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MgB₂ superconducting joints for persistent current operation

Abstract

High-performance superconducting joints are essential for realizing persistent-mode magnets. Herein, we propose a concept and fabrication of such superconducting joints, which yielded reliable performance in the operating temperature range of 4.2-25 K. MgB₂-MgB₂ joints in magnets are known to result in deterioration of localized electrical, thermal, and mechanical properties. To overcome these problems, the ends of the two wires are inserted into a pellet press, which is then filled with a mixture of unreacted magnesium and boron powders, followed by heat treatment. The critical current capacity and joint resistance were precisely evaluated by the standard four-probe method in open-circuit and by field-decay measurements in a closed-loop, respectively. These joints demonstrated up to 66% of the current-carrying capacity of unjoined wire at 20 K, 2 T and joint resistance of $1.4 \times 10^{-12} \Omega$ at 4.2 K in self-field.

Keywords

current, persistent, operation, joints, superconducting, mgb2

Disciplines

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MgB₂ superconducting joints for persistent current operation

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Abstract

High-performance superconducting joints are essential for realizing persistent-mode magnets. Herein, we propose a new process and fabrication of such superconducting joints, which yielded reliable performance in the operating temperature range of 4.2 K to 25 K. MgB₂-MgB₂ joints in magnets, are known to result in deterioration of localized electrical, thermal, and mechanical properties. To overcome these problems, the ends of the two wires are inserted into a pellet press, which is then filled with a mixture of unreacted magnesium and boron powders, followed by heat treatment. The critical current capacity and joint resistance were precisely evaluated by the standard four-probe method in open-circuit and by field-decay measurements in a closed-loop, respectively. These joints demonstrated up to 66% of the current-carrying capacity of unjoined wire at 20 K, 2 T and joint resistance of $< 1.4 \times 10^{-12} \Omega$ at 4.2 K in self-field.

1. Introduction

Magnetic resonance imaging (MRI) is the key technology for diagnosing critical injuries and diseases. In commercially available MRI systems, superconducting magnets based on niobium titanium (NbTi), are used for producing the high and precise magnetic fields required under persistent-mode for better image quality. It is well known that superconducting MRI magnets are currently operated in expensive liquid helium (LHe) bath at 4.2 K. The soaring LHe prices and possible shortages have increased the demand for LHe-free MRI magnets more than ever [1]. Magnesium diboride (MgB_2), which was found to be superconducting in 2001 [2], is considered as a promising candidate for LHe-free operation in MRI due to its relatively low material and fabrication costs compared to high temperature superconductors. In addition, its transition temperature of 39 K allows it to operate at higher temperatures up to 25 K [3-6]. Owing to these benefits [7, 8], there have been many recent reports on MgB_2 -conductor-based MRI magnets [9-14]. In fact, *PARAmed* has already commercialized LHe-free MRI systems, called as “open sky MRI”. These MRIs are not operated in persistent-mode, however. Therefore, a precise power supply is needed, which leads to high-cost operation. In general, MRI magnets are operated in the persistent-mode to retain magnetic field stability throughout the spherical imaging volume, to keep the long-term drift rate of the magnetic field under 0.1 ppm h^{-1} , and to maintain overall stable operation [15]. The development of a highly reliable and consistent superconducting joint technique for MgB_2 conductors is considered to be the most critical challenge for its wide application in the MRI market.

Several superconducting joint techniques for both *in-situ* and *ex-situ* MgB_2 conductors have been reported [13, 16-23]. The first successful joint addressed splicing MgB_2 and NbTi for an MgB_2 coil operated at 4.2 K under persistent mode, reported in 2005, Hitachi, even if measurement resolution were not high enough to conclude that their joint was indeed superconducting. A real MgB_2 - MgB_2 joint was reported in 2006 by ASG superconductor, based on a field decay measurement of a closed MgB_2 loop. Nardelli and co-workers [24] further developed and reported the lowest joint resistance as low as $10^{-14} \Omega$ at 20 K for *ex-situ* multifilament MgB_2 tape. Most recently in 2013, Ling et al reported their joint concept for *in-situ* monofilament MgB_2 wires, resulting in consistent critical current capacities [12], although their closed-loop coil fabricated via the “wind and react” method achieved joint resistance of $1.3 \times 10^{-10} \Omega$ at 15 K in self-field, which needs to improve by at least an order of magnitude for practical MRI application. It is thus necessary to further improve the state-of-

the-art superconducting joint, to give it low joint resistance in an order of 10^{-12} Ω for persistent-mode MgB_2 magnet operation.

