Transactions of the Materials Research Society of Japan 33[2] 315-318 (2008)

Superconducting Properties of In-situ processed MgB_2 Tapes Prepared by a Hot Pressing

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In-situ powder-in-tube processed MgB_2 tapes have been prepared through a hot pressing and conventional heat treatment. Magnesium hydride MgH_2 and amorphous B powder mixed with SiC powder addition was encased in a carbon steel tube to form a core/sheath composite. The composite was fabricated into a square rod by grooved-rolling, then into a monocore tape 4mm in width and 0.5mm in thickness by flat rolling. The hot pressing was performed under 100 MPa in the temperature range of 630-800℃ for 1-10 h in Ar gas atmosphere.

The combination of hot pressing during the heat treatment and the addition of SiC nano-sized powder appreciably enhanced the core Jc for the MgB_2 tapes. The core Jc at 4.2 K for the 5 mass% SiC addition tape hot pressed at 630℃ for 10 h was 125 A/mm^2 at 10 T, 500 A/mm^2 at 7 T and close to 1,000 A/mm^2 at 5 T, respectively. Hot pressing prevented voids due to the volume reduction in MgB_2 synthesis reaction, and strengthened the linkage among MgB_2 grains. The addition of SiC nano-sized powder enhanced the core Jc, especially the Jc at high magnetic field due to the improvement of Hc_2 and refinement of the MgB_2 grains.

Key words: MgB_2 superconductor, powder-in-tube, in-situ process, hot pressing, critical current

1. INTRODUCTION

Since the discovery of superconductivity at critical temperature Tc of 39 K in MgB_2 [1], many research and development of conductors as well as thin films on the new superconductor have been done. In particular, improvement of critical current Ic and critical current density Jc in MgB_2 superconducting wires and tapes have carried out for practical applications.

There are two different methods for fabricating MgB_2 wires and tapes by the powder-in-tube (PIT) process. First is the so-called ex-situ process, in which a commercially available MgB_2 powder is utilized as raw material. Second is the in-situ process, in which a mixed powder of Mg and B is used to synthesize MgB_2 superconductor. Many papers have been already published on the ex-situ PIT process [2]-[5] and in-situ PIT process [6]-[10]. The former process is simpler since no heat treatment is necessary to synthesize MgB_2, while the superconducting properties depend sensitively on the properties of the starting MgB_2 raw powder. The latter process is more suitable to introduce effective addition of nanopowders and produce high performance under magnetic field, while it is difficult to obtain homogeneous and dense MgB_2 structure due to the synthesis reaction.

In this paper the effect of SiC nanopowder addition to the MgB_2 core and hot pressing on the enhancement of the Ic and Jc in steel-sheathed in-situ PIT processed MgB_2 tapes has been studied relating with the improvement in their microstructures.

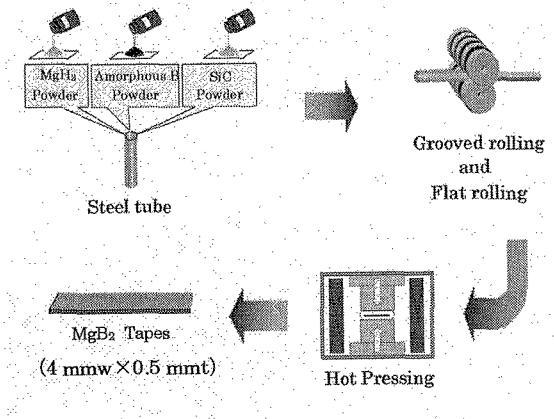


Fig. 1 Preparation procedure of the in-situ PIT processed MgB_2 tapes by a hot pressing.

2. EXPERIMENTAL

Fig. 1 illustrates the preparation procedure of the in-situ PIT processed MgB_2 tapes by a hot pressing. Magnesium hydride MgH_2 and amorphous B powder mixed with and without 5 mass% SiC powder was encased in a carbon steel tube outside diameter of 8 mm and inside diameter of 4.5 mm to form a core/sheath composite. The composite was fabricated into 1.8 mm square rod through grooved-rolling, then into a monocore tape 4 mm in width and 0.5 mm in thickness through flat rolling. The hot pressing (H.P.) was

Table 1 Grain size and purity of starting MgH_2 and amorphous B raw powders, and SiC additional powder.

Powder	Grain Size	Purity
MgH_2	< 45 ìm	98 %
B (amorphous)	< 1 ìm	97 %
SiC	30 nm	95 %

performed under 100 MPa in the temperature range of 630-800℃ for 1-10 h in Ar gas atmosphere. The heat treatment with no pressing (H.T.) was similarly performed.

The grain size and the purity for the starting MgH_2 and amorphous B raw powders, and additional SiC ceramic powder are listed in Table 1. MgH_2 was used to avoid oxidized Mg powder such as MgO. The MgH_2 is thermally decomposed at near 450℃ into Mg metal to react with B powder. SiC added to the MgB_2 core is nano-sized powder.

