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High performance, advanced-internal-magnesium-infiltration (AIMI) MgB₂ wires processed using a vapor-solid reaction route

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Abstract

MgB₂ superconducting wires made using a Mg infiltration method have reached a higher performance than either *in-situ* or *ex-situ* mixed powder based routes. Indeed, very high layer J_c coupled with whole-strand J_e (critical current per total strand cross section) exceeding 10^4 A cm⁻² at 4.2 K, 10 T have been found for monocoil MgB₂ wires. However, previous multicore infiltration route wires have not reached their potential for J_e due to partially reacted and non-uniform MgB₂ layers. This study shows that 18-core MgB₂ AIMI wires processed using a low temperature route can attain higher and more uniform J_e values due to a more uniform MgB₂ reaction layer. The formation of fully reacted, uniform MgB₂ layers is attributed to the switch from a liquid-solid to a vapor-solid reaction route.

Keywords

MgB₂ wire; advanced internal magnesium infiltration (AIMI); vapor-solid reaction route; engineering critical current density

1. Introduction

MgB₂ superconducting strands are promising for magnet applications due to the high transition temperature (39 K) [1], moderate coherence length [2, 3], and low anisotropy [3–6] of the MgB₂. In order to develop practical MgB₂ superconducting wires, most efforts have focused on powder-in-tube routes where either an unreacted Mg + B mixture was placed in a tube (*in-situ*) [7–9] or already formed MgB₂ powders were filled into a tube (*ex-situ*) [9, 10]. Such wires have had a relatively good performance, but they retained significant porosity. A different approach was developed where liquid Mg infiltrated into a B powder layer, this reactive-liquid-infiltration (RLI) technique was shown to give fully dense MgB₂, but large grain sizes associated with the technique led to low layer J_c [11]. Such an infiltration approach was further developed under the name of internal-Mg-diffusion (IMD) which used smaller B powders (micron-sized) and SiC addition [12–14] and the

advanced-internal-magnesium-infiltration (AIMI) which optimized B powder type (nano-sized, amorphous, and C pre-doped B powder) and filament diameter [15, 16]. All of these infiltration techniques start by surrounding a Mg rod with B powder inside of a metallic tube, which acts as a chemical or physical barrier or both. This forms a filament or subelement which can be restacked to form a multifilamentary composite. After final wire drawing, the conductor must be given a reaction heat treatment (HT) during which time the Mg reactively infiltrates into the B powder to form highly dense MgB_2 layer. In the AIMI-processed wires, Mg was able to percolate more deeply into B layer by using nano-sized, ‘fluffy’, and low-density B powder. MgB_2 layers with typical thickness of up to $50\text{ }\mu\text{m}$ were achieved with a HT of $675\text{ }^\circ\text{C}/1\text{ h}$ in monocoil AIMI wire and as a result, high overall J_{cs} were obtained. Relevant to increasing the level of conversion, or reaction depth, Ma *et al* [17] and Grivel *et al* [18] have reported that the infiltration of the Mg into the B powder layer could be increased by minor Cu addition. Based on this investigation, a Cu coated Mg rod was used to fabricate an IMD-processed monocoil MgB_2 wire HT at $600\text{ }^\circ\text{C}$ for 20 h, achieving high J_{c} of 10^4 A cm^{-2} at 4.2 K, 11 T which is comparable to the highest J_{c} obtained by AIMI monofilament [19, 20], an impressive result.

