Effects of carbon concentration and filament number on advanced internal Mg infiltration-processed MgB$_2$ strands

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Abstract

In this paper we show that an advanced internal Mg infiltration method (AIMI) is effective in producing superconducting wires containing dense MgB$_2$ layers with high critical current densities. The in-field critical current densities of a series of AIMI-fabricated MgB$_2$ strands were investigated in terms of C doping levels, heat treatment (HT) time and filament numbers. The highest layer $J_c$ for our monofilamentary AIMI strands was $1.5 \times 10^5$ A cm$^{-2}$ at 10 T, 4.2 K, when the C concentration was 3 mol% and the strand was heat-treated at 675 $^\circ$C for 4 h. Transport critical currents were also measured at 4.2 K on short samples and 1 m segments of 18-filament C doped AIMI strands. The layer $J_c$s reached $4.3 \times 10^5$ A cm$^{-2}$ at 5 T and $7.1 \times 10^4$ A cm$^{-2}$ at 10 T, twice as high as those of the best powder-in-tube strands. The analysis of these results indicates that the AIMI strands, possessing both high layer $J_c$s and engineering $J_e$s after further optimization, have strong potential for commercial applications.

Some figures may appear in colour only in the online journal

1. Introduction

Extensive efforts have been made to improve the transport properties of superconductors based on MgB$_2$ since the discovery of its superconducting properties in 2001 [1]. Typical MgB$_2$ powder-in-tube (PIT) wires, consisting of a MgB$_2$ core surrounded by a chemical barrier and a hard outer sheath, can be made by either the ex situ or the in situ routes [2]. Through the ex situ technique, it is possible to develop homogeneous MgB$_2$ wires with high powder packing densities over long lengths [3]. Since the powder fill consists of pre-reacted MgB$_2$, the wires can be used either as-formed or after a sintering heat treatment (HT) at temperatures of around 800–900 $^\circ$C [4, 5]. The in situ wires need to be heat-treated in order to react the mixed Mg and B powders. Typical HT temperatures are 650–800 $^\circ$C [6, 7], although lower temperatures, even below the melting point of Mg, have been used [8]. High porosity and weak connectivity are critical issues associated with conventional in situ PIT MgB$_2$ wires [9], even though they have some of the highest $J_c$ values present in the literature [10, 11].

An interesting variant of the in situ PIT route is the ‘internal Mg diffusion’ (IMD) process or the ‘reactive liquid Mg infiltration’ (Mg-RLI) process initiated by Giunchi et al [12, 13]. The conductor is formed by Mg from the central rod diffusing into the surrounding B precursors in certain conditions and then reacting into a MgB$_2$ hollow cylinder. Whereas the PIT process produces a porous MgB$_2$ core, the IMD process provides a dense MgB$_2$ layer with excellent longitudinal and transverse connectivities [14–16]. To calculate the critical current densities of these IMD wires, it is important to take care with the definition of the areas to which the critical current, $I_c$, is normalized. Three areas are commonly used depending on the application [17]:

1. The layer critical current density, $J_c$, is defined by using the cross-sectional area of MgB$_2$ in the composite.
All the other components, including the central hole in IMD wires, are ignored. (2) The non-barrier critical current density, non-barrier $J_c$, takes into consideration the cross-sectional area of everything within the chemical barrier. It is best used for direct quality comparison between different types of MgB$_2$ wires. (3) The engineering critical current density, $J_e$, adopts the cross-sectional area of the entire strand. This is an important parameter for engineering designs.

