinal non-uniformity. S1-A and S1-B have similar core structures as seen in Figures 5.4(d) and (f), but there is a notable difference in J_{ce} values.

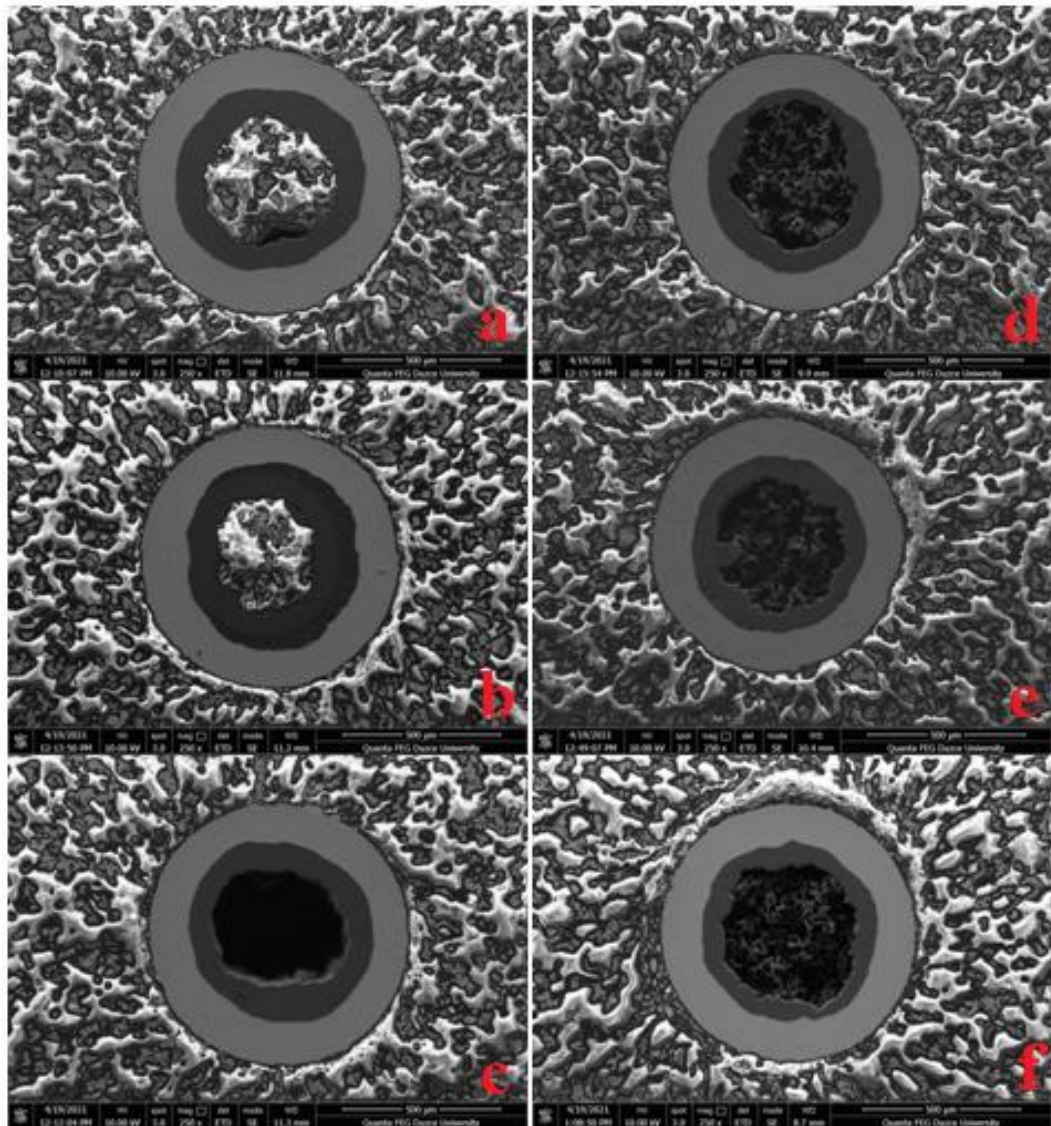


Figure 5.5. Scanning electron microscope images (for 250 $\times$ magnification) taken from the polished cross-sectional areas of S-1A, S-2A and S-1B wires (a-b-c) and RD-1A, RD-2A, and RD-1B (d-e-f) wires.

SEM images taken from the polished core surfaces of S2-A and RD-2A wires at 2000 $\times$ magnification are given in Figure 5.6. S-2A and RD-2A wires subjected to heat treatment at 700 °C for 1h have unreacted boron regions, layered and partial, respectively. It is possible to separate regions where the reaction completely occurs from the regions where the superconducting MgB₂ structures are not fully formed. In regions where the MgB₂ structure is not good, spills occur during the polishing and washing process, while the regions where the superconducting structure is well formed have a hard and smooth surface. It is seen that the rapid reaction of Mg with nano-sized B at 700 °C causes a dense MgB₂ structure in the S-2A wire, which prevents further diffusion of Mg towards the boron layer, as seen in Figure 5.6 (a). Since the dense MgB₂ structure in the interior prevents the diffusion of magnesium, Mg cannot reach the regions close to the sheath, and an unreacted boron layer is left, which causes resistance in current transfer, a decrease in the critical current density and quenching at high currents (Shimida Y. *et. al*, 2011). The unreacted boron layer observed in the S-2A wire does not apply to the RD-2A wire, instead there are small regions of partially unreacted boron.

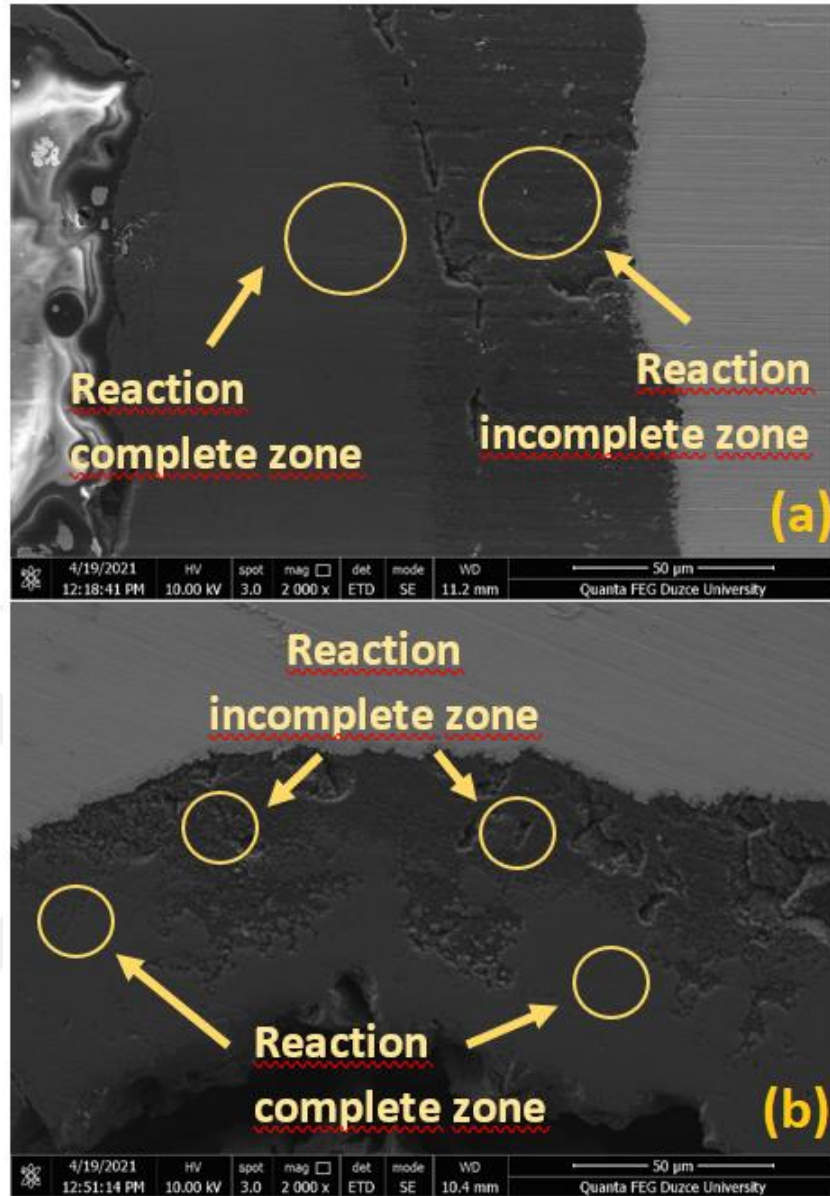


Figure 5.6. SEM images of (a) S-2A and (b) RD-2A wires obtained at 2000 $\times$ magnification from regions having structural differences in the wire core.

In Figures 5.7 and 5.8, SEM images obtained with 100.000 $\times$ magnification, are given from the regions where the reaction occurred and partially occurred in the S-2A and RD-2A wires, respectively. It is obviously seen in Figure 5.7(a) that there are many small unreacted boron clusters which reflect themselves as black spots in the dense MgB_2 layer. On the other hand, spherical boron grains are visible in the regions where MgB_2 formation is not completed as shown in Figures 5.7(b) and 5.8(b). The reason for the formation of boron clusters in S-2A wire core is MgB_2 formation may surround some boron and Mg cannot reach these boron regions, so

the reaction of boron with Mg in this region is prevented. As shown in Figure 5.8(a), a large number of boron clusters observed in the dense MgB_2 layer of S-2A are less in RD-2A. This is thought to be due to the fact that RD-2A wire has magnesium in powder form and the thickness of dense MgB_2 ring layer in RD-2A is thinner than S-2A. In (RD) IMD wires, source Mg is filled in powder form, in which case the Mg is in the form of small spherical granules. During wire production, these granules elongate and turn into fine Mg fibers dispersed in boron powder. In standard IMD production, a magnesium rod used at the wire center elongates during drawing and form a thin Mg wire. When the two cases are evaluated in terms of reaction kinetics, it can be thought that the fine Mg fibers dispersed in B powder find more diffusion paths and diffuse more outward, thus reacting with more B. Since Mg diffusion did not develop uniformly at every point in the (RD) IMD wires, some unreacted B regions are remained (Figure 5.6(b)).

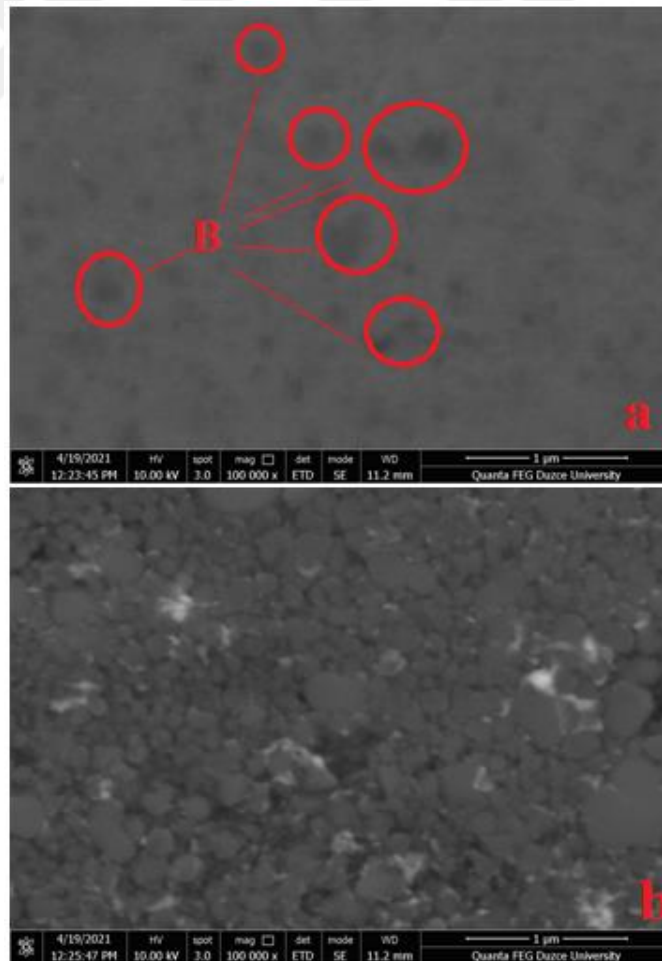


Figure 5.7. SEM images obtained at 100.000× magnification from the (a) reaction complete and (b) reaction incomplete core regions of S-2A sample.

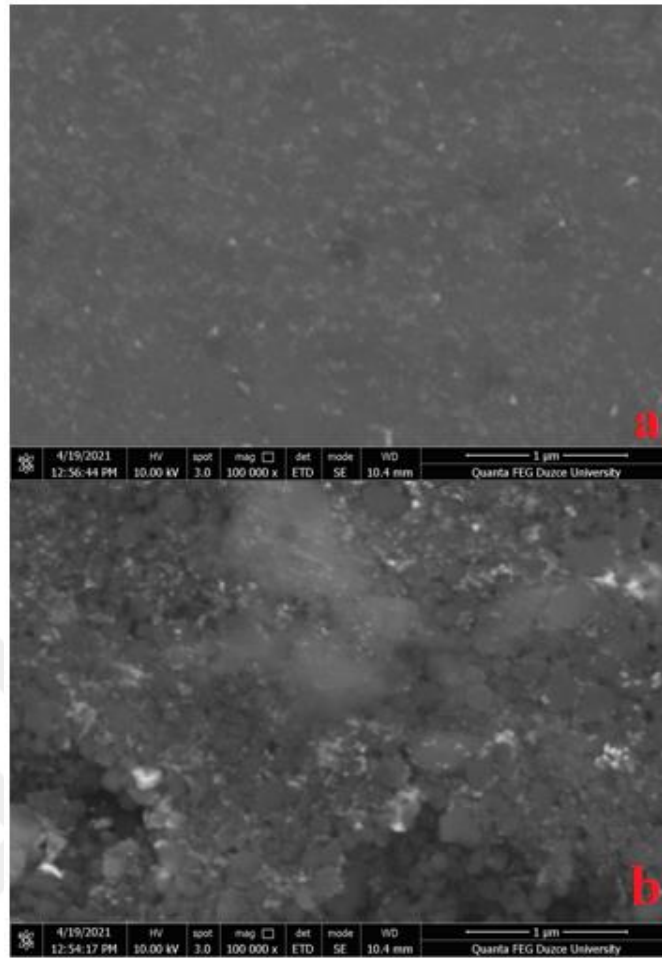


Figure 5.8. SEM images obtained at 100.000× magnification from the (a) reaction complete and (b) reaction incomplete core regions of RD-2A sample.

Elemental mapping analysis for S-2A and RD-2A wires was also performed using scanning electron microscopy as shown in Figures 5.9 and 5.10. It was realized that magnesium concentration increases in regions where the reaction is completed and decreases considerably in regions where the reaction is incomplete. For boron, on the other hand, the opposite picture of magnesium is obtained. Regions where the reaction is not completed in the RD-2A sample are less than in S-2A sample. The phase difference is clearly seen when looking at the picture in which all element maps are brought together in Figures 5.9 and 5.10. It is seen that the boron and oxygen concentrations are dominant in the regions close to the edges. The reason why only Mg is seen in the regions where the reaction takes place is related to the fact that the atomic diameter of Mg is quite large compared to B.

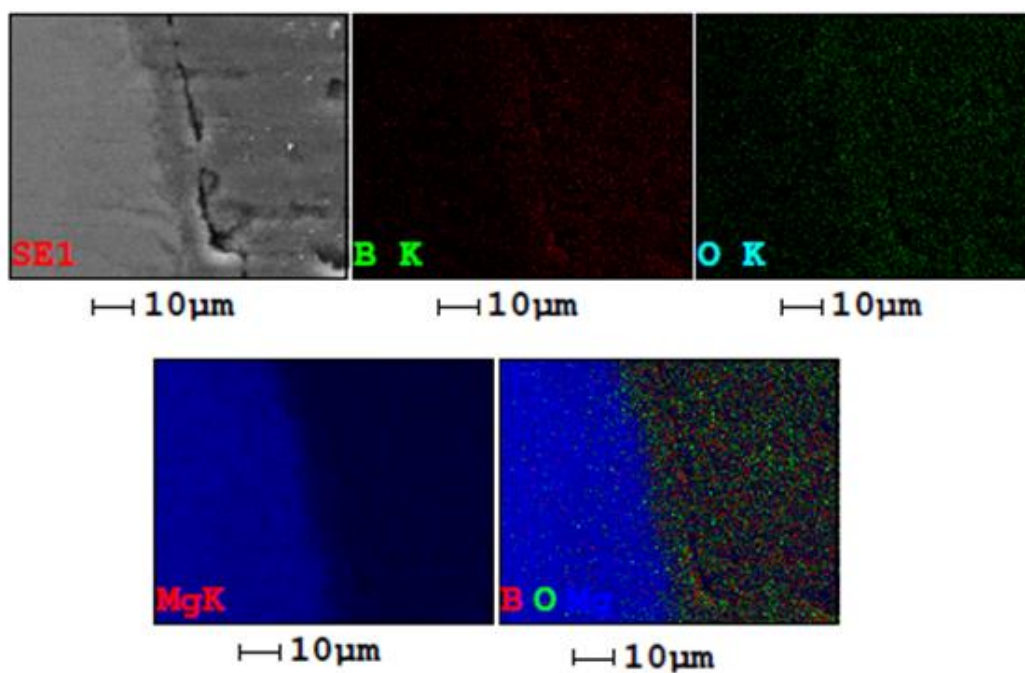


Figure 5.9. Elemental mapping analysis for S-2A sample at 2000 $\times$ magnification.

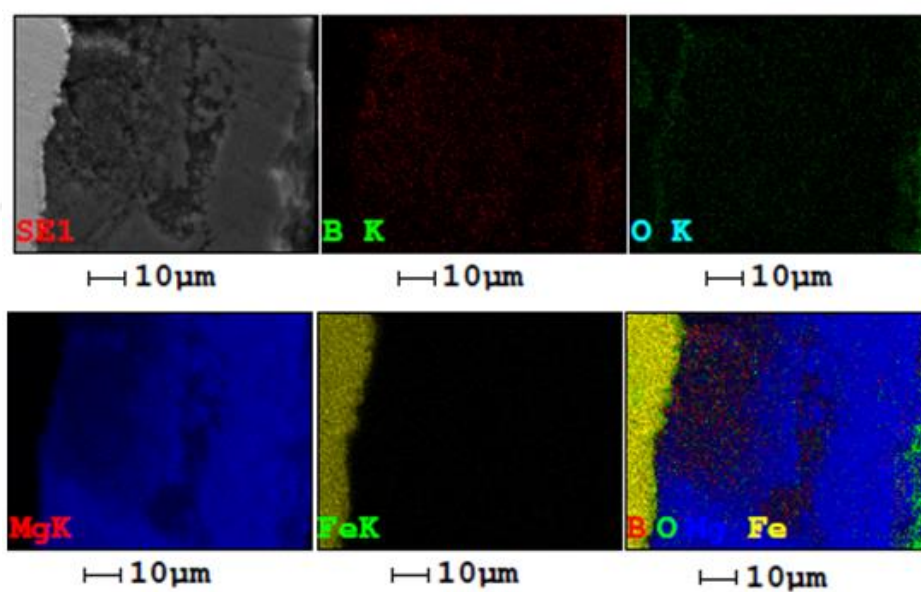


Figure 5.10. Elemental mapping analysis for RD-2A at 2000 $\times$ magnification. Color codes: Boron-Red, Magnesium-Blue, Iron-Yellow and Oxygen-Green.

5.2 Investigation of electrical and electromechanical properties of IMD MgB₂/Fe wires in terms of production methods.

5.2.1 Current - voltage measurements with fast current ramp

The results of the transport measurements of RD-1B, S-1B, and P-1B wires at various temperatures above 30 K are shown in Figures 5.11(a) to (d). The $E - I$ curves were obtained by initially setting the temperature to 36 K and then applying the current incrementally within 1 second. Then the temperature was lowered by 1K and the measurements were repeated. The $E - I$ characteristic curves were used to calculate the I_c values for self-field using the 1 $\mu\text{V}/\text{cm}$ criterion. RD-1B wire showed a very abrupt voltage peaks at low applied currents but these peaks were suppressed when the applied current was increased towards I_c value (see Figure 5.11(a)). No physical damage (burning) was observed even at high applied currents; that is, RD-1B wire was measured without any degradation in its I_c value over all temperature ranges. In Figures 5.11(b) and (c), showing the $E - I$ curves of S-1B and P-1B wires, it is seen that the voltage peaks are lower than those of RD-1B wire at low currents, but there are some voltage fluctuations above the noise level. However, in the $E - I$ curves of the P-1B sample measured at 34 K, voltage distortions are observed at high applied currents close to I_c . This behavior, which is indicative of the quenching effect, arises as a result of the inability to increase the current further. As a result, P-1B wire was totally burned when it was tried to measure again at 34 K. When $I - V$ measurements were performed on S-1B wire at 32 K, a comparable but moderate quenching effect around I_c was also observed. It seems that, the current contacts may be responsible from the quenching at small wires. S-1B and P-1B wires were damaged at high currents even if the entire current was applied for a short duration of 1s.

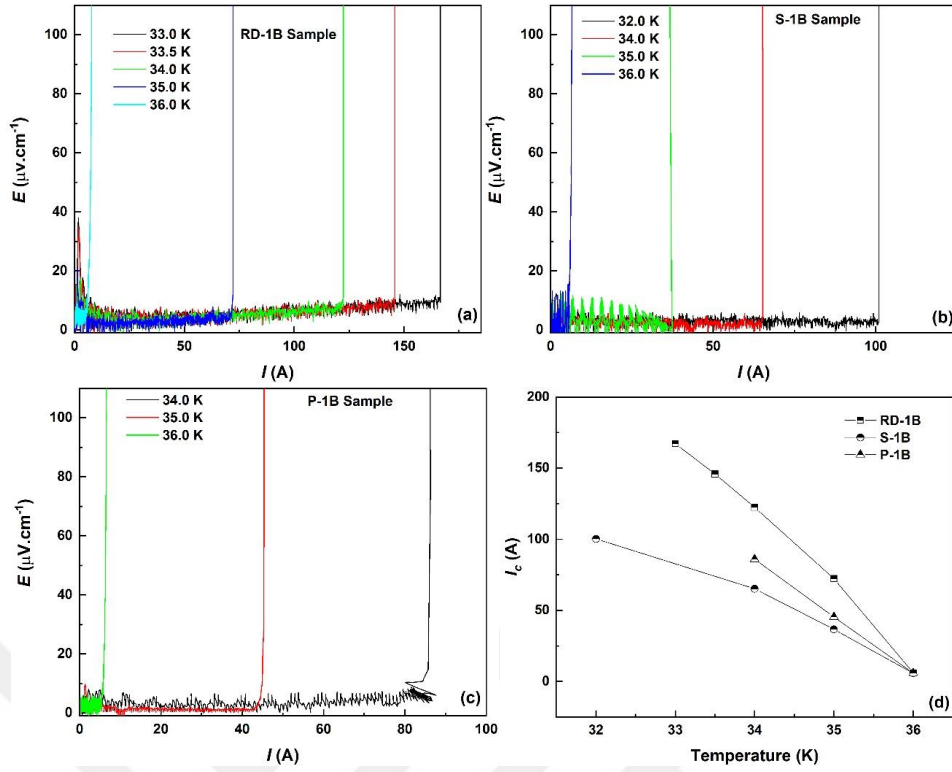


Figure 5.11. $E - I$ curves of (a) RD-1B, (b) S-1B, and (c) P-1B wires. (d) $I_c(T)$ behavior of RD-1B, S-1B, and P-1B wires in the self-field.

The $I - V$ measurements performed in self-magnetic field reveal that Fe sheath is insufficient to prevent quenching effects even below the critical currents of IMD wires (see Figure 5.12). The greatest I_c of 167 A at 33 K was recorded for RD-1B wire without any quenching. IMD wires appear to be more prone to quenching, at least for these three wires, as previously mentioned in the study of (Kováč *et al*, 2020). The IMD/PIT hybridization in (RD) IMD wire contributes to compensating the low thermal stability of MgB_2/Fe IMD wire in order to counteract the detrimental effects of quenching. This may be achieved by sharing the applied current by dense MgB_2 layer and the central porous MgB_2 core. Although the S-1B wire has the highest J_c under the external magnetic field, as shown in Figure 5.14, the (RD) IMD wire appears to be able to carry higher currents in the absence of the external magnetic field. In this respect, it has been shown that the porous structure in the middle of the (RD) IMD wire is able to compensate the loss of J_c observed in the (S) IMD wires, but the J_c of porous MgB_2 structure still needs to be improved under the external magnetic field.

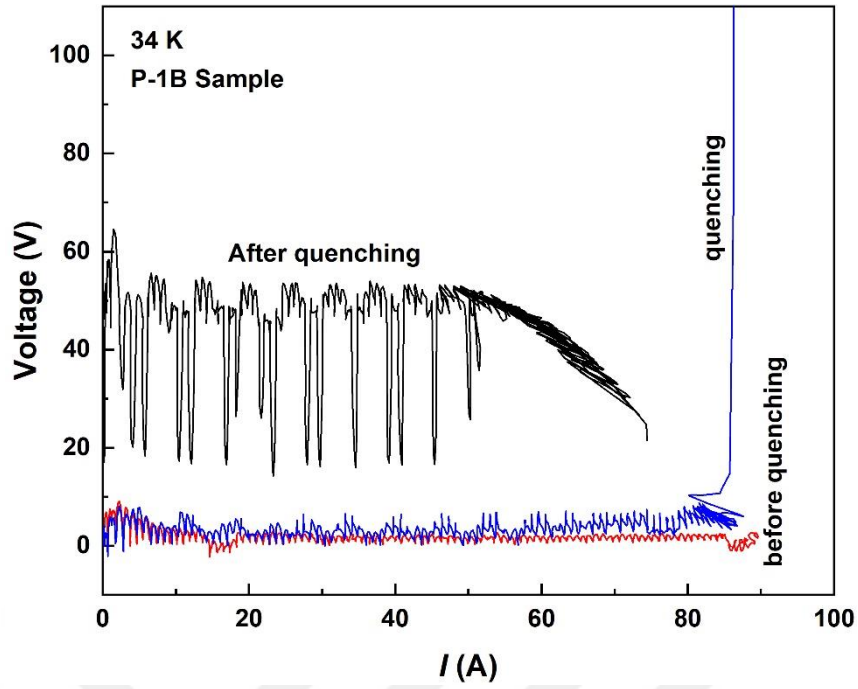


Figure 5.12. Quench development for P-1B wire at 34 K under self-magnetic field.

5.2.2 Current - voltage measurements of bent wires

In this section, the electromechanical properties of wires produced by different methods are examined. In Figures 5.13, 5.14, and 5.15, the $E - J_e$ curves of RD-1B, S-1B, and P-1B, obtained under the external magnetic field at 25 K, are given. The $I - V$ measurements were performed on both straight and bent wires. Bending diameters were determined as 350 mm, 300 mm, 250 mm, 200 mm, 175 mm, 150 mm, 125 mm, and 100 mm, respectively. The $E - J_e$ curves were obtained for the bending diameter of 200 mm or less as seen in Figures 5.13 to 5.15. This is because the $E - J_e$ measurements were made by regarding the first degradation in critical current of the straight wire due to the bending. The straight lines show the $E - J_e$ curves of the samples before bending, while the dashed lines show the $E - J_e$ curves after the bending in Figures 5.13, 5.14, and 5.15. It is seen that RD-1B and S-1B were not adversely affected when these wires are bent to a diameter of 175 mm. However, when they are bent up to a bending diameter of 150 mm, critical current values begin to deteriorate. The smaller the bending radius is the greater the amount of degradation in the critical current. When the $E - J_e$ curves of P-1B given in Figure 5.15 are examined before and after the bending process, it is seen that P-1B has a critical bending diameter of 175 mm. When the $E - J_e$ measurement results

obtained for all samples are compared, it has been observed that the P-1B is most affected by the bending process.

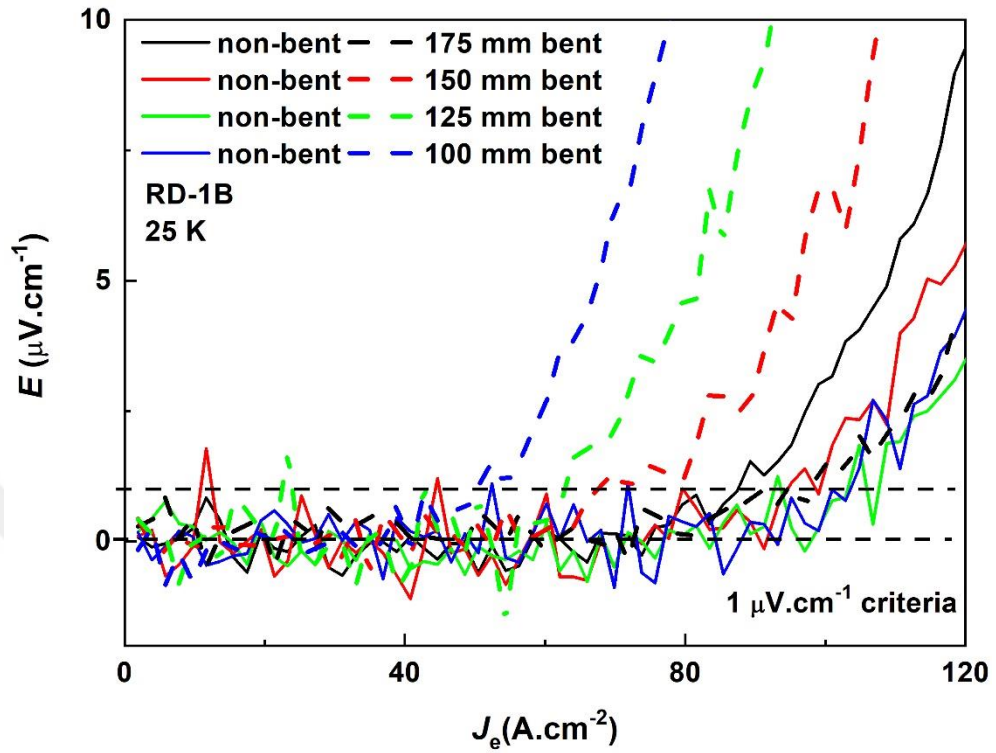


Figure 5.13. $E - J_e$ curves of RD-1B at 25 K effect of 4.7 T external magnetic field before and after bending.

