Regular article

Superior transport J_c obtained in *in-situ* MgB$_2$ wires by tailoring the starting materials and using a combined cold high pressure densification and hot isostatic pressure treatment

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ABSTRACT

The best J_c performance (8×10^4 A·cm^{-2} at 10 T, 4.2 K) has been obtained in our work among all the *in-situ* powder-in-tube (PIT) MgB$_2$ wires reported so far by tailoring their boron and magnesium precursors, as well as employing appropriate high pressure during the manufacturing process. Moreover, the second stage densification using the special HIP has been applied to commercial long-length wire. Our work suggests that the economical starting precursors and the combination of cold and hot densification techniques could represent a promising alternative for industrial and economical production of practical MgB$_2$ wires with excellent J_c.

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1. Introduction

The superconductivity at 39 K that was discovered in MgB$_2$ with its simple binary chemical composition has attracted much interest in its fabrication techniques and practical applications due to its low density, good compositional tolerance, and easy fabrication methods [1]. Its electrical applications are largely determined by its current carrying capability, *i.e.* the critical current density (J_c) of the superconductor at the service temperature. Therefore, many researchers have been trying to improve the J_c of MgB$_2$ since its discovery in 2001. Although considerable progress has been made towards improving the fabrication and performance of MgB$_2$ during this period [2,3,4], the critical current density in MgB$_2$ is still not satisfactory compared to the expectations for its large-scale application.

Strong pinning strength and connections between grains are the critical factors determining the performance of the upper critical field (B_{c2}) and in-field J_c in the Type II superconductors. Flux pinning strength

in MgB$_2$ is closely associated with its elementary pinning force and grain size [5,6]. On the other hand, grain connectivity is generally suppressed by voids, an insulating oxide phase, and imperfect connections between grains [6]. Yamamoto et al. [7] investigated this issue by designing a percolation model and showed that the grain connectivity could be defined as a function of the packing factor. Accordingly, significant improvement might be achieved by increasing the in-field J_c if it were possible to enhance both the flux pinning strength and the grain connectivity at the same time. It is actually difficult in practice, however, to balance the connectivity and flux pinning strength of MgB$_2$ during the preparation process. A variety of processing techniques have been investigated to improve J_c in MgB$_2$ superconductors in the last 15 years, including irradiation [8,9], chemical doping [10–16], and ball milling [17–20]. Among them, carbon doping has been proved to be the currently most effective way of improving J_c, especially at high fields [11,13]. Nevertheless, all these techniques have focused on enhancing flux pinning by engineering grain boundaries or introducing nano-impurities and lattice defects, but they have tended to neglect and even worsen the associated issues of connectivity.

In particular, the corresponding issue of connectivity plays a more vital role in determining the J_c performance in the low field region in

MgB$_2$ wires because the low density that currently exists within the filament cores has been a serious obstacle in reaching high J_c values for *in-situ* MgB$_2$ wires. In addition, the reaction between magnesium and boron to form MgB$_2$ involves a volume contraction that produces a final density limited to about 50% of the theoretical density (2.36 g/cm^3) [6]. Consequently, the typical effective cross-sectional superconducting area in MgB$_2$ wire is only about 10% [21]. According to Rowell [21], the porosity of the wires strongly affects the grain connectivity of the grains in the finished wires, thus resulting in decreased wire performance. Several attempts have been undertaken to enhance the mass density of *in-situ* filaments by applying high pressure during the reaction heat treatment, by methods such as hot isostatic pressure (HIP) and other forms of hot pressing [22–27]. Cold densification was also applied as an alternative to the application of pressure at high temperature [28–30]. None of these high pressure processes, however, has the potential to be applied on long wire lengths. Very high mass densities in MgB$_2$ wires (up to 100%) can be produced without external pressure by using the *infiltration method*, originally proposed by Giunchi et al. [31]. Hur et al. [32] and Togano et al. [33] applied this technique to a wire configuration and found very high critical current densities in ring shaped MgB$_2$ filaments. The hollow centre of these filaments, however, introduces new homogeneity problems and a small superconducting filling factor, both of which require further development.

The application of cold high pressure densification (CHPD) on long wires, leading to significant enhancement of J_c, was undertaken at the University of Geneva by Flükiger et al. [34] and Hossain et al. [35], on binary and alloyed MgB$_2$ wires, respectively. Since no degradation of J_c was observed at the overlapping pressure zones between two pressed regions when compared to short wire lengths, these authors extended the CHPD method to lengths exceeding 10 m, a first step towards industrial lengths [36].