However, in order to obtain high J_{cs} and J_{cs} for multicore AIMI wires, a complete MgB_2 reaction is required for all subelements. Li *et al* performed a detailed investigation of the reactive mechanism of Mg into the B layer, developing a quantitative model [21]. However, this model was made to describe reactions above the melting point of Mg (i.e. during a liquid-solid reaction route). His model had two stages. In Stage I, molten Mg percolates out from the core, while it locally reacts with the B to form an MgB_2 layer which grows some tens of microns thick while it continually densifies. Once the layer becomes 100% dense, we transition to Stage II, during which the Mg would have to diffuse through the now 100% dense MgB_2 layer in order to reach the porous B powder region outside of the reacted MgB_2 shell. The diffusion of Mg through the reacted MgB_2 layer is quite slow ($D = 10^{-17}\text{ m}^2\text{ s}^{-1}$ at $675\text{ }^\circ\text{C}$ [21, 22]), effectively stopping the MgB_2 reaction at the end of Stage I. Further heat treatment time is not effective in increasing the MgB_2 fill factor; the next $1\text{ }\mu\text{m}$ of reaction layer would take years [21]. In the AIMI design, the thickness of the precursor B layer was therefore designed to be at or below the limiting reaction depth of the Stage I process, in order to maximize conductor utilization. This in principle allows a $675\text{ }^\circ\text{C}/1\text{ h}$ heat treatment to completely transform the precursor B layers into MgB_2 leading to high conductor performance. However, in practice, even reducing the precursor B layer thickness below $30\text{ }\mu\text{m}$ still allowed for some partially reacted MgB_2 subelements in the multicore AIMI wire, effectively suppressing the MgB_2 fill factor and thereby limiting the J_{cs} and J_{cs} of the multicore AIMI wire [16]. Given the formation of the dense MgB_2 layer during Stage I, further HT will not improve the reaction completeness. In addition, extending reaction time might further decrease the layer J_{cs} via grain coarsening or oxidation [16]. It would be beneficial for engineering applications if we could develop a simple route to induce full MgB_2 conversion in all subelements while simultaneously retaining a high layer J_{cs} . In the present work, we show that a vapor-solid reaction route, where Mg vapor reacts with solid B powders, is effective for achieving this goal. In this vapor-solid reaction route the wires were reacted at a temperature below Mg’s melting point ($650\text{ }^\circ\text{C}$), for example $625\text{ }^\circ\text{C}$ for 8–14 h, and the high vapor pressure of the solid Mg allows for a viable reaction route to form MgB_2 ,

with the Mg vapor penetrating deeply into the B powder compact while it is locally reacting to form MgB_2 .

In order to demonstrate this route, an 18 filament, multicore AIMI wire was fabricated, and then liquid-solid and vapor-solid reaction routes were compared. The wire consisted of subelements in which Mg rods were surrounded with 2 mol% C doped B powder (typical B powder size 10–100 nm [16]) provided by Specialty Materials Inc. (SMI) with an additional 2 wt% Dy_2O_3 additive. The Mg rod and B powder, surrounded by a Nb chemical barrier, constituted the subelement. Eighteen of these subelements were stacked into a Monel outer sheath, and the composite was wire drawn to 0.84-mm outer diameter. We included two additions, C and Dy_2O_3 as they are the most effective dopants known to increase the J_c of MgB_2 at 4.2 ~ 20 K [7, 23–29]. For AIMI-processed wires, it was found that the optimized C and Dy_2O_3 concentrations are 2 mol% and 2 wt% respectively, based on B powder; therefore, we applied same C and Dy_2O_3 concentrations on the 18-core AIMI wires in this work.

2. Experimental details

The B powders were pre-doped with 2 mol% C and were 10–100 nm in size, provided by Specialty Materials Inc. (SMI). The synthesis process of the B powder is described in [30]. Dy_2O_3 powders (99.9%, 100 nm) were added to the B at a 2 wt% level (based on B powder). The 18-core AIMI wires were fabricated by following process [16]. (1) The initial billet consisted of a Mg rod of 3.0 mm diameter inside a larger Nb tube with a substantial layer of B powder between them (B thickness specified an atomic ratio of Mg:B of 1.2:2.0). (2) A moncore composite was obtained by drawing the initial billet into an intermediate size. (3) An assembly was made by restacking 18 of the moncore composites and one Nb central filament into a Ni-Cu alloy (Monel) tube, after which it was finally cold-worked (wire drawn) into a multicore wire with an outer diameter of 0.84 mm. Cold-working was performed using typical wire manufacturing equipment, primarily conical die drawing. Some samples were processed with a vapor-solid reaction route (625 °C/8 ~ 14 h) and the other samples were processed with a liquid-solid reaction route (675 °C/1 ~ 4 h). The fabricated wire was cut into a number of segments; both short segments approximately 5-cm long (short samples), longer segments about 1 meter long, the latter helically wound around cylindrical barrels (barrel samples). One set was reacted at 625 °C for 8, 10, and 14 h, to explore the vapor-solid reaction. The second set was reacted at 675 °C for 1, 2, and 4 h, to give the baseline liquid-solid reaction. The heat treatment was conducted in a tube furnace with flowing argon gas. The wires were ramped into a target temperature in about 80 min, kept at the target temperature for certain times, and were furnace-cooled into room temperature. Table 1 shows the sample type, sample length and HT conditions.