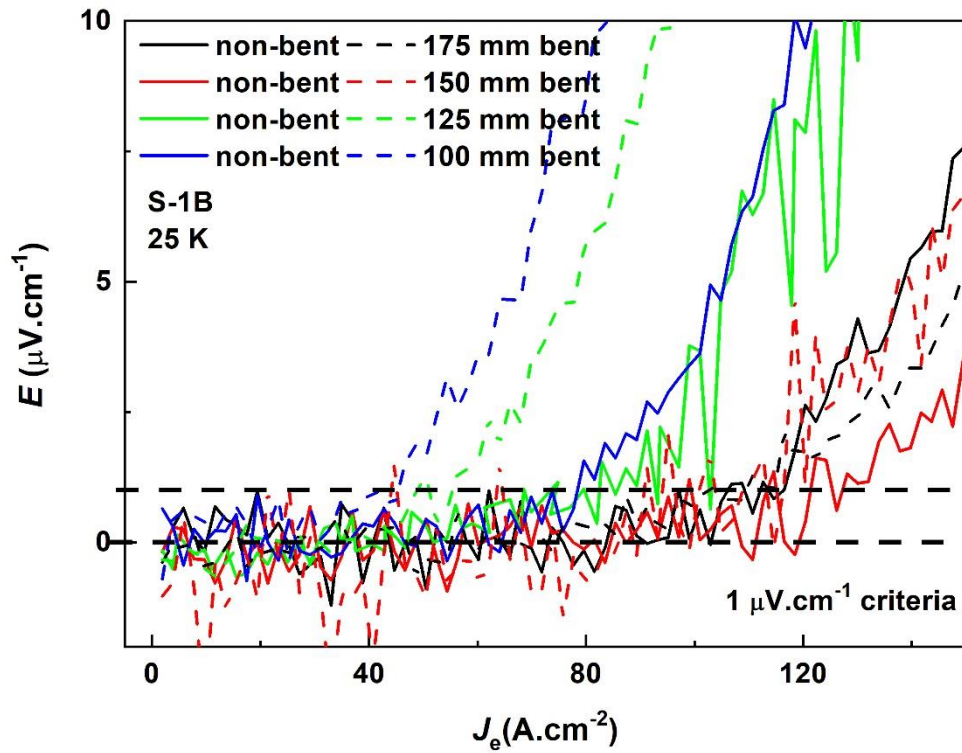


Figure 5.14. $E - J_e$ curves of S-1B at 25 K effect of 5.2 T external magnetic field before and after bending.

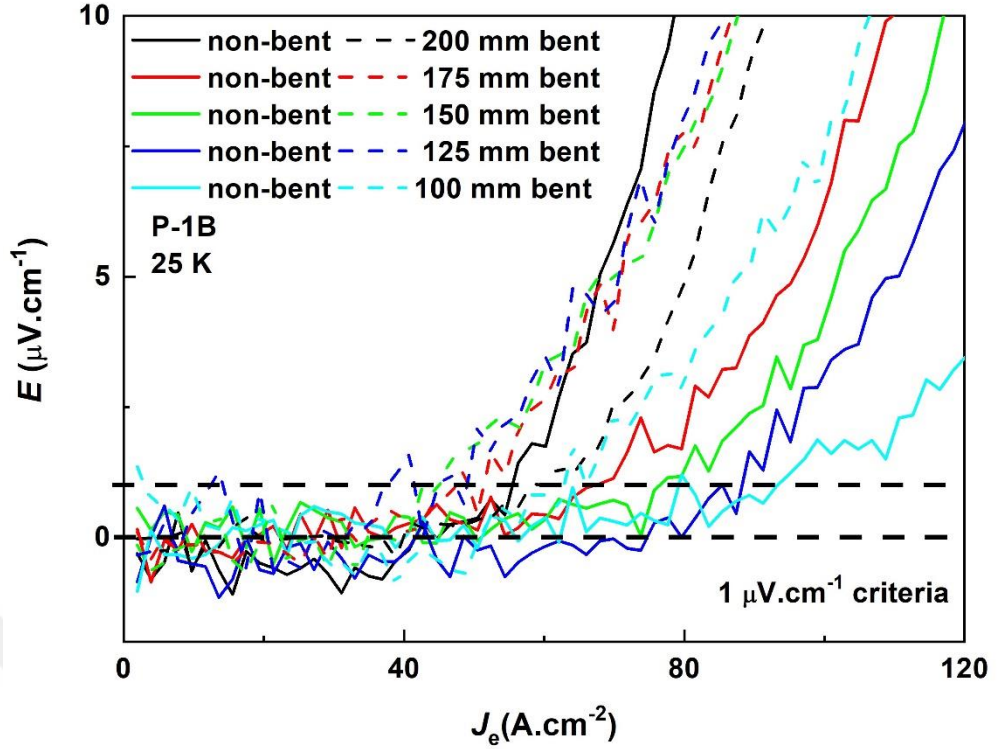


Figure 5.15. $E - J_e$ curves of P-1B at 25 K effect of 4.7 T external magnetic field before and after bending effect.

The variations of the J_c and J_{ce} values obtained from the $I - V$ measurements of the produced wires according to the samples are given in Figures 5.16 and 5.17. J_c values are calculated by dividing the critical current value to the MgB_2 core cross-sectional area and J_{ce} values are determined by dividing the critical current value to the total cross-sectional area of the wire. When the results given in Figure 5.16 are examined, if an evaluation is made between RD-1B and P-1B under the same external magnetic field, it is seen that P-1B has a higher J_c value than RD-1B but, RD-1B has more uniform J_c behavior than P-1B. This shows that RD-1B has a more homogeneous structure than P-1B. S-1B has the largest critical current density among the samples because S-1B has already the same level of J_c as the others under 5.2 T. In Figure 5.17, it is seen that S-1B has the largest J_{ce} values and RD-1B has a more uniform J_{ce} than S-1B and P-1B. When the core thickness values given in Figure 5.18 are examined, it is clearly seen that the variations in core thickness are greatest in P-1B and least in RD-1B. It is also worth noting that RD-1B has the thinnest uniform MgB_2 ring structure but the lowest J_c value because the central porous MgB_2 superconducting core area was also taken into account in J_c calculation.

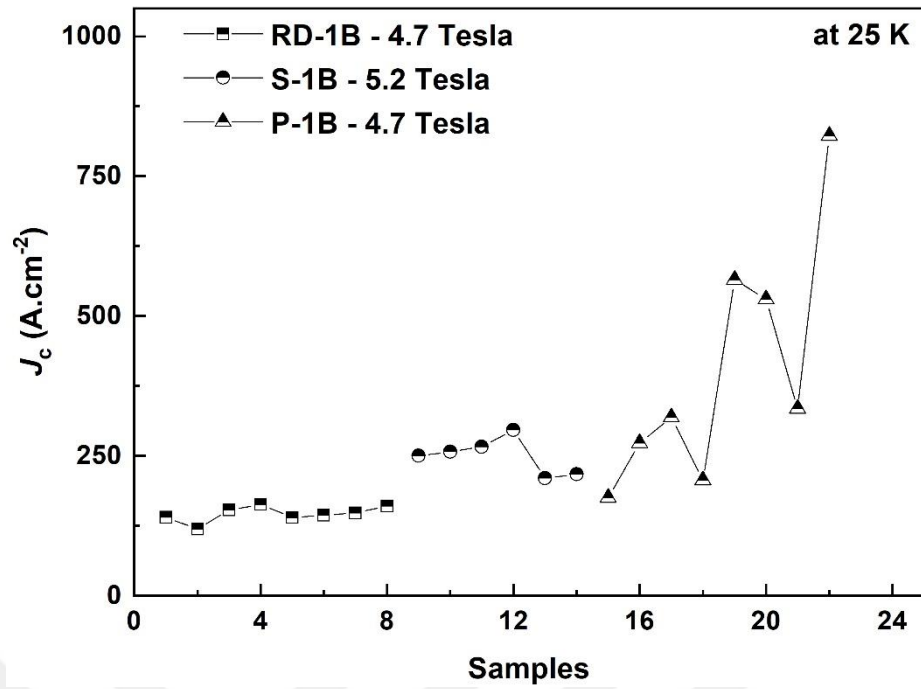


Figure 5.16. Critical current density values of multiple parts taken from RD-1B, S-1B and P-1B.

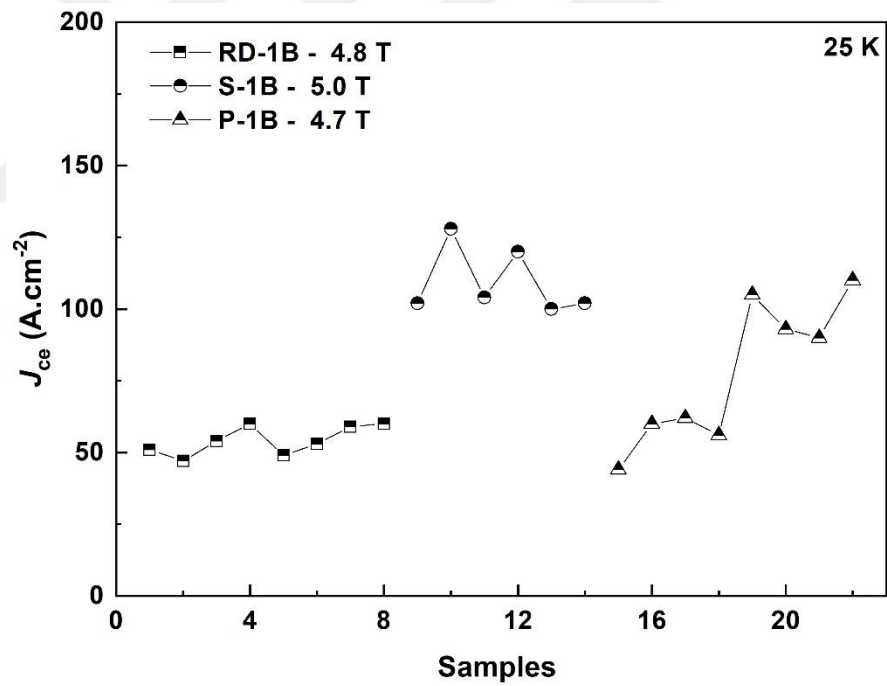


Figure 5.17. Engineering critical current density values of multiple parts taken from RD-1B, S-1B and P-1B.

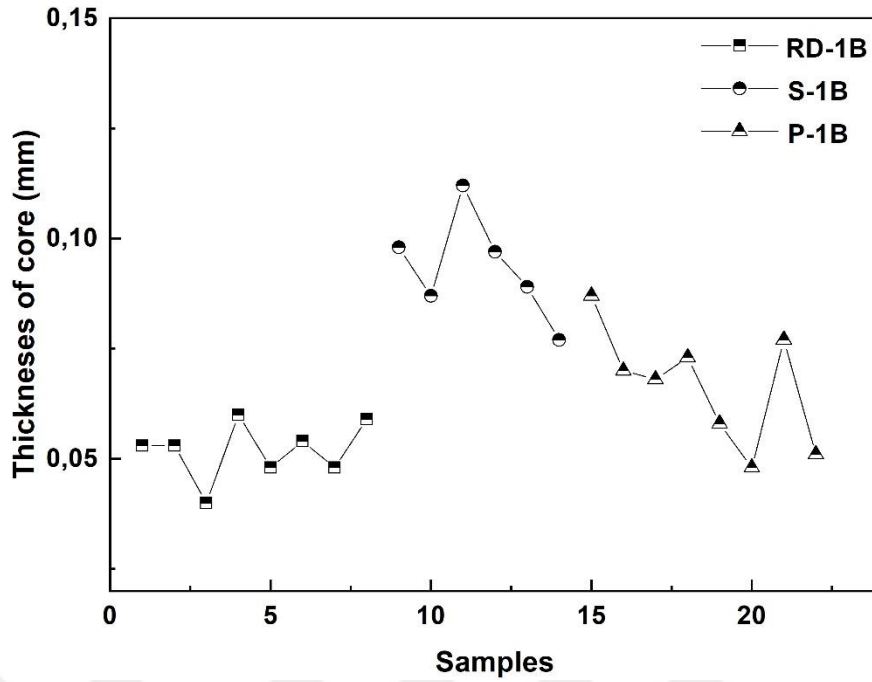


Figure 5.18. Ring core thickness values of multiple parts taken from RD-1B, S-1B and P-1B.

The normalized critical current value ($I_{c,after}/I_c$) versus the bending strain tolerance curves for the RD-1B, S-1B, and P-1B are shown in Figure 5.19. The normalized critical current value is obtained by dividing the critical current value after bending ($I_{c,after}$) to the value before bending. The bending strain tolerance is calculated using the formula given in Equation 1.

$$\varepsilon_B = \varphi/d \quad (1)$$

In this equation, ε_B represents the bending strain tolerance, φ (0.81 mm) represents the diameter of the wire sample, and d represents the bending diameter. Bending diameters are between 350 mm and 100 mm (K. Katagiri *et al*, 2005). The results shown in Figure 5.19 show that P-1B can maintain its I_c value up to a tolerance value of 0.40. The RD-1B and S-1B have a bending tolerance value of 0.46.

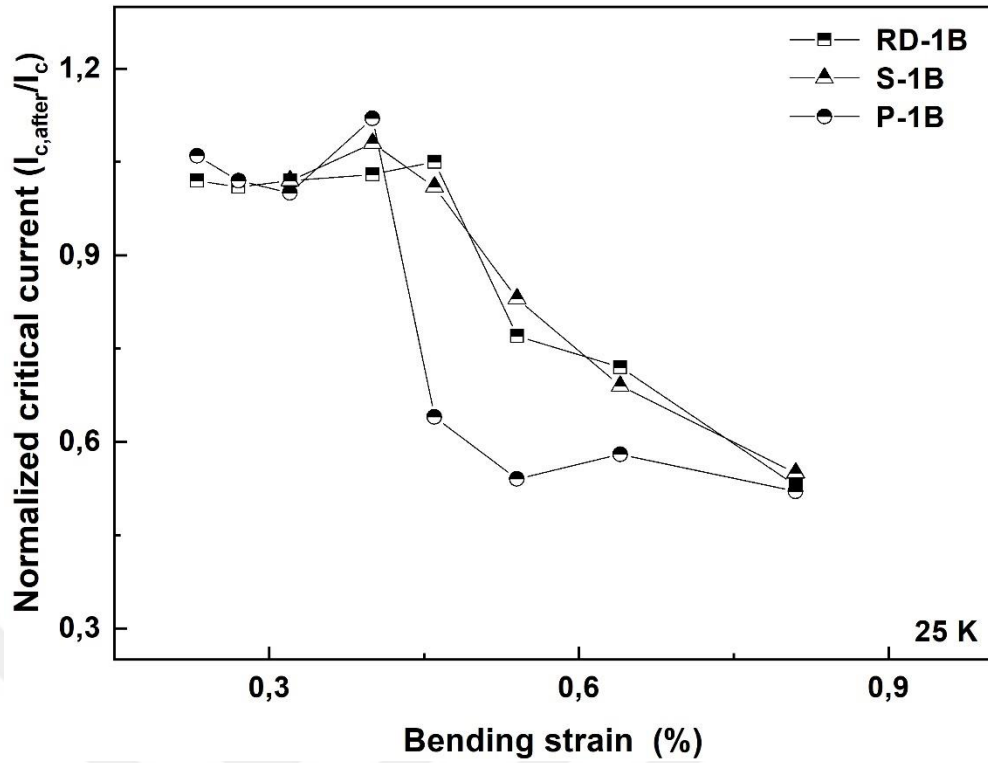


Figure 5.19. Normalized critical current vs. Bending strain characteristic curves of RD-1B, S-1B and P-1B.

5.2.3 Surface morphology analyses

Surface morphology analyses of the samples are shown in Figures 5.20-5.22. The surface images were obtained using SEM after the polishing the wire samples. The cross-section and lateral section of bent RD-1B, S-1B, and P-1B are given in Figure 5.20. SEM analysis was performed on RD-1B and S-1B wires bent to 150 mm and on P-1B wire bent to 175 mm. These bending diameters are the diameters in which the bending tolerance of the wires are first exceeded. The SEM images taken from the lateral surfaces of the samples confirm that the wires get the bending effect as can be seen in the right column in Figure 5.20. When the SEM images taken at 250× magnification from the cross-sectional surface of the samples in the left column in Figure 5.20 are examined, it is seen that there are porous MgB_2 structure in the center of the RD-1B as shown in Figure 5.20(a). The RD-1B and S-1B have a large hole in their centers and the holes are filled with conductive bakelite during polishing. One of the obvious differences between the samples shown in Figure 5.20 is the IMD core layer thickness of the samples. It is seen that the thickest core belongs to the standard IMD wire, namely the S-1B.

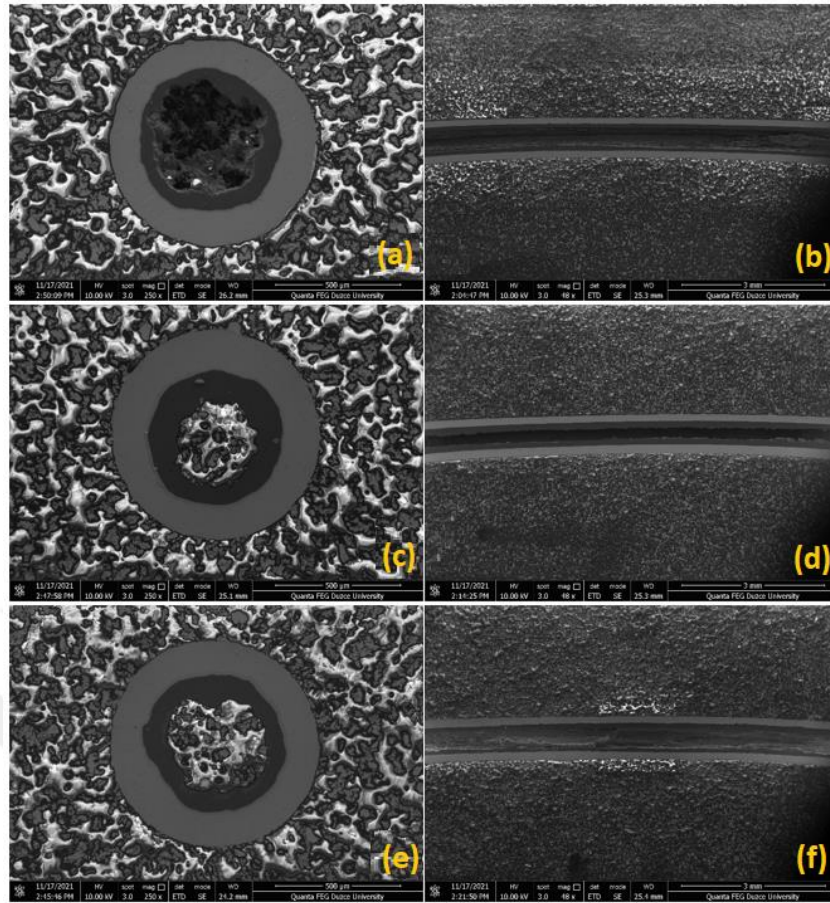


Figure 5.20. SEM images of the bent samples taken from cross-sections (left column) and lateral sections (right column). (a)-(b) RD-1B, (c)-(d) S-1B, and (e)-(f) P-1B.

The images given in Figure 5.21 were obtained from the lateral sections of the bent samples, at 200x magnification (left column) and at 1000 $\times$ or 2000 $\times$ (right column) magnifications. In the SEM images taken by zooming (Figure 5.20(b), (d) and (e)), the cracks caused by bending in the core region of the wires are clearly seen. Crack formation is consistent with the measurement results in which the bending tolerances of the wires were determined because the SEM images in Figure 5.20 were obtained from the bent wires in which the critical current value first degraded. When the crack patterns are examined, it is observed that cracks occur in transverse direction for RD-1B and S-1B, as seen in Figures 5.21(b) and (d), respectively. However, P-1B has longitudinal cracks as shown in Figure 5.21(f). These cracks in the core of the samples restrict the current flow paths and reduce the current carrying capacity of the wires.

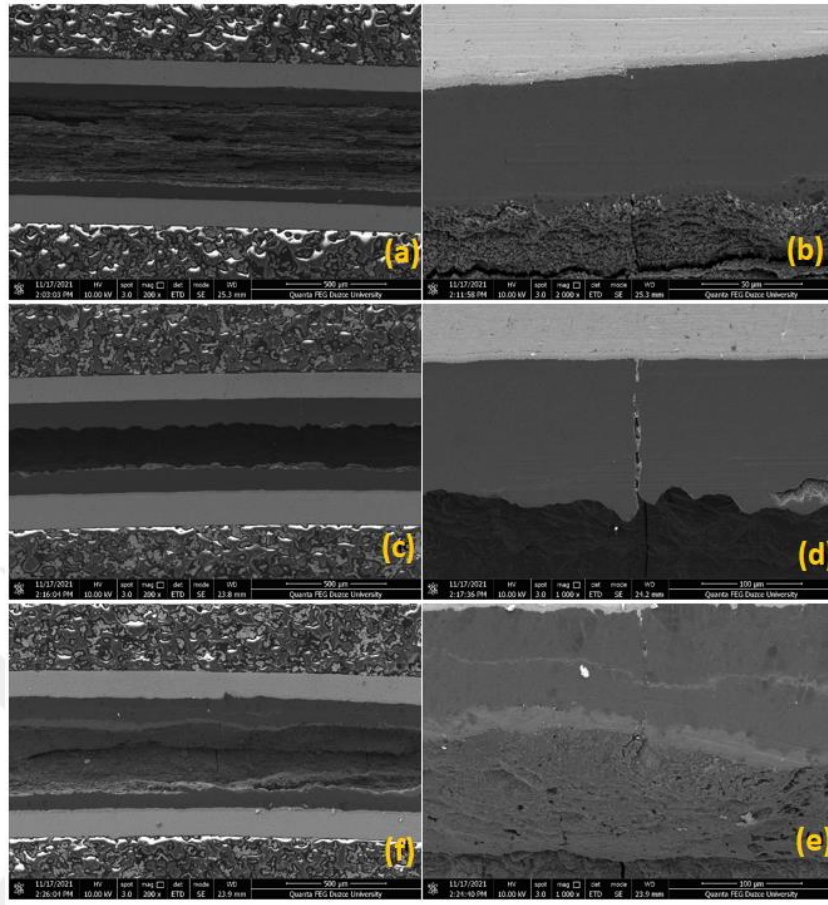


Figure 5.21. SEM images taken from the lateral sections of the bent wires at 200× (left) and 1000× or 2000× (right) magnifications. (a)-(b) RD-1B, (c)-(d) S-1B, (e)-(f) P-1B.

In Figure 5.22, images obtained from the μm lateral sections of the wire samples using an optical microscope at 5× magnification (left column) and 20× magnification (right column) are represented. Different from the SEM images given in Figure 5.21, the optical microscopy images give an information about core density in terms of the darkness of the orange color. A dense MgB_2 core has a more reflective feature and seems dark orange color (S. Ye *et al*, 2016). When the wires are evaluated in terms of the color, RD-1B and S-1B have denser cores than P-1B. On the other hand, it is clearly seen that the core thickness of S-1B is quite thick compared to RD-1B and P-1B. Considering the measurement results in Figures 5.3(a), 5.16 and 5.17, the thick dense MgB_2 layer in S1-B seems to be beneficial for J_c and J_{ce} values under the external magnetic field, but it does not constitute a mechanical advantage. In the images in the right column of Figure 5.22, the cracks

in the core region of the wires are clearly visible, and in Figure 5.22(b), the gap formed by the cracks in the core of RD-1B is approximately 2-3 μm . The images of S-1B and P-1B given in Figure 5.22(d) and (f) show that the size of the cracks in the superconducting core is 6-7 μm and 8-10 μm , respectively. With these results, the widest crack is observed in P-1B, the narrowest crack occurs in RD-1B.

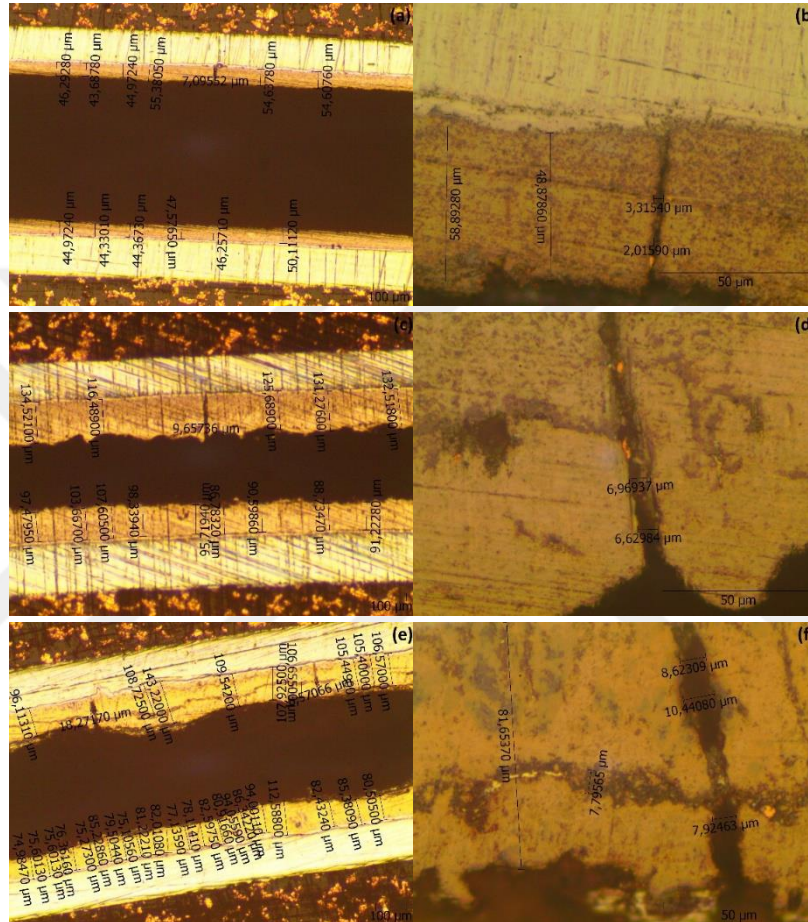


Figure 5.22. Optical microscope images of the wires taken at 5 $\times$ magnification from the lateral section and 20 $\times$ magnification from the crack regions. (a)-(b) RD-1B, (c)-(d) S-1B, (e)-(f) P-1B.

The hardness values obtained by applying different forces to the core regions of the (RD) and (S) IMD wires are given in Figures 5.23(a) and (b). As seen in Figure 5.23(a), as the annealing temperature or time increases, the hardness values increase. The wire with the highest hardness value under 1.961 N load is RD-1B. It was observed that the hardness increased more with the increase of annealing time than the increase in annealing temperature. The unreacted boron region, denoted as RD-2A-2 in Figure 5.23(a), and the MgB_2 core region had

different hardness under the same load in RD-2A wire. The hardness value obtained from the unreacted boron region of the superconducting core is lower than the hardness value of the reacted regions. In addition, the inset picture in Figure 5.23 (a) shows the regions where the hardness measurements were performed, and it is observed that indentation trace is not clear on the soft black boron region. In Figure 5.23(b), it is observed that hardness values for (S) IMD wires increase with the increase in annealing temperature, but not affected much by the annealing time. S-2A has the highest hardness value under the load of 1.961 N. The inset picture in Figure 5.23(b) shows that the core region has different structural formations. There is a significant difference in the hardness values obtained under the same load from the S-2A-2 wire core. The hardness value taken from the region where the core formation is completed is five times higher than the hardness value taken from the region where the formation is not completed in S-2A wire. In general, it has been observed that there is no significant difference between hardness of (S) IMD and (RD) IMD wires. Annealing of (S) IMD wire at annealing temperatures above 650 °C appears to be beneficial in increasing the core hardness but prevents the proper diffusion of Mg.

