The Role of CHPD and AIMI processing on enhancing $J_C$ and transverse connectivity of in-situ MgB$_2$ strand

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Abstract. Research into in-situ MgB$_2$ strand has been focused on improvements in $J_C$ through reduction of porosity. Both of cold-high-pressure-densification (CHPD) and advanced-internal-magnesium-infiltration (AIMI) techniques can effectively remove the voids in in-situ MgB$_2$ strands. This study shows the nature of the reduced porosity for in-situ MgB$_2$ strands lies on increases in transverse grain connectivity as well as longitudinal connectivity. The CHPD method bi-axially applying 1.0 GPa and 1.5 GPa yielded 4.2 K $J_{CM\parallel s}$ of $9.6 \times 10^4$ A/cm$^2$ and $8.5 \times 10^4$ A/cm$^2$ at 5 T, respectively, with compared with $6.0 \times 10^4$ A/cm$^2$ for typical powder-in-tube (PIT) in-situ strand. Moreover, AIMI-processed monofilamentary MgB$_2$ strand obtained even higher $J_C$s and transverse grain connectivity than the CHPD strands.

1. Introduction

MgB$_2$ superconducting strands are promising to the practical magnetic application due to its high transition temperature $T_C$ (39 K) [1], high coherence length [2, 3], and low anisotropy of upper critical fields ($B_{C2}$) [3–6]. The powder-in-tube (PIT) in-situ MgB$_2$ strands were fabricated by filling a mixture of Mg and B powder into a non-reactive metallic tube and then being cold-worked into wires or tapes. The PIT strands have large amount of voids elongated along longitudinal strand axis which were left behind by molten Mg powders after heat treatment [7]. The present of voids tends to limit the number of the continuous current path in the in-situ MgB$_2$ strand and therefore suppress the current-carrying-capacity of the strand.

The CHPD technique can effectively increase the transport properties of PIT in-situ MgB$_2$ strands by eliminating the pores [8–11]. The pre-reacted powder-in-tube composite was bi-axially cold-densified at room temperature to increase the mass density of the Mg + B mixture. In this case, the cold-densified MgB$_2$ strand can obtain higher grain connectivity after heat treatment. Additionally, the Mg reactive – liquid – infiltration (RLI) process, initiated by Giunchi et al [12], also has the ability to eliminate the pores and produce a dense MgB$_2$ layers in in-situ MgB$_2$ strands. For RLI process, a Mg rod is inserted axially into a boron-filled metallic tube. After wire drawing the heat treatment (H. T.), MgB$_2$ layer is formed through the reactive diffusion of Mg into B layer. Since Mg is totally separated
with precursor B layer before H. T., the RLI process can totally eliminate the “Mg-site porosity” from MgB$_2$ layer but induce the formation of a big hole at the central region of the strand [13]. Furthermore, since the molar volume of MgB$_2$ (17.46 cm$^3$/mol) is twice as that of two B atoms (9.18 cm$^3$/mol), the volume expansion associated with the reactive transformation from 2B to MgB$_2$ enables even better connections between MgB$_2$ grains during heat treatment [13]. Our group named our RLI-processed strands as advanced – internal – magnesium – infiltration (AIMI) strands due to their optimized strand architecture and high $J_{cS}$ [13].

The previous researches mostly focused on investigating the effect of CHPD and AIMI techniques on the transport properties along longitudinal strand axis of in-situ MgB$_2$ strands, such as transport $J_{cS}$ and longitudinal grain connectivity. However, Shi and Susner pointed out that high anisotropic grain connectivity exists in PIT in-situ strands, which was resulted from the elongated voids between elongated MgB$_2$ stringers [7]. Therefore longitudinal transport properties are different with the transport properties along transverse strand axis for MgB$_2$ superconducting wires. In continuing along these lines, the transport properties along transverse strand axis were investigated for the 2.0 mol% C-doped in-situ MgB$_2$ strands in this study. The anisotropic connectivity of the PIT strands results in the differences between perpendicular magnetic $J_{c}$ ($J_{CM|\perp}$) and parallel $J_{c}$ ($J_{CM||}$). The influence of aspect ratio (S = length/diameter) on transverse and longitudinal $J_{c}$ were investigated for the PIT strand (P00). The CHPD-processed strands were P10 (1.0 GPa cold-pressing) and P15 (1.5 GPa cold-pressing). According to the previous results of our group, the CHPD technique increased the transport $J_{c}$ of the monofilamentary PIT in-situ MgB$_2$ strand from $3.0 \times 10^4$ A/cm$^2$ to $3.6 \times 10^5$ A/cm$^2$ at 4.2 K and 10 T due to decreased porosity [11] and AIMI-processed MgB$_2$ strands attained the 4.2 K, 10 T transport layer $J_{cS}$ of $1.0 \sim 1.5 \times 10^5$ A/cm$^2$ [13-16]. In this study, we compared the $J_{CM||s}$ and transverse connectivity of the CHPD- and AIMI-processed strands with those of the P00 strand at 4.2 K and 20 K. The relationship between porosity and transverse flux pinning force density $F_{p||}$, which is $J_{CM||} \times B$, for the in-situ MgB$_2$ strands was also discussed.