Transport I_c s of the wires were tested in a pool of boiling liquid helium (4.2 K) as a function of magnetic field, B , applied perpendicular to the wire axis (along the helical axis of the winding) up to 13 T using a four-point measurement technique. The transport I_c s were determined from the V – I curves of the short samples and barrel samples using the gauge lengths of 5 mm and 50 cm, respectively and an electric field criterion of $E_c = 1.0 \mu\text{V cm}^{-1}$. It should be noted that three kinds of J_c can be defined: (i) a layer J_c which is equal to I_c

normalized with the area of MgB_2 layer; (ii) a non-barrier J_c defined as I_c divided with the area inside chemical barrier; and (iii) a J_e defined as I_c normalized with whole strand area.

3. Results and discussion

3.1. Microstructures and MgB_2 layer formation

Figure 1(a) shows a back-scattered SEM (BSE) image of the whole multicore wire showing all eighteen filaments after a HT of 675 °C/1 h, representative of the liquid-solid reaction route. Figures 1(b)–(c) show higher magnification BSE images of the subelements in cross section after such a higher temperature (675 °C for 1 h (b) and 2 h (c)) liquid-solid reaction. It can be clearly seen that unreacted, B-rich regions still exist. Even though the wire architecture was designed to keep the B layer thickness (about 20 μm average) below the previously measured reaction depth limit of about 50 μm , some unreacted regions persist. We also observed MgB_2 layers with varying thicknesses along longitudinal wire axis as shown in figure 1(d). We can observe that the final reaction MgB_2 layer thickness can vary both around the subelement, as well as along it for this liquid-solid reaction route.

One possible explanation is as follows. Figure 2 schematically shows the progression of a liquid-solid reaction. At shorter times in Stage I, molten Mg both percolates into and locally reacts with the B powder compact. However, as Stage I continues, the molten Mg shrinks into several distinct segments (Mg ‘bubbles’ form) due to the consumption of Mg and surface tension. Subsequently, these molten Mg segments can only infiltrate into the local B regions they are immediately adjacent to. This leads, effectively, to a non-uniform distribution of molten Mg during the latter times of Stage I, and finally induces the variable thickness of the MgB_2 layer which we observe.

In comparison, figures 3(a)–(b) show that full MgB_2 reactions were accomplished in all subelements of wires reacted with a vapor-solid route (625 °C/8 h). That this is truly a vapor-solid and not a solid-solid reaction can be clarified by noting that the atomic diffusion coefficient (D_p) of Mg into individual C-doped B powders at 625 °C is estimated to be $7.0 \times 10^{-18} \text{ m}^2 \text{ s}^{-1}$ using the parameters given in [22], leading to a solid-solid reaction depth of 0.63 μm for a HT of 625 °C/8 h (using $L_p = \sqrt{2D_p t}$). However, figure 3(b) shows that the thicknesses of MgB_2 layers in wires HT at 625 °C/8 h reach up to 30 μm , clearly indicating a Mg vapor-B powder solid reaction mechanism. More importantly, these results show the vapor-solid route can induce full transformation of the precursor B layers into MgB_2 layers and therefore generate a high MgB_2 fill factor for multicore AIMI wires. According to the model of gas diffusion in porous media, the diffusivity D_k of Mg vapor into a porous B powder compact at 625 °C (898 K) is estimated to be $2.95 \times 10^{-8} \text{ m}^2 \text{ s}^{-1}$, calculated from the Knudsen Diffusion expression [31]