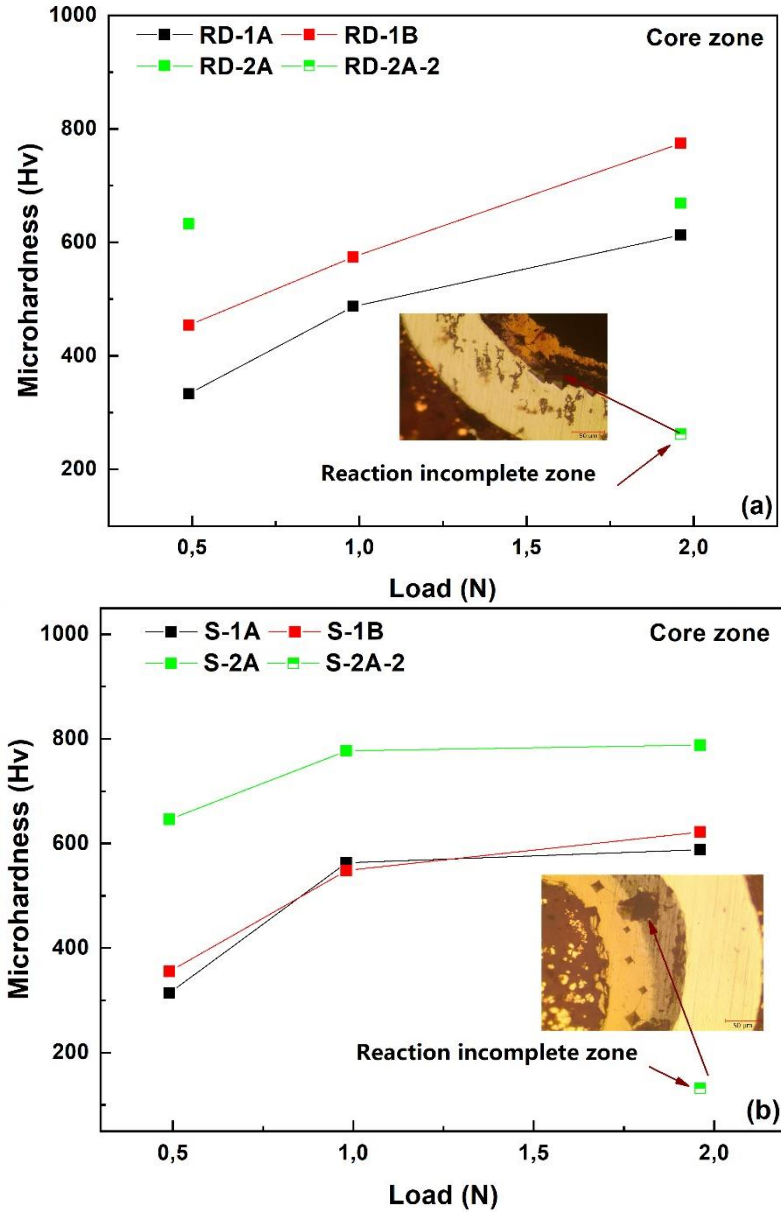


Figure 5.23. Hardness values obtained from the superconducting core regions of the wires under different forces. (a) (RD) IMD MgB_2/Fe wires. (b) (S) IMD MgB_2/Fe wires.

In Figure 5.24(a) to (c) and (d) to (f), optical microscope images of the indentation traces formed in different regions during the hardness measurements of the (RD) and (S) IMD wires are given, respectively. In the images, the indentations in the hard regions appear to be small in size and have regular diamond shape. In the soft-core regions, it was observed that the indentation trace took an irregular shape after the applied load was released. The applied load also caused fine cracks extending from the edge of some indentations in hard MgB_2 core region.

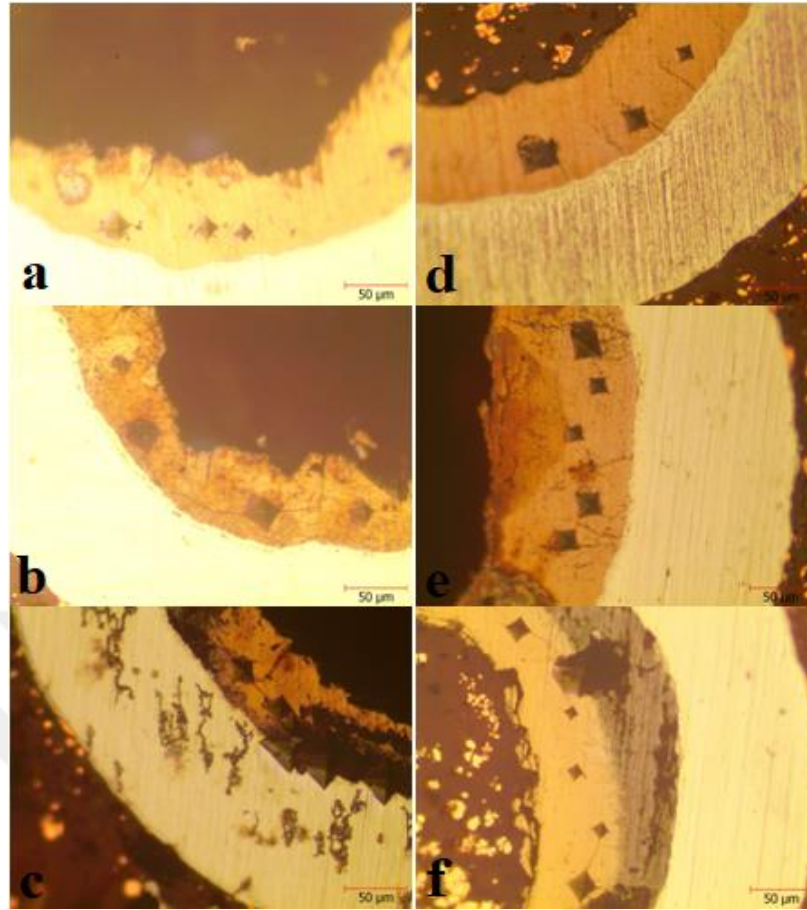


Figure 5.24. Optical microscope images of the indentations on the RD-1A (a-b-c) and S-1A (d-e-f) samples.

5.3 Experimental results of MgB_2 wires produced by Three Hole-IMD method

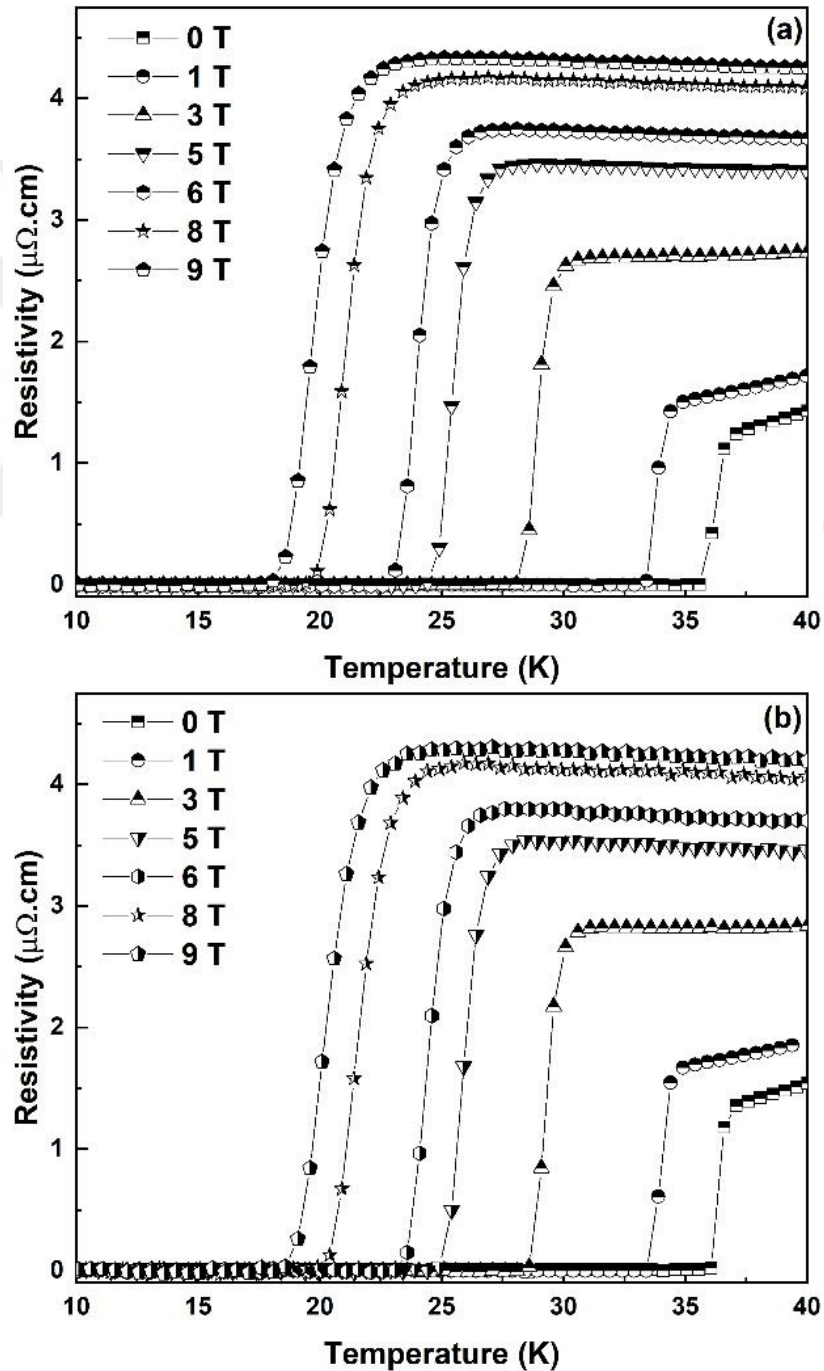
Table 5.2 presents the production properties of wires manufactured employing the three-hole internal magnesium diffusion (TH-IMD) technique. The TH-IMD method was designed to improve MgB_2 core structure. This methodology involves strategically positioning three thin rod-shaped magnesium sources in a triangular configuration within the boron, ensuring efficient Mg diffusion throughout the entire boron layer, resulting in a homogeneous MgB_2 core structure.

Table 5.2. The letters A, B, and C in the wire codes indicate the annealing times of 60, 120, and 240 minutes, respectively. “TH” stands for MgB₂/Fe Three Hole-IMD wires. Heat treatment temperatures of 650 °C and 700 °C are coded as 1 and 2, respectively.

Sample Name	Diameter (mm)	Synthesis Temperature (°C)	Synthesis Time (min.)	Filling Factor (%)	Cross sectional Area (cm ²)	Thickness of Fe ₂ B/FeB (μm)
TH-1A	0.81	650	60	54.72	1.10x10 ⁻³	8.0
TH-1B	0.81	650	120	46.72	1.00x10 ⁻³	10
TH-1C	0.81	650	240	47.68	1.03x10 ⁻³	15
TH-2A	0.81	700	60	53.23	1.07x10 ⁻³	12

TH-IMD wires with a diameter of 0.81 mm were produced by cold drawing. Various heat treatment conditions were studied for these wires with a new type core structure, and the effects of temperature and time were investigated. The applied heat treatment parameters are detailed in Table 5.2. The resistivity-temperature ($\rho - T$) curves of TH-IMD MgB₂/Fe wires, subjected to different heat treatment conditions, are depicted in Figures 5.25(a) - (d). Although there is no significant difference in the critical temperature values of TH-IMD wires depending on the heat treatment condition, it is noteworthy that the normal state resistivity of the TH-2A sample, annealed at 700 °C for 1 hour, shows a lower value compared to other TH-IMD wires. The normal state resistivity of the TH-1C sample, annealed at 650 °C for 4 hours, was found to be higher than that of the other samples. The $R - T$ measurements of the wires revealed the existence of the magnetoresistance effect with increasing the applied magnetic field from 0 T to 9 T. This behavior is particularly evident in wires heat treated at 650 °C for 1 and 2 hours, as illustrated in Figures 5.25(a) and (b). The variations in normal state resistivity of the wires under external magnetic field are due to the presence of unreacted Mg remaining in the structure after heat treatment. The unreacted Mg significantly influences the resistivity of the MgB₂ wire, and the normal state resistivity of MgB₂/Fe wire can vary depending on the amount of Mg in the core structure (R. Zeng *et al*, 2008). In Figures 5.25(a), (b), and (d), it is evident that the normal state resistivity under self-field of the wires subjected to relatively short heat treatment durations is three times lower than that of TH-1C wire heat-treated for 4 hours. The increase in the normal state resistivity of the wires with increasing applied magnetic field is attributable to

the magnetoresistance properties of Mg (R. T. Webber, 1956; K. H. Kim *et al*, 2002). The $R - T$ measurements conducted under magnetic field revealed that low-temperature heat treatment is a bit more advantageous in obtaining relatively high T_c , B_{irr} , and B_{c2} values in the wires. $R - T$ measurements were conducted up to 9 T, and the curves depicting B_{irr} and B_{c2} values in relation to temperature are presented in Figure 5.26. It can be seen that there is a very small difference between B_{irr} values of the wires even under a high magnetic field of 9 T.



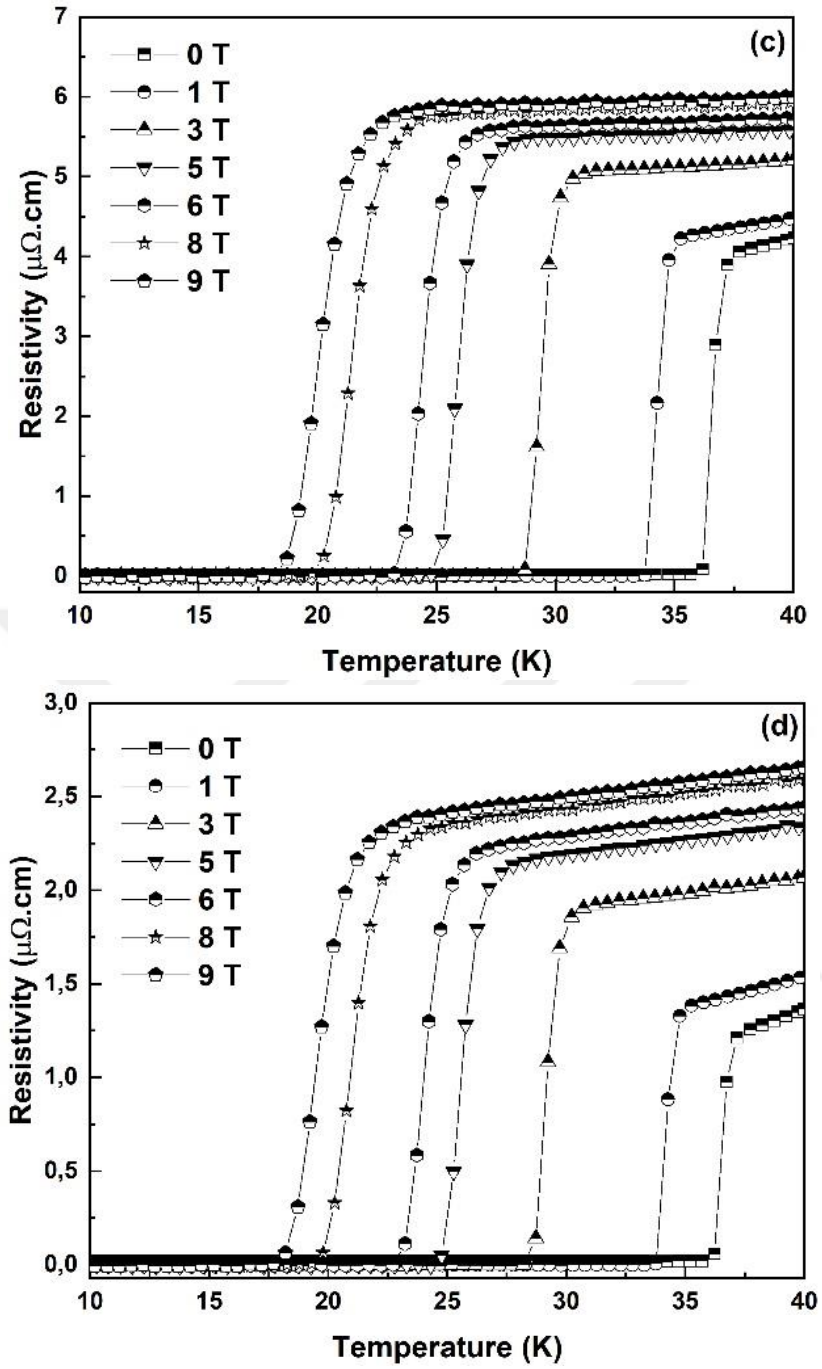


Figure 5.25. $R - T$ curves of TH-IMD wires under external magnetic field. (a) TH-1A, (b) TH-1B, (c) TH-1C, and (d) TH-2A. A direct current of 100 mA was applied.

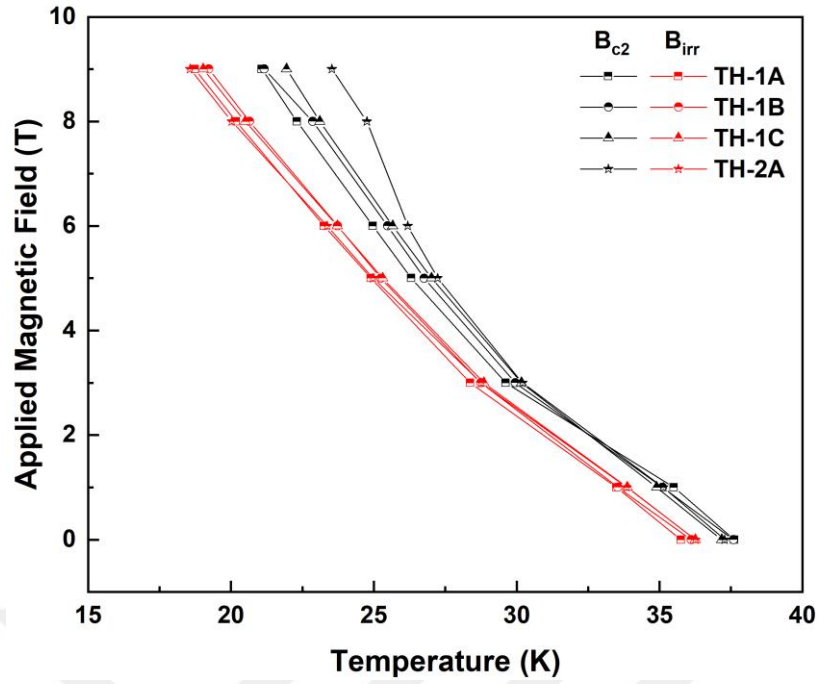


Figure 5.26. B_{irr} (red color) and B_{c2} (black color) curves of TH-IMD samples.

The I_c and J_c values obtained from $I - V$ measurements under various external magnetic fields at 4.2 K are presented in Figures 5.27(a) and (b), respectively. Notably, the TH-1A, TH1B, and TH-1C wires, subjected to low-temperature heat treatments at 650 °C, have higher I_c and J_c values for magnetic fields above 6 T than that of TH-2A wire. The I_c values of TH-1A, TH1B, and TH-1C wires are almost the same and approximately 90 A under 6 T at 4.2 K. It is also worth noting that the I_c (or J_c) value of TH-2A wire is higher than other wires below 7 T.

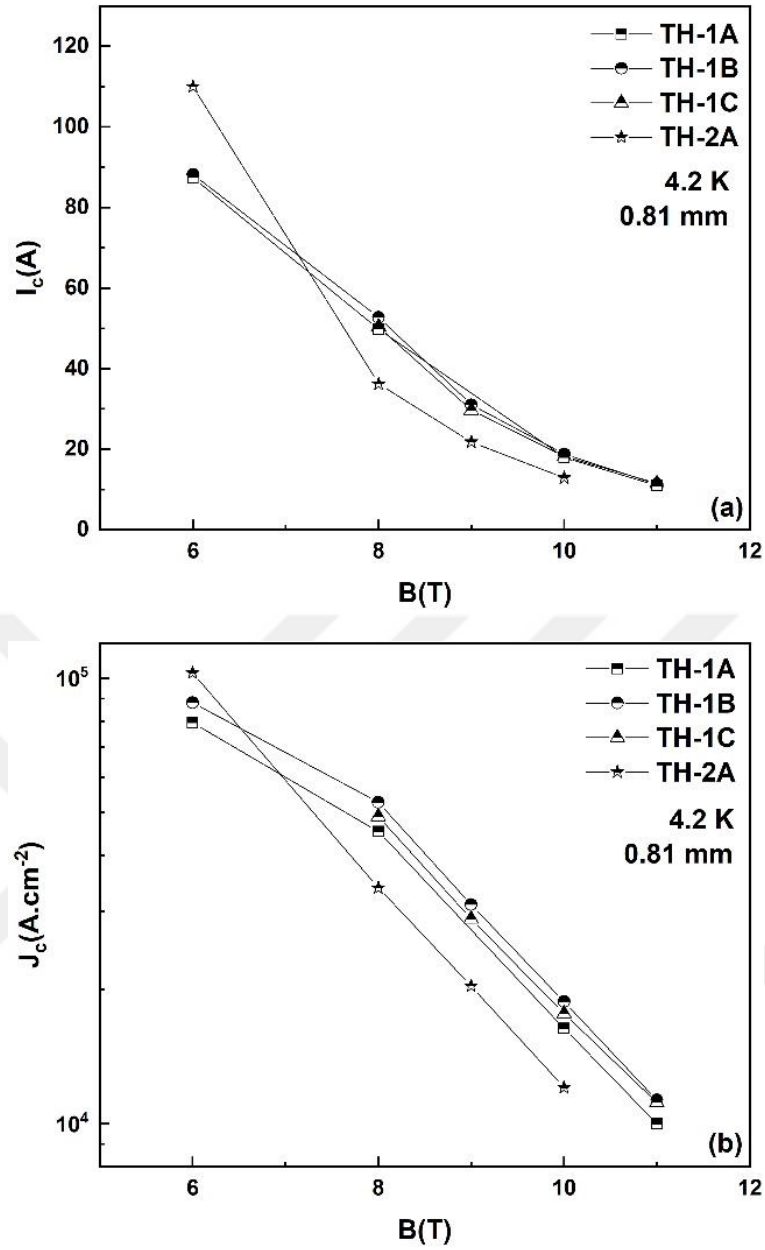
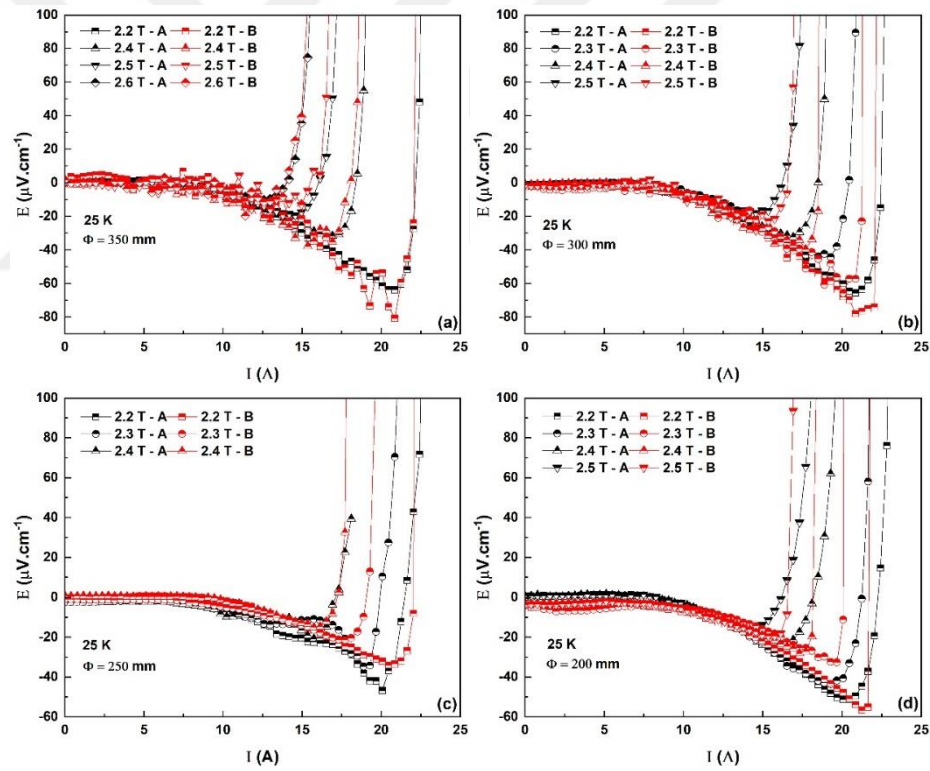


Figure 5.27. Magnetic field dependent (a) I_c and (b) J_c values obtained from $I - V$ curves at 4.2 K.

When the electrical properties of wires subjected to various heat treatment conditions were evaluated, it was observed that all samples treated at 650 °C had equivalent transport and superconducting properties. Therefore, electromechanical measurements were performed on wires annealed at 650 °C for 1 hour such as TH-1A. Figures 5.28(a) - (h) illustrate the $E - I$ curves obtained for straight and bent wires under various external magnetic fields at 25 K. The $I - V$ measurements were performed on different segments of the TH-1A wire. The bending diameters were specified as 350 mm, 300 mm, 250 mm, 200 mm, 175 mm, 150 mm, 125 mm, and

100 mm, respectively. In Figures 5.28(a) - (h), the red lines denote the $E - I$ curves of the wires prior to bending, while the black lines represent the $E - I$ curves subsequent to the bending process. It can be seen from Figures 5.28(a) to (d) that there is no degradation in the critical current value of TH-1A wire until the bending diameter reaches to 175 mm. When the bending diameter was gradually reduced from 175 mm to 100 mm, the degradation in the critical current of TH-1A wire gradually increased. Electromechanical measurement results show that TH-IMD wire is resistant to bending. As seen in Figure 5.28(h), there is a loss of at most 50 % in the critical current value of the TH-IMD wire after bending to a diameter of 100 mm. In other words, the current carrying capability of the wire is not completely or largely lost.



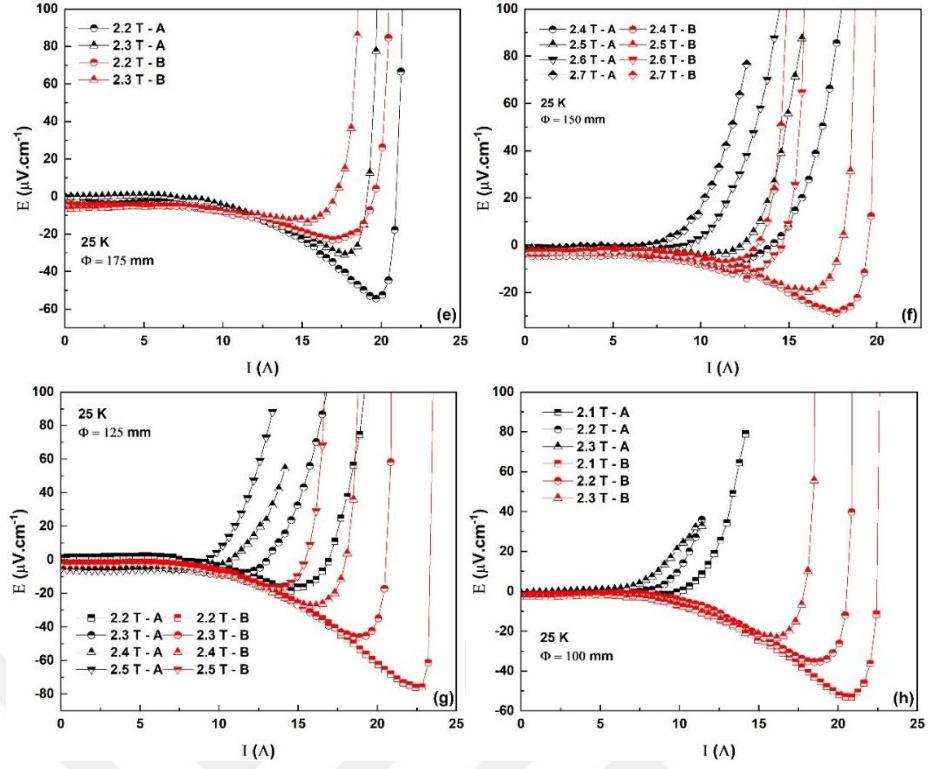


Figure 5.28. $E - I$ curves of straight and bent TH-IMD wires at 25 K.

Figure 5.29 shows the variation of the normalized critical current value of the wires according to the bending strain. The normalized critical current value is obtained by dividing the critical current value of the samples after bending ($I_{c,bent}$) by the value before bending ($I_{c,nonbent}$). The bending diameters are between 350 mm and 100 mm (K. Katagiri *et al.*, 2005). The results in Figure 5.19 show that TH-IMD wire has a bending tolerance value of 0.46 which is very promising compared to that of mono core PIT MgB_2 wires (Byeongha Y. *et al.*, 2019). The bending strain versus I_c , which behaves reversibly and linearly before I_c deteriorates, is similar to the behavior in the reversible region observed in tensile strain (G. Nishijima *et al.*, 2012), because the outer side of the wire is subjected to the tensile effect in the bending process.