In the previous sections, it was mentioned that HIP is not suitable for the production of wires with industrial length due to several reasons: a) the possibility of Ar gas being trapped inside the filament, b) the limitation of applied gas pressure to 0.2 GPa, and c) the size of the pressure chamber being too small for reacting wires in km lengths. In order to avoid these difficulties, a new type of HIP device was recently constructed at the Institute of High Pressure Physics in Poland, and HIP was successfully applied on long lengths of wires [37]. This new toroid-type isostatic hot press enables the production of very long lengths of MgB$_2$ wire. Second stage densification using the toroidal HIP chamber after CHPD has been applied on optimized CHPD treated wires under the collaboration between the University of Wollongong, Australia and the Institute for High Pressure Physics, Unipress, Poland.

In the present work, both the CHPD and the HIP techniques were applied to *in-situ* PIT MgB$_2$ wires to increase mass density and further promote grain connectivity. In combination with the usage of optimized carbon-encapsulated boron as precursor, the highest J_c has been obtained for our *in-situ* CHPD + HIP PIT wires, and this result is very comparable to the performance of second generation MgB$_2$ wires produced by the Advanced Internal Magnesium Infiltration (AIMI) technique [38].

2. Experimental details

C-encapsulated amorphous boron powder (with a C percentage of about 2.30 wt.%) was obtained from Pavezyum Advanced Chemicals (PAVEZYUM) as the boron precursor. They produce carbon encapsulated amorphous boron powder through pyrolysis of a mixed gas consisting of diborane gas (B$_2$H$_6$), hydrogen, and hydrocarbon (C$_x$H$_y$) under inert conditions [39]. Then, the boron precursor was mixed with magnesium coarse powder (99.9%, 150 μm) in a molar ratio of Mg + 2B. MgB$_2$ wires were fabricated by the *in-situ* powder in-tube (PIT) process. The mixed Mg + 2B powder was firstly packed into metal tubes with an Nb barrier and a Monel outer sheath. Then, the tube was drawn to wire with 0.83 mm outer diameter (OD).

After that, pressure (about 1.8 GPa) was applied to the MgB$_2$ wires using the CHPD device (Fig. 1(a)) at room temperature after drawing, just before the final reaction heat treatment. It is clear from Fig. 1(a) that high pressure on the wire is uniformly applied from four sides *via* hard metal anvils at room temperature. After CHPD treatment, it was found that the cylindrical wires had been deformed into cuboid wires with a significant reduction of volume [34,35]. Consequently, a sizable enhancement of the mass density in MgB$_2$ wires can be obtained by CHPD, as reported in our previous studies [34,35].

After CHPD treatment, HIP (about 1.4 GPa in pressure) was applied to MgB$_2$ wires at 700 °C for 20 min. The CHPD-treated carbon-encapsulated MgB$_2$ wires were placed in the toroidal molten-salt-based HIP chamber (designed and built at IHPP, Poland) for further densification during the reaction [37]. In this device (Fig. 1(b)), the pressing medium consists of molten salt (specific eutectic mixtures of salts (*i.e.* NaCl and KCl) along with boron nitride (BN)) instead of gas. BN is used for its high chemical stability and plasticity. In this process, a long wire sample is kept inside the salt bed in a sealed high pressure chamber. At the desired temperature the solid salt mixture melts in the high pressure chamber, which is tightly closed. External forces on the upper cover are directly transmitted to the pressure chamber before it reaches the reaction temperature of 700 °C. This new toroid type isostatic hot press enables the production of very long lengths of MgB$_2$ wire. A schematic illustration of the first prototype of the liquid-salt-based HIP machine is shown in Fig. 1(b).

The morphology of the carbon-encapsulated boron precursor and the as-prepared MgB$_2$ wire samples was observed by scanning electron microscopy (SEM) on a JEOL JSM-7500FA, as well as by transmission electron microscopy (TEM) on a TEM with electron energy loss spectroscopy (EELS). The transport J_c values were measured as a function of applied magnetic field in a 15 T magnet at 4.2 K in a He flow cryostat using the four-probe technique, with currents up to 200 A.
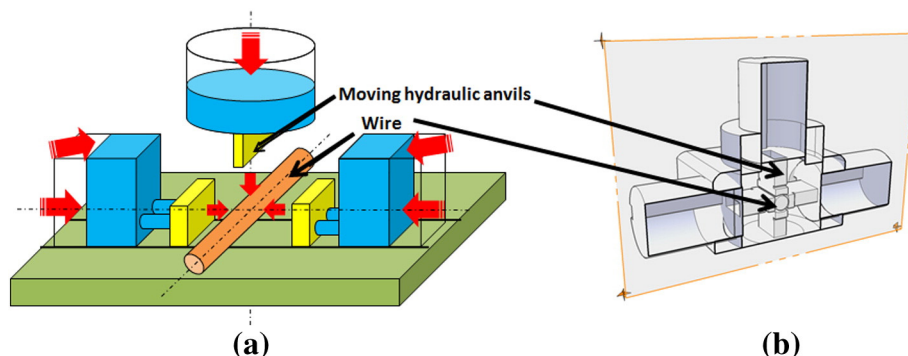

Fig. 1. Schematic illustrations and photographs of (a) CHPD [31,32] and (b) HIP [37] devices.