In this study, therefore, of a joint technique for unreacted *in-situ* MgB_2 conductor with monofilament, test results at different temperatures and magnetic fields that include comparison of the critical current (I_c) of the joint with the wire, the closed-loop coil, and the microstructural analysis of the joints are presented in detail.

2. Experimental details

Monofilament MgB_2 wire was fabricated by a powder-in-tube (PIT) technique, using an *in situ* process. The detailed method for fabricating wires was reported elsewhere [8]. Magnesium (99 %, 325 mesh) from Sigma Aldrich and amorphous boron (98.8 %, ~ 400 nm) powder from Pavezyum were used as the starting materials with the stoichiometric composition of $\text{Mg}:\text{B} = 1:2$. The mixed powder was packed into an iron (Fe) tube with an outer diameter (O.D.) of 6.30 mm and an inner diameter (I.D.) of 4.11 mm. The composite wire was swaged and subsequently drawn to an O.D. of 1.00 mm.

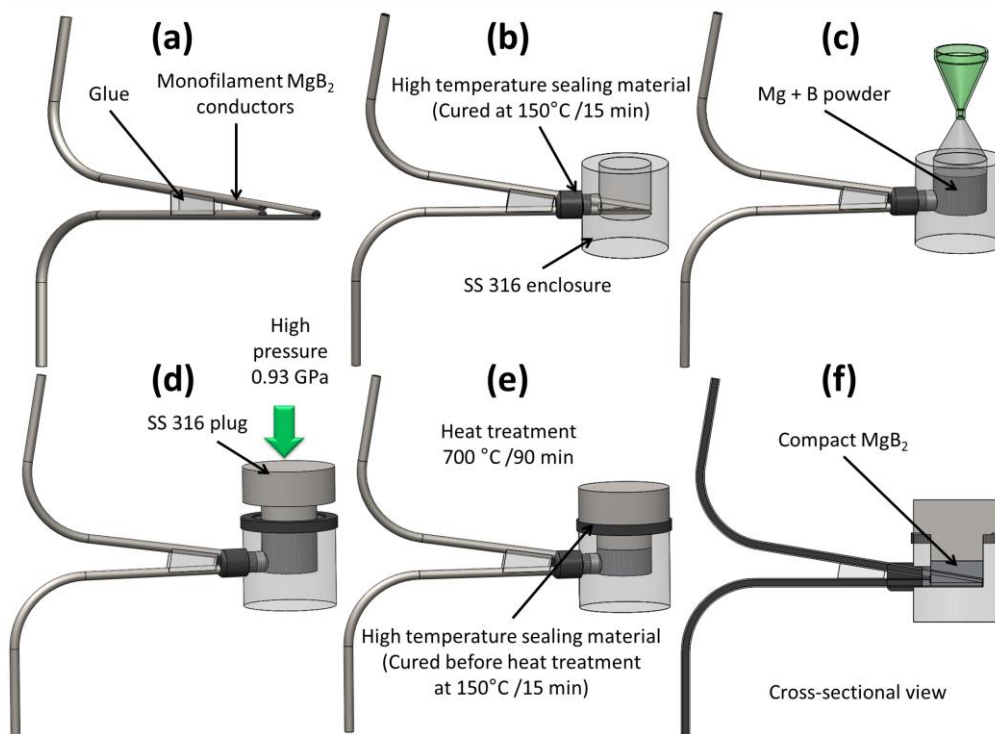


Figure 1. Joint configuration and fabrication steps for unreacted monofilament MgB_2 wires.