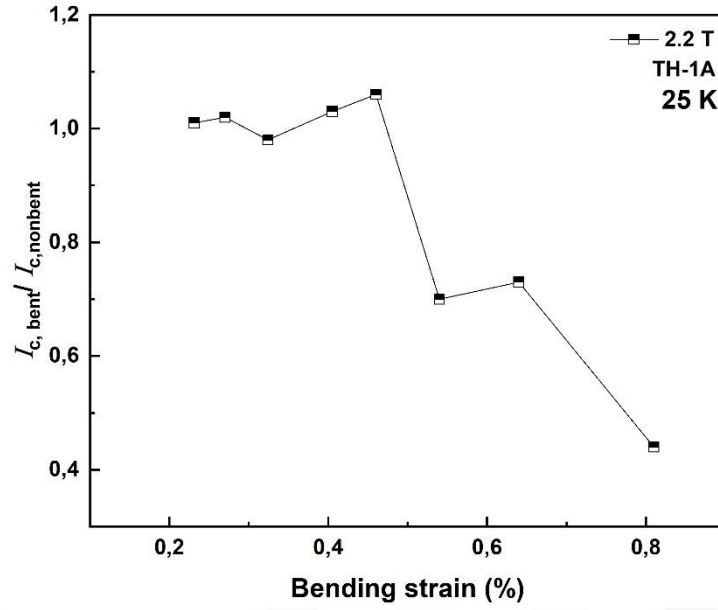
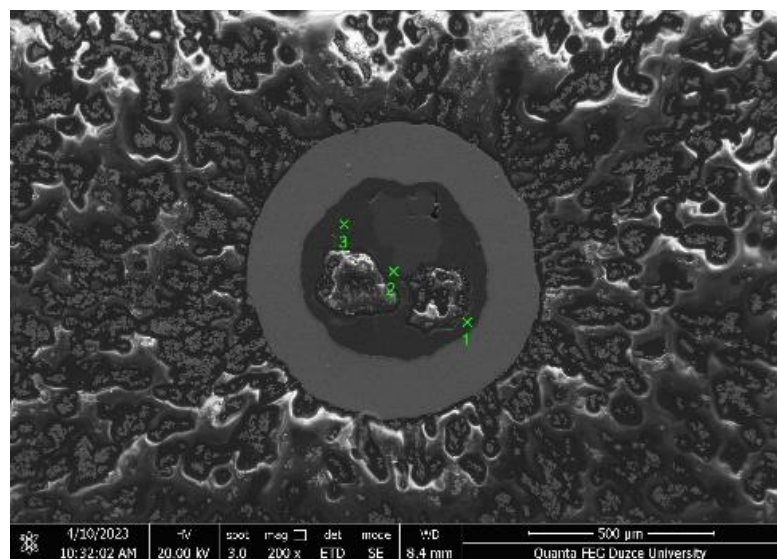


Figure 5.29. Normalized critical current vs. bending strain values of TH-IMD wire.

Figures 5.30(a)-(d) present the results obtained using the point Energy Dispersive X-ray Analysis (EDAX) method within the core region of all wires. In this specific analysis, the presence of magnesium in the TH-IMD samples following the reaction was examined through point analysis at three distinct locations. Upon careful examination of the obtained data, it was clearly seen that magnesium diffuses effectively from the initial boron interface region to the outermost boundary, facilitating the uniform formation of the MgB_2 structure in the wire core.



EDAX ZAF Quantification (Standardless)

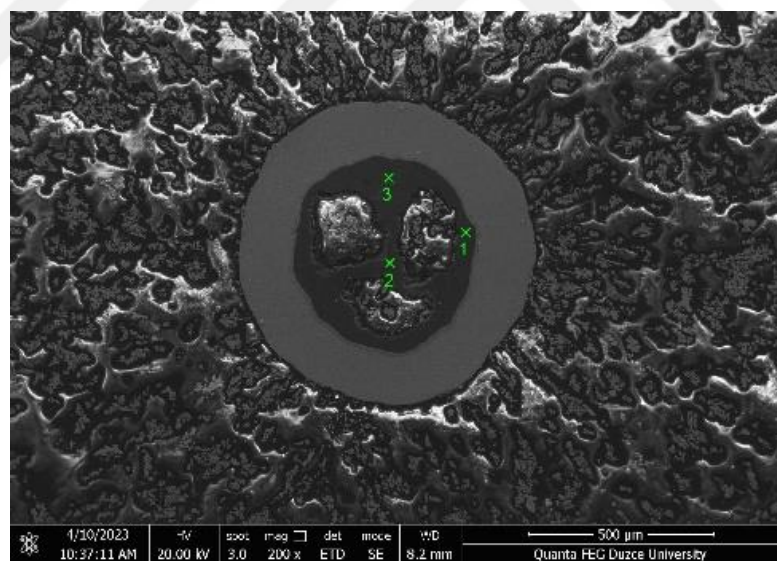
Element Normalized

SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
B K	23.17	40.41	0.0168	0.9934	0.0732	1.0001
MgK	76.83	59.59	0.7502	1.0024	0.9741	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
B K	15.85	13.03	19.03	1.22
MgK	6277.31	29.75	0.59	211.00

(a)



EDAX ZAF Quantification (Standardless)

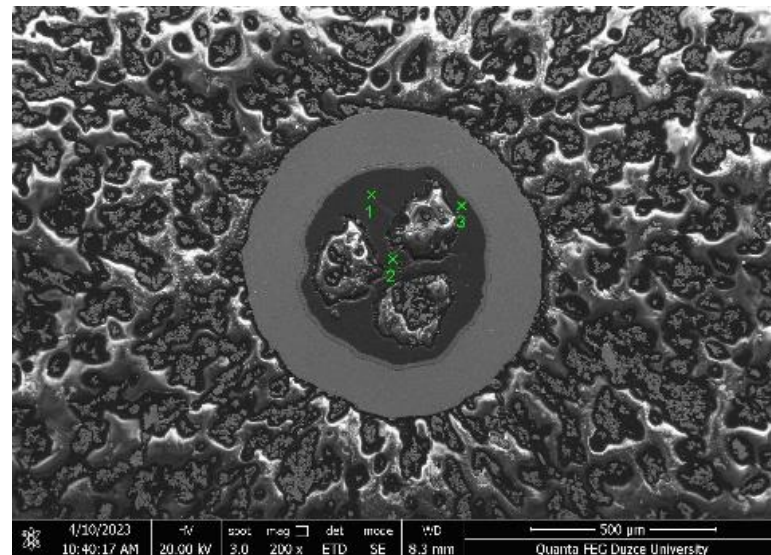
Element Normalized

SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
B K	27.05	45.46	0.0206	0.9937	0.0766	1.0001
MgK	72.95	54.54	0.7095	1.0028	0.9697	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
B K	21.33	8.71	13.98	2.45
MgK	6534.28	27.52	0.59	237.47

(b)

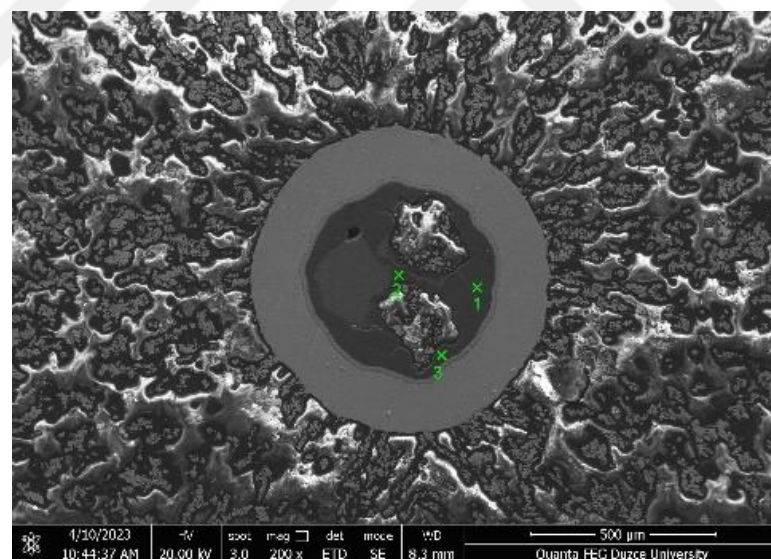


EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
B K	24.26	41.87	0.0179	0.9935	0.0741	1.0001
MgK	75.74	58.13	0.7387	1.0025	0.9728	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
B K	18.41	9.79	16.16	1.88
MgK	6775.16	34.95	0.59	193.86

(c)



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

Element	Wt %	At %	K-Ratio	Z	A	F
B K	24.31	41.94	0.0179	0.9935	0.0742	1.0001
MgK	75.69	58.06	0.7381	1.0025	0.9728	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
B K	18.83	10.96	16.55	1.72
MgK	6904.19	30.74	0.59	224.57

(d)

Figure 5.30. Point - EDAX analysis from cross-section of all TH-IMD wires of (a) TH-1A, (b) TH-1B, (c) TH-1C, and (d) TH-2A.

Figures 5.31 shows the surface morphology analysis results of the wires. The surface images were taken after the samples were polished in bakelite molds. Figure 5.31 presents images obtained from the cross-section of the wires using Scanning Electron Microscope. As clearly seen in Figures 5.31(a) and (d), the presence of unreacted magnesium is observed in one of the wire holes, especially in wires subjected to a relatively short heat treatment time of 1 hour. MgB_2 core structure was successfully obtained under all heat treatment conditions of the wires. Another important observation in Figures 5.31(a) and (d) is that there is a significant increase in the FeB- Fe_2B phases with increasing heat treatment temperature and time.

Figures 5.32(a – b) and (c – d) present optical microscope images obtained from the longitudinal section of the wires at $5\times$ and $20\times$ magnifications, respectively. In Figure 5.32(b), which contains images of the TH-1A sample bent to a diameter of 350 mm, it is seen that the bending process does not cause any fracture in the core region. In Figure 5.32(a), when the wire is bent up to a diameter of 150 mm, it is seen that many cracks occur in the core, and these cracks occurring after bending are thought to cause a 25 % decrease in the critical current value. When the transverse cracks in the core seen in Figure 5.32(c) are examined at $20\times$ magnification (see Figure 5.32(d)), it is evident that these cracks intersect only on one side of the core. This observation implies that the current carrying capacity can be preserved by 75 % due to the distinctive design featuring a three-hole core.

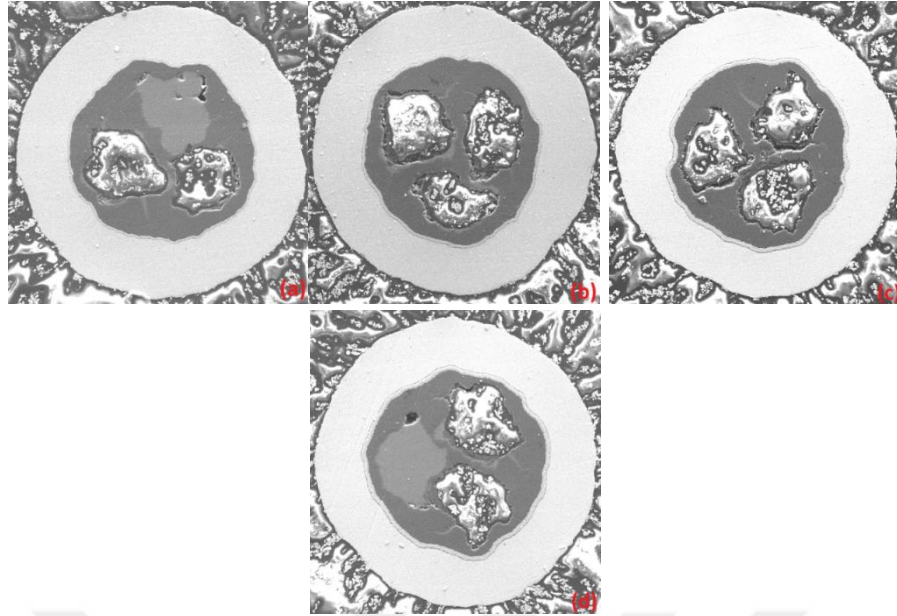


Figure 5.31. Cross-sectional SEM images of TH-IMD samples, which were (a) TH-1A, (b) TH-1B, (c) TH-1C, and (d) TH-2A.

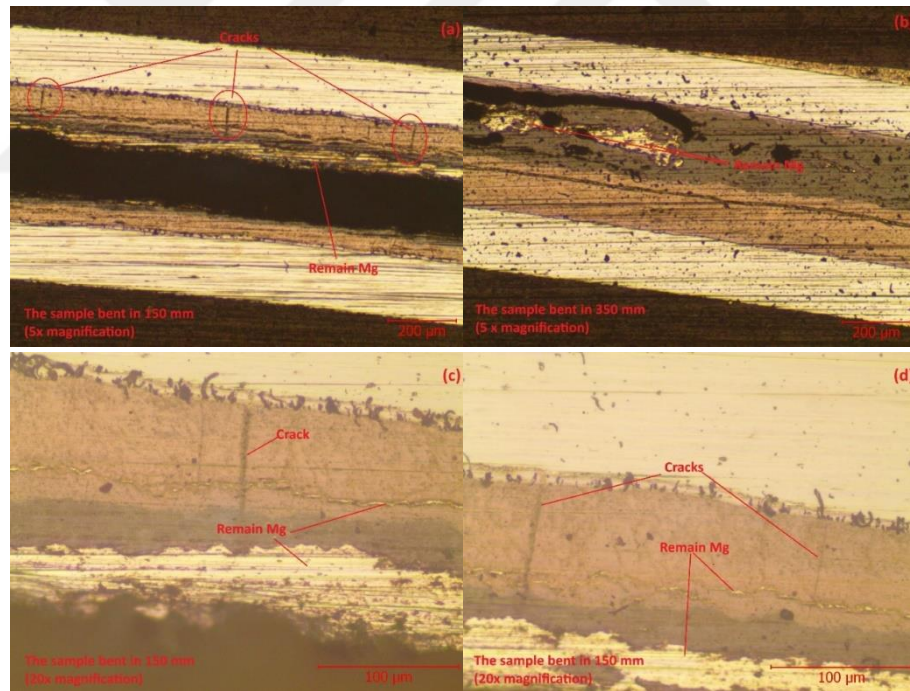


Figure 5.32. Optical microscope images taken from longitudinal section of TH-1A wire pieces after bending to (a) 150 mm and (b) 350 mm diameter (at 5× magnification), and (c)-(d) crack pictures at 20× magnification for the wire bent to 150 mm.

5.4 Experimental results of MgB₂ wires produced by half-EMD method

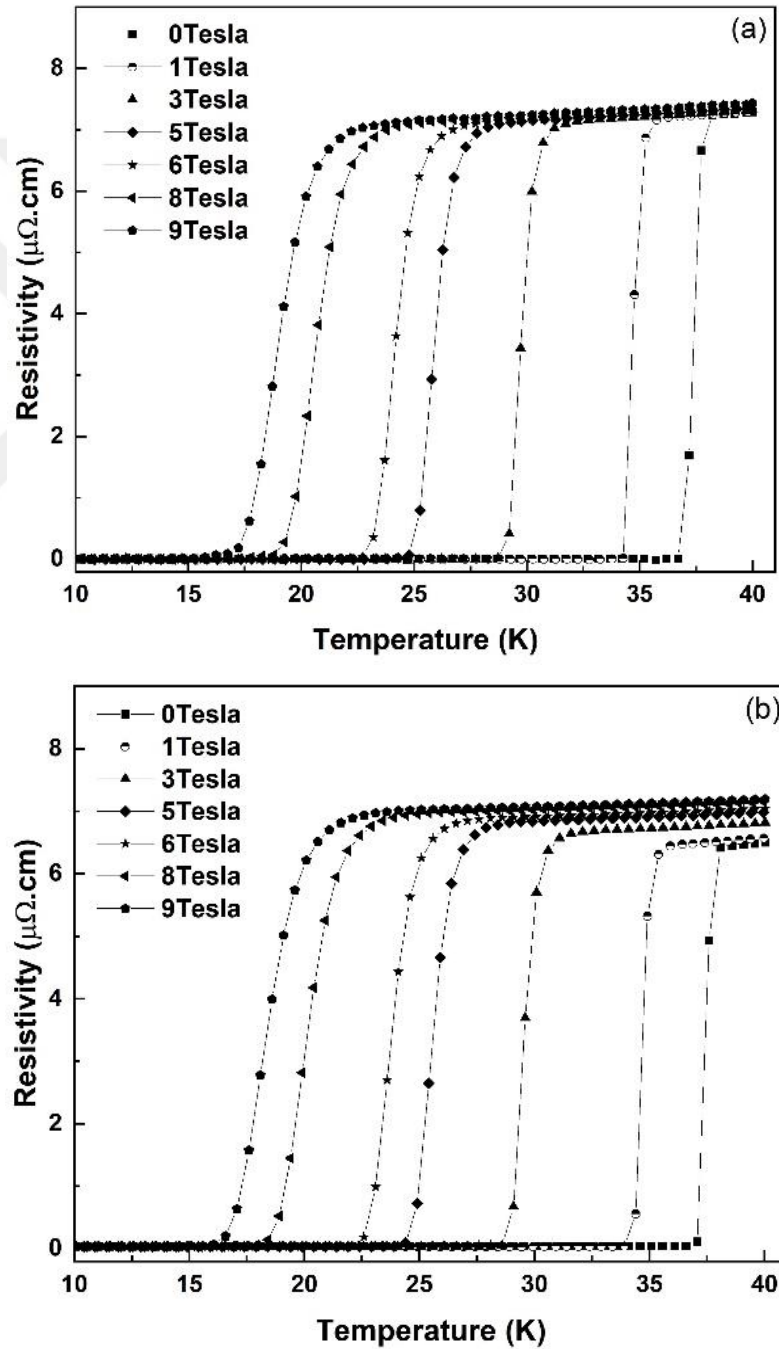
Table 5.3 presents some production details for the wires manufactured by the half-external magnesium diffusion (H-EMD). This technique is designed to make an MgB₂ wire with a semicircular MgB₂ core structure, which can provide advantages in terms of joining. The H-EMD wire is initially composed of semicircular columns of magnesium and boron before being subjected to the heat treatment procedure.

Table 5.3. The letters A, B, and C in the wire codes indicate the annealing times of 60, 120, and 240 minutes, respectively. “HE” stands for MgB₂/Fe half-EMD wires. The heat treatment temperatures of 650 °C and 700 °C are coded as 1 and 2, respectively.

Sample Name	Diameter (mm)	Synthesis Temperature (°C)	Synthesis Time (min.)	Filling Factor (%)	Cross sectional Area (cm ²)
HE-1A	0.81	650	60	25.40	1.31×10 ⁻³
HE-1B	0.81	650	120	25.70	1.30×10 ⁻³
HE-1C	0.81	650	240	23.96	1.22×10 ⁻³
HE-2A	0.81	700	60	26.07	1.33×10 ⁻³

The H-EMD wires with a diameter of 0.81 mm were produced using cold drawing process. The effects of annealing temperature and time on the transport properties of the wires were investigated by applying different heat treatment conditions on H-EMD wires with a unique core design. Heat treatment conditions were carried out as given in Table 5.3. The $R - T$ curves of H-EMD MgB₂/Fe wires, which were heat treated under different conditions, are given in Figures 5.33(a) - (d). The H-EMD wires have almost the same superconducting critical temperature as a function of the external magnetic fields from 0 to 9 T. It is observed that the normal state resistivity of H-EMD wires varies between 6 and 8 $\mu\Omega\cdot\text{cm}$ depending on the applied external magnetic field, except for the HE-2A wire, which has a slightly lower resistivity than the other samples. Magnetoresistance effects are also prominent in the HE-2A wire (C. U. Jung *et al*, 2002). The $R - T$ measurements made under external magnetic field reveal that the T_c , B_{irr} , and B_{c2} values of wires

produced at 650 °C are better than the wire annealed at 700 °C. A sharp transition to the superconducting phase was observed for all wire samples. The upper critical magnetic field and irreversibility magnetic field values were determined at 90 % and 10 % of ρ (40 K). The B_{irr} and B_{c2} values of the wires as a function of temperature were shown in Figure 5.34. While no significant difference is observed between the B_{irr} and B_{c2} values of the wires, it is observed that the B_{c2} value of the HE-2A wire differs negatively from the others under magnetic fields above 3 T.



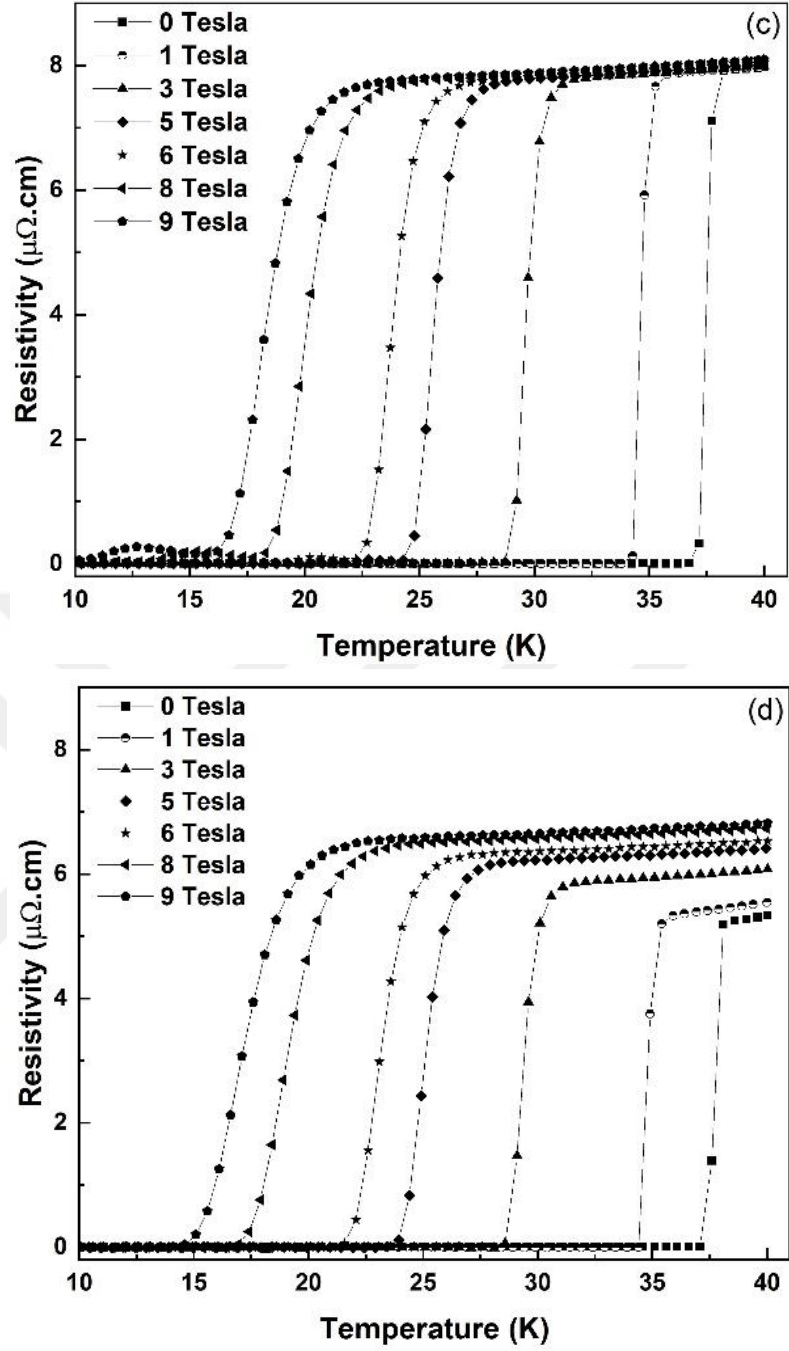


Figure 5.33. $R - T$ curves of H-EMD wires of (a) HE-1A, (b) HE-1B, (c) HE-1C, and (d) HE-2A under various external magnetic fields.

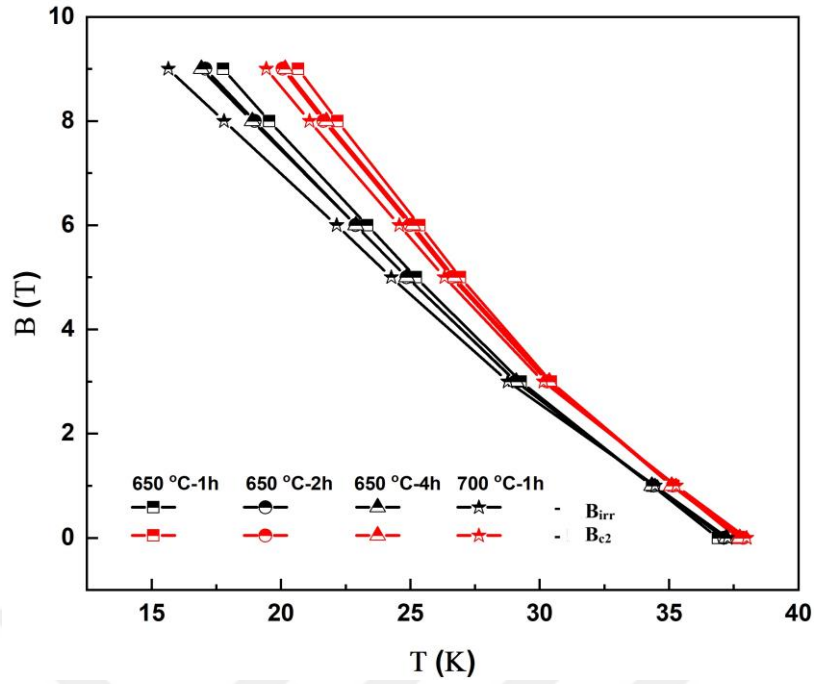


Figure 5.34. B_{irr} (red color) and B_{c2} (black color) curves of the wires.

The I_c and J_c values obtained from $I - V$ measurements under various external magnetic fields at 4.2 K are given in Figures 5.35(a) and (b), respectively. It is observed that HE-1A wire has the highest I_c of 70 A under 6 T (or J_c) value among the wires. HE-2A wire has the lowest I_c (or J_c) value especially under 10 T external magnetic field. Additionally, when the annealing time at 650 °C was extended, a slight decrease was observed in the I_c and J_c values of the wires. Similar differences are also observed between the I_c and J_c curves of the wires obtained from $I - V$ measurements at 20 K and 25 K, as shown in Figures 5.36(a) and (b).

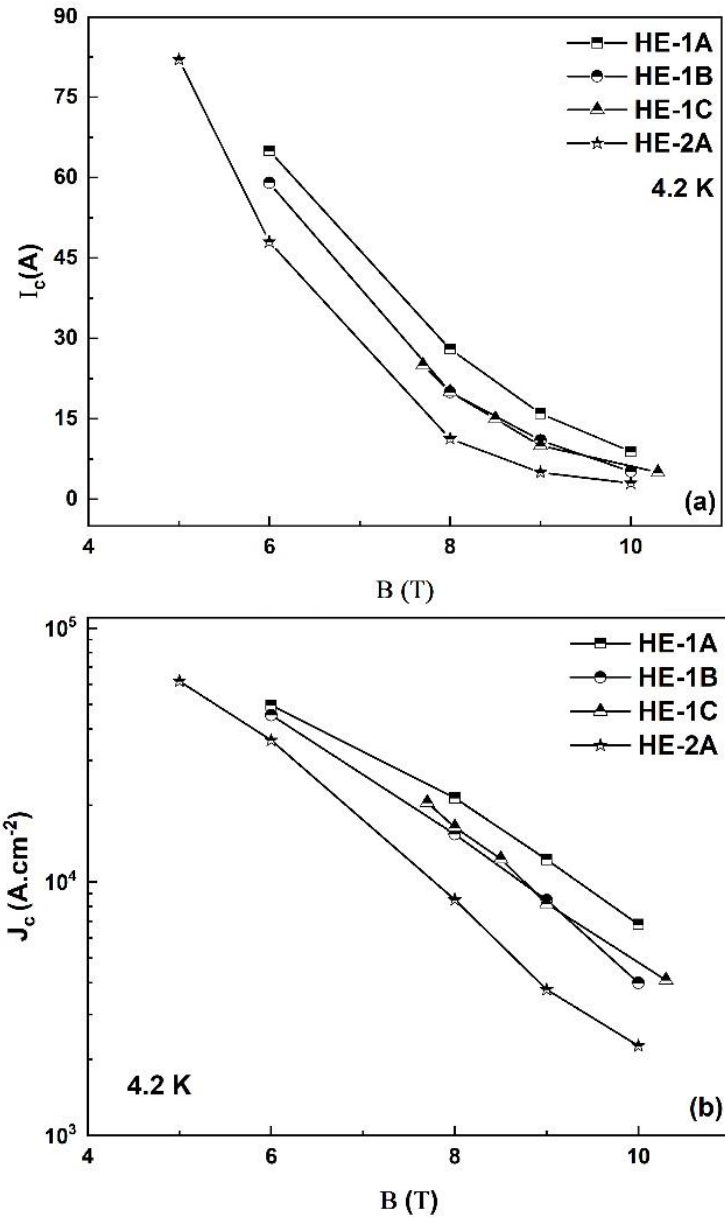


Figure 5.35. (a) $I_c(B)$ and (b) $J_c(B)$ values obtained from $I-V$ curves at 4.2 K.

