Overview of MgB\textsubscript{2} wires fabricated by Sam Dong Co., Ltd.

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Synopsis: MgB\textsubscript{2} is a known superconductor material competing with the commercially popular low-temperature superconductor of Nb–Ti. The key advantages of MgB\textsubscript{2} include a relatively high transition temperature of 40 K, low material cost, and large upper critical field, with potential in various superconducting applications in a cryogen-free environment. This review will summarize the state-of-the-art MgB\textsubscript{2} various conductors fabricated by Sam Dong Co., Ltd., Republic of Korea and provide clear insights to practical users.

Keywords: carbon doping, electromagnetic property, in-situ process, mechanical property, MgB\textsubscript{2} superconducting wires (Some figures in this article may appear in colour only in the electronic version)

1. Introduction

Since the discovery of superconductivity in 2001, magnesium diboride (MgB\textsubscript{2}) has been regarded as a potential candidate for replacing the low-temperature superconductors, such as Nb–Ti and Nb\textsubscript{3}Sn\textsuperscript{1,2). Combined with its low-cost precursor, simple crystalline structure, and easy fabrication, a relatively high transition temperature of 40 K enables its cryogen-free operation between 10–20 K for various engineering applications. In particular, the key applications such as nuclear magnetic resonance (NMR)\textsuperscript{3), magnetic resonance imaging (MRI)\textsuperscript{4-6), power transmission cables \textsuperscript{7), and superconducting magnetic energy storage (SMES)\textsuperscript{8-10) are highly desirable.

Moreover, MgB\textsubscript{2} has significant advantages over other high-temperature superconductors (HTSs) such as YBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{7 (YBCO) coated conductor and Bi\textsubscript{1.8}Pb\textsubscript{0.2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x (BSCCO-2223) tape for commercial expansion. These include: 1) round (or rectangular shape) conductor, 2) anisotropy-free conductor, 3) cost-effective conductor (ratio of performance to price), and 4) long length conductor >5 km\textsuperscript{11).}

Despite the mentioned advantages of MgB\textsubscript{2}, it has drawbacks because of the in-field performance and wire inhomogeneity. It is known that carbon is a very effective dopant for enhancing high-field performance. A substituted carbon in a boron (B) lattice increases the impurity scattering, resulting in an enhanced upper critical field (B\textsubscript{c2})\textsuperscript{12-14). Presently, the performance of carbon doped MgB\textsubscript{2} conductor is comparable to that of Nb–Ti at 4.2 K. The Powder-in-Tube (PIT) method is still limited due to an inhomogeneous powder packing into a metallic tube when we consider a long-length conductor of more than 3 km. Therefore, it is necessary to develop a wire fabrication process. Alternatively, a continuous form process has been suggested. This review will summarize our research progress on various MgB\textsubscript{2} conductors over the past six years at Sam Dong Co., Ltd.\textsuperscript{15, 16) Republic of Korea. In addition, we offer a practical guideline to the users for development of various superconducting devices.

2. History of Sam Dong R&D

Sam Dong Co., Ltd. (hereafter Sam Dong) was established in 1977, fabricating oxygen-free high conductivity (OFHC) copper and various metal-based wires. Particularly, the company has been supplying the highest quality magnet wire products for heavy industries. To meet international customer requirements, Sam Dong also established entities in Tennessee (2007), Ohio (2009), in the USA and Poland (2018), EU. As a new vendor for customized MgB\textsubscript{2} superconducting wires, Sam Dong seeks both experimentally and theoretically multi-faceted and highly collaborative multidisciplinary areas in materials science, physics, cryogenics, and electrical and mechanical engineering within and beyond the EU/Korean/Australian universities, institutes, and industries. In 2017, the company first supplied our
commercial MgB$_2$ conductors to Korean and Japanese institutes. Recently, we provided wires to a European company for a Big Science project and Japanese university for MgB$_2$ SMES program.

In the last six years, three Korean government grants were awarded to us: (1) Development of fabrication techniques of metal–alloy superconductor composites wires, (2) Development of a commercial nano-carbon doping process using glycerin, and (3) Scalable integration of MgB$_2$ superconducting wires considering cost-effectiveness and industrial competitiveness. The three grants were funded to a total of 1.3 million $.