$$D_k = \frac{\varepsilon d}{\tau} \left(\frac{8RT}{\pi M_{\text{Mg}}} \right)^{\frac{1}{2}} \quad (1)$$

in which the porosity ε of B powder layer is about 30 %, the tortuosity τ is given by equation (2) [32, 33]

$$\tau = 1 - 1.66 \ln \epsilon \quad (2)$$

The average pore diameter d is on the order of 10^{-9} m, molar mass of Mg, M_{Mg} is 24.31 g mol^{-1} , and the gas constant R is $8.314 \text{ J mol}^{-1} \text{ K}^{-1}$. Therefore, the diffusion distance of Mg vapor into porous B powder layer can reach larger than $200 \mu\text{m}$ within 1 s. Thus, for the vapor-solid reaction route, the MgB_2 layer formation seems to be mainly limited by the sublimation rate of Mg vapor. The space between B particles was filled with air before reaction. Therefore, the pressure in the pre-reacted AIMI wire should be close to 1 atm. The rate of sublimation W_{Mg} for Mg is on the order of $10^{-7} \text{ g cm}^{-2} \text{ s}^{-1}$ at 625°C (898 K) and $0.1 \sim 1 \text{ atm}$ [34]. The amount of volatilized Mg per second can be calculated by equation (3)

$$n_r = \frac{2\pi r L W_{\text{Mg}}}{M_{\text{Mg}}} \quad (3)$$

where r is the average radius of Mg rod for the subelements in the 18-core AIMI wire ($4.4 \times 10^{-3} \text{ cm}$), L is the length of the AIMI wire. Assume the length L is 100 cm; the amount of volatilized Mg per second in each subelement of the AIMI wires is about $1.13 \times 10^{-8} \text{ mol s}^{-1}$. On the other hand, the average amount (mole) of B particle for each subelement is expressed as equation (4)

$$n(\text{B}) = \frac{AL(1 - \epsilon)\rho_{\text{B}}}{M_{\text{B}}} \quad (4)$$

in which A is the transverse cross-sectional area of porous B powder layer of each subelement ($4.10 \times 10^{-6} \text{ cm}^2$), ρ_{B} is the density of B powder (2.37 g cm^{-3}), and M_{B} is the molar mass of B (10.81 g mol^{-1}), so the $n(\text{B})$ of each subelement is about $6.3 \times 10^{-4} \text{ mol}$. Then we can see that an amount of Mg of $n(\text{Mg}) = n(\text{B})/2 = 3.15 \times 10^{-4} \text{ mol}$ is required to consume all of the B powders of each subelement. Therefore, a time of $n(\text{Mg})/n_r = 7.7 \text{ h}$ is required to fully react the MgB_2 . In other words, based on high Mg vapor diffusivity into porous B layer and relatively lower MgB_2 formation rate, Mg vapor is able to fully percolate into B layer before the complete formation of the dense MgB_2 layer. The above diffusivity and sublimation rates of Mg vapor are estimates, but give a full reaction time quite close to the observed 8 h required to fully react our samples. As we can see in the optical images of the longitudinal cross-sections for wires reacted at $625^\circ\text{C}/8 \text{ h}$ (figures 3(c)–(d)), highly uniform MgB_2 layers were obtained using this vapor-solid reaction route. Since the Mg vapor, unlike the shrinking molten Mg of the liquid-solid route, was able to percolate deeply into the B powder layer, it was possible to form uniform and homogeneous MgB_2 phase throughout the subelements and the whole of the AIMI wire. We can expect that such uniform MgB_2 layers will lead to improved transport performance in the wire.