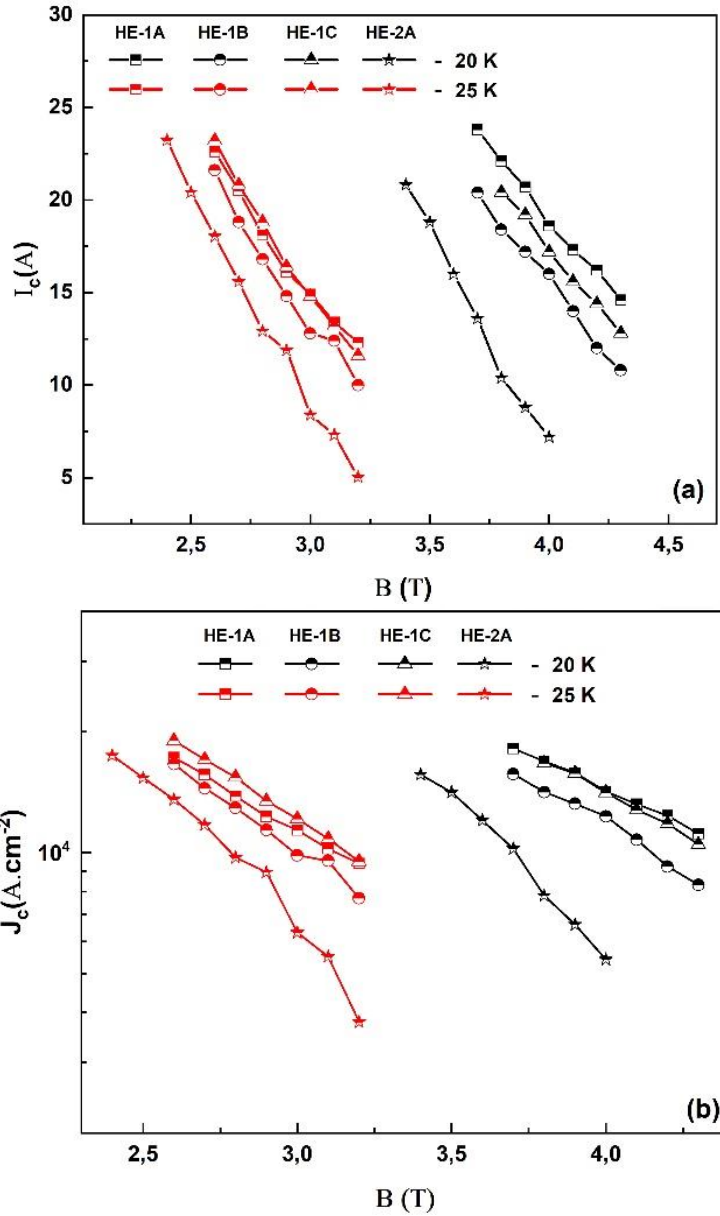
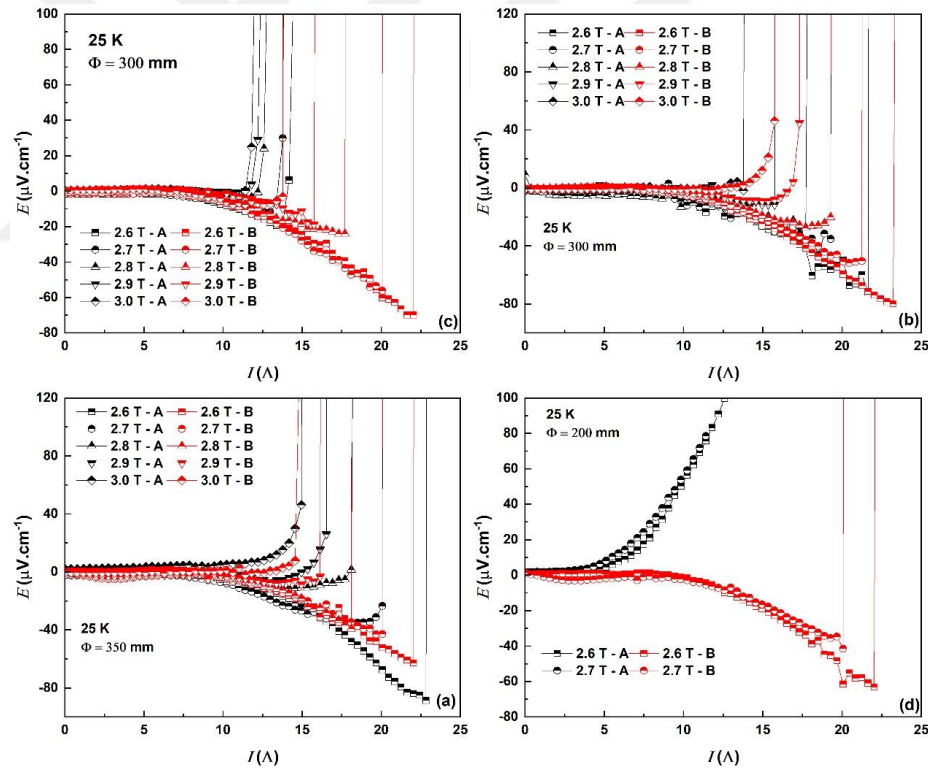


Figure 5.36. (a) I_c (B) and (b) J_c (B) values obtained from $I - V$ curves at 20 K and 25 K.

Figures 5.37(a) – (f) show the $E - I$ curves obtained before and after bending the HE-1A wires to the specified diameters. Since HE-1A wire has the best transport properties among the wires, electromechanical experiments were carried out on this wire. The bending process of the reacted wires was done at room temperature. $I - V$ measurements were performed on different parts of the HE-1A wire under various external magnetic fields at 25 K. The bending diameters were determined as 350 mm, 300 mm, 250 mm, 200 mm, 175 mm, and 150 mm, respectively. In Figures 5.37(a) – (f), the red-colored lines show the $E - I$ curves of the samples before

bending, while the black-colored lines belong to the $E - I$ curves after bending. It is observed that these samples were not adversely affected by the 350 mm bending diameter, as the $E - I$ curves before bending almost overlap with the $E - I$ curves after bending. However, as the bending diameter decreases from 300 mm to 150 mm, the voltages occurring before the bending process occur at lower current values after the bending process, revealing that the critical current value of the HE-1A sample begins to degrade below the 300 mm bending diameter. This degradation in the critical current value gradually increases as the bending diameter decreases. According to the results of the electromechanical measurements, H-EMD wire is highly affected by the bending process, as seen in Figure 5.37(f), where the current carrying performance decreases by 90 % after the bending process for a bending diameter of 150 mm.



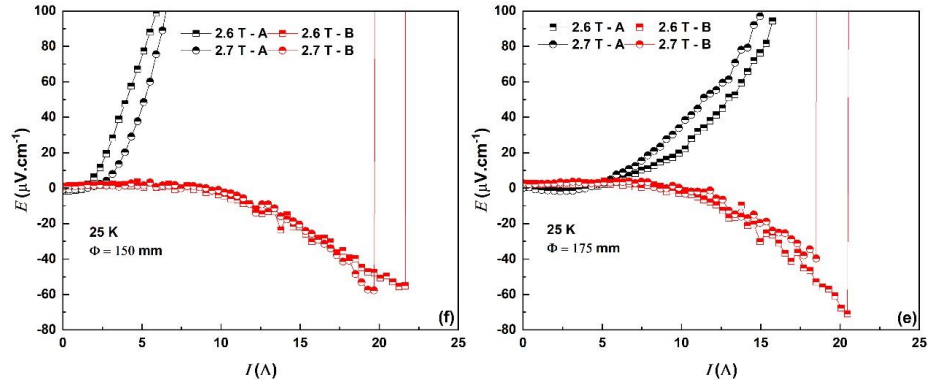


Figure 5.37. $I - V$ curves of straight and bent wires at 25 K.

Figure 5.38 shows the variation of the normalized critical current value of the HE-1A wire against the bending strain. The normalized critical current value is obtained by dividing the critical current value of the samples after bending ($I_{c,bent}$) by the value before bending ($I_{c,nonbent}$). The bending diameters are between 350 mm and 150 mm (K. Katagiri *et al*, 2005). The results in Figure 1.6 show that the H-EMD wire has a very low bending tolerance value of < 0.3 .

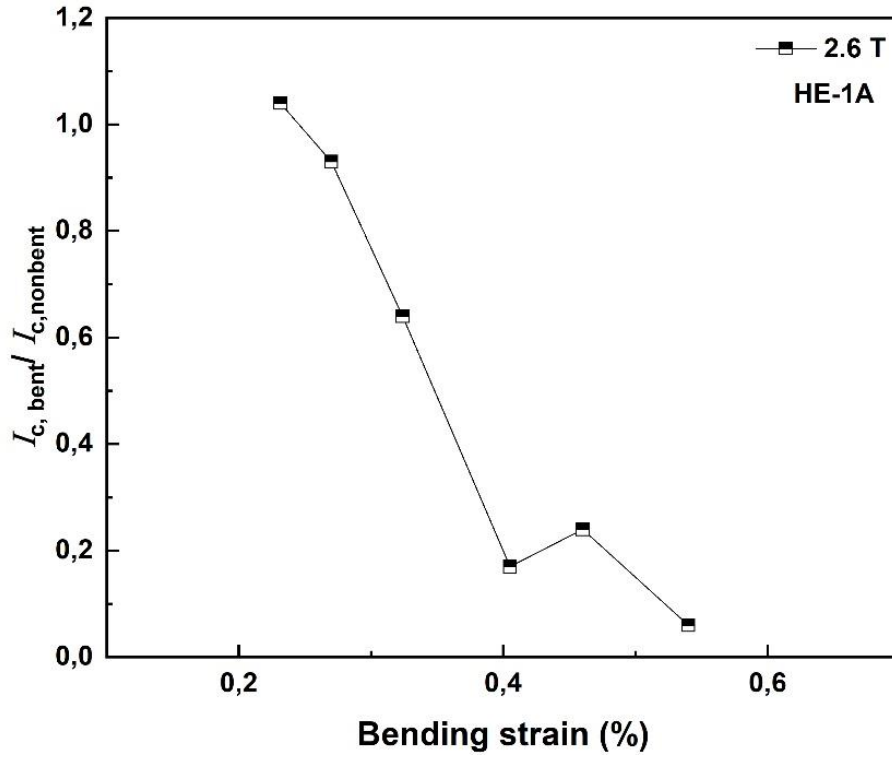


Figure 5.38. Normalized critical current vs. bending strain values for HE-1A wire.

Figure 5.39 shows the results of XRD analysis of HE-1A, HE-1B, HE-1C, and HE-2A wires. The superconducting MgB_2 cores were extracted by stripping the iron sheath and then the MgB_2 core was ground and pulverized in an agate. The sheath stripping process in both IMD and H-EMD wires is quite difficult due to the bonding formed in the core/sheath interface region after heat treatment, so the amount of MgB_2 powder required for XRD differ from wire to wire. This difference can be seen from the difference in the intensities of the peaks of the samples. It is seen in Figure 5.39 that the MgB_2 phase is formed in all H-EMD samples. There is unreacted Mg phase in all samples, but the HE-2A sample heat-treated at 700 °C for 1 hour was found to contain more unreacted magnesium than the others. The obtained XRD results are consistent with the transport properties of the wires. The fact that the HE-2A wire has the lowest normal state resistance in $R - T$ measurements and the increase in normal state resistance with increasing the external magnetic field can be explained by the presence of relatively high amount of unreacted Mg phase. $I - V$ measurements performed under a magnetic field are also compatible with $R - T$ measurement results for the same reason.

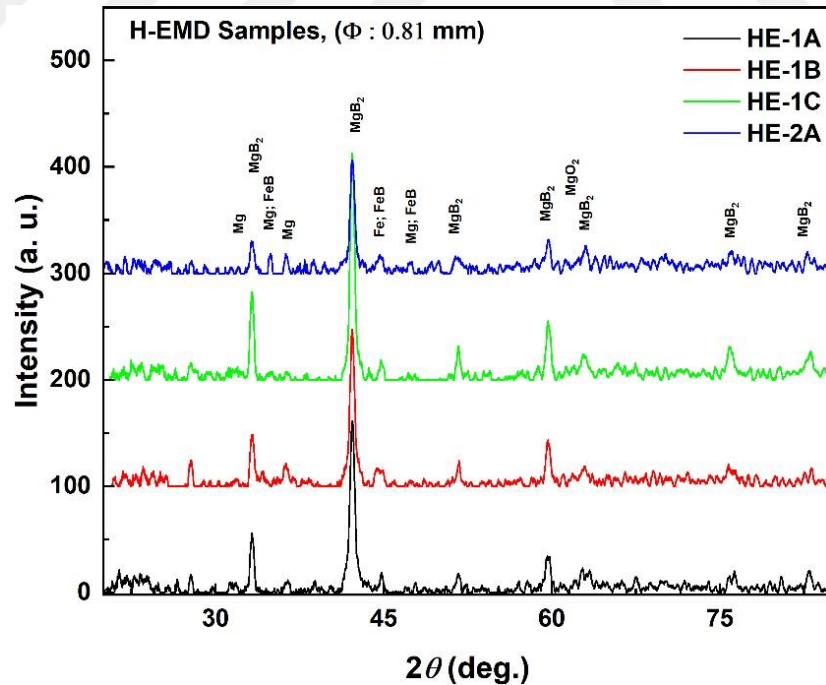
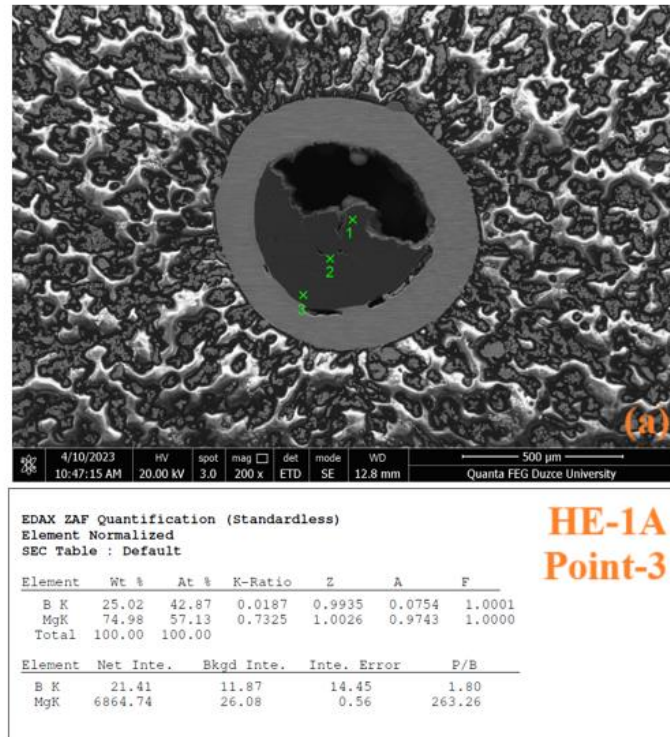
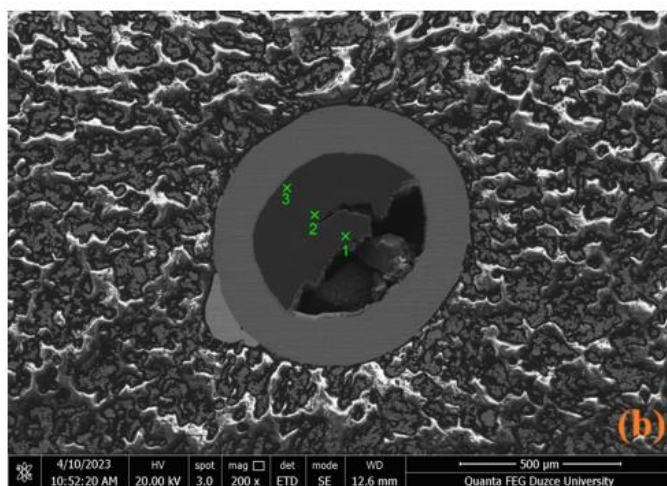


Figure 5.39. XRD patterns for H-EMD wires.

Figures 5.40(a) - (d) illustrate the results achieved through the point energy dispersive *X*-ray analysis (EDAX) method within the core region of all samples. In this particular analysis, the presence of Mg or Mg diffusion was assessed through point analysis from innermost to outermost in the core region of H-EMD samples. The results revealed that magnesium diffusion was achieved even in places where the core was thickest and MgB₂ phase was formed. In Figure 5.40(d), it can be seen that the half region where the compressed Mg powder was located before the reaction is not completely empty after the reaction. This situation can be considered as the cause of relatively high Mg-peaks in the XRD pattern of HE-2A sample. The rapid reaction of nano boron with Mg occurring at 700 °C may be slowed down the diffusion of Mg into the boron region.



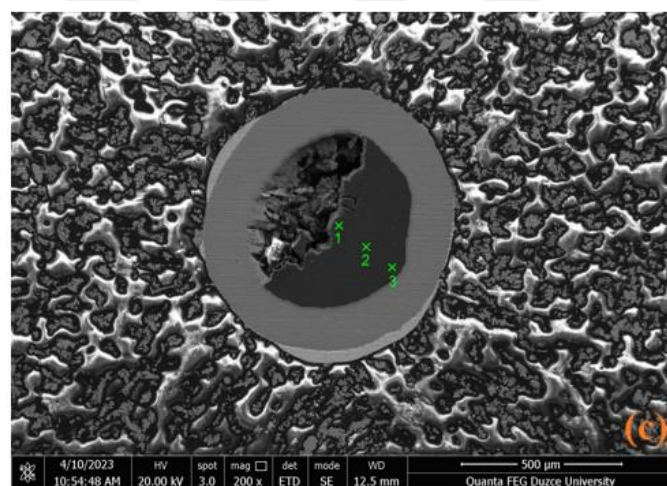


EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

**HE-1B
Point-3**

Element	Wt %	At %	K-Ratio	Z	A	F
B K	20.35	36.48	0.0144	0.9931	0.0715	1.0001
MgK	79.65	63.52	0.7816	1.0021	0.9791	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
B K	14.81	9.47	19.33	1.56
MgK	6574.06	17.48	0.61	376.00



EDAX ZAF Quantification (Standardless)
Element Normalized
SEC Table : Default

**HE-1C
Point-3**

Element	Wt %	At %	K-Ratio	Z	A	F
B K	23.42	40.75	0.0172	0.9934	0.0740	1.0001
MgK	76.58	59.25	0.7491	1.0025	0.9759	1.0000
Total	100.00	100.00				

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
B K	19.19	7.80	14.12	2.46
MgK	6878.56	38.58	0.56	178.28

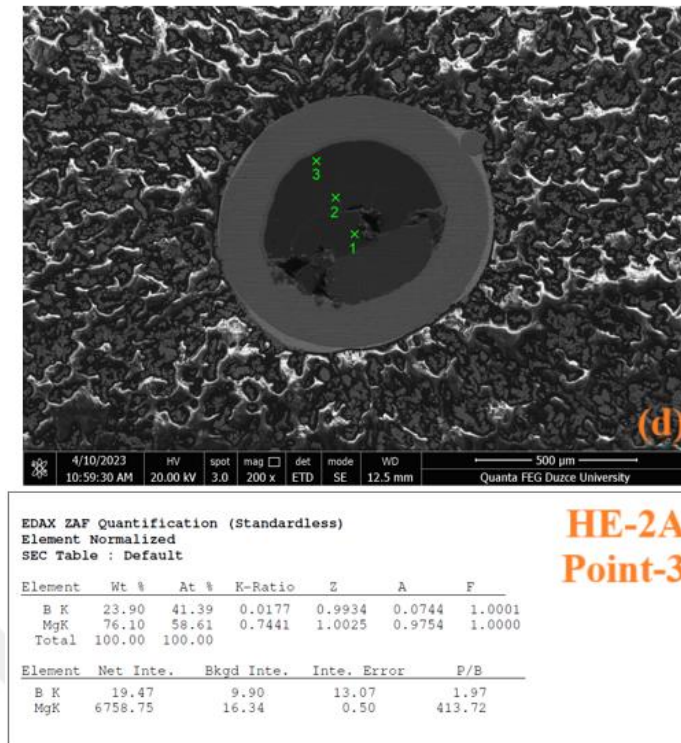


Figure 5.40. Point - EDAX analysis from cross-section of all H-EMD wires of (a) HE-1A, (b) HE-1B, (c) HE-1C, and (d) HE-2A.

Figures 5.41 and 5.42 show the surface morphology examinations of the samples. The surface images were obtained after polishing the samples in bakelite molds. Figures 5.41 and 5.42 show the images obtained from the longitudinal section of the samples using an optical microscope at $5\times$ and $20\times$ magnifications, respectively. The significance of the optical microscope images is that there is a difference in the orange color tone depending on the density of the superconducting core of the samples. Density difference in superconducting cores is important in terms of the MgB_2 phase formation because dense MgB_2 has a more reflective property (S. Ye *et al.*, 2016). When HE-1A sample is evaluated in terms of this fact, it can be said that the sample has a dense core. In the optical images of HE-1A sample bent to 350 mm diameter in Figure 5.41(a), it is seen that the sample is not negatively affected by the bending process, while in Figure 5.41(b), when the sample is bent to 150 mm diameter, it is seen that multiple cracks occur in the core. These cracks explain the 90 % degradation in the critical current value after the sample was bent to 150 mm. When the transverse cracks in the core, shown in Figures 5.42(a) and (b), are examined at $20\times$ magnification, it can be seen that these net cracks on the core destroy the current paths at a high rate.

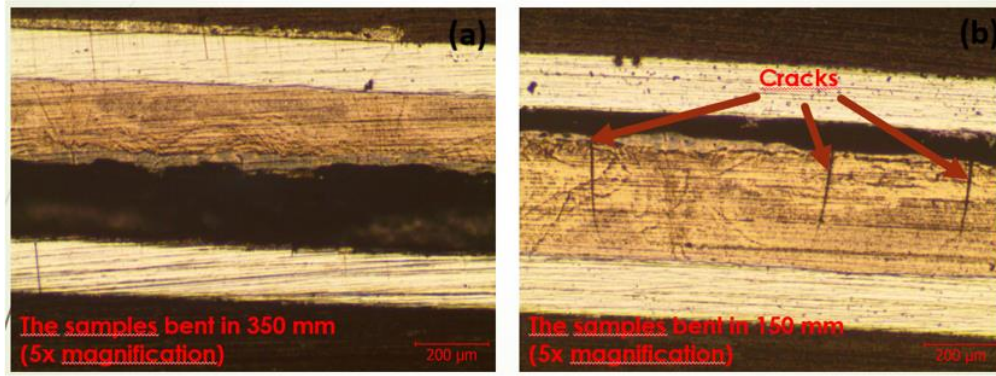


Figure 5.41. Optical microscope images taken from longitudinal section of HE-1A wire pieces after bending to (a) 350 mm and (b) 150 mm diameter (5× magnification).

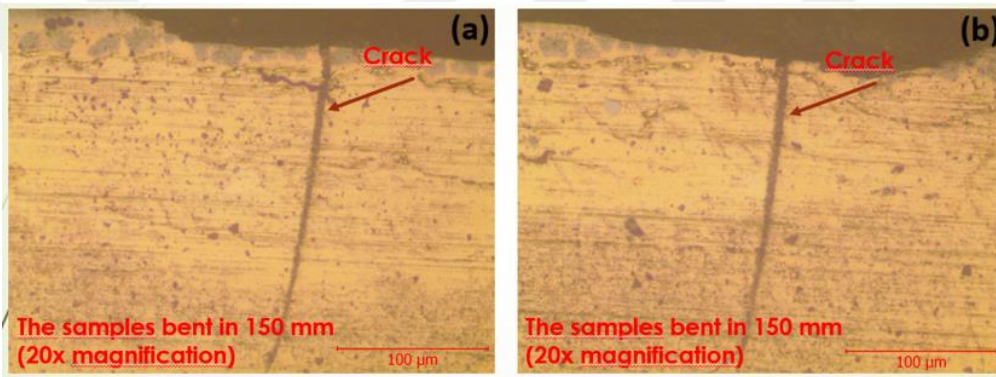


Figure 5.42. Optical microscope images taken from longitudinal section of HE-1A wire pieces after bending to 150 mm diameter (20× magnification).

5.5 Experimental results of the joints

In this section, the results of comparative studies on joining both reacted and unreacted standard IMD and half-EMD wires of 0.81 mm diameter with a continuation-type superconducting joints (G. D. Brittles *et al*, 2015) are presented. The joint mold, which has a small diameter of 5.0 mm, contains Mg and B powders compressed into semicircular columns within composite Cu/Nb metal sheaths. Additionally, the joint resistance measurements were performed on single-turn coils which were constructed using unreacted half-EMD and standard IMD wires. A total of four single-turn coils were made, closed with termination-type joints called R-1, R-1H, R-2, and R-2H, were made. The R-1/R-1H and R-2/R-2H joints were made using half-EMD and standard IMD wires, respectively. The I_c measurements of H coded joints were performed by applying high current in 20 K He vapor and $I - V$

measurements were made in three different ways; under the external magnetic field applied from low to high value (LTH), under the external magnetic field applied from high to low value (HTL), and in the field cooling (FC).

5.5.1 Electrical and structural measurement results for joints

Figures 5.43(a) and (b) show $I - V$ curves of the continuation-type joints, which join the unreacted standard IMD and half-EMD wires, under different external magnetic fields at 25 K, respectively. $I - V$ measurements were done using a pulse-train current source. In this way, a transport current up to a maximum of 24 A could be applied to the samples without causing significant heating inside the cryostat system. When Figures 5.43(a) and (b) are examined, it is seen that the superconductivity of the joint produced using standard IMD wires deteriorates under 2.5 T just before reaching 24 A. However, the joint produced using half-EMD wires does not break its superconductivity under the same external magnetic field. It is seen that the joint prepared using half-EMD wire has better transport properties than the joint prepared using standard IMD wire. Figures 5.44(a) and (b) show $I - V$ curves obtained at 25 K under different external magnetic fields for the joints for reacted standard IMD and half-EMD wires, respectively. The wires heat treated at 650 °C for 4 hours were joined using continuation type. The joint was compressed horizontally using a hydraulic press, applying a load of 1 ton, and annealed at 800 °C for 3 hours. The second heat treatment at a high temperature of 800 °C is to ensure phase formation in the joint region and to provide a good connection between the reacted wires and the joint core (X. H. Li *et al*, 2008). Figure 5.44(a) shows the steadily growing $I - V$ behavior of the joint for the reacted standard IMD wires under external magnetic field. This may be due to weak coupling between the wires and the joint region, thus causing current to flow along a resistive path across the contact surfaces. As seen in Figure 5.44(b), the voltage does not increase continuously with the applied current, but the voltage increases rapidly after the current reaches a critical value. The joint for reacted half-EMD wires is capable of carrying current and has better transport properties than the joint for reacted standard IMD wires. The half-EMD wire is thought to provide an effective contact surface to ensure the connection between the wires and the joint core. The design of half-EMD wires became more pronounced when the reacted wires were joined.

The results show that it is possible and beneficial to take advantage of the half-EMD core design, which aims to make the surface area of the thin MgB_2 ring formed in standard IMD wires more effective in terms of joining.

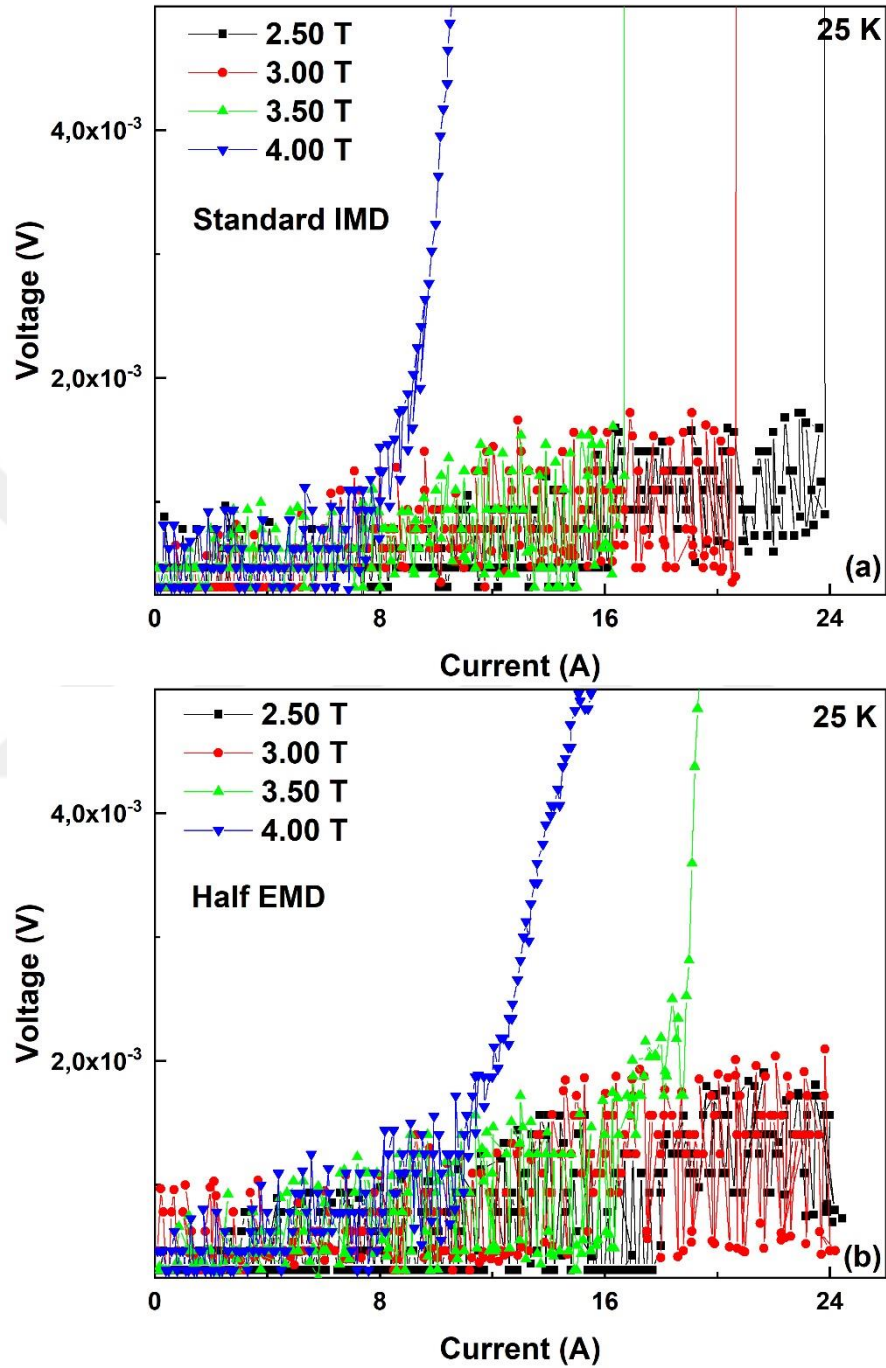


Figure 5.43. $I - V$ curves of joints joining the unreacted (a) standard IMD wire, (b) half-EMD wire.