3. Powder-in-Tube (PIT)

A Sam Dong MgB$_2$ superconducting wire is fabricated using the Powder-in-Tube (PIT) method. Owing to the brittle nature of the ceramic, using a metallic tube easily enables a long-piece wire of MgB$_2$\textsuperscript{17, 18}. Based on the type of initial raw material, the PIT process can be classified into \textit{in-situ} (using a mixed powder of raw magnesium (Mg) and B) and \textit{ex-situ} (using as-formed MgB$_2$ powder). Sam Dong strategically uses the \textit{in-situ} approach, with a subsequent heat-treatment for commercial conductors. To elaborate, a relatively low-reaction temperature below 700°C is applied to form an MgB$_2$ phase, resulting in small grains. This occurs because of the grain boundary pinning\textsuperscript{19-21}. As the grain size decreases, electromagnetic performance is enhanced. Moreover, the high-field performance is improved by adding carbon because it is the most effective dopant to improve high-field critical current. It is to be noted that resultant wire performances are also affected by the manufacturing process or initial material. It is well known that the conventional PIT approach causes wire inhomogeneity, i.e., a sausaging effect along the longitudinal direction, while fabricating a long-piece wire. To overcome this issue, we need to rationally design a starting powder using ball-milling and core density (i.e., porosity) via cold or hot isotropic press. In terms of mass production, our company puts great effort into precursor powders.

4. Precursor MgB$_2$ Powders

\textbf{Figures 1} (a) and (b) show the powder X-ray diffraction (XRD) and the corresponding scanning electron microscope (SEM) images of the precursor Mg (purity 99.5%), and B (purity 95–97%). As shown, Mg has a spherical shape fabricated using a spray pyrolysis and well-crystalline phase. It is to be noted that the MgO phase exists as an impurity. Mg tends to easily oxidize in air according to the Gibbs-free formation energy\textsuperscript{22}). B also has a spherical shape, but a typical amorphous phase. This can be attributed to the fabrication method, i.e., decomposition of B gas source. In addition, small amounts of B$_2$O$_3$ and H$_2$BO$_3$ are present as impurities\textsuperscript{23, 24}. In particular, B$_2$O$_3$, which has a relatively low decomposition temperature of 450°C, reacts with the starting material Mg to form MgO. Although the function of MgO remains unclear, most of the MgO particles are agglomerated at the boundaries of MgB$_2$ grains. This barrier can interrupt the current flow. However, small sized MgO particles (< 20 nm) can act as flux pinning centers to enhance the low-field electromagnetic performance\textsuperscript{25, 26}. Figures 1 (c) and (d) show the particle size distributions for Mg and B, respectively. Mg possesses an average particle size of approximately 10 μm and B of 1 μm or less (approximately 300–400 nm in size), indicating a substantial difference in the particle sizes. We expect that the size difference between the two powders causes an agglomeration after mixing the powder. Therefore, we need more efforts to obtain a narrow size distribution of as-prepared powder through the ball-milling process\textsuperscript{27-30}.

\textbf{Figure 2} shows the Rietveld refinement of the XRD diffraction pattern for sintered MgB$_2$ superconducting core. After heat treatment, Mg and B were effectively crystallized as MgB$_2$ along with a small fraction of MgO. The MgB$_2$ lattice parameters of $a$ and $c$-axes were calculated to be 3.0830 and 3.5210 Å, respectively. As a result of quantitative analysis, the phase composition was determined to be 85 wt.% MgB$_2$ and 15 wt.% MgO. In particular, the MgO is affected by the purity and size of Mg used, fabrication environment of the raw material powder, and atmosphere of MgB$_2$ heat treatment. To further improve the critical current characteristics of the MgB$_2$ superconducting wire, it is essential to reduce the proportion of the MgO impurity, i.e., the MgB$_2$ fraction should be maximized. Sam Dong is currently conducting research on a pretreatment process for removing MgO from Mg, resulting in the enhancement of electrical properties.

\textbf{Figures 3} (a) and (b) present a photograph of the wound wire of a 1 km-scale 18-multifilament MgB$_2$ and the corresponding cross-sectional image, fabricated by Sam Dong. The wire was fabricated with a final diameter of 0.83 mm using the PIT method. Presently, various wires up to 3 km in length can be fabricated with uniform properties in the longitudinal direction. The internal structure of the
superconducting wire consists of 18 MgB₂ superconducting filaments and one copper (Cu) filament at the center. For the outer sheath, Monel, which is an alloy of Cu and nickel (Ni), was used to enhance the mechanical strength of the wire, and Cu was used as the inner matrix for electrical and thermal stability. In particular, Cu imparts an excellent formation property for the conductor fabrication. Moreover, the applied Monel and Cu exhibit excellent physical bonding characteristics during fabrication.