Numerous attempts have been made to develop high performing IMD wires, including doping [18], adding extra Mg into B layers to assist the MgB$_2$ layer growth [19], optimizing HT conditions [20] and adjusting filament numbers and wire geometries [21, 22]. Adding different kinds of chemicals into wires, such as SiC [21] and liquid aromatic hydrocarbon [23], were proved to achieve layer $J_s$ and engineering $J_e$ which are much higher than in undoped IMD wires. For example, Kumakura et al [21] fabricated a series of 10 mol% SiC doped MgB$_2$ wires using the IMD process. A high layer $J_c$ of $1.1 \times 10^5$ A cm$^{-2}$ was attained at 4.2 K and 10 T for one of their monofilamentary wires heat treated at 600°C. By using C doped nano-sized amorphous B powders, our group also made IMD wires with a good layer $J_c$ of $1.0 \times 10^5$ A cm$^{-2}$ obtained at 4.2 K and 10 T [16]. Togano et al [20] prepared a series of multifilamentary IMD wires. They considered the 19-filament IMD wires as optimal candidates for high performing strands, because the fine filaments in the 19-filament IMD wires enabled a thinner MgB$_2$ layer and hence were more suitable for obtaining full MgB$_2$ phase transformation. A good layer $J_c$ of $9.9 \times 10^4$ A cm$^{-2}$ was obtained for their 19-filament IMD wire at 4.2 K and 10 T. However, given the low ‘MgB$_2$ fill factor’, (i.e. the effective MgB$_2$ cross-sectional area fraction in the whole strand), this wire only achieved an engineering $J_e$ of less than $3 \times 10^3$ A cm$^{-2}$ at 4.2 K and 10 T, even though the B in it has been nearly fully transformed into MgB$_2$.

Nevertheless, because the maximum MgB$_2$ layer thickness was always limited to 20–30 μm [20], the $J_s$ of all of these IMD wires were actually lower than state-of-the-art PIT wires. To overcome the limitations of MgB$_2$ layer thickness in previous IMD wires, our group studied the MgB$_2$ layer growth mechanism associated with the Mg diffusion route. In a recent report [24], our efforts in choosing optimized B powder types, various wires diameters, HT conditions and wire constructions finally led to high performing wires with maximum layer $J_c$ of $1.1 \times 10^5$ A cm$^{-2}$ and maximum engineering $J_e$ of $1.7 \times 10^4$ A cm$^{-2}$ at 4.2 K and 10 T. The $J_s$ for those samples were much higher than those of best-of-class PIT strands. To indicate the substantial advances in the capabilities of these ‘second generation’ MgB$_2$ strands, and to point to the large quantitative difference in the practical properties between these conductors and previous wires, we describe these ‘$J_e$-optimized’ strands as ‘advanced internal Mg infiltration’ (AIMI) wires.

Although it was widely reported that dopants, especially C and carbides, were beneficial in enhancing the layer $J_c$ and engineering $J_e$, the effect of the C doping level on these properties has not been systematically investigated in either early IMD-synthesized wires or our new AIMI wires. So it is necessary to figure out the optimal C concentration for the AIMI wires. Moreover, considering the excellent layer $J_c$ and engineering $J_e$ properties of monocoire AIMI strands, it is also essential to develop multifilamentary AIMI strands for application. In addition, most reports about diffusion- or infiltration-processed wires to date have focused on short wires (e.g. ≪1 m total length); only a few measurements have been made on long strands [22] although a 1 m long undoped Mg-RLI monofilament wire fabricated by Giunchi et al had an engineering $J_e$ of over $1.0 \times 10^4$ A cm$^{-2}$ at 4.2 K and 5 T [25]. Though the AIMI wires have higher layer $J_s$ than their PIT counterparts, many other characteristics such as engineering $J_e$, MgB$_2$ fill factor and thermal stability need to be optimized in long wires before this manufacturing process is applied commercially.