Figure 1 shows the joint configuration and fabrication steps for unreacted monofilament MgB_2 wires. As shown in figure 1(a), the metallic sheath material of the two wires was partially peeled off using mechanical polishing until the MgB_2 core was exposed. After

removing the metallic sheath, exposed cores of the wires were aligned and made to face each other using conventionally available super glue. The two aligned wires were then fixed in a suitable SS316 enclosure having an inner bore diameter of 6 mm, using a high temperature sealing material from Coptaltite, as shown in figure 1(b) [25]. The curing of the sealing material was carried out at 150 °C for 15 min in a drying oven. For the next step, mixed powder from the same batch (Mg + 2B) used for wire fabrication was then packed into the enclosure bore, as shown in figure 1(c). The packed powder density was estimated to be $1.96 \text{ g cm}^{-3} \pm 4 \%$. As shown in figure 1(d), sealing material was applied on the top edge of the enclosure to hermetically seal the enclosure to avoid Mg evaporation during the heat treatment process. For compacting Mg + 2B powder to make close contact with the wire core, $\sim 0.93 \text{ GPa}$ of pressure was applied, using a suitable SS316 plug. Again, the curing of the sealing material was carried out at 150 °C for 15 min in the drying oven. Finally, the joints were heat treated in high purity argon (Ar) inert atmosphere at 700 °C for 90 min as shown in figure 1(e). The joints were allowed to cool down naturally to room temperature (RT) before removal from the furnace. Figure 1(f) shows a longitudinal cross-sectional view of the as-prepared joint specimen after a final heat treatment.

The I_{cs} of the joints were measured using an American Magnetics Superconducting (AMS) magnet with DC currents up to 200 A (since 200 A was the limit of the power supply), different temperatures up to 25 K, and magnetic fields in the range of 0 – 2 T, using the standard four-probe method with the criterion of $1 \mu\text{V cm}^{-1}$.

To observe the longitudinal cross-section, the joints were cut from the appropriate location in the centre of the enclosure. Scanning electron microscopy of all the joints was conducted using a JEOL low-vacuum scanning electron microscope (SEM).

To measure the joint resistance, a small closed-loop coil was fabricated using the same conductor through the “wind and react” method and evaluated using the field-decay method. The selected specifications of the closed-loop coil are listed in table 1. Inductance was calculated using the standard formula because insulation was not used in the closed-loop coil [26]. Later, the two ends of the wire were connected using same joining technique mentioned earlier. The entire coil assembly was heat treated in high purity Ar inert atmosphere at 700 °C for 90 min.

Table 1. Selected specifications of the closed-loop coil.

I.D. (mm)	O.D. (mm)	Height (mm)	Turn s	Inductance (L, μH)
20	24	15	28	$14 \pm 7 \%$

To determine joint resistance, a field-decay measurement was carried out. The field-decay measurement setup is shown in figure 2. A Hall sensor with 0.1 G sensitivity was installed at the bottom of the closed-loop coil to measure the magnetic field generated by the coil. The cryogenic CernoxTM SD package temperature sensor was also installed using Apiezon® N grease and Kapton® tape for thermally well-coupled with the joint to monitor the temperature [27], during the closed-loop coil measurement as well as short joint measurement (not shown here). The test probe was then inserted into the bore of the variable temperature insert (VTI) of the AMS magnet. First, a magnetization test of the closed-loop coil was carried out to check for any magnetization effect on the coil at RT because Fe was present in the coil as a wire sheath. But, we did not observe any noticeable magnetization effect. External magnet field was applied up to 1.42 T and then decreased to zero with a ramp-down rate of $\sim 0.17 \text{ T s}^{-1}$. A similar magnetic field and ramp-down rate applied during the actual field-decay measurement to induce current in the closed-loop. Owing to the limitations of our equipment, the field-decay measurement was carried at 4.2 K.

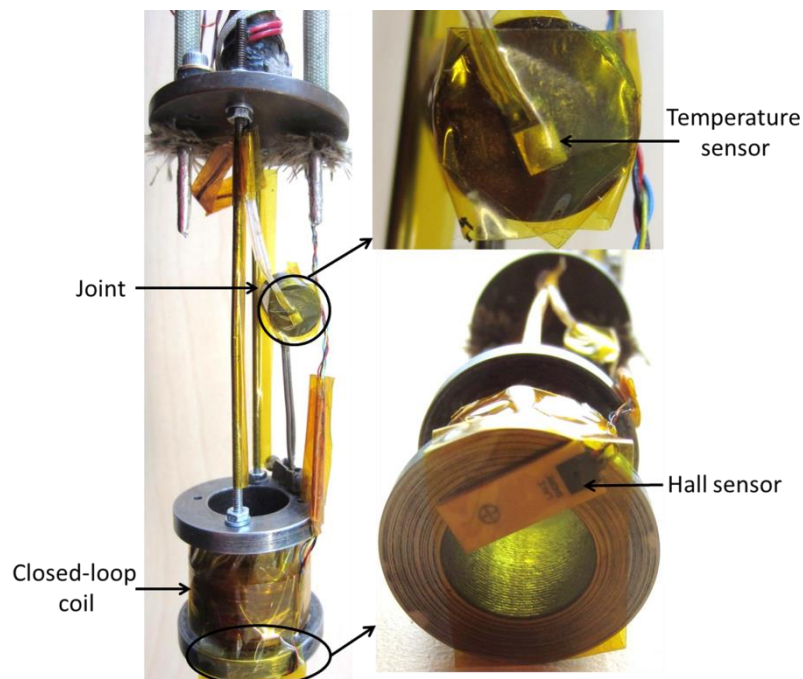


Figure 2. Field-decay measurement setup.

3. Results and discussion

To evaluate the current-carrying capacity of the jointed MgB₂ conductor, firstly, we carried out I_c measurements, as shown in figure 3. Unjoined and jointed wires (inset of figure 3) were heat treated under the same conditions for comparative study. As can be seen in the figure,

the I_c values of the unjoined wire under 2 T were measured to be 64 A and 155 A, respectively, at 25 K and 20 K. After mechanical pressing and heat treatment, the jointed wires showed electrical performance deterioration, leading to $\sim 35\%$ I_c degradation. The n -values, however, which are extracted from the power law of voltage-current characteristic, did not show any noticeable difference between the jointed and unjoined wire. Regardless of the presence or absence of the joint, a very sharp transition appeared near the criterion of $1 \mu\text{V cm}^{-1}$. Compared to low temperature superconductors such as NbTi and niobium tin (Nb_3Sn), however, the n -value of the MgB_2 conductor still shows a relatively smooth transition from the superconducting state to the resistive one. In particular, it is well-known that both the I_c and the n -value are close to the microstructures.

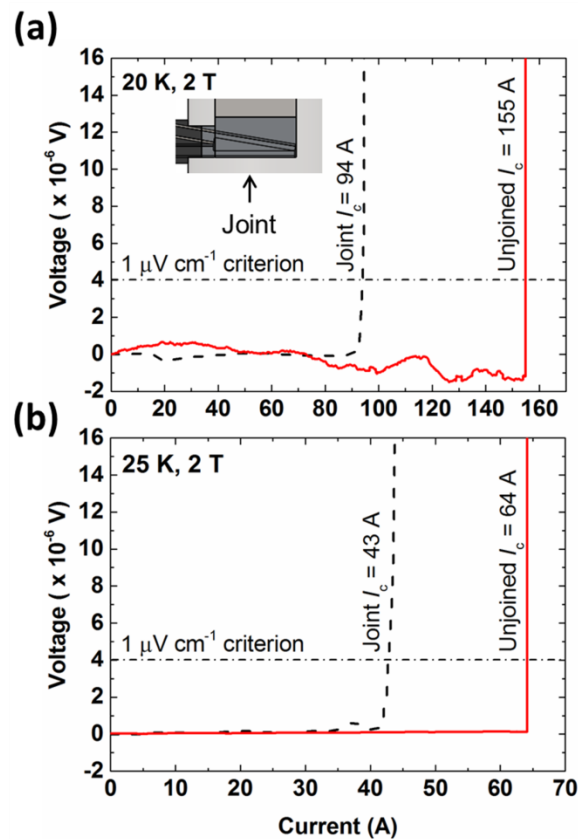


Figure 3. Current and voltage characteristics of the jointed (inset figure) and unjoined wires at (a) 20 K, 2 T, and (b) 25 K, 2 T. The distance between voltage taps was 4 cm.

To determine the reason for the current deterioration, a detailed microstructure analysis was conducted using SEM. Figure 4 shows schematic representation of a longitudinal cross-section of a joint and SEM images of the specified areas in a schematic. As has been described, core-exposed MgB_2 wires were put into Mg + 2B powders contained in a SS316 enclosure. Thus, we expect that some porous regions might still exist, even after pressing and heat treatment processes. The total length of the wire inside joint was ~ 5.63 mm and direct

MgB₂ core to core contact length was ~ 2.38 mm. The remaining length was connected through porous bulk MgB₂ between two cores. Figure 4(a) shows schematic representation of a longitudinal cross-section of a joint (not scaled). Figure 4(b) depicts a bulk region formed by the Mg + 2B powders between wire cores. We observed that porous microstructure exists. On the other hand, the interface between wire cores has a denser structure with less porosity, as can be seen in figure 4(c). Most importantly, as can be seen in figure 4(d), the interface between the bulk and the wire core shows different microstructure. This might result in microstructural defects, such as micro-/macro-cracks.

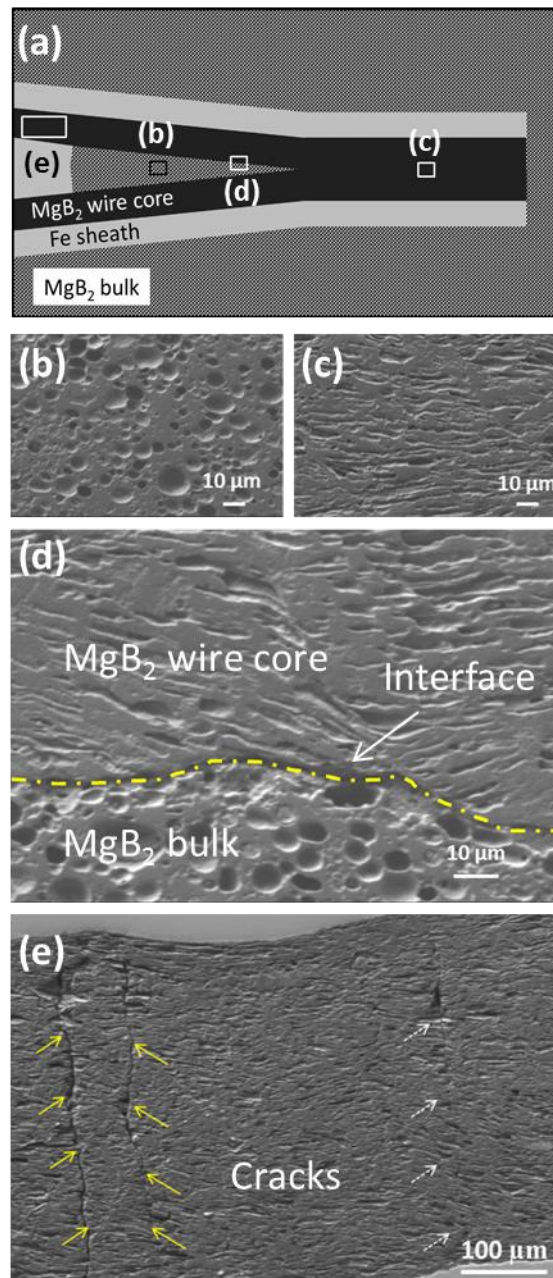


Figure 4. (a) Schematic representation of a longitudinal cross-section of a joint (not scaled), SEM images of the jointed wires area represented in a schematic: (b) bulk MgB₂ region,

interfaces between (c) MgB₂ wire cores, and (d) MgB₂ bulk and wire core, and (e) cracks in the joint (yellow and white arrows are showing macro and micro cracks, respectively).

To examine this point, we carried out further microstructure analysis. It is worth noting that pressure was vertically applied to the MgB₂ wires. As expected from this, micro-/macro-cracks were observed, as can be seen in figure 4(e). External pressure helps MgB₂ formation in the Mg + 2B bulk area, but this can induce microstructural defects, resulting in degradation of current-carrying capacity. In fact, by avoiding the cracks, the performance of the joints can be further improved. During MgB₂ formation, micro-/macro-cracks were not healed during the heat treatment process. As a result, we need to determine the optimized pressure conditions.