**Fig. 1** XRD diffraction patterns of (a) Mg and (b) B powders. The insets are SEM images of the Mg and B powders, respectively. Particle size distribution of (c) Mg and (d) B powders.

**Fig. 2** Rietveld refined XRD patterns of MgB₂ core.

**Fig. 3** (a) Wound wire image and (b) cross-sectional image of a 1 km-scale commercial MgB₂ wire fabricated by Sam Dong.
the mechanical processing. Additionally, debonding can be minimized because of similar thermal expansion coefficients during heat treatment. Niobium (Nb) as a barrier was an unavoidable material to suppress the reaction between Cu and Mg, during the heat-treatment of MgB₂. For superior electrical properties, a relatively high fill factor is one of the key factors to increase the transport current. In our company, we currently increase the fill factor to a maximum of 20% in 18-multifilament MgB₂ wire.

5. Electromagnetic Properties

Critical current ($I_c$) or critical current density ($J_c$), which was determined by using the cross-sectional area of the wire cores of a superconducting wire is the most important parameter to evaluate the wire performance. Figure 4 shows the magnetic field dependence of $I_c$ and $J_c$ characteristics for an 18-multifilament MgB₂ superconducting wire at different operating temperatures, from 4.2 to 30 K. This wire was sintered at 650°C for 30 min under an argon (Ar) flow. $I_c$ performance was evaluated from the direct transport current using the standard four-probe method with the criterion of 1 $\mu$V/㎝. At 4.2 K and 3 T, $I_c$ was found to be 270,000 A/㎝² ($I_c \sim 240$ A), and at 20 K and 2 T, $I_c$ was estimated to be 84,000 A/㎝² ($I_c \sim 73$ A). As mentioned above, $I_c$ values are proportional to the fill factor. Therefore, as the wire diameter or superconducting core area increase, $I_c$ may increase. Our wire with a 20% fill factor, increases $I_c$ over 300 A at 4.2 K and 3 T and over 100 A at 20 K and 2 T.

6. Carbon Doping

Our significant research efforts for the past six years have led to noticeable outcomes with improvement in the in-field performances of MgB₂ conductors. Carbon is known to be a very effective dopant for enhancing the high-field $J_c$, even comparable to the low-temperature superconductor, Nb–Ti, at 4.2 K. When carbon is doped into MgB₂ lattices, atomic carbons partially substitute into B sites or induce B vacancies, causing lattice distortion in the MgB₂ structure. These structural defects increase the impurity scattering, thus improving the $B_{c2}$ and consequently improving the critical current capability in a high magnetic field (> 5 T). For this purpose, our company strategically selected solid-hydrocarbon, i.e., pyrene (C₁₆H₁₀), as a dopant. Pyrene exists in a colorless solid state, has a melting point of 145–148°C, and decomposes into molecular carbon below the formation temperature of MgB₂ phase. We expect that the active carbon is much easier into MgB₂ lattice during sintering process. Figure 5 shows $J_c$ characteristics of our doped and undoped MgB₂ wires. At 4.2 K and 10 T, $J_c$ of the doped wire was estimated to be 21,000 A/㎝², which is approximately ten times greater than that of the undoped wire. Even if there is a $J_c$ crossover between the two wires at 20 K and 3 T, the carbon doped wire exhibits a superior in-field performance. According to literature, poor transport properties at low field are related to porosity. The reaction between starting materials causes approximately 30% volume reduction, causing unavoidable porosity. For quantitative analysis, a 3-dimensional X-ray tomography was performed. The analysis revealed that the void fraction
is diminished by 10% due to the homogeneous malic-acid doping, approaching the theoretical limit of 30%, which exactly corresponds to the increase in the measured low-field critical current. This is comparable to that of Nb–Ti. With this consideration, we put extensive efforts to further enhance the low-field $J_c$ performance for practical applications. Recently, MgB$_2$ superconducting systems have shown significant potential to operate in a liquid hydrogen environment, at around 20 K.

7. Design and Fabrication of Various MgB$_2$ Conductors

A superconducting conductor is the key element for design considerations of various superconducting devices. The traditional system operation was immersed and operated in liquid helium (wet-coolant). However, this often causes technical difficulties for users who need to access the system in the event of an operational failure. Alternatively, GM (Gifford-McMahon)-cryocooler (dry-coolant) is suggested and utilized because of its cost-effectiveness and easy operability. Regardless of coolant, the safety of superconducting systems is regulated by the wire design, i.e., filament number, Cu fraction, sheath materials, etc. With these considerations, our MgB$_2$ conductors are designed and manufactured to meet the requirements of different users.