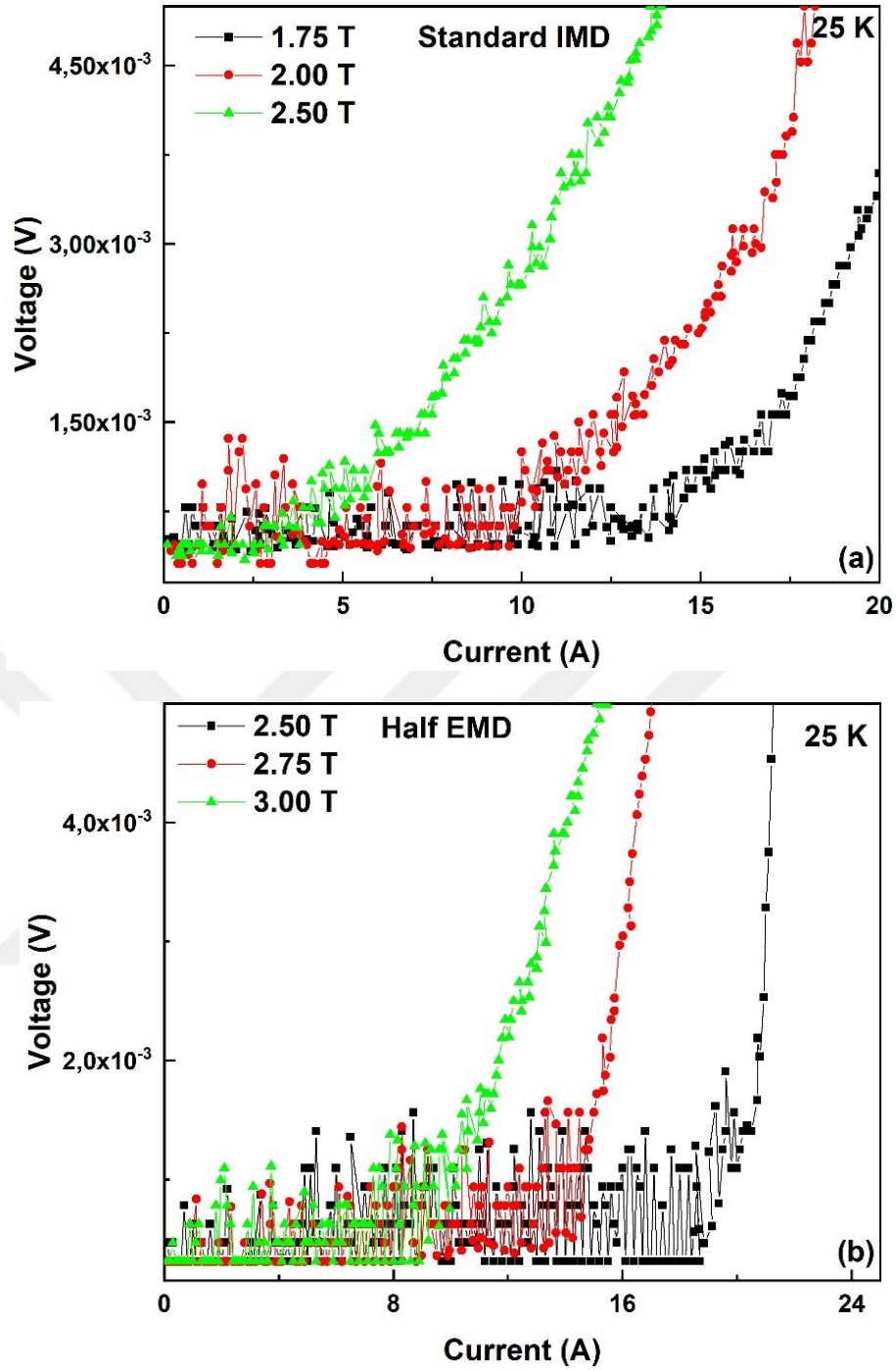


Figure 5.44. $I - V$ curves of joints for reacted (a) standard IMD wire, (b) half-EMD wire.

Figure 5.45 presents the magnetic field dependent I_c values of straight wires fabricated using both the standard IMD and half-EMD methods. The I_c values for these wires were determined from the $I - V$ curves obtained under various magnetic fields at temperatures of 4.2 K, 20 K, and 25 K. The I_c values of half-EMD wires are higher than that of standard IMD wires under equivalent magnetic field strengths and temperatures. It can be seen in Figure 5.45 that this difference is

evident under high magnetic fields at 4.2 K, but the $I_c(B)$ behavior is similar for both wires around 25 K and 3 T. This behavior indicates that joining half-EMD wires shows better transport properties than joining standard IMD wires is due to the design advantage of the half-EMD wire.

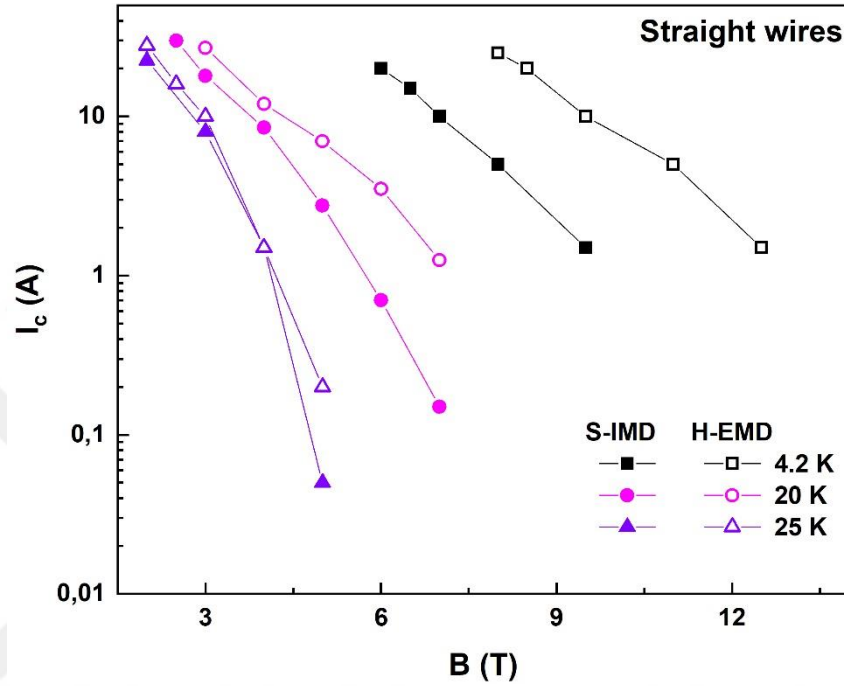


Figure 5.45. The magnetic field dependent critical current values of straight standard IMD and half-EMD wires.

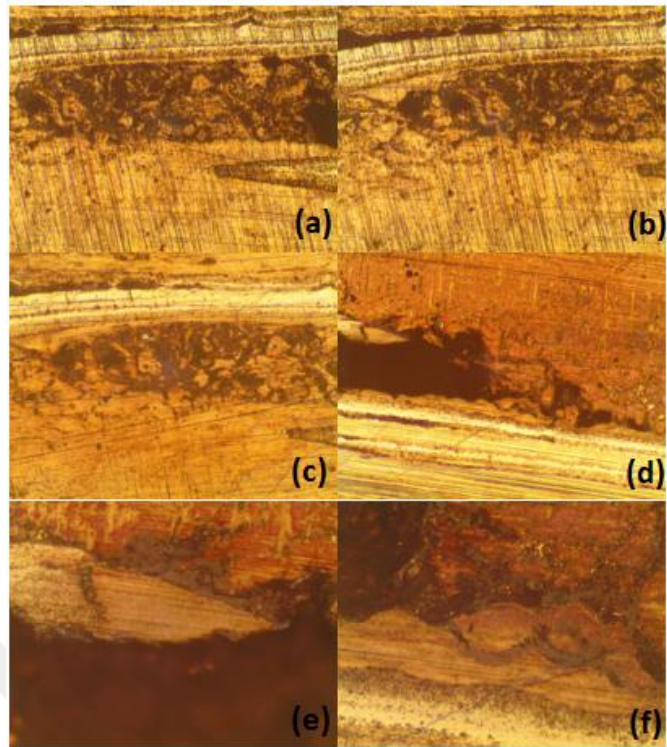


Figure 5.46. Optical microscope images obtained at different magnifications for the joint prepared using reacted standard IMD wire. (a-b-c-d) $5\times$ magnification, (e-f) $20\times$ magnification.

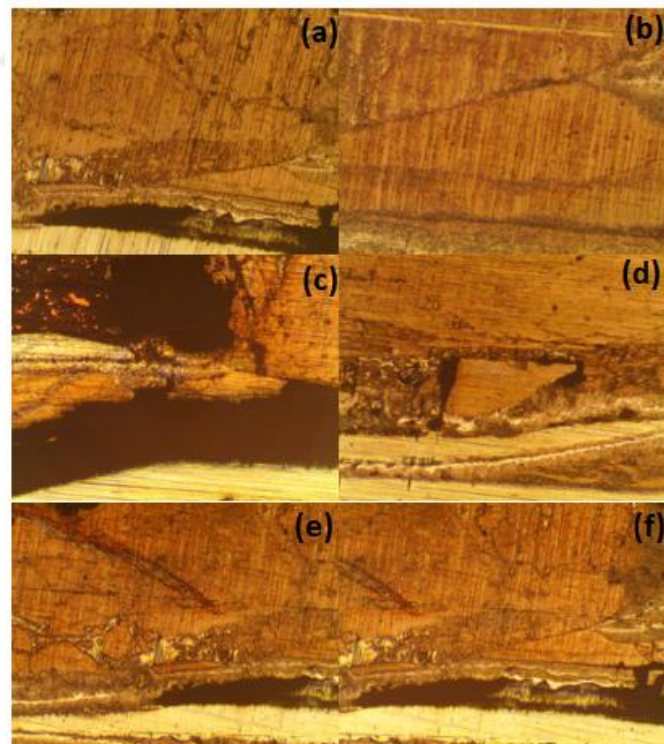


Figure 5.47. Optical microscope images obtained at different magnifications for the joint prepared using reacted half-EMD wire are given. The image in Fig. 5.47(b) was taken at $20\times$ magnification and the other images were taken at $5\times$ magnification.

Figures 5.46 and 5.47 show optical microscope images of the joint regions produced using standard IMD and half-EMD wires, respectively. Figure 5.46 shows that there are broken areas in the joining section. These breaks are likely to occur during polishing. However, it can be seen in Figure 5.47 that this type of fragmentation is less at the joints of half-EMD wires. The joining surface between the half-EMD wires and the joint region is wider and smoother than that of the joint for standard IMD wires. The images obtained support the difference between the transport properties of the joints. The wide and dense superconducting MgB₂ core structure of half-EMD wires provided better current transfer within the joint.

5.5.2 Closed loop joint resistance measurements

A closed superconducting loop was obtained by joining the ends of a superconducting loop using the termination joint as shown in Figure 5.48. The induced circulating supercurrents that occur when the initially applied magnetic field is decreased to zero cause some magnetic field to be trapped in the closed superconducting loop. The trapped magnetic field decays over time due to the resistive nature of the joint. The resistance value of the joint is calculated using the equation given below (D. Patel *et al*, 2015).

$$B = B_0 e^{-(R/L)*t} \quad (2)$$

B is the instant magnetic field at time t , B_0 is the initial trapped magnetic field, L is the inductance of the superconducting closed loop, t is the decay time and R is the resistance of the joint. Theoretically, the magnetic field value trapped in a resistance-free superconducting closed loop is expected to be maintained indefinitely. However, if the MgB₂ grain connectivity is not fully achieved between the tips of the MgB₂ superconducting wires and the joint core region, resistance occurs. In the presence of joint resistance, power on a superconducting closed loop is dissipated in time and magnetic field trapped inside loop decays in time. For this reason, the resistance values of the joints must be $<10^{-13} \Omega$ in order to operate a magnet produced using MgB₂ superconducting wire in persistent current (PC) mode

(D. Patel *et al*, 2016). Magnetic flux trapping inside a superconducting loop can be achieved in two ways: The first way is the field cooling method; superconducting loop is cooled below its critical temperature value in the presence of external magnetic field. When the temperature is lower than the critical temperature of the superconductor, the magnetic field is turned off and a magnetic field is trapped inside the loop due to the circulating supercurrent. The second method is to create a dynamic effect on the closed loop in the superconducting state by first increasing the external magnetic field and then decreasing it to zero.

Four single closed loops with superconducting joints were made using different unreacted wires; i. half-EMD wire (joint R-1/R-1H), and ii. standard IMD wire (joint R-2/R-2H). The loops shown in Figures 5.48(a) and (b) were obtained using unreacted half EMD and standard IMD wires, respectively. The tips of the wire loops were cut with an angle of 15° in order to increase contact surface inside the joint. These tips were placed in two 0.8 mm diameter holes drilled in the compressed B powder in the joint mold. Then, a vertical load of 1 ton was applied to the side surface of the joint mold to compress the joint core without breaking the wire ends. Finally, the closed loops with joint were heat treated at 650°C for 6 hours. After the formation of the superconducting MgB_2 phase, a magnetic field sensor was placed in the center of the loop perpendicular to the external magnetic field in the cryostat system, as shown in Figure 5.48.

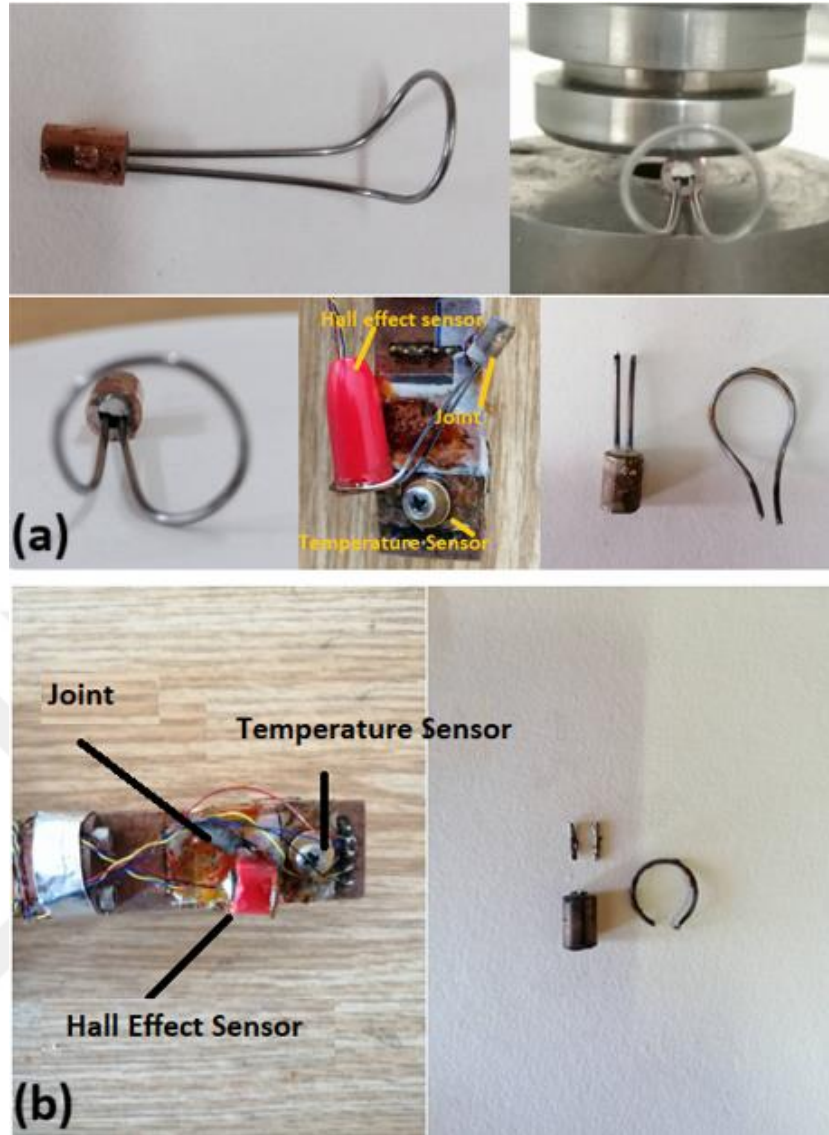


Figure 5.48. Images belong to the joints named (a) R-1 and (b) R-2 and their placement to the sample holder of the cryogenic measurement system.

Figures 5.49(a) and (b) show the process for magnetic field decay measurements in the single turn coils by dynamic effect. The blue line in Figure 5.49 indicates the time variation of the external magnetic field generated by a superconducting magnet. The black line is the magnetic field measured using a Hall sensor placed in the center of the single turn superconducting coil. Magnetic field decay measurements were performed at a constant temperature of 20 K, as indicated by red line in Figures 5.49(a) and (b). The external magnetic field was first increased from zero to 1 T or 1.5 T at 0.01 T/s and held at these values to ensure the stability of the magnetic field. The external field was then reduced to zero at the same rate of 0.01 T/s. The Hall sensor reads the magnetic field that is the sum of

external and induced magnetic fields in the center of the loop. It is seen in Figs. 5.50(a) and (b) that after the external magnetic field was reduced to zero, magnetic flux of ~ 97 mT and 10.5 mT remained in the R-1 and R-2 joints, respectively. The magnetic field decay measurements revealed that the magnetic field within the loop with R-1 joint is significantly greater than that of the loop with R-2 joint. This implies the supercurrent circulating in the loop with R-1 joint is larger than the induced supercurrent in the loop with R-2 joint. Furthermore, upon careful examination of Figures 5.51(a) and (b), it becomes evident that the magnetic field confined within the loop with R-1 joint suddenly drops towards zero with increasing the control temperature. The magnetic field trapped in the R-2 jointed loop gradually decreases until 1.1 mT, which is considered as the remnant field in the source superconducting magnet. The quench behavior observed in the loop with R-1 joint by a controlled increase in working temperature reveals the circulation of large supercurrents. The joint resistance values were determined by monitoring the magnetic field decay in the loops for 24 hours. Figure 1.8 shows that the magnetic field value inside the loop with R-1 joint decreases from 97.44 mT to 96.15 mT during 24 hours at a constant temperature of 20 K. The magnetic field value in the loop with R-2 joint varies between 10.3 mT and 10.4 mT for about 16 hours and then decreases by fluctuations. The single loop inductance values of R-1 and R-2 joints are 0.019 μ H and 0.014 μ H, respectively. Using the inductance values in Equation 2, the resistances for R-1 and R-2 joints were calculated as $9.58 \times 10^{-15} \Omega$ and $2.43 \times 10^{-15} \Omega$, respectively. Figures 5.51(a) and (b) show the quenching behavior of the R-1 and R-2 joints, respectively.

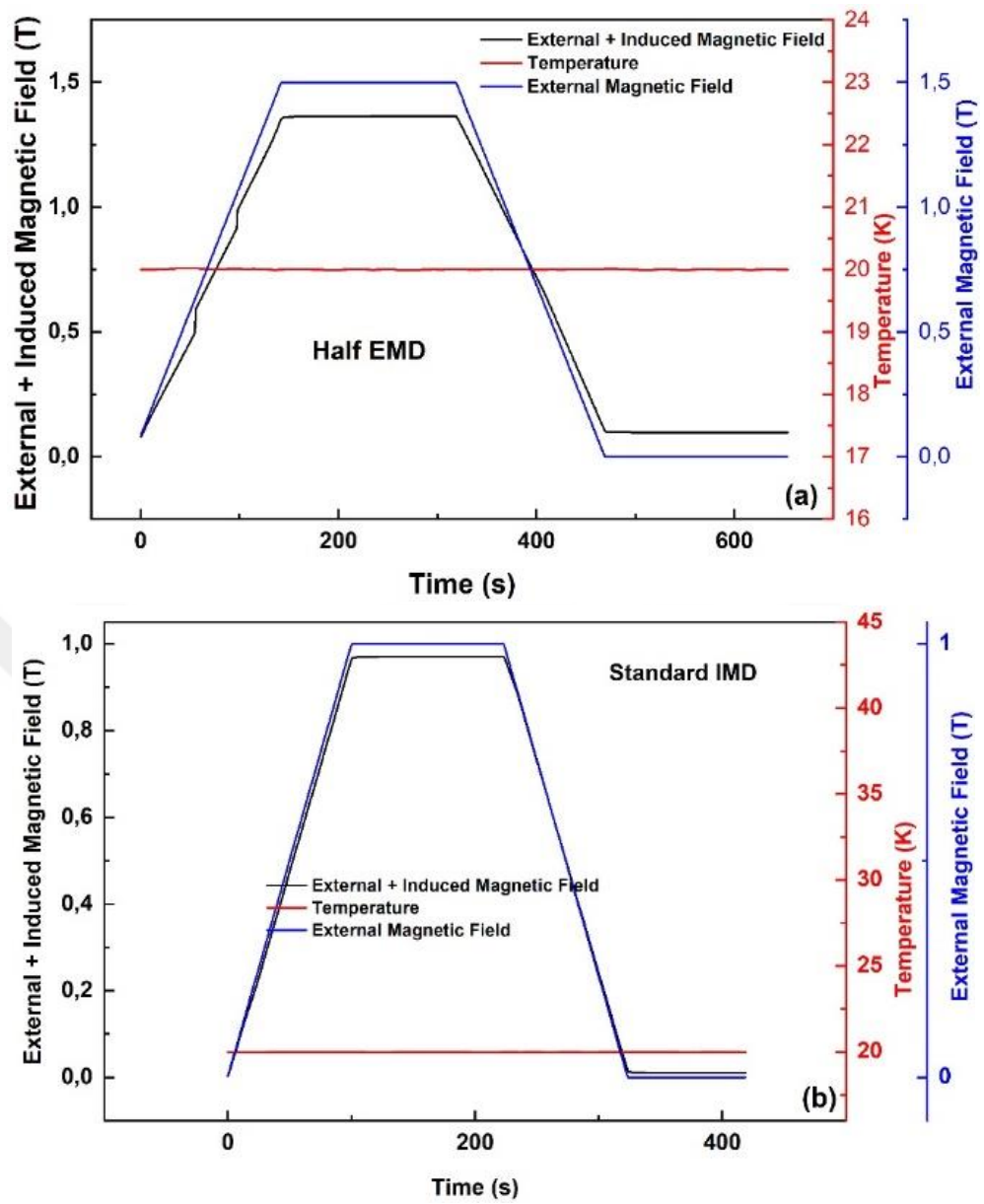


Figure 5.49. Experimental processes for magnetic field decay measurements to determine the resistance of superconducting joints of (a) R-1 and (b) R-2.

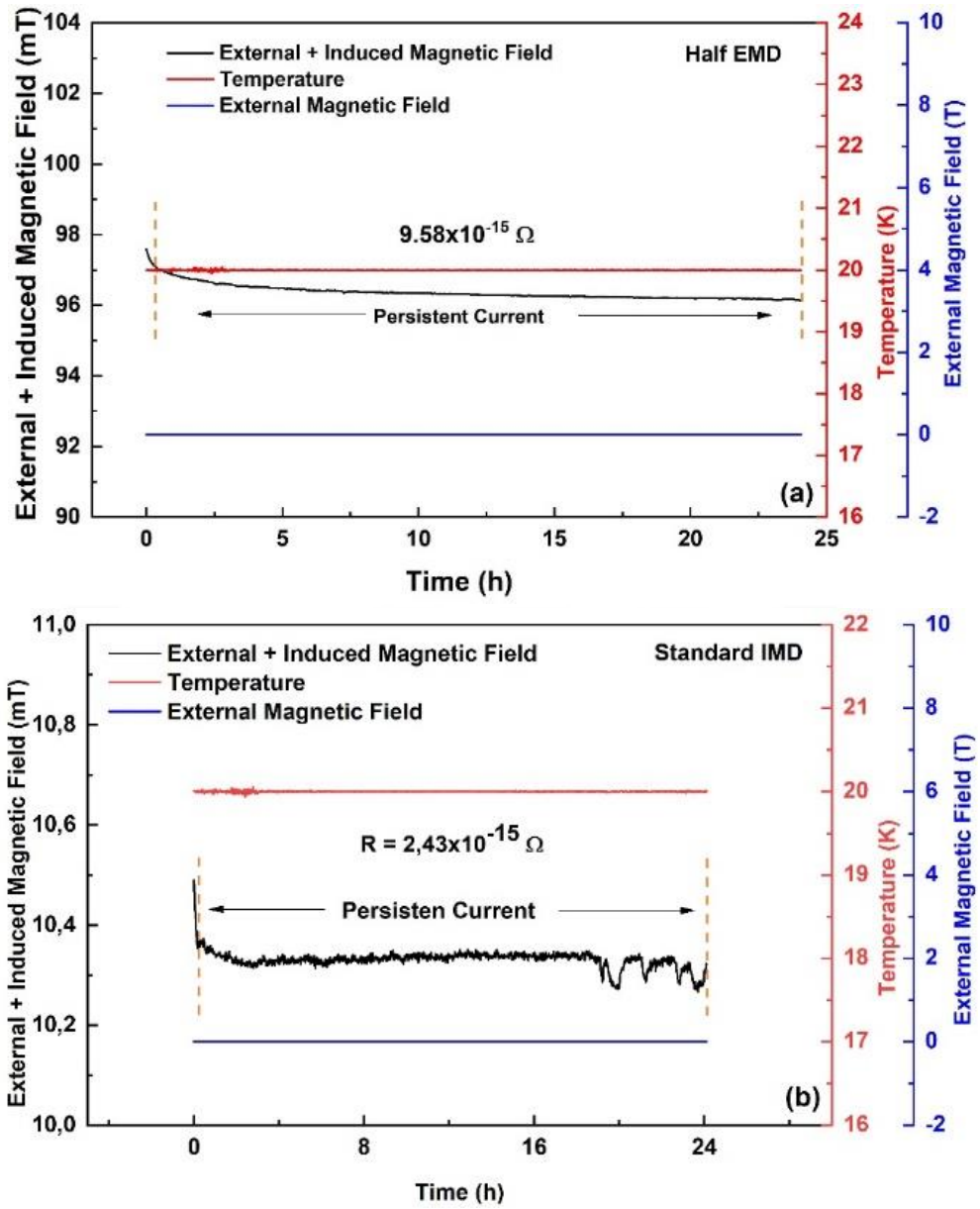


Figure 5.50. The magnetic field decay measurement graphs for superconducting loops closed with joints of (a) R-1 and (b) R-2.

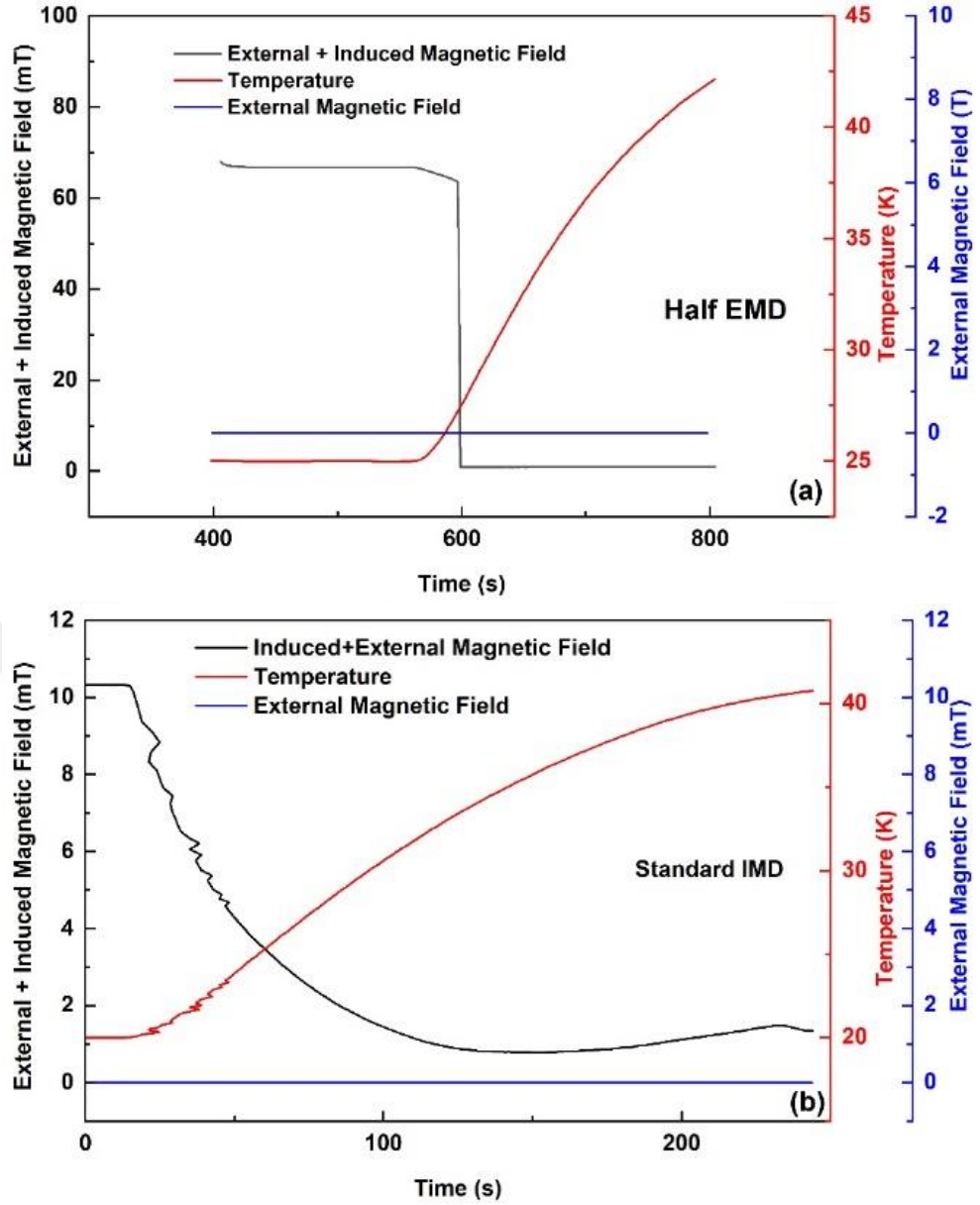


Figure 5.51. Quenching behavior resulting from a controlled increase in the temperature of superconducting loops closed with (a) R-1 and (b) R-2 joints.