For this study a group of 3 and 4 mol% C doped multifilamentary AIMI strands were fabricated and compared with the previously reported 2 mol% C monocoire strand. The transport critical currents were measured and the results are reported in terms of layer $J_s$ and non-barrier $J_s$ after the appropriate cross-sectional areas were measured by scanning electron microscopy (SEM). The effects of C doping level on the layer $J_s$ and microstructures of the multifilamentary wires have been studied. Multifilamentary strands of 18 sub-elements were also prepared using the same C doping concentrations. Their respective values of layer $J_c$ non-barrier $J_c$ and engineering $J_e$ were then compared with a set of conventional multifilamentary PIT strands at 4.2 K. In particular, the 2 mol% C doped 18-filament AIMI wires were made into 1 m long strands for the purpose of large scale characterization of the superconducting properties of the AIMI strands.

2. Experimental details

2.1. Sample preparation

In this paper, three groups of MgB$_2$ strands, including (i) monofilamentary AIMI strands, (ii) multifilamentary AIMI strands and (iii) typical multifilamentary PIT strands, were manufactured by Hyper Tech Research Inc. (HTR). All strands used the C doped B powder produced by the plasma assisted reaction between BC1$_3$, H$_2$ and a suitable amount of CH$_4$ (Specialty Materials Inc., SMI) [26]. This powder was mostly amorphous, 10–100 nm in size, with C doping levels ranging from 2 to 4 mol%.

The wire fabrication procedure was as follows. (1) The strand geometry of the monofilamentary AIMI strands was based on our previous studies of 2 mol% C doped ‘2G-IMD’ strands [24] and 2–4 mol% C doped PIT strands [27]. The starting billet was a Mg rod positioned along the axis of a B-filled double tube of Nb and Monel; the B is doped with either 3 or 4 mol% C. Then the billet was drawn to 0.55 mm outer diameter (OD). (2) To fabricate multifilamentary AIMI strands, the initial billet, of the same geometry as that of monofilamentary AIMI strands, was drawn to an intermediate-sized monofilament. Then 18 of these monofilaments and one central Cu–Ni alloy filament were restacked into a Monel
The microstructures of the MgB$_2$ wires, including the areas of the reacted MgB$_2$ layers, were characterized by scanning electron microscopy (SEM) and compositions quantified using energy dispersive x-ray spectroscopy (EDS). SEM observation was carried out using a FEI (Philips) Sirion field-emission source SEM and a Quanta 200 SEM equipped with EDS. Transport voltage–current measurements were performed on all samples at 4.2 K in a pool of boiling liquid He in transverse magnetic fields, $B$, of up to 13.5 T. Two types of samples were studied in this work. (1) ‘Short’ straight samples 50 mm long, with a gauge length of about 5 mm. (2) ‘ITER-barrel’ samples made with 1 m long segments helically wound onto 32 mm diameter Ti–Al–V alloy holders [28]. The gauge length was 500 mm. In both cases, the transport critical current was determined under an electric field criterion of 1 $\mu$V cm$^{-1}$.

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2.2. Characterization

The ‘Filament to strand fraction’ in the non-barrier area fraction in the whole strand, i.e. ‘Filament to strand fraction’ = the cross-sectional area of everything within the Nb chemical barrier area (not including Nb)/overall wire area.

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with the EDAX EDS system. Microstructures were also characterized using an Olympus PME-3 optical microscope (OM) to obtain better contrast between MgB$_2$ and B layers.

3. Results

3.1. Monofilamentary AIMI strands

Figure 1 shows the layer $J_c$ of the 3 and 4 mol% C doped monofilamentary AIMI strands at 4.2 K in a magnetic field of 5–13.5 T. A previously reported 2 mol% C doped ‘2G-IMD’ strand heat treated at 675 °C for 1 h [24] is also included in this plot for comparison. As the C doping level increases from 2 mol%, the layer $J_c$ of the AIMI strands are firstly enhanced at 3 mol% C and then suppressed when the C concentration is 4 mol%. The 3 mol% C doped sample A3 achieves the highest layer $J_c$ of $1.5 \times 10^5$ A cm$^{-2}$ at 10 T. Also, it is noted that the layer $J_c$s of all samples, except A5, increase as the strands are heat treated longer. Take the 3 mol% C doped strands for example; after 1 h HT, the layer $J_c$ at 10 T is $1.3 \times 10^5$ A cm$^{-2}$ for A1. It increases to $1.4 \times 10^5$ A cm$^{-2}$ after 2 h HT and finally attains $1.5 \times 10^5$ A cm$^{-2}$ after 4 h HT.