3. Results and discussion

Fig. 2 presents schematic diagrams of the process for preparing MgB$_2$ wires and the corresponding microstructures of boron or magnesium within the wires at different stages. As shown in the TEM image of the C encapsulated boron precursor in Fig. 2(a), the boron particles are very small (about 150–200 nm in size) and in the amorphous state. Observing more carefully, it can be found that these boron particles are homogeneously coated by some kind of thin layer. In combination with the EELS results, it is clear that the coating layer is carbon-rich, which indicates that the carbon is indeed mainly concentrated at the surfaces of the B particles and encapsulates them uniformly during the pyrolysis process, as we reported in our previous study [39]. Compared to other techniques for introducing carbon doping, such as solid state mixing and the chemical solution route [11–13], the significant advantages of using this C-encapsulated B as a precursor are that an appropriate level of carbon substitution can be achieved more homogeneously in the finally sintered MgB$_2$ wires at low cost and that the whole process is precisely controlled [39]. SEM images of the elongated fibrous Mg in the PIT MgB$_2$ wires after drawing and after CHPD treatment are presented in Fig. 2(b) and (c), respectively. Obviously, the coarse Mg original particles in the PIT wires are gradually deformed into Mg fibres as the size of the wires is decreased to the final diameter by drawing. Uchiyama et al. [40] reported that such behaviour of Mg in the PIT wires exists because magnesium is more ductile than boron, and during cold-working, the magnesium powder can be easily elongated along the wire direction, resulting in such fibrous structure within the wire core. During the process of Mg elongation, it is very important how well the nano-boron is uniformly and homogeneously distributed and diffused on the surfaces of the elongated Mg grains with minimum energy. Therefore, the application of CHPD prior to heat treatment is very crucial, not only for the uniform distribution of nano-boron particles, but also for minimizing the voids and empty spaces after drawing. After the CHPD process, this deformation of Mg is further enhanced, and the Mg fibres are elongated (see the element map of Mg within the wires after CHPD in Fig. 2(c)). Finally, the diameter of these Mg fibres can be reduced down to approximately 40 μm. Elongated voids with reduced size can still exist in the original positions of the elongated Mg fibres after the heat treatment due to the volume shrinkage as a result of the chemical reaction between Mg and B, and may be further minimized during the HIP process. In this way the shape and direction of the void can be effectively controlled and aligned along the wire direction using the coarse Mg as starting material, as discussed in our previous work [41]. Consequently, the electrical current can percolate easily with less obstruction in this kind of microstructure and thus improve the grain connectivity as well as the current carrying capacity.