3.2. Transport J_{cs} and n -values

The field dependence of transport measurements of I_{cs} and J_{cs} for 1-m long helical wires is given in figure 4 for both liquid-solid (675°C) and vapor-solid (625°C) reactions. The vapor-solid reacted samples achieved higher I_{cs} (corresponding to higher J_{cs}) than the liquid-solid reacted samples at all applied fields. The 4.2 K, 7 T I_{cs} of the wires heat-treated

at 625 °C for 8, 14 h reached 181 A and 180 A, respectively, which are higher than the I_c of 157 A for the wire heat-treated at 675 °C/1 h. The J_c s of these two vapor-solid reacted wires obtained 1.02×10^4 A cm⁻² at 10 T and 4.2 K, which is a record for multifilamentary MgB₂ wires of any kind [13, 14, 16, 35, 36]. The I_c/J_c enhancements induced by the vapor-solid state reaction route are attributed to the fact that high layer J_c s and large MgB₂ fill factors were simultaneously achieved by using vapor-solid reaction route. Although the highest layer J_c s were achieved by the wire reacted by 675 °C/1 h as can be seen from the layer J_c comparison in figure 3(c), the presence of unreacted B-rich regions in many subelements of this wire decreased its MgB₂ fill factor and thereby suppressed its I_{cs} or J_{cs} . The I_{cs} and J_{cs} of the wire reacted at 675 °C for 4 h became even lower which is consistent with the results reported in [15, 16]. In other words, the extended HT time is harmful to the transport properties of the multicore AIMI wires processed with 675 °C. On the contrary, extended reaction time has no effect on the J_{cs} and J_e s of the wires reacted at 625 °C, as shown in figure 4(d). The multicore AIMI wires synthesized with vapor-solid reaction exhibit high J_c and J_e repeatability. An analysis of the wires HT at 625 °C shows the 5-cm short wire reacted at 625 °C for 14 h achieved the highest 4.2 K, 10 T J_e of 1.1×10^4 A cm⁻² and the lowest J_e is 9.9×10^3 A cm⁻², obtained by the 1-m long wire heat-treated at 625 °C for 10 h. The mean value of the 4.2 K, 10 T J_e s of all the wires reacted at 625 °C, $\langle J_e \rangle$, is 1.04×10^4 A cm⁻². The standard deviation (371 A cm⁻²) in the J_e s was 3.6% of the mean J_e value. Therefore, the multicore AIMI wires synthesized by the vapor-solid reaction route (HT at 625 °C) have a very narrow J_e variance, apparently from the formation of uniform subelements in the wires. This result confirms that the vapor-solid reaction route generate the subelements with high uniformity in the AIMI wires.

The n index is another measure of the filament (subelement) uniformity of superconducting wires. The narrow resistive transition of the $V-I$ curve is associated with high n -value extracted from the empirical equation: $V/V_c = (I/I_c)^n$. As the critical currents of the filaments are close to their intrinsic limits, the filaments yield high n -values and steep $n-B$ plots of superconducting wire [37]. When the critical currents were limited by extrinsic factors, notably non-uniform filaments, the extrinsic factors can drop n -values at relatively lower magnetic fields, yielding a flat $n-B$ plot [37]. Figure 5 shows the n -value versus B at 4.2 K for all wires. It can be seen that the vapor-solid reaction route gives much higher n -values and steeper $n-B$ plots than the liquid-solid reaction route. The n -values of the wires reacted by 625 °C/8 and 14 h are larger than 30 at 5 T; however, the n -values of the wires reacted at 675 °C are lower than 18 at all applied fields. In general, the n -value reflects the width (σ) of the critical current (I_c) distribution for a superconducting wire, where $n \propto 1/\sigma$ [38]. For the 1-meter helical AIMI wires in this study, the n -values of the wires reacted at 625 °C are 1.56 times those of the wires reacted at 675 °C at 4.2 K and 8 T; thus, the vapor-solid reaction route give a 56% narrower 8 T I_c distribution than the liquid-solid route.