2. Experimental

2.1. Sample preparation

A series of pre-reacted powder-in-tube (PIT) in-situ strands, typically 0.834 mm diameter, with a Nb barrier and a Cu outer sheath were fabricated by Hyper Tech Research, Inc. (HTR). Two PIT strands (designated P10 and P15) were bi-axially densified with 1.0 GPa and 1.5 GPa at room temperature, respectively. The other strand (designated A00) manufactured through AIMI technique were also provided by HTR. The AIMI-processed strand, with 0.55 mm diameter, has a Nb barrier and a Monel outer sheath. The powders used for the present strands were 2 mol% C-doped amorphous B (10 – 100 nm) from Specialty Materials Inc. (SMI). The cold–densed PIT strands were heat-treated at 675 ℃ for 1 h and the AIMI strand was heat – treated at 625 ℃ for 16 h. The specification and heat treatment (H. T.) conditions of the strands are presented in table 1.

2.2. Transport and Magnetic Measurements

The transport $I_{c}$ ($I_{CT}$) test was conducted in perpendicular magnetic field up to 13 T in a pool of liquid Helium at 4.2 K on the MgB$_2$ strands with a total length of 50 mm and a gauge length of 5 mm. The electric criterion used for determining $I_{CTS}$ is 1.0 µV/cm. The magnetizations versus perpendicular and parallel magnetic fields ($M – H$) loops were measured by a Quantum Design Model 6000 Physical Property Measuring System (PPMS) for all strands with a sample length of 3 – 5 mm.
Table 1. Strand specification and H. T. conditions

| Strand Name | Strand Type | CHPD (GPa) | H. T. (h\(^o\)/C) |
|-------------|-------------|------------|-------------------|
| P00         | PIT         | 0.0        | 1/675             |
| P10         | PIT         | 1.0        | 1/675             |
| P15         | PIT         | 1.5        | 1/675             |
| A00         | AIMI        | 0.0        | 16/625            |

3. Results

The \( J_{CT} \)s of the PIT strands were the transport critical current normalized by MgB\(_2\) core area. As shown in Figure 1(a) the MgB\(_2\) core of the typical PIT in-situ strand is a solid cylinder. Figure 1(b)-(d) shows the shape of MgB\(_2\) cores for the CHPD- and AIMI-processed strands are cuboid and hollow cylinder, respectively. The \( J_{CT} \) of the AIMI–processed strands, which is also named as transport layer \( J_C \), were calculated by dividing the \( I_{CT} \) by the area of annulus MgB\(_2\) layer. Values of magnetic \( J_C \) \( (J_{CM,\perp} \text{ and } J_{CM,\parallel}) \) for the MgB\(_2\) strands were extracted from the full \( M – H \) loops heights \( \Delta M \), using the standard Bean model equations [7, 17]:

For the PIT in–situ wire P00:

\[
\text{Perpendicular Magnetic } J_C: \quad J_{CM,\perp} = \frac{3\pi \Delta M}{8R_0} \tag{1}
\]

\[
\text{Parallel Magnetic } J_C: \quad J_{CM,\parallel} = \frac{3\Delta M}{2R_0} \tag{2}
\]

Here \( R_0 \) is the radius of the cylinder MgB\(_2\) core in the PIT wire.