Fig. 3 shows the microstructure of the superconducting core within the MgB$_2$ wire after HIP treatment and after traditional sintering for comparison. It is clear that the amount and size of the voids are reduced, and the density during sintering has been further increased in the HIP treated MgB$_2$ wire compared to traditional sintered wire (see Fig. 3(a) and (b)). It should be understood that the isostatic pressure is employed synchronously during the reaction process between Mg and B, which can further compact the superconducting core and compensate for the whole volume shrinkage. HIP without the CHPD stage is not very effective because high energy may be needed for the diffusion between the starting materials, which can destroy the linear fibrous structure and cause a mismatch of the boron distribution among the Mg grains. So, the application of HIP on the densified filament is obviously more beneficial, because minimum diffusion energy is needed to form homogeneous grains. In addition, comparing Fig. 3(c) with (d), clear and regular lattices are observed throughout whole MgB$_2$ grains in the traditional heat-treated wire, while some locally modulated nanostructure exists within the MgB$_2$ grains in the wire treated by HIP. The presence of this modulated nanostructure could be attributed to the microstrain resulting from HIP treatment. Based on the above results, usage of coarse Mg particles as precursor, together with CHPD and HIP treatment, is capable of dramatically increasing the mass density and decreasing the amount and size of voids, as well as controlling their shape and alignment. All these factors are beneficial for the connections between grains. Moreover, carbon-encapsulated amorphous boron precursor can introduce sufficient homogeneous carbon substitution into the boron, and the combination of CHPD and HIP treatment can significantly improve the grain connectivity, increase the uniformity in the MgB$_2$ filament, and increase the number of dislocations (Fig. 3). Gajda et al. [42] demonstrated considerable enhancement of J_c in both the high and low field regions after HIP treatment, which was due to the contribution of point and surface pinning centres, respectively, as well as the considerable reduction of voids. To confirm this, the transport critical current density (J_c) was measured, and the corresponding magnetic field dependence of the transport critical current density at 4.2 K for the CHPD and HIP co-treated MgB$_2$ wires with carbon encapsulated amorphous boron precursor is presented in Fig. 4. As references, the magnetic field dependence of the transport critical current density (J_c) for a traditional PIT MgB$_2$ wire, an internal Mg diffusion (IMD) MgB$_2$ wire, a solely CHPD treated PIT MgB$_2$ wire, and a solely HIP treated PIT MgB$_2$ wire is also presented in Fig. 4. Obviously, the values of J_c for the CHPD and HIP co-treated MgB$_2$ wire are four times higher than for the
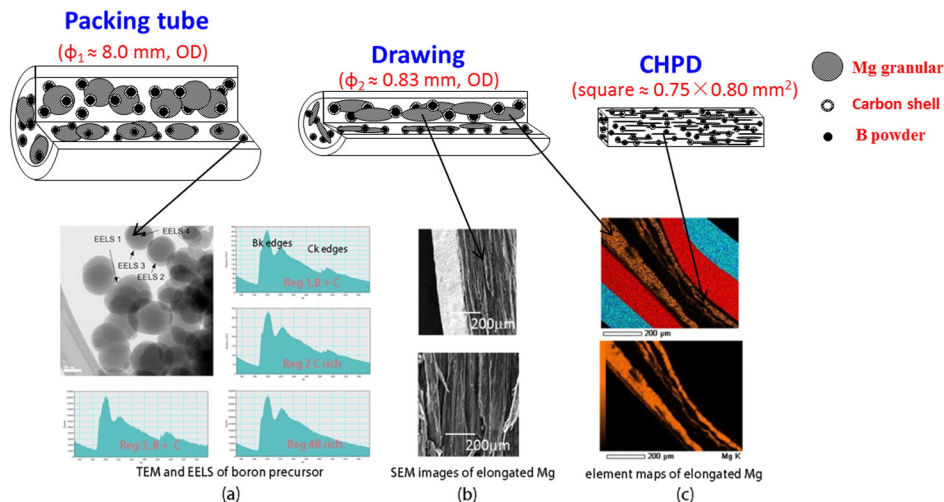

Fig. 2. Schematic diagrams of the process for preparing MgB$_2$ wires and the corresponding microstructures of the boron or magnesium within the wires at different stages.
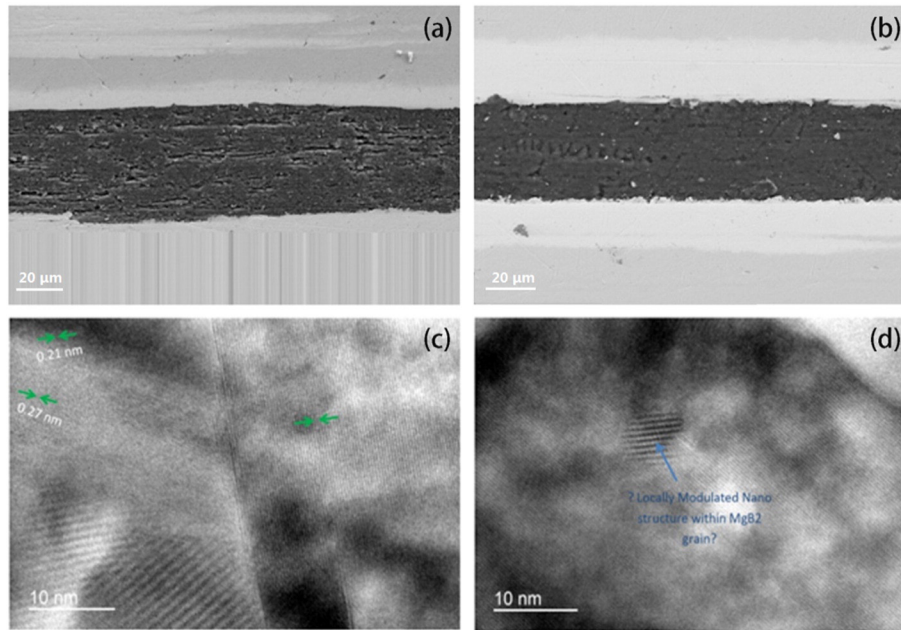
Fig. 3. (a, b) SEM and (c, d) TEM images of the microstructure of the superconducting core within the MgB_2 wire after HIP treatment.