4. Conclusions

In summary, we show that highly dense and uniform MgB₂ layers and the highest J_c of 18-core AIMI MgB₂ wires ever observed have been achieved using a vapor-solid reaction route. Compared to the liquid-solid state reaction route, the vapor-solid reaction route generated a higher diffusivity of Mg into porous B particle layer and reduced somewhat (beneficially)

the rate of local MgB_2 layer formation, allowing the Mg vapor to fully percolate into B powders, resulting in full MgB_2 layer formation. At the same time, the layer J_c s of the vapor-solid state synthesized AIMI wires were maintained at comparable values to those of the liquid-solid state synthesized wires. Therefore, the vapor-solid state reaction route led to higher J_c and J_e performance than the liquid-solid state reaction route and to very substantially increased n values and J_e repeatability (the standard deviation is only 3.6% of the mean value for J_e at 10 T and 4.2 K). Considering the enhanced percolation of the Mg vapor into the porous B powder layers, we can now return to a larger precursor B powder layer and thickness (or smaller Mg rod) re-optimizing the strand and subelement geometry to obtain an even larger MgB_2 area fraction. We expect correspondingly higher J_c s may be achieved using such re-optimized geometries in conjunction with the vapor-solid state route in AIMI wires.

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Figure 1.

Back-scattered SEM images for the 18-core AIMI wires: (a) a transverse cross-section of the strand showing the 18 subelements after HT at 675 °C for 1 h, (b) the MgB_2 layers and B-rich regions of the wire HT at 675 °C for 1 h, (c) the MgB_2 layers and B-rich regions of the wire HT at 675 °C for 2 h, and (d) a longitudinal cross section for one subelement of the wire HT at 675 °C for 1 h. A variable thickness MgB_2 layer is observed. The white specs seen in (b)–(d) are Dy-rich inclusions (verified by EDS, not presented here).

Figure 2.

Schematic diagram of molten Mg infiltration into B compact during the HT condition of 675 °C/1 h; Before HT: Mg rod (blue) is surrounded (adjacent to) a B powder (orange) layer; During the Stage I of MgB₂ layer formation: (i) molten Mg rapidly reacted with B to form a thin and dense MgB₂ layer (yellow), (ii) molten Mg shrinks into several segments due to Mg consumption and surface tension, (iii) non-uniform distribution of Mg source induces the MgB₂ layer with variable thicknesses.

Figure 3.

Back-scattered SEM images: (a) a transverse cross section of the wire showing the 18 subelements after HT at 625 °C for 8 h, (b) full-reacted MgB_2 layers of the wire after HT at 625 °C for 8 h. Optic images: (c) a longitudinal cross section of the wire after HT at 625 °C for 8 h, and (d) a longitudinal cross section for one subelement of the wire HT at 625 °C for 8 h. A uniform MgB_2 layer is observed.

Figure 4.

(a) Critical current I_c versus magnetic field B at 4.2 K for 1-m long wires, (b) J_c versus magnetic field B at 4.2 K for 1-m long wires, (c) layer J_c and non-barrier J_c versus magnetic field B at 4.2 K for 1-m long wires, and (d) the field dependence of layer J_c , non-barrier J_c , and J_e at 4.2 K for a number of wires processed with vapor-solid reaction route, showing high repeatability.

Figure 5.
Field dependence of n -values at 4.2 K.

Table 1.

Sample type, sample length and HT conditions of all multicore wires.

Samples	Sample Type	Sample length (cm)	HT, ($^{\circ}\text{C h}^{-1}$)
5 cm–625 $^{\circ}\text{C}$ –8 h	Short	5	625/8
1 m–625 $^{\circ}\text{C}$ –8 h	Barrel	100	625/8
5 cm–625 $^{\circ}\text{C}$ –10 h	Short	5	625/10
1 m–625 $^{\circ}\text{C}$ –10 h	Barrel	100	625/10
5 cm–625 $^{\circ}\text{C}$ –14 h	Short	5	625/14
1 m–625 $^{\circ}\text{C}$ –14 h	Barrel	100	625/14
1 m–675 $^{\circ}\text{C}$ –1 h	Barrel	100	675/1
5 cm–675 $^{\circ}\text{C}$ –2 h	Short	5	675/2
1 m–675 $^{\circ}\text{C}$ –4 h	Barrel	100	675/4