X-ray diffraction (XRD) scans of the MgB_2 core were performed, after crushing into powders, using Co-Kα line. The microstructures of the MgB_2 core were observed using an optical microscope and scanning electron microscope (SEM), respectively. The Ic at 4.2 K of the tape specimens was measured by a four-probe resistive method, the criterion of the Ic measurement being 1 μV/cm. The magnetic field was always applied perpendicular to the specimen current, and parallel or perpendicular to the tape surface at the time of the Ic measurement. The core Jc was calculated by dividing Ic by the cross-sectional area of the MgB_2 core. The transport currents at 4.2 K up to 12 T were measured by the facilities of National Institute for Materials Science.

3. RESULTS and DISCUSSION

Figs. 2(a) and (b) illustrate optical micrographs of the cross-section for the 5 mass% SiC addition MgB_2 tapes heat-treated at 630℃for 10 h. Many voids (black parts) are observed in the core of the tape heat-treated with no pressing shown in Fig. 2(a). On the other hand, almost no void is observed in the core of the tape hot pressed under 100 MPa seen in Fig. 2(b). The cross-sectional area of the core for hot-pressed tapes, being 0.35 mm^2 in area, decreases by about 40% compared to that of the tape heat treated with no pressing. The reduction in MgB_2 core area almost corresponds to the area of all voids formed during MgB_2 synthesis reaction.

SEM structures of the fractured MgB_2 core in the tapes heat-treated at 630℃ for 10 h with no SiC addition and no pressing, and with 5 mass% SiC addition and hot pressing at 100 MPa are shown in Figs.3(a) and (b), respectively. Fig. 3(a) indicates that various-sized voids shown by arrows were formed in the core after the MgB_2 synthesis. The structure in Fig. 3(b) shows that MgB_2 core consists of fine grains including submicron-sized grains, and much less voids are formed. Thus, hot pressing during heat treatment prevents void formation due to volume reduction in MgB_2 synthesis and form the denser structure to strengthen the linkage among MgB_2 grains. Furthermore, SiC nanopowder addition in the core may enhance the refining of MgB_2 grains since the SiC nanopowder depresses the MgB_2 grain growth during hot pressing process.

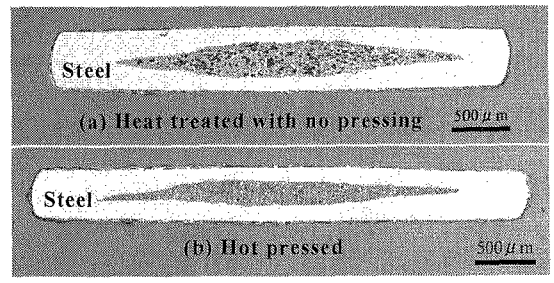
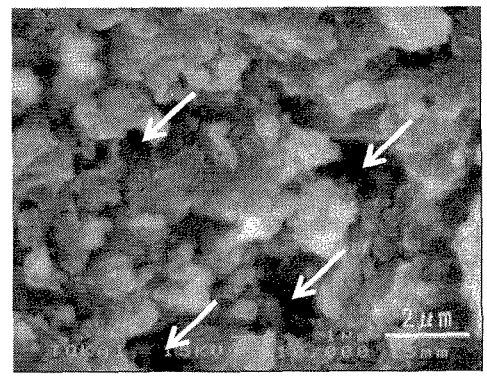
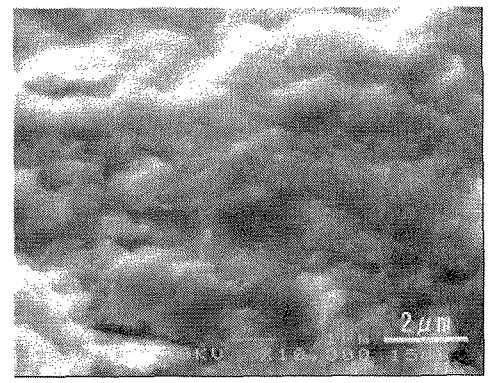


Fig. 2 Optical micrographs of the cross-section for the 5% SiC addition MgB_2 tapes heat-treated at 630℃ for 10 h. (a) Heat-treated with no pressing, (b) Hot pressed at 100 MPa.



(a) Heat treated with no pressing



(b) Hot pressed at 100 MPa

Fig. 3 SEM micrographs of the cross-section of the fractured MgB_2 core. (a) Heat treated with no pressing, (b) Hot pressed at 100 MPa.

Fig. 4 illustrates XRD patterns of the MgB_2 core powder for 5 mass% SiC addition tapes hot-pressed at 630-800℃ for 10 h. MgB_2 phases are synthesized and dominant in the whole range of 630-800℃. The iron boride such as FeB reacted with steel sheath increases with increasing temperature of hot pressing. The formation of FeB may deviate from the nominal composition ratio of Mg:B=1:2 in MgB_2 synthesis. On the other hand, the magnesium silicide such as Mg_2Si is formed with decreasing temperature of hot pressing.

Fig. 5 shows magnetic field dependence of Ic and core Jc at 4.2 K in 5 % SiC addition tapes hot-pressed at 630-800℃ for 10 h. The highest Ic at 4.2 K and magnetic field up to 12 T was obtained in the tape