After cutting the ring parts shown in Figure 5.48, resistance-temperature ($R - T$) measurements were performed on the joints. Then, after the joint parts were cut and removed, $R - T$ measurements for the remaining wires were made using the four-probe contact method. The $R - T$ curves of the joints and one of the wire pieces were obtained by slowly heating working temperature from 15 K to 45 K. Figures 5.52(a) and (b) show the $R - T$ curves of the joints and one of the wires (joint terminal). It can be seen that the normal state resistance of the R-1 (Fig. 5.52(a)) joint is 5 times higher than the normal state resistance of its wire. Also, the $T_{c,offset}$

value of the joint is about 2.5 K lower than that of the wire. It is also seen in Fig. 5.52(b) that the normal state resistance of the R-2 joint is 3 times higher than the normal state resistance of its wire. As the temperature increases from 15 K to $T_{c,offset}$ value, there is a gradual increase in the resistance value of R-2 joint. There is a slight increasing trend in the resistance value of the samples as the temperature increases from 15 K to their $T_{c,offset}$ values. This situation is more evident in the joints that are larger in mass than the wires. This situation is thought to be related to the heating - cooling balance occurring on the samples in the He gas contact closed cycle cryostat system.

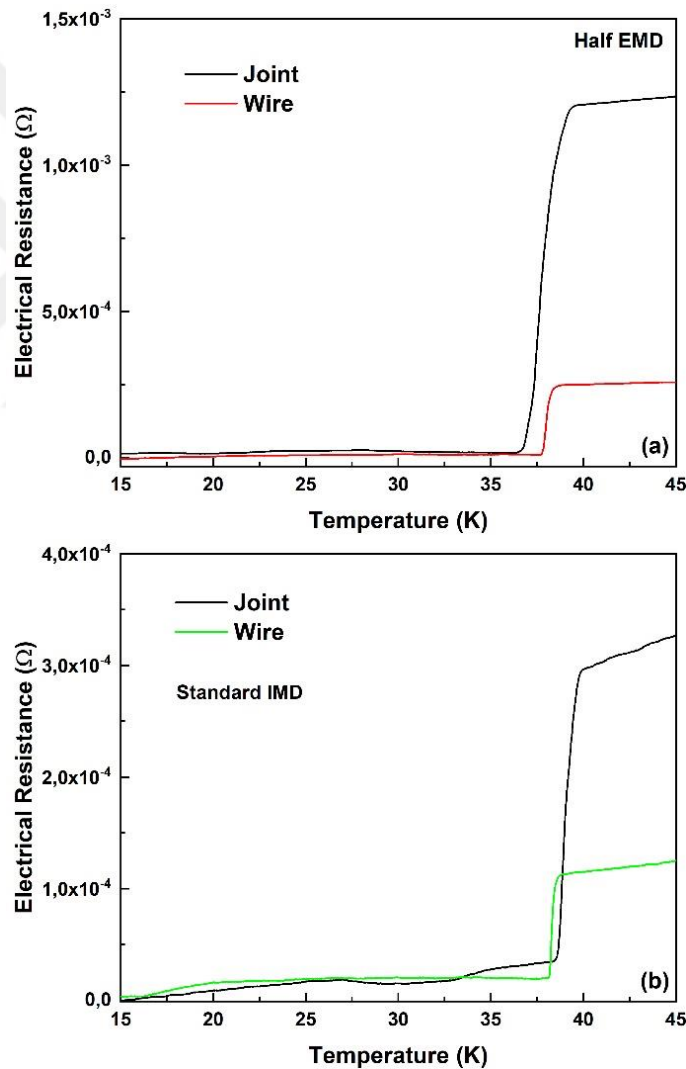


Figure 5.52. $R - T$ curves of the wires used as joint terminals and superconducting joints (a) half EMD wire / R-1 and (b) standard IMD wire / R-2, respectively.

Figures 5.53(a) and (b) show the images of the joints R-1 and R-2 taken with an optical microscope. These images were taken from the contact surface of the MgB_2 core of joint and wire tips in the joint region. It is seen in Figs. 5.53(a) and (b) that the MgB_2 core formation in the wire and joint region has been successfully achieved. When the interface where the wire and joint core meet in the R-2 joint is examined, it is observed that there are broken pieces caused by the thin MgB_2 core structure of the standard IMD wire. On the other hand, it is clearly seen in Figure 5.53(a) that the wire and joint contact surface is successfully formed for the R-1 joint. However, upon closer examination of the core connection interfaces in the contact region, variations in color shade suggest a slightly looser core formation at certain places compared to surrounding regions. This discrepancy can be attributed to the drilling of holes for wire tip insertion into the B powder region of joint mold, causing disruption of the structure and a subsequent decrease in density within this region. A low-density core structure formed at the interface between the wire and the joint region may cause a quench effect when a high current is transported.

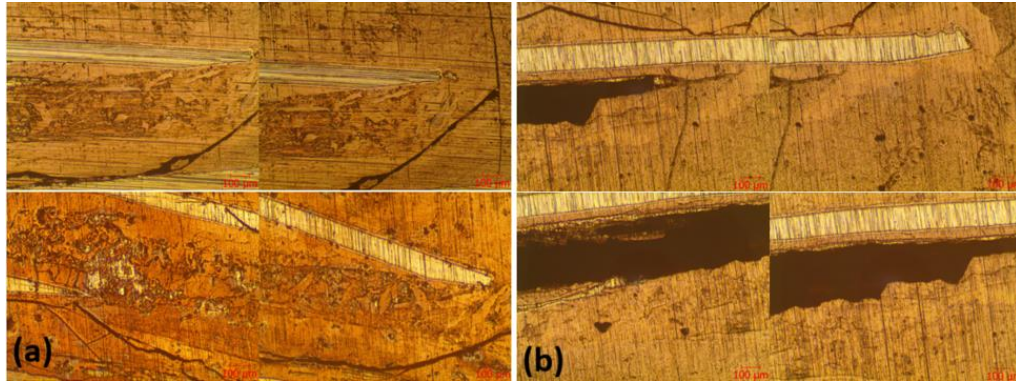


Figure 5.53. Optical microscope images for joints of (a) R-1 and (b) R-2 obtained at $5\times$ magnification.

Figures 5.54(a) and (b) show images of the R-1H and R-2H termination-type joints, which joins half-EMD and standard IMD wires, respectively. The experimental processes were carried out the same as R-1/R-2 joints. After the superconducting MgB_2 phase formation was achieved, a magnetic field sensor was placed in the center of the ring and placed in the measurement system perpendicular to the external magnetic field as shown in Figures 5.54(a) and (b) (top). The I_c measurement setup was shown in Figs. 5.54(a) and (b) (bottom). The current-voltage measurements were carried out using high transport current in helium vapor

medium at 20 K under the external magnetic field. Some important parameters which belong to the R-1H and R-2H joints are given in Table 5.4.

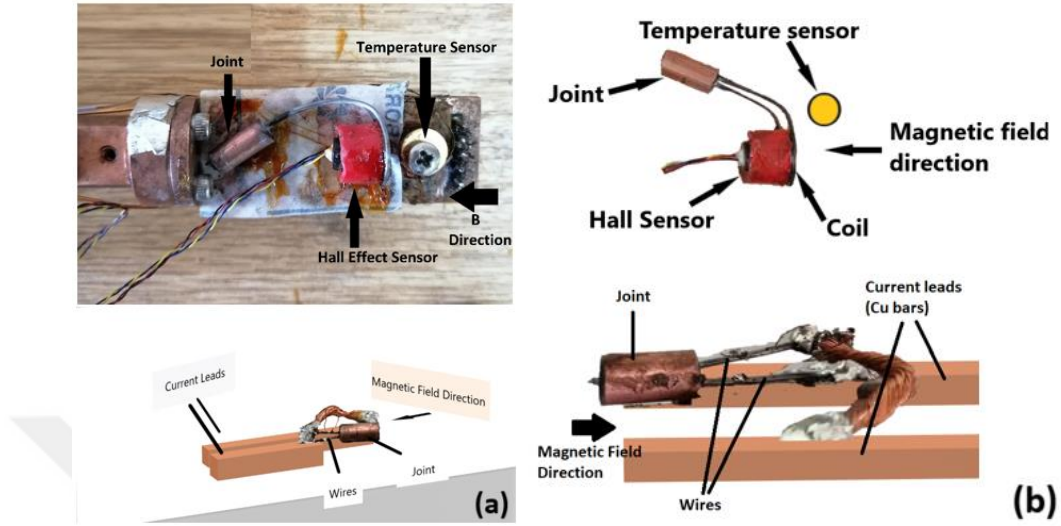


Figure 5.54. Images of joints (a) R-1H and (b) R-2H placed on the sample holder. Magnetic field decay measurement setup (top). Transport I_c measurement setup (bottom).

In Figures 5.55(a) and (b), the results of magnetic field decay measurements conducted on the joints R-1H and R-2H were presented, respectively. Initially, the joints were subjected to a cooling process down to a temperature of 20 K in the absence of an external magnetic field. Subsequently, an external magnetic field was incrementally raised from 0 to 1 T, with a rate of 0.01 T/s. Following the attainment of a stable magnetic field at 1 T, this level was sustained for a duration of couple of minutes. Subsequently, the magnetic field generated by the superconducting magnet was reduced back to 0 T at the same rate. Precise temperature control was followed throughout the measurement period by maintaining a constant cryostat temperature at 20 K with a variation of approximately 20 mK. Notably, a total magnetic field of approximately 24.46 mT and 9.7 mT were measured within single turn superconducting coils with R-1H and R-2H joints, respectively. Figure 5.55 shows that the magnetic field inside the loop decays rapidly at first, but then slows down. The results obtained from the magnetic field decay measurement exhibit that initial field of 24.46 mT decays to 24.29 mT in the loop with R-1H joint and 9.7 mT decays to 9.594 mT in the loop with R-2H joint for 12 h. It is worth noting that

the rapid initial drop in magnetic field strength may be attributed to the interplay between the critical current and the n -value, both of which are sensitive to magnetic field variations (D. Patel *et al*, 2015)

Table 5.4. Some important parameters for the R-1H and R-2H joints.

Parameter	R-1H	R-2H
Wire diameter (mm)	0.81	0.81
Loop inner diameter (mm)	10.30	8.55
Loop outer diameter (mm)	11.92	10.17
Coil inductance (μ H)	0.01948	0.0157
Coil constant (mT)	0.121	0.146
Wire type	in-situ mono/ EMD	in-situ mono/ IMD
Number of turns	1	1
Winding method	Wind & react	Wind & react

Based on the calculations, the electric current circulating within the closed loop approximates 200 A and 65 A, resulting in the generation of magnetic fields of 24.29 mT and 9.594 mT for joints R-1H and R-2H, respectively. For the joints R-1H and R-2H, over a 12 hours duration, the magnetic field monitored within the coil exhibited decreases of 0.17 mT and 0.017 mT, respectively. The calculated resistance values for the R-1H and R-2H joints stand at $3.14 \times 10^{-15} \Omega$ and $6.44 \times 10^{-16} \Omega$, respectively, signifying an exceptionally low resistance level among joints fabricated using monofilament MgB_2 wires. This remarkably low resistance value of 10^{-15} - $10^{-16} \Omega$ renders the R-1H and R-2H joint highly suitable for the operation of an MRI magnet in Persistent Current (PC) mode.

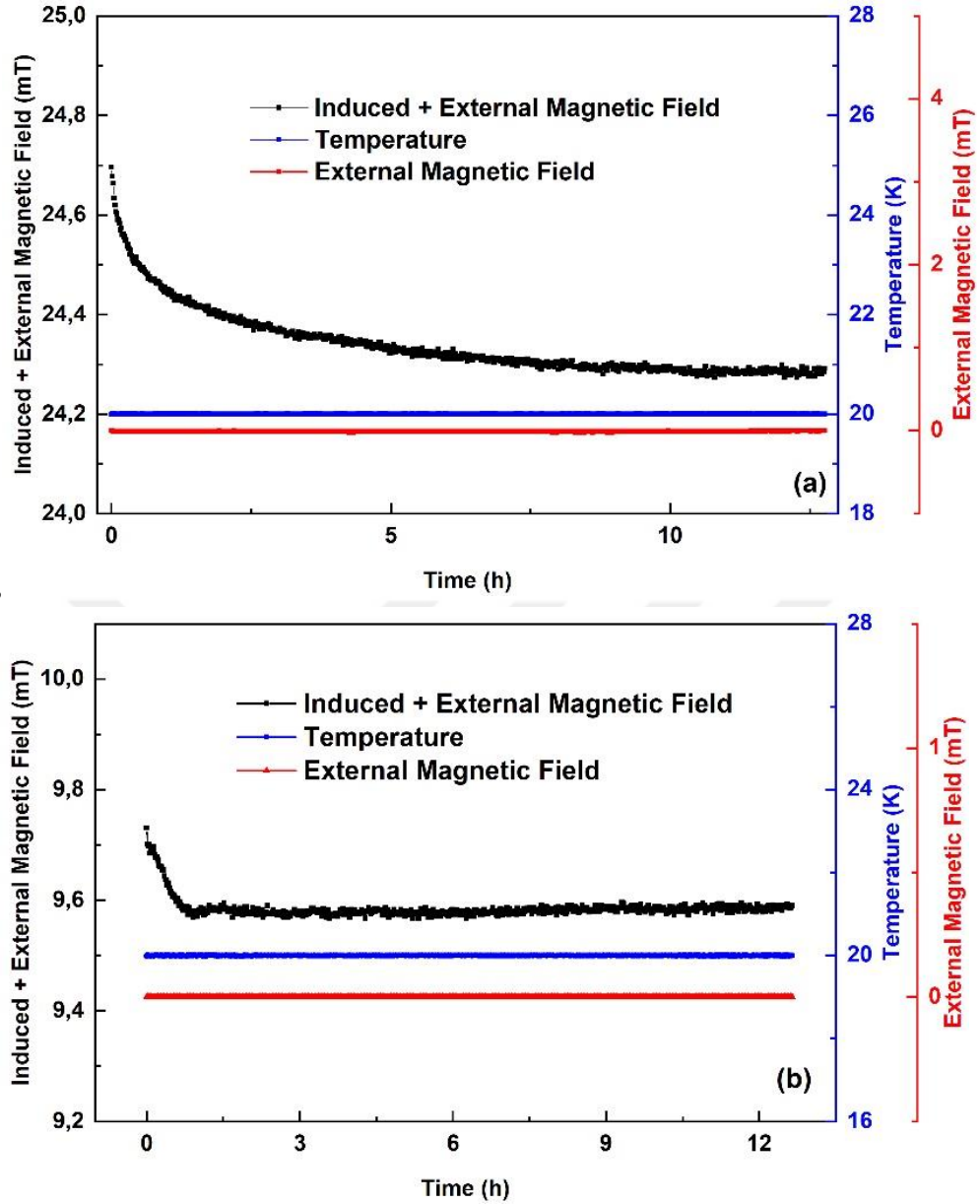


Figure 5.55. Magnetic field decay measurement results for the superconducting loops with joints of (a) R-1H and (b) R-2H.

After the removal of the circular segment of the loop with a cutting process, the transport I_c measurements were conducted on the joints. These I_c measurements were done at 20 K in the presence of an externally applied magnetic field which is oriented perpendicular to the transport current direction within the joint region. I_c measurements were performed in two different ways: first, by increasing the magnetic field from a lower value to a higher value (LTH), and second, by decreasing the magnetic field from a higher value to a lower value (HTL). In both scenarios, the cooling procedures for the joint were done in the absence of external

magnetic field. The $I - V$ characteristics of the joints at a temperature of 20 K, varying in response to alterations in the external magnetic field, are visually presented in Figures 5.56 and 5.57 for the joints R-1H and R-2H, respectively. Fig. 5.56(a) demonstrates that the R-1H joint exhibits superconducting behavior when subjected to a current value of >10 A under a magnetic field strength of 1.5 T, which is increased low to high magnetic field value at a temperature of 20 K. However, Fig. 5.56(b) the R-1H joint is not superconducting above the current value of 5 A under 1.5 T when the magnetic field value decreased from high value to low value. The R-1H joint, which was produced using half EMD wire, is quietly affected by magnetic field due to the semi cylindrical MgB_2 core structure of half-EMD wire. It was not observed any significant difference on the superconducting behavior of the R-2H joint when the measurement method is changed. It can be seen in Figs. 5.57(a) and (b) that the joint exhibits superconducting behavior when subjected to a current of 7 A under a magnetic field strength of 1.5 T at 20 K for LTH or HTL measurement methods. Notably, a negative voltage peak is observed in $I - V$ curves during the transition from superconducting to normal states for R-2H joint, but this phenomenon is not consistently observed for all magnetic field strengths (D. Patel *et al*, 2016).

In addition to the Zero Field Cooling (ZFC) procedure, $I - V$ measurements were also performed, in which the joints were cooled from 50 K to 20 K under a magnetic field, called the Field Cooling (FC) procedure. Figures 5.56(c) and 5.57(c) depict the $I - V$ characteristics of the R1-H and R2-H joints at temperature of 20 K under 2.5 T and 2 T, respectively, for all measurement methods. In particular, a significant decrease in the I_c of the R-1H joint was observed in the FC condition compared to the $I - V$ measurement results obtained from the LTH or HTL procedures, as seen in Figure 5.56(c). However, there is no visible change in $I - V$ characteristics of the R-2H joint at 20 K, as illustrated in Figure 5.57(c). This behavior may be attributed to no magnetic field is trapped within the joint bulk during FC process. However, the decrease in the I_c of the R-1H joint for FC process under a magnetic field of 2.5 T reveals that the magnetic field start to penetrate into the joint. These findings hold significant implications for the comprehensive characterization of superconducting joints and their application in magnetic field-related technologies (D. Patel *et al*, 2021).

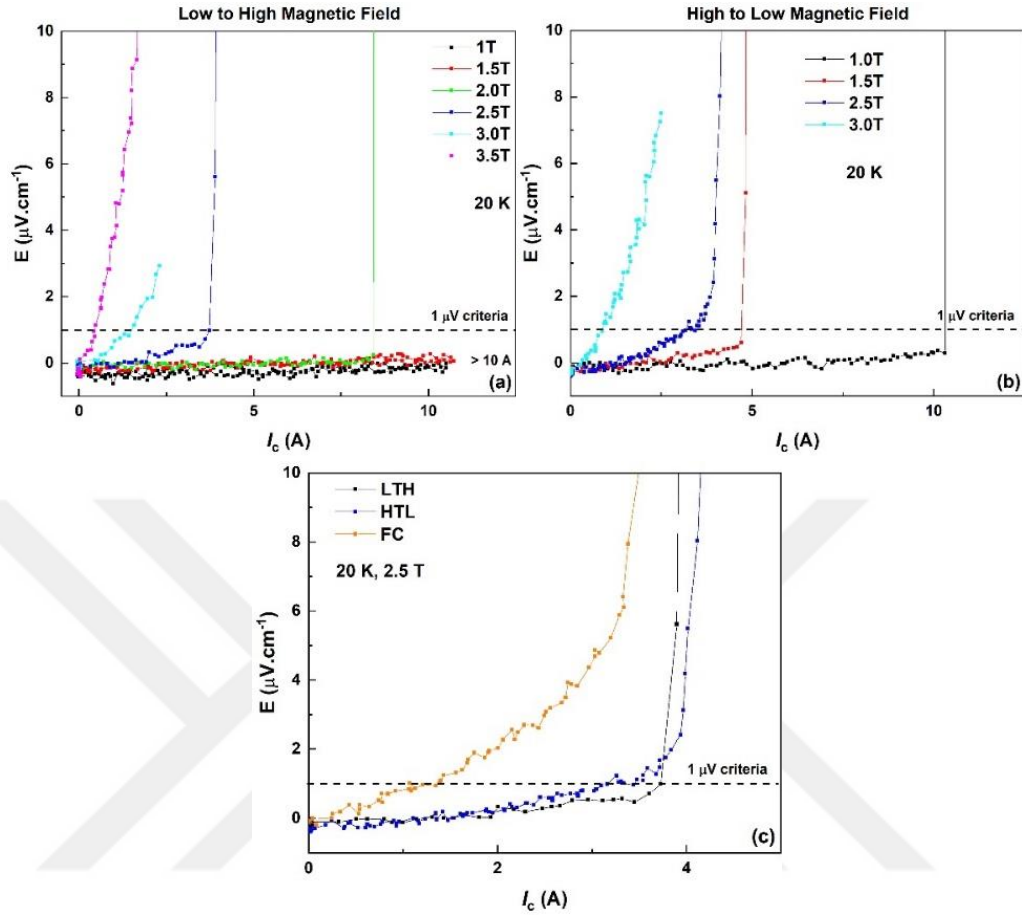


Figure 5.56. $I-V$ curves of the R-1H joint under different external magnetic fields. (a) and (b) LTH and HTL cases under ZFC at 20 K, respectively. (c) $I-V$ curves for three different measurement methods at 20 K under 2.5 T.

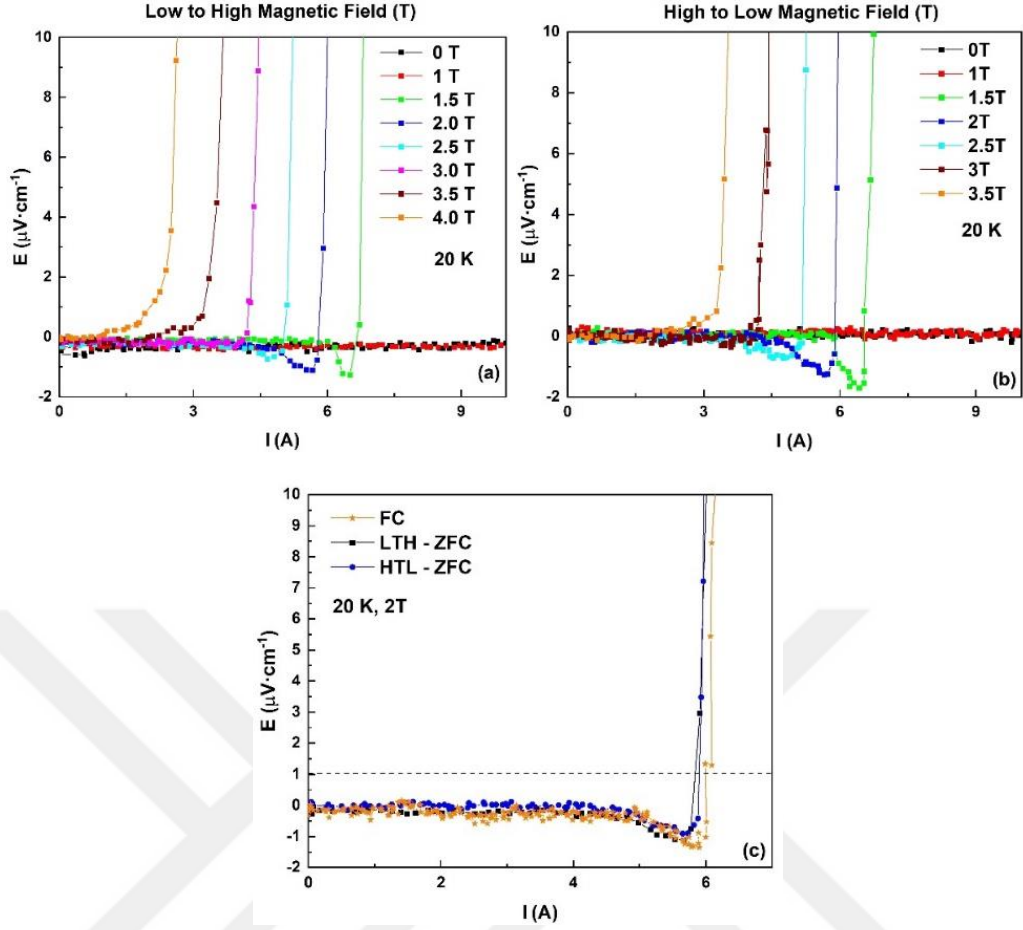
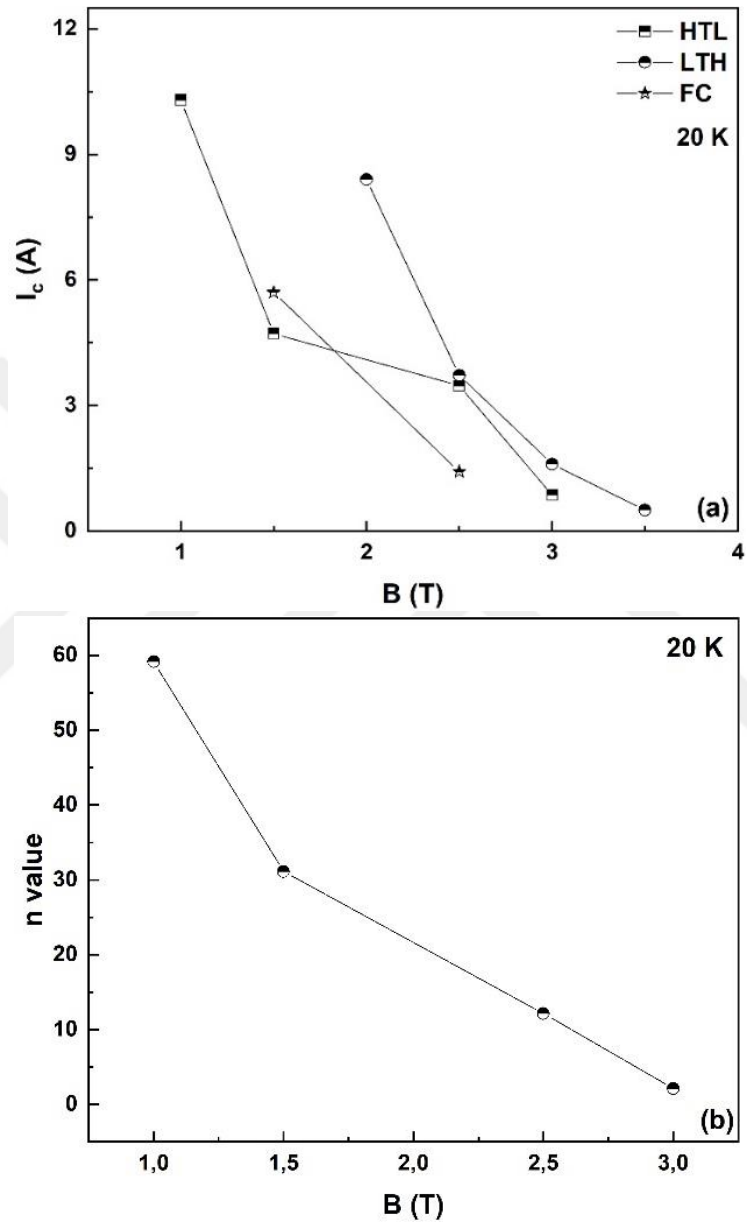


Figure 5.57. $I-V$ curves of the R-2H joint under different external magnetic fields. (a) and (b) LTH and HTL cases under ZFC at 20 K, respectively. (c) $I-V$ curves for three different measurement methods at 20 K under 2 T.

In Figures 5.58(a) and (c), the I_c values for the R-1H and R-2H joints were presented as a function of the applied magnetic field at 20 K. The results demonstrate that while there is no significant difference in the I_c values of the R-2H joint, a significant difference exists in the I_c values of the R-1H joint. Specifically, the I_c for the R-2H joint remains consistently around 7 A, as seen in Fig. 5.58(c). Furthermore, the n -values indicate the performance difference between the superconducting R-1H and R-2H joints such as 59 and 66 at 20 K under magnetic field of 1.0 T and 1.5 T as shown in Figures 5.58(b) and (d), respectively. These n -values were determined by fitting the $I-V$ curves in the electric field range of -1.5 to $1 \mu\text{V}\cdot\text{cm}^{-1}$ because the superconducting transition for the R-2H joint initiates with negative voltage values. Obtaining a high n -value is of great importance for the safety and efficiency of superconducting coils. Notably, the n -values for the R-1H and R-2H joints under magnetic field of 1 T are promising. The

n -value lower than 30 at 20 K is accepted insufficient for the operation of superconducting magnets in PC mode (D. Patel *et al*, 2014).