For the densified wires P10 and P15:

\[
\text{Parallel Magnetic } J_C: \quad J_{CM,\parallel} = \frac{2\Delta M}{b(1-\frac{b}{3a})} \tag{3}
\]

Here \( a, b \) are both lengths of the transverse cross – sectional area of cuboid MgB\(_2\) core, \( a > b \).
For the AIMI wire A00:

\[
\text{Parallel Magnetic}\ J_c: \ J_{(CM)} = \frac{3\Delta M}{2} \left( \frac{R_0^2 - R_i^2}{R_0^2 - R_i^2} \right)
\]

(4)

Here \( R_i \) is the inner diameter of the annulus MgB\(_2\) layer and \( R_0 \) is the outer diameter of the annulus MgB\(_2\) layer.

Figure 1. (a) Back scattered SEM images of (a) strand P00, (b) strand P10 (1.0 GPa cold pressure), (c) strand P15 (1.5 GPa cold pressure), and (d) AIMI strand A00.
3.1. Transport and magnetic critical current densities, $J_{CT}$ and $J_{CM}$

Figure 2(a) shows the $J_{CT}$ and $J_{CM\perp}$ versus $B$ at 4.2 and 20 K for the strand P00. It can be seen that $J_{CM\perp}$s agree with $J_{CT}$ at low fields, whereas the bifurcation of $J_{CT}$ and $J_{CM\perp}$ happened at high fields. Moreover, $J_{CM\perp}$s were greatly affected by the aspect ratio $S$, especially at high magnetic fields. The relationships among $J_{CT}$, $J_{CM\perp}$, and aspect ratio were fully discussed in ref [7] and [18]. Therefore, the values of $J_{CM\perp}$ are not only determined by the intrinsic properties but also by the extrinsic properties of the in-situ MgB$_2$ strands. As shown in Figure 2(b) $J_{CM\parallel}$s were independent on the aspect ratio $S$. Based on this result, we can know the values of $J_{CM\parallel}$s as well as $J_{CT}$ are merely determined by the intrinsic properties of the in-situ MgB$_2$ strands. The MgB$_2$ macrostructure of the reacted PIT in-situ strand is characterized by elongated polycrystalline MgB$_2$ fibers and elongated pores partially separating the fibers [7, 11, and 18]. The $J_{CT}$s and $J_{CM\parallel}$s can represent the longitudinal and transverse connectivity of the MgB$_2$ fibers, respectively. In summary, we can investigate the effect of CHPD and AIMI technique on the current–carrying–capacity of the in-situ MgB$_2$ strands through $J_{CM\parallel}$s as well as $J_{CT}$.

Figure 3 shows the $J_{CT}$ and $J_{CM\parallel}$ versus $B$ at 4.2 and 20 K for all strands. The CHPD technique significantly enhanced $J_{CM\parallel}$s of PIT in-situ strands at 4.2 K. Strand P10 and P15 attained $9.5 \times 10^4$ A/cm$^2$ and $8.5 \times 10^4$ A/cm$^2$ at 4.2 K and 5 T, with respect to $6.0 \times 10^4$ A/cm$^2$ for strand P00. 4.2 K $J_{CT}$s were slightly enhanced by the CHPD technique. Therefore, the increases in $J_{CT}$ and $J_{CM\parallel}$ indicated that the both longitudinal and transverse connections between MgB$_2$ fibers were enhanced. With the formation of high density MgB$_2$ layer, strand A00 attained 4.2 K, 10 T $J_{CT}$ of $9.4 \times 10^4$ A/cm$^2$, which is 180% higher than those of CHPD-processed strands. On the other hand, the AIMI strand obtained 5 T $J_{CM\parallel}$s of $3.1 \times 10^5$ A/cm$^2$ at 4.2 K and $1.7 \times 10^4$ A/cm$^2$ at 20 K, which are about 240% higher than those of CHPD strands.