traditional PIT MgB_2 wire without any treatment. The J_c performance of the solely CHPD treated wire and the solely HIP treated wire are also both higher than for the traditional PIT MgB_2 wire, whereas they are inferior compared to the CHPD and HIP co-treated wire. Neither CHPD nor HIP treatment alone is sufficient to increase the mass density. Only a collaborative effort using both CHPD and HIP treatment can effectively enhance the mass density, minimize the voids, and enhance the J_c overall in both the high and low magnetic field regions due to the uniform carbon substitution (Figs. 3 and 4). Generally, CHPD treatment alone compacts the original particles in the PIT MgB_2 wire, deforms these particles, and reduces the space between them, leading to an increase in the original packing density and filling factor. It has little effect, however, on the formation of voids as a result of the reaction between Mg and B during

heat-treatment. On the other hand, HIP treatment can make up for this limitation of CHPD treatment and reduce such voids.

It is worth noting that the J_c performance of the CHPD and HIP co-treated PIT MgB_2 wires in our work is close to that of IMD MgB_2 wires reported in previous studies [38]. Although the IMD MgB_2 wires exhibit the highest J_c values among all the MgB_2 wires reported to date [38], there are still serious shortcomings in the process for scaling-up these IMD second generation (2G) MgB_2 wires for applications. During the IMD process, the Mg diffuses into the B layer, forming a hollow wire, and there is only a thin layer of MgB_2 in the interior of the sheath tube (see its cross-section in the left inset of Fig. 4). As a result, the filling factor is very low (generally approximately 18%), and thus, the corresponding engineering critical current density (J_e) performance is not as remarkable as their J_c performance. Moreover, their mechanical performance is inferior due to this hollow structure, especially when the wire is twisted into coils, which is essential to fabricate various practical superconducting devices. On the contrary, the cores of the CHPD and HIP co-treated PIT MgB_2 wires in our work are full of solid superconducting MgB_2, and thus, the filling factor is very high (see the cross-section of this wire in the right inset of Fig. 4). One can imagine that this structure in our as-prepared wires leads to higher J_e as well as better mechanical properties than in IMD MgB_2 wires.
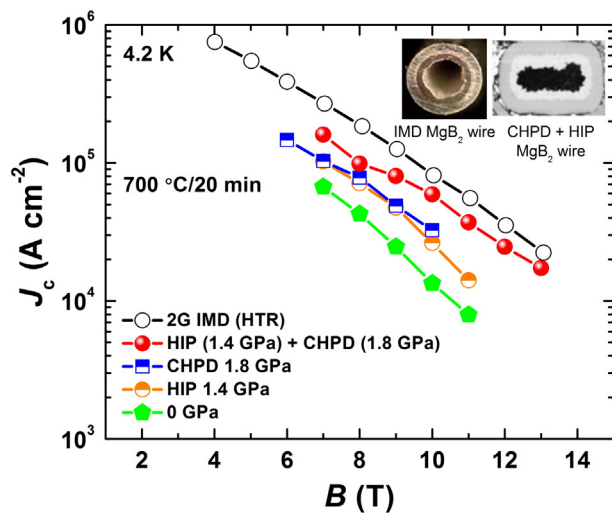
4. Conclusions

In summary, various techniques, including CHPD, HIP, and the usage of coarse Mg particles and carbon-encapsulated amorphous boron precursor were collaboratively employed to improve the J_c performance of PIT MgB_2 wires in the present work. Consequently, the best J_c performance was obtained among all the PIT MgB_2 wires reported so far, due to their improved grain connectivity and enhanced flux pinning strength. Our work suggests that the economical starting precursors and the combination of cold and hot densification techniques could represent a promising alternative for industrial and economical production of practical MgB_2 wires with excellent J_c.

Fig. 4. Magnetic field dependence of the transport critical current density (J_c) at 4.2 K for the CHPD and HIP co-treated MgB_2 wires with carbon encapsulated amorphous boron precursor and course Mg powder. For comparison, the magnetic field dependence of the transport critical current density (J_c) for a traditional PIT MgB_2 wire, an internal Mg diffusion (IMD) MgB_2 wire [38], a solely CHPD treated PIT MgB_2 wire, and a solely HIP treated PIT MgB_2 wire is also presented here. The insets are photographs of cross-sections of (left) the IMD MgB_2 wire and (right) the MgB_2 wire prepared in this work.

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