Although moderately increasing the C concentration from 2 to 3 mol% is effective in improving the layer $J_c$, the transverse cross-sectional view of the strands shows that the MgB$_2$ layer growth is suppressed for 3 mol% C doped strands and thus the MgB$_2$ layer thickness becomes relatively narrower than the 2 mol% C strands. Figure 2 shows the OM and back scattered SEM images of strand A3. They are taken from the same transverse cross section of the wire. Because of better contrast, the OM picture is helpful to discern the MgB$_2$ and B-rich layers. As indicated in figure 2(a), the orange (or purple) annulus is the MgB$_2$ layer and the outside dark area is the B-rich region. The MgB$_2$ layer looks dense but its layer thickness is limited—only 8–25 µm, which is less than a quarter of the maximum layer thickness of the previously reported 2 mol% C sample [24] under the same HT condition. The suppression of the MgB$_2$ layer growth caused by the carbide doping has also been reported by other groups [21], but the underlying mechanism is still unclear. Consequently not only the area of MgB$_2$ and central Mg but also the area occupied by the non-superconducting B-rich region is taken into account as the total area to calculate the non-barrier $J_c$s of the wires. Therefore the non-barrier $J_c$s of these samples are reduced compared with the fully reacted AIMI strands.

Figure 3 shows the field dependence of the non-barrier $J_c$s for all of the monofilamentary strands. Sample A3 has the highest non-barrier $J_c$ of $2.4 \times 10^4$ A cm$^{-2}$ at 10 T. This value is lower than that of the fully reacted 2 mol% C doped AIMI strand, but is still comparable to the non-barrier $J_c$s of most PIT wires [16]. Given the high layer $J_c$s of the 3 mol% C doped AIMI strands, there might be a great improvement in non-barrier $J_c$s once the full MgB$_2$ reaction could be realized.
Figure 3. Field dependence of the non-barrier $J_c$ of the 3 and 4 mol% C doped monofilamentary AIMI strands at 4.2 K. A fully transformed 2 mol% C doped ‘2G-IMD’ strand is included [24].

3.2. Multifilamentary AIMI and PIT strands

Figure 4(a), a transverse cross-sectional SEM image of strand B1, represents the strand geometry of all multifilamentary AIMI samples. In the transverse direction, all subfilaments are about 100 µm in size and uniformly deformed. Mg and dispersed powders are found existing in the ‘prior-Mg’ holes in the centers of filaments. Between the Nb barriers and the holes are the reacted MgB$_2$ layers with a thickness of 0–30 µm. After increasing the contrast and brightness of figure 4(a) by 50%, and thus the contrast difference within the reaction layers, it is found that only in some filaments has the B powder become fully transformed to MgB$_2$. This is exemplified by the OM picture (figure 4(b)), in which some filaments have gray B-rich areas outside the yellow MgB$_2$ circulars, indicating partial transformation. The structure of these multifilamentary strands is also examined longitudinally. Tubular MgB$_2$ layers are seen attached to the Nb barriers in each filament. These MgB$_2$ filaments are uniform in diameter along the longitudinal direction. However, Nb barrier breakages can be seen at some locations. It is also noted that for every multifilamentary AIMI strand some residual Mg remains in the region of the break. EDS, employed to analyze the composition of the remaining Mg region for strand B3 and B5, detects the presence of Mg and Cu in an atomic ratio of about 4:5, implying that Cu has leaked into the filaments.