*Figure 2.* (a) $J_{CT}$ and $J_{CM\perp}$ versus $B$ at 4.2 and 20 K (b) $J_{CM\parallel}$ versus $B$ at 4.2 and 20 K for strand P00.
3.2. Transverse Flux Pinning, Porosity and Transverse Grain Connectivity

Figure 4 shows the $F_{p||}/F_{p,max||}$ versus $B/B_{irr}$ for all strands, where $F_{p||} = J_{CM||} \times B$. It can be seen that the peak pinning occurred at $b = B/B_{irr||}$ close to 0.2 at 4.2 and 20 K, which is in agreement with the Dew-Hughes/Kramer model [19, 20]. In other words, the dominant pinning centers for the all strands are also grain boundaries for the direction along transverse strand axis. According to the previous work [11], the densified wires have decreased porosities, the values of porosity are $\sim 50\%$ ($p = 0$) and $\sim 30\%$ ($p = 1.0$ or $1.5$ GPa). Since the Mg-site porosity was totally eliminated for AIMI–processed strands, the porosity of the AIMI strand is close to 0. Therefore, both CHPD and AIMI processes enable the resulting MgB$_2$ phases to be denser and more connected. For the discussion, it can be concluded that the enhanced $J_{CT}$ and $J_{CM||}$ in densified wires and AIMI wire is correlated with lower porosity (higher grain connectivity).

The connectivity $K$ defined by Rowell [21] can represent the grain connectivity of MgB$_2$ strands. The connectivity $K$ is calculated by the equation:

$$K = \frac{\Delta \rho_{SC}}{\Delta \rho}$$

Here $\Delta \rho$ is the difference between the sample’s resistivity at 300 K and the sample’s resistivity at 40 K and $\Delta \rho_{SC}$ is the resistivity difference for an ideal single crystal. However, transverse connectivity $K_{||}$ is difficult to be determined for the MgB$_2$ wires. It has been reported that the maximum flux pinning force densities of fully–connected MgB$_2$ superconductor, where grain connectivity $K = 100\%$, are estimated to be 90 GN/m$^3$ at 4.2 K and 22 GN/m$^3$ at 20 K [22]. Therefore, we can roughly estimate the transverse grain connectivities $K_{||}$s of the in-situ MgB$_2$ strands by normalizing the $F_{p,max||}$ with the $F_{p,max}$ of fully-connected MgB$_2$. Table 2 shows the transverse maximum flux pinning force densities $F_{p,max||}$ at 4.2 K and 20 K and estimated transverse grain connectivities for all the strands. The connectivity of 5% is achieved by the 2 mol% C-doped PIT strand, P00. The cold–densification increases the
connectivity of PIT strand by 20%. The AIMI strand A00 obtained the highest transverse grain connectivities of 20%.

Figure 4. (a) $F_{p||}/F_{p,max||}$ versus $B/B_{irr}$ for all strands at 4.2 K, (b) $F_{p||}/F_{p,max||}$ versus $B/B_{irr}$ for all strands at 20K

Figure 5. Relationship between porosity and transverse flux pinning force densities for in–situ MgB$_2$ strands
Table 2. Transverse flux pinning force densities and estimated connectivity of the strands.

| Strand Name | $F_{p,max}$, 4.2 K (GN/m$^3$) | $F_{p,max}$, 20 K (GN/m$^3$) | Transverse Connectivity, $K_{||}$ % |
|-------------|-----------------|-----------------|-----------------|
| P00         | 4.3             | 1.1             | 5.0             |
| P10         | 6.3             | 1.4             | 6.0             |
| P15         | 5.6             | 1.3             | 6.0             |
| A00         | 19.6            | 4.1             | 20              |

4. Conclusion

$J_{\text{CM}}$ is merely determined by the intrinsic properties of in-situ MgB$_2$ strands, so the current-carrying capacity of in-situ MgB$_2$ strands can be represented by $J_{\text{CM}}$ as well as $J_{\text{CT}}$. The CHPD of 1.0 GPa and 1.5 GPa enhanced the 4.2 K, 5 T $J_{\text{CM}}$ from $6.0 \times 10^4$ A/cm$^2$ to $9.6 \times 10^4$ A/cm$^2$ and $8.5 \times 10^4$ A/cm$^2$, respectively. AIMI strand attained the highest $J_{\text{CT}}$ and $J_{\text{CM}}$ at 4.2 and 20 K due to the formation of a high dense MgB$_2$ layer. By eliminating the voids in in-situ MgB$_2$ strands through CHPD and AIMI technique, better connections between MgB$_2$ grains along transverse strand axis can be obtained in in-situ MgB$_2$ strands.
5. References

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