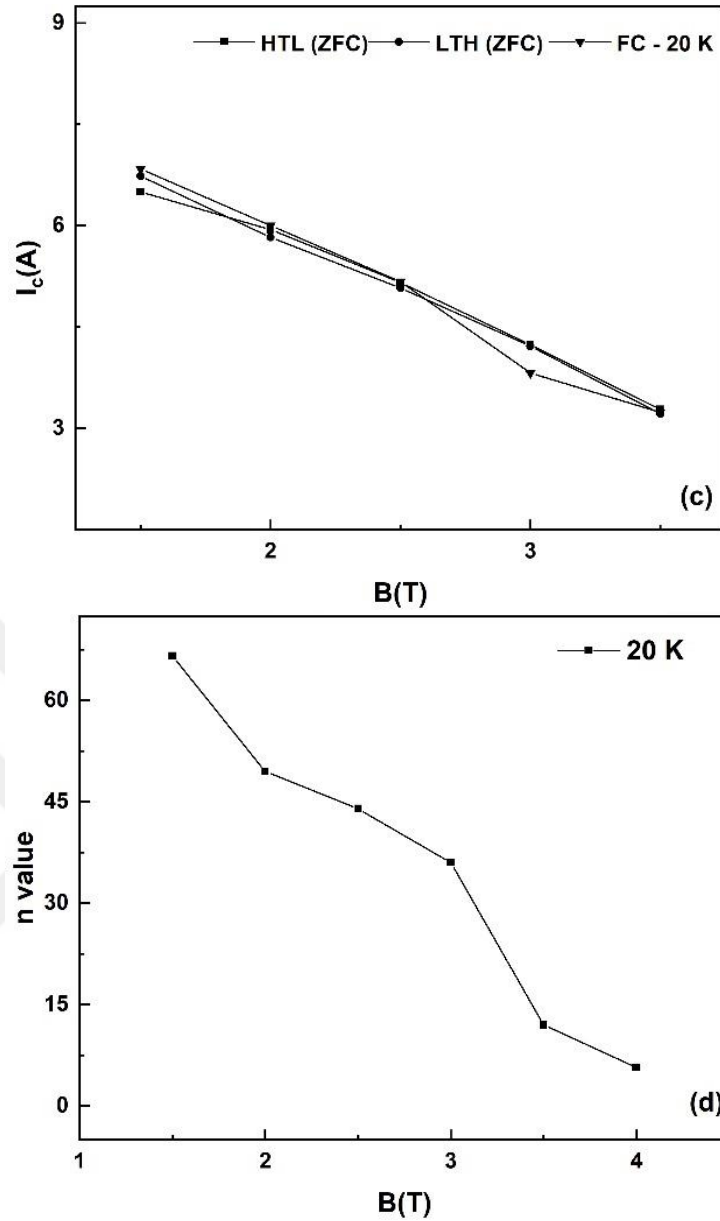


Figure 5.58. The magnetic field dependent I_c and the n -value obtained from $I - V$ curves of the R-1H joint (a-b) and R-2H joint (c-d).

Figures 5.59 and 5.60 illustrate $I - V$ characteristics of joints and wire segments cut from these joints, at 20 K under magnetic fields of 0 T and 1.5 T. Figure 5.59 shows that voltages increase linearly with increasing current and a sudden jump in voltage occurs at a critical current value. This type of $I - V$ behavior may be caused by the MgB_2 wires forming the joint input/output ends being too short. In Figure 5.59, the I_c for the wire segment was determined as 31.30 A, slightly higher than the I_c value of 29.86 A measured for the R-1H joint at 20 K. The I_c of the joint is 4.61 % lower than the I_c of the wire measured at 20 K and under 0 T magnetic field. In fact, the transport I_c value of the joint is considerably lower than

the induced current of 200 A calculated using the results of magnetic field decay experiments. This significant degradation in I_c can be attributed to the excessive heating effect of the circulating high supercurrent in the quenching experiments where the ring was heated rapidly from 20 K to 40 K. Figure 5.60(a) shows that the I_c of the wire is 58 A and the joint has 62 A at 20 K. Importantly, the I_c of the joint is very close to the calculated induced current of 65 A, arising from the magnetic field trapped within the joint loop. A 6.5 % difference between I_c of the wire and joint was observed. This minor reduction in I_c of the wire can be attributed to less heating effects induced by supercurrents during quench measurements. The reason of the slightly lower I_c of the wire than that of the joint at 20 K under 0 T may be related with the larger volume of the joint mold in terms of thermal effects (W. Goldacker *et al*, 2004, H. Yetiş *et al*, 2022). This is because the I_c of the joint is 20 % lower than the I_c of the wire at 20 K under 1.5 T as seen in Fig. 5.60(b).

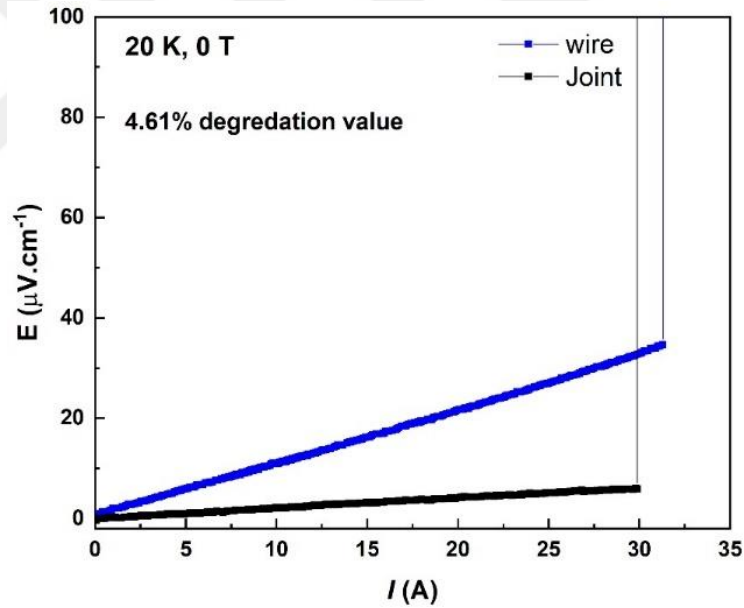


Figure 5.59. $I - V$ curves of the R-1H joint and joined (terminal) wire under 0 T at 20 K.

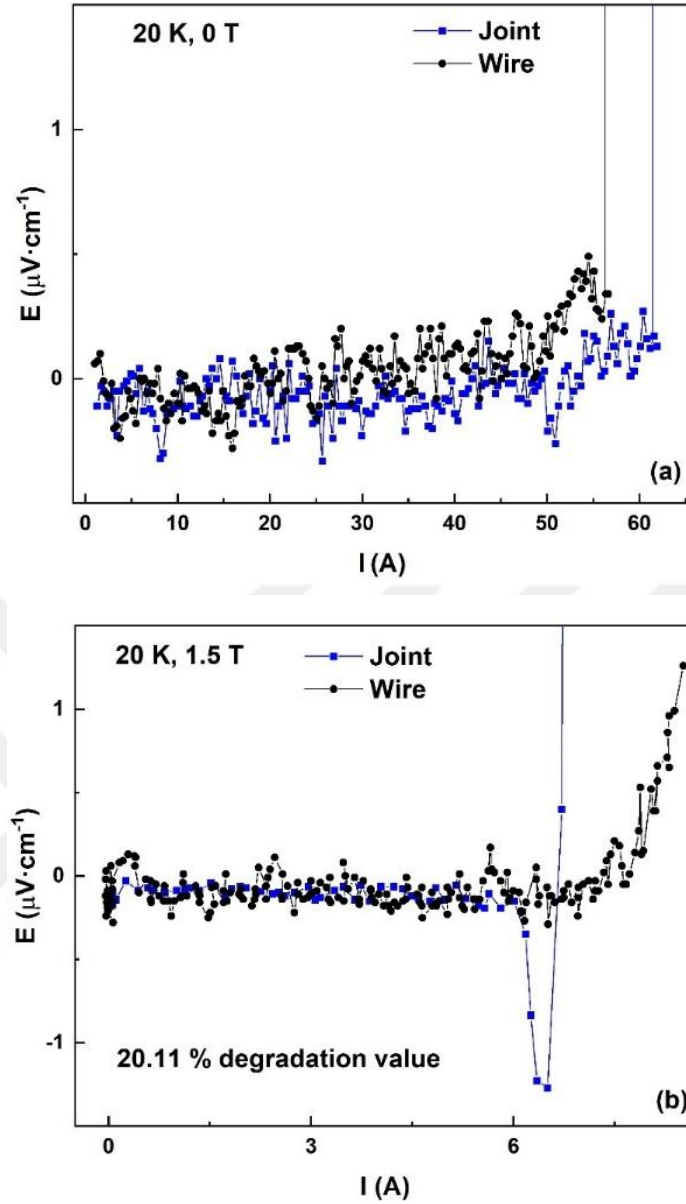


Figure 5.60. $I - V$ curves of the R-2H joint and joined wire (a) 0 T at 20 K, (b) 1.5 T at 20 K.

Following the completion of DC transport measurements, the joint underwent longitudinal cutting operations, as shown in Figure 5.61. Remarkably, the mechanical strength of the joint was such that it could not be damaged during the cutting procedures, allowing intact longitudinal sections to be obtained suitable for subsequent scanning electron microscopy (SEM) examinations. The formation of the MgB_2 phase within the joint mold was carried out by IMD method, and a high-density interconnection structure was obtained between the wires. Examining

the longitudinal sections near the wires aims to reveal the MgB_2 structure between these wire elements.

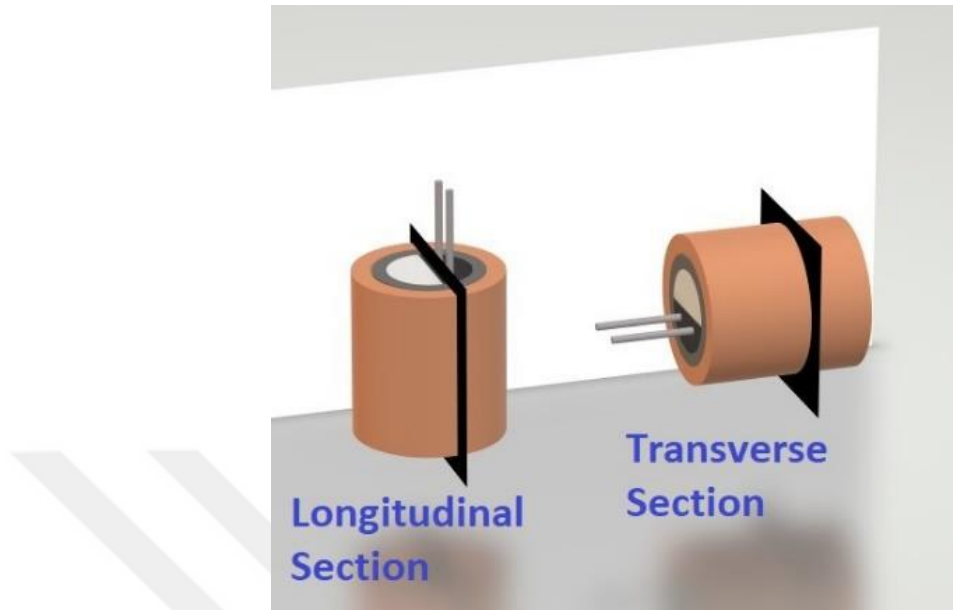


Figure 5.61. The schematic picture represents the cutting processes for the joint in transverse and longitudinal directions.

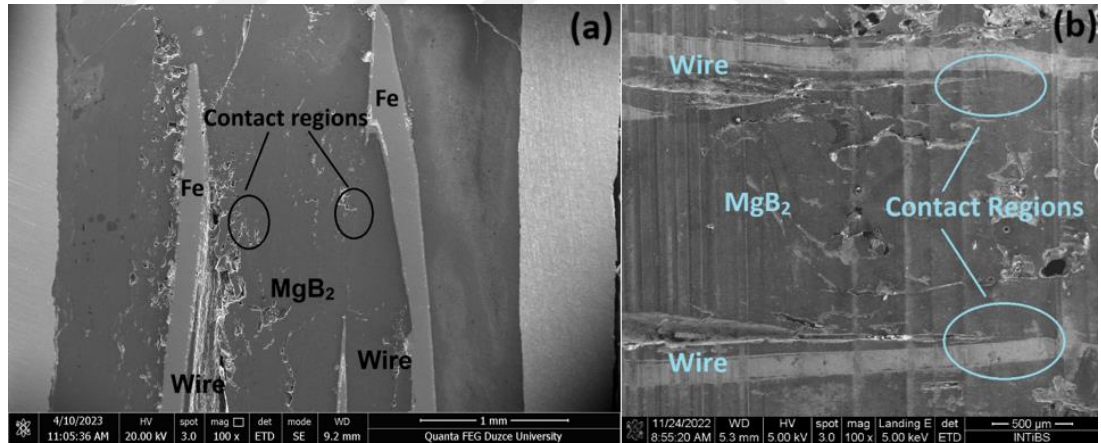
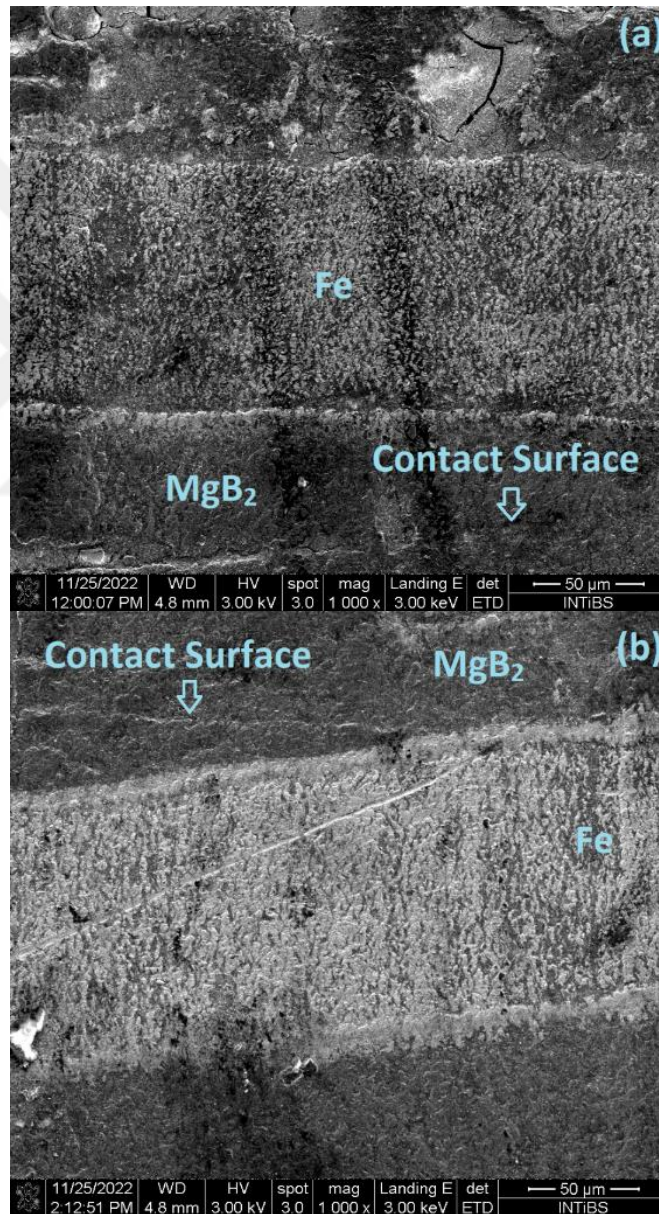


Figure 5.62. The images taken using SEM device from the longitudinal sections of the (a) R-1H and (b) R-2H joints (100 $\times$ magnification).

The SEM images taken from the longitudinal section reveal an arrangement where the angled surfaces of the wire ends are oriented toward each other, as shown in Figure 5.62. The measurement of the maximum distance between these wire ends is approximately 1.6 mm for the R-2H joint, while for the R-1H joint this distance is approximately 1 mm. The slight curvature or narrowing observed along the wire

ends is attributed to volumetric contraction that occurs during the chemical reaction between Mg and B within the joint mold. This suggests that the connectivity between the wires and the core material is significantly strengthened.

Figures 5.63(a), (b), (c), and (d) provide convincing evidence of an almost perfect structural fit between the wires and the joint core. In other words, the MgB_2 phase formed shows a remarkable similarity everywhere. This structural perfectness accounts for the extremely low joint resistances quantified as $3.14 \times 10^{-15} \Omega$, and $6.44 \times 10^{-16} \Omega$.



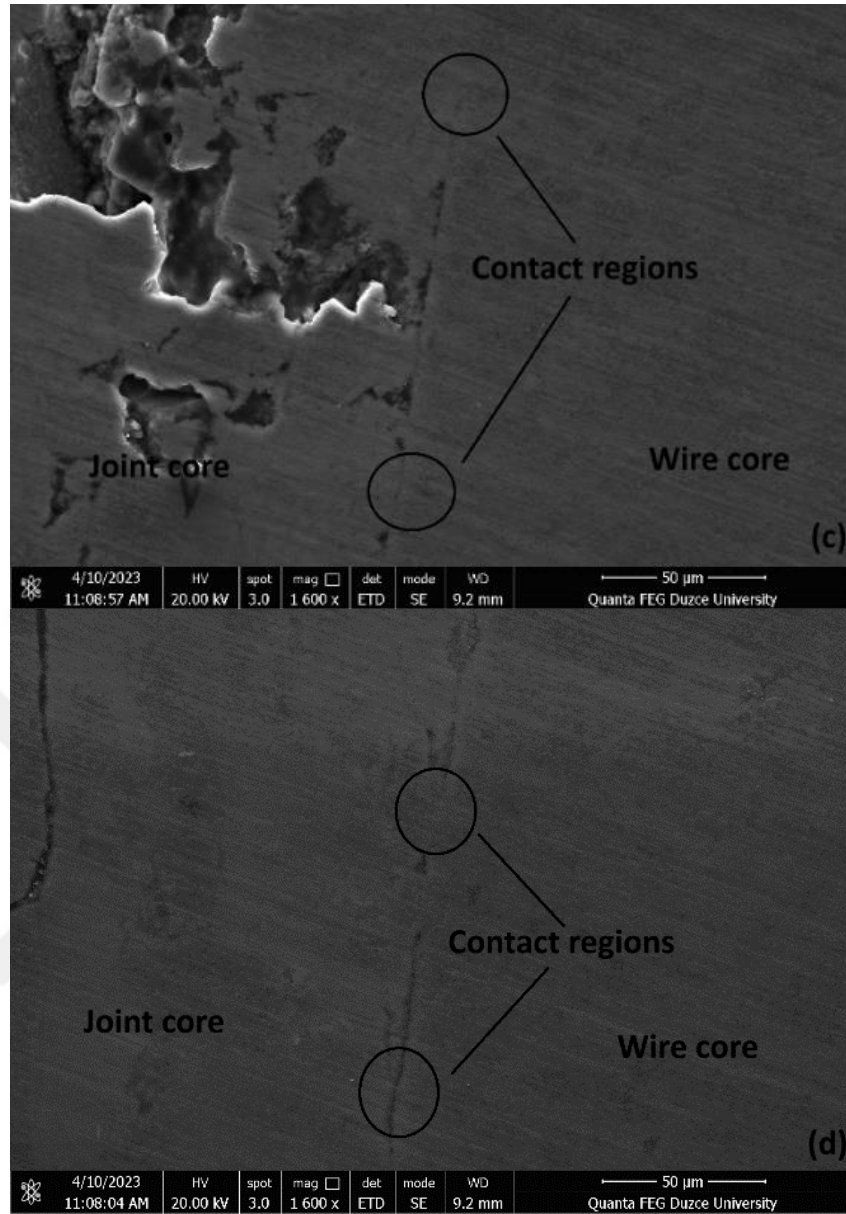


Figure 5.63. SEM images of R-2H joint (1000× magnification) taken from the longitudinal section of the wire-joint bulk connection region, which is close to the wire ends. (a) The tip of the top wire, (b) the tip of the bottom wire. The images (c) and (d) of R-1H joint (1600× magnification) taken from the wire-bulk connection region, which is close to the wire end.

The resistance values for the single turn closed loops with joints were calculated using the Eqn. 2 after the magnetic field decay measurements were completed. The resistance values of the joints produced using half-EMD, and S-IMD MgB₂ wires are as follows; (R-1) $9.58 \times 10^{-15} \Omega$ – (R-1H) $3.14 \times 10^{-15} \Omega$, and (R-2) $2.43 \times 10^{-15} \Omega$ – (R-2H) $6.44 \times 10^{-16} \Omega$, respectively. According to these results, the same level of resistance values is achieved for all single turn closed

loops. This novel joining methodology demonstrates the reproducibility of the joints. When the joints produced in the study were compared to each other, the joint with the lowest resistance value was obtained for the closed loop produced using standard IMD wire. However, it has been observed that the trapped magnetic field value in the loop closed by this joint is also low. This situation is directly related to the low supercurrent circulating in the closed loop. From this perspective, it can be seen that a higher magnetic field is trapped in the closed loop produced using half-EMD wire compared to the loop prepared by standard IMD wire. The loop produced by half EMD wire has a low joint resistance and a high current carrying capacity, making these wires stand out for the use of superconducting coils in case of PC mode. Another interesting observation is that the I_c of the R-2H joint does not change regardless of whether the magnetic field is applied in an increasing (HTL) or decreasing (LTH) manner. In a study by Patel et al. (D. Patel *et al*, 2021), a significant decrease in the I_c of the joint was noted in the $I - V$ measurement made by reducing the magnetic field from 5 T to zero at 20 K. This decrease in I_c was explained by the presence of trapped magnetic field within the superconducting joint. This drawback causes difficulties in terms of using such superconducting joints in superconducting coil systems. The designed wire and joints designed in this study offers an improved sensitivity in determining the I_c of the joint under the external magnetic field. Moreover, considering the current results obtained for the joint resistance, these are the lowest values obtained to date for joining unreacted mono-core IMD wires, which are quite suitable for the operation of a superconducting magnet in PC mode (D. Patel *et al*, 2016).

6. CONCLUSIONS

In this thesis study, various wires were produced with distinct core designs based on the internal Mg diffusion technique. In addition to the standard Internal Magnesium Diffusion (S-IMD) wire which is produced using Mg rod, alternative wires were manufactured using Mg-rich Mg + B tablets (RD-IMD), powder Mg (P-IMD), and three thin Mg rods (TH-IMD). The RD-IMD method, proposed as an alternative to the S-IMD method, aims to produce wire samples using Mg-rich Mg + B tablets instead of using Mg rod. The primary objective of this approach is to increase the engineering critical current density in the IMD wires. This increase is achieved by increasing the proportion of MgB_2 core in the wire by forming a porous MgB_2 structure instead of a hole in the wire center. A dense MgB_2 layer surrounding the porous MgB_2 structure was obtained by diffusion of excessive amount of Mg particles in the center. Thus, both a dense MgB_2 ring layer similar to S-IMD wire and a porous MgB_2 structure in place of a central hole were obtained. In the P-IMD method, wire samples were prepared using magnesium powder instead of Mg rod. The P-IMD method offers important advantages such as freedom in core dimensions, ability to determine Mg particle size and purity, mechanical deformation benefits and low cost. Since the size of Mg rod is a limiting factor, the production of IMD wires using magnesium powder also allows the production of long wires. The only drawback for the P-IMD method may be the increase in oxygen content since the use of spherical Mg particles instead of Mg rod increases total interaction surface. TH-IMD, another innovative wire preparation method, involves placing three thin Mg rods in boron to form a triangle. This type of core design aims to reduce the diffusion path of Mg in boron and uniformly spread the melted Mg into the boron layer during heat treatment.

Studies on superconductor jointing of wires have been carried out with the realization of wire production. At this stage, H-EMD wires were also produced with a filling method containing Mg powder in the longitudinal semicircle and B powder in the other semicircle. The layer thickness of the MgB_2 ring formed in IMD wires is very thin. H-EMD wires are designed to provide an effective contact surface for the joint. The H-EMD method enabled the superconducting core of the IMD wire to be formed in one region and a more effective current path was obtained.

Structural analyses of S-IMD, RD-IMD, and P-IMD wires showed that increase at annealing time improved core homogeneity. All three methods utilized amorphous nano boron powder in the core, but differences emerged in the thickness of the MgB_2 layer. J_{ce} - B curves indicated that RD-IMD wire has higher J_{ce} values than that of P-IMD wire. S-IMD wires had the highest J_{ce} values under various external magnetic fields. The thickest MgB_2 layer of S-IMD wire provided obtaining a high J_{ce} value under the external magnetic field. On the other hand, RD-IMD wire exhibited superior critical current in self magnetic field as compared to S-IMD and P-IMD wires. Fast $I - V$ measurements showed that RD-IMD wire had the I_c value of 167 A at 33 K without quenching. It is considered that the current is shared between the porous MgB_2 structure and non-porous dense MgB_2 ring structure in the RD-IMD wire. The porous structure in the center of RD-IMD wire acted as a stabilizer at high currents. The thicker MgB_2 layer in S-IMD samples caused to obtain highest J_{ce} values under external magnetic fields but was found not capable to carry high currents. Additionally, S-IMD and P-IMD wires were quenched at high currents. The heat generated at high currents due to the low thermal stability of the Fe sheath could not be distributed quickly. Electromechanical tests of wires produced by three different methods revealed that S-IMD and RD-IMD samples were superior to P-IMD samples with a bending strain tolerance of 0.46 %.

Monofilament TH-IMD wires with a diameter of 0.81 mm were heat treated at 650 °C for 1, 2, and 4 h and at 700 °C for 1 h. TH-IMD wires exhibited a narrow superconducting phase transition and had $T_{c, \text{onset}}$ initial values close to 37 K in self-field. $I - V$ measurements performed at 4.2 K revealed that TH-IMD wires have excellent transport J_c values and the J_c value as high as $5.0 \times 10^4 \text{ A.cm}^{-2}$ was obtained under 8 T for TH-IMD wire annealed at 650 °C for 1h. This means that the I_c value is approximately 55 A under 8 T (90 A under 6 T) at 4.2 K. Undoped TH-IMD MgB_2 wires showed superior current carrying capacity under high magnetic fields. TH-IMD wires were found to be less sensitive to heat treatment time than heat treatment temperature. It has been demonstrated that it is possible to produce high-quality superconducting MgB_2 wire by TH-IMD method. Detailed examination on the lateral section of the TH-IMD wire at three different locations within the MgB_2 core by point Energy Dispersive X-ray Analysis (EDAX) showed that there was a uniform core structure in all regions of the wire. This uniformity

was supported by the similarity in the atomic mass percentage of Mg in the EDAX analysis results. Electromechanical tests performed on the wires produced by the TH-IMD method showed that the three-hole core structure is resistant to bending effects with a bending tolerance value of 0.46 %.

Monofilament H-EMD wires with a diameter of 0.81 mm were heat treated at 650 °C for 1, 2, and 4 hours and at 700 °C for 1 h. As similar to TH-IMD wires $R - T$ measurements showed that H-EMD wires exhibited an exceptionally narrow phase transition width ($\Delta T = T_{c,onset} - T_{c,offset}$) below 1 K in self-field. $I - V$ measurements performed at temperature of 4.2 K showed that the H-EMD wire heat treated at 650 °C for 1 h had a remarkable J_c value of 1.5×10^4 A.cm⁻² under the external magnetic field of 8 T. It is understood that undoped H-EMD wires have high current carrying capacity, similar to TH-IMD wires. EDAX analysis revealed that Mg diffused effectively throughout the core, even in regions where the core was thickest. Electromechanical tests performed on wire produced by the H-EMD process revealed that the semi-cylindrical core shape is sensitive to bending effects, with a bending tolerance value of less than 0.3 %.

The superconducting joining of the produced H-EMD and S-IMD wires were done and the joints were characterized using transport and magnetic field decay measurements. The field trapped inside the superconducting loop is used to calculate joint resistance by Equation 2. The resistance values for joints of H-EMD and S-IMD MgB₂ wires are as follows: (R-1) 9.58×10^{-15} Ω, (R-1H) 3.14×10^{-15} Ω, and (R-2) 2.43×10^{-15} Ω, (R-2H) 6.44×10^{-16} Ω, respectively. The fact that the joints have resistance values close to each other shows that the joints are reproducible. When the obtained joint resistance values are compared with the results of other studies, the lowest resistance value ever has been achieved in joining unreacted monocoire IMD wires. This resistance levels are low enough to maintain a magnet in a persistent current mode (D. Patel *et al.* 2016).

It is observed that the joint, joining the standard IMD wires, has the lowest resistance value. However, a lower magnetic field is trapped within the superconducting loop formed using S-IMD wire than in the loop formed using H-EMD wire. This means that induced supercurrents circulating in the H-EMD loop is higher than that in S-IMD loop. It is obvious that the H-EMD loop closed by a single joint can carry high currents at 20 K. Another important result is the unchanged I_c of the R-2H joint regardless of whether the magnetic field is first

increasing (HTL) or decreasing (LTH) in $I - V$ measurements. Patel *et al.* observed that the I_c value obtained by decreasing the magnetic field from 5 T to zero was lower than the I_c value obtained by increasing the magnetic field from 0 T to 5 T in $I - V$ measurements of a large joint performed at 20 K under magnetic field (D. Patel *et al*, 2016). The small size joint may allow the I_c of the joint to be determined precisely under the external magnetic field and may be particularly advantageous for in large superconducting coils.



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