The areas of the MgB$_2$ layers are measured based on the transverse SEM images. Since both B and Mg are light atoms, the weak contrast in SEM figures between MgB$_2$ and other borides or B makes measurement difficult even when the contrast and brightness of the pictures are adjusted. So OM images, with better phase contrast, are used to help distinguish the MgB$_2$ region. Table 2 lists the MgB$_2$ areas as well as the MgB$_2$ fill factors of all multifilamentary AIMI samples obtained using this method. It is important to note that the MgB$_2$ fill factors of the present AIMI strands are only 9.2–14.6%, significantly lower than those of PIT strands.

The field dependences of the layer $J_c$s at 4.2 K for all the multifilamentary AIMI strands are shown in figure 5. Those of the two best performing PIT strands, P3 and P4, are also included for comparison. The best performing strand in magnetic fields below 10 T is the 2 mol% C doped strand B1. At 5 T its layer $J_c$ reaches $4.3 \times 10^5$ A cm$^{-2}$; at 10 T it is $7.1 \times 10^5$ A cm$^{-2}$ which is twice as high as that achieved by the best PIT strand (i.e. the 3 mol% C doped strand P3). The 4 mol% C doped strand B5 exhibits the highest layer $J_c$s at high fields above 10 T, although its low-field $J_c$s are lower than those of other strands. Its layer $J_c$ attains $2.8 \times 10^4$ A cm$^{-2}$ at 13 T. Compared with B1 and B5, the 3 mol% C doped strand B3 shows good layer $J_c$s in all applied fields. But unlike its monofilamentary counterpart A1, a slightly heavier doping in multifilamentary AIMI strands does not result in an appreciable layer $J_c$ enhancement as compared with 2 mol% C doped AIMI wires. Secondly, it is noticeable that all the strands heat treated for 2 h (i.e. B2, B4 and B6) show deteriorated layer $J_c$s compared to their counterparts heat treated for 1 h, i.e. B1, B3 and B5, respectively. For

Figure 4. Transverse cross-sectional (a) SEM and (b) OM images of the 2 mol% C doped multifilamentary AIMI strand B1.
Table 2. MgB$_2$ areas and MgB$_2$ fill factors of multifilamentary AIMI strands.

| Sample name | MgB$_2$ area ($\mu$m$^2$) | Error ($\mu$m$^2$) | MgB$_2$ fill factor (%) | Error (%) |
|-------------|---------------------------|-------------------|------------------------|----------|
| B1          | 71 600                    | 2700/2200         | 13.2                   | 0.5/0.4  |
| B2          | 77 800                    | 3900/1200         | 14.4                   | 0.7/0.2  |
| B3          | 70 800                    | 700/1500          | 13.1                   | 0.1/0.3  |
| B4          | 70 500                    | 1300/3700         | 13.0                   | 0.2/0.7  |
| B5          | 52 900                    | 3100/300          | 9.8                    | 0.6/0.1  |
| B6          | 65 400                    | 2500/2500         | 12.0                   | 0.5/0.5  |

Figure 5. Field dependence of the layer $J_c$s for multifilamentary AIMI strands at 4.2 K; data from P3 and P4 are included for comparison. Here B1 and B2 are 1 m long ‘ITER-barrel’ type strands.

Figure 6. Field dependence of the non-barrier $J_c$s for multifilamentary AIMI strands at 4.2 K; data from P3 and P4 are included for comparison. Here B1 and B2 are 1 m long ‘ITER-barrel’ type strands.

Figure 7. Field dependence of the engineering $J_c$s for multifilamentary AIMI strands at 4.2 K; data from P3 and P4 are included for comparison. Here B1 and B2 are 1 m long ‘ITER-barrel’ type strands.