Figure 6 shows the cross-sectional views of various commercial MgB$_2$ wires fabricated by Sam Dong. The detailed specifications for the corresponding wires are illustrated in Table 1. As mentioned before, composites of various conductors consist of Monel, Cu, Nb, and MgB$_2$ filaments. Up to 54 core filaments are stacked, which are further increased according to the requirement. The architecture of the multifilament is important for creating another current pathway. If the supercurrent flow is interrupted in a damaged filament, it can flow through the neighbor filaments. Therefore, the Cu fraction is increased up to 74% for thermal and mechanical stabilities. A relatively high Cu fraction provides certain safeguards against any mechanical damage when an external strain/stress is applied to the wire.

Figure 7 shows $I_c$ and $J_c$ characteristics of the corresponding wires. The $I_c$ performance is proportional to the superconducting area. Among wires, the SD-C54 and SD-M36 wires exhibit the best $I_c$ values of 181 and 154 A, respectively, at 4.2 K and 4 T. The SD-C54 wire exhibits a relatively high $I_c$ value despite the lower area fraction because the MgB$_2$ core sufficiently compresses the inner superconducting cores in the drawing process due to several filaments. In contrast, at 4.2 K and 4 T, the SD-M06 and SD-M07 wires exhibit the lowest $I_c$ values at 64 and 62 A, respectively. This is because the MgB$_2$ fractions of these wires reached a fill factor of only 9 and 10%, respectively. For alternating current (AC) applications, our wires are fabricated as a braided or twisted conductor. In this case, both the outer sheath and Cu fraction should be

| Outer sheath | Wire   | Number of filaments | Composition ratio (area %) |
|--------------|--------|---------------------|----------------------------|
| Monel sheath |        |                     | Monel | Cu | Nb | MgB$_2$ |
|              | SD-M06 | 6                   | 32    | 48 | 11 | 9       |
|              | SD-M07 | 7                   | 34    | 44 | 12 | 10      |
|              | SD-M18 | 18                  | 33    | 30 | 21 | 16      |
|              | SD-M30 | 30                  | 33    | 31 | 23 | 13      |
|              | SD-M36 | 36                  | 32    | 23 | 29 | 16      |
|              | SD-M54 | 54                  | 35    | 29 | 21 | 15      |
| Copper       |        |                     |       |    |    |         |
|              | SD-C24 | 24                  | -     | 74 | 15 | 11      |
|              | SD-C30 | 30                  | -     | 65 | 21 | 14      |
|              | SD-C54 | 54                  | -     | 62 | 24 | 14      |
considered in advance.

8. Mechanical Properties

Under a cryogenic environment, mechanical issues related to structural deformation of the MgB2 conductors exists. These issues are mainly due to the thermal stress derived from temperature gradient in the cooling chamber or Lorentz force from an external magnetic field, which eventually destroys the system38, 39). For this purpose, we performed a tensile test under different temperatures and magnetic fields. Figure 8 shows the relationship between $I_c$ and the tensile strain of an 18-multifilament MgB2 wire at 20 K and 2 T. The irreversible strain ($\varepsilon_{irr}$) of our 18-multifilament wire with a final diameter of 0.83 mm is approximately 0.25%. To find out permeable strain, we need to design our wire architectures based on accurate evaluation of the mechanical properties. From the viewpoint of supplier, a hard metallic tube can be considered for a robust mechanical property. It is well known that Monel, a Cu–Ni alloy, is commonly used for enhancing the deformable property. Monel has a similar thermal expansion coefficient as Cu. During the sintering process, the adhesion layer is an important factor to suppress the debonding between the outer Monel and inner Cu.

9. Conclusion

For the past six years, our manufacturing facilities have been successfully installed to fabricate various MgB2 wires for practical applications. In particular, several filaments, various sheath materials, and a high ratio of MgB2-core-to-Cu were considered to meet the requirements of our customers. Presently, we can fabricate up to 3 km in length and the superconducting property of multifilament wire with carbon doping at 4.2 K is comparable to the low-temperature superconductor, Nb–Ti. However, we need to further consider the following points: (i) wire homogeneity, (ii) mechanical property, bending/torsion/tensile, and (iii) Nb-free architecture. Sam Dong aims to contribute high-quality wires to the industry.

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