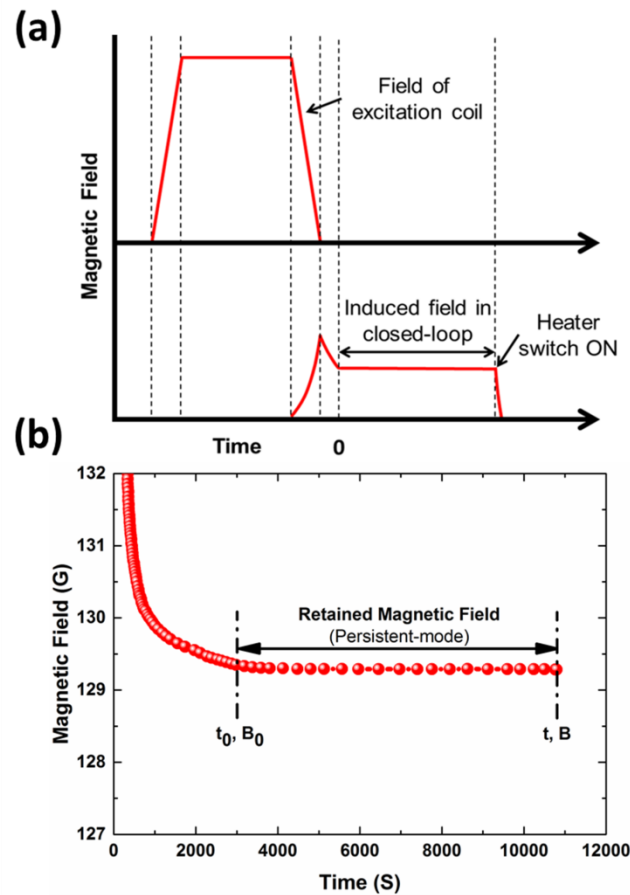


Figure 5. (a) The schematic of the operating procedure of the field-decay measurement method. (b) Time decay curve of the captured magnetic field (y-axis field is the induced field in the closed-loop coil).

As was described above, superconducting joint resistance between MgB₂ wires can be evaluated by field-decay measurements, i.e., the decay of the induced current in the superconducting closed circuit. The decay behaviour normally has two stages as shown in the

schematic of the operating procedure of the field-decay measurement method in figure 5(a). The first stage shows exponentially decreasing current with a high decay rate, due to the lower n -value and the small difference between the induced current and I_c of the closed-loop coil [26, 28]. In second stage, the decay is very slow and depends on the joint resistance. The field-decay measurement results obtained from our system are shown in figure 5(b). During entire field-decay measurement period, the joint was exposed to only self-field at 4.2 K. The magnetic field was allowed to stabilize for about 3000 s, and at this point, was considered to be the initial magnetic field B_0 at time t_0 for the resistance estimation. About 0.1 gauss decay in retained magnetic field was observed in 7764 s. The retained magnetic field after 0.1 gauss decay was 129.3 G which correspond to 30.8 A current based on FEM analysis.

The joint resistance was estimated from temporal decay of magnetic field in the time constant of $L - R$ circuit:

$$B = B_0 e^{-\left(\frac{R}{L}\right)t}$$

where B is the magnetic field at time t , B_0 is the magnetic field at time t_0 , L is the inductance of the closed-loop coil, R is the joint resistance, and t is the decay time in seconds. The joint resistance was estimated to be $< 1.4 \times 10^{-12} \Omega$ at 4.2 K. Based on our experience, the same joint technique will be applied to make an MgB₂ based persistent current switch (PCS). For practical MRI application, however, the joint technique without any electromagnetic performance deterioration will be a major challenge for long-term operation.

4. Conclusions

We fabricated and evaluated superconducting joints and determined their joint resistance with unreacted MgB₂ wire. The I_c results for the jointed wires demonstrated consistent performance, which is promising from the viewpoint of “wind and react” magnets. Despite the performance consistency in the joint performance, about 60 to 66 % of the current was retained after the joint was installed. A field-decay measurement of the closed-loop coil was also conducted to estimate the joint resistance, which is about $< 1.4 \times 10^{-12} \Omega$. Optimisation of the wire cutting, heat treatment conditions, and powder density in the joint is required, however, for further performance enhancement. The SEM observations showed very good MgB₂ core to core contact in the joint, but some cracks were also induced in this region. These should be avoided for reliable joint processing.

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