4. Discussion

The C or carbide doping concentration has been reported to have a great impact on the critical current densities of the
conventional MgB$_2$ PIT wires [29, 30]. This effect still works for monofilamentary AIMI strands. Monocore AIMI strands achieve maximum layer $J_c$s when the C doping concentration is in the vicinity of 3 mol%. The layer $J_c$ is suppressed when the C doping level deviates from this optimal value. This is in agreement with our group’s previous results that the PIT strands behave best when 3 mol% C is mixed into the wires [16, 27]. Proper carbon doping is proved to enhance the upper critical field $B_{c2}$, decrease the anisotropy ratio $\gamma$ and provide extra flux pinning centers [27, 31, 32], all of which help to obtain higher layer $J_c$s. Nevertheless, if too much C is doped it might deteriorate the connectivity and thus reduce the transport layer $J_c$s [33]. As a consequence, relatively low layer $J_c$s are obtained for 4 mol% C samples when the C doping level is above 3 mol%. Compared with monofilamentary AIMI strands, the multifilamentary AIMI strands possess finer subfilaments and thus more easily suffer from degradations during the process of wire fabrication. The Nb barrier breakage observed in B1 and Cu leakage detected in samples B3 and B5 would cause a certain level of deterioration in layer $J_c$s, so the layer $J_c$s of all multifilamentary AIMI strands with any C doping level are lower than those of their monofilamentary counterparts. For the same reason, these extrinsic defects such as Cu contamination have such negative effects on multifilamentary wires that no obvious layer $J_c$ improvement is observed even when the C doping level is optimal (i.e. 3 mol%).

The effect of HT time on the layer $J_c$ is quite opposite for monofilamentary and multifilamentary AIMI strands. A longer HT time, such as 4 h, is desired for monofilamentary AIMI strands, while a shorter period of HT is preferred in multifilamentary strands. The average B precursor layer thickness in multifilamentary strands is less than 20 $\mu$m, so the whole layer quickly transforms into MgB$_2$ within 1 h during the HT. Extending the HT time is not helpful for MgB$_2$ layer growth but could only cause grain coarsening, oxidation or Cu contamination, which are harmful for the strands [34]. In contrast, the average layer thickness of B precursor in monofilamentary AIMI strands is thicker than 50 $\mu$m. So Mg is not able to diffuse deeply into the B layer within a short time. According to the MgB$_2$ layer growth mechanism in AIMI wires discussed in an early paper [24], a partially reacted MgB$_2$ layer is formed at the very beginning of HT, so moderate $J_c$s are obtained for A1. As HT continues, the MgB$_2$ layer becomes thicker, better transformed and well connected. Consequently, A2 and A3 achieve improved $J_c$s and $J_e$s.

The above difference also implies that multifilamentary AIMI MgB$_2$ wires are more advantageous for obtaining high layer $J_c$s and engineering $J_e$s than monocore AIMI wires. They could be fully reacted within a short time because the B precursor layers become narrower and thus the Mg diffusion distance is shortened. As the HT is completed more quickly, grain coarsening can be prevented and other detrimental effects like oxidation limited to a low level. Also, since the maximum MgB$_2$ layer thickness is always limited to a very small value (for example, 25 $\mu$m for 3 mol% C doped monofilamentary AIMI strands), it is desirable to reduce the thickness of the B precursor layer below 25 $\mu$m, which can be realized in multifilamentary strands.

5. Summary

The in-field critical current densities of a series of advanced internal Mg infiltration-processed MgB$_2$ strands have been investigated in terms of the C doping level, HT time and filament number. The best monofilamentary AIMI strand A3,
with 3 mol% C and under 4 h HT at 675 °C, achieves high layer $J_c$ of $1.5 \times 10^5$ A cm$^{-2}$ and non-barrier $J_c$ of $2.4 \times 10^4$ A cm$^{-2}$ at 10 T and 4.2 K. In multifilamentary AIMI strands, we have not retained the full layer $J_c$, increase due to some defect structure. Even so, their layer $J_c$s, non-barrier $J_c$s and engineering $J_c$s are still better than, or at least comparable to, those of the best PIT strands. The 1 m long version of the multifilamentary strand shows that this kind of advanced MgB$_2$ strand is uniform and thermally stable, capable of being utilized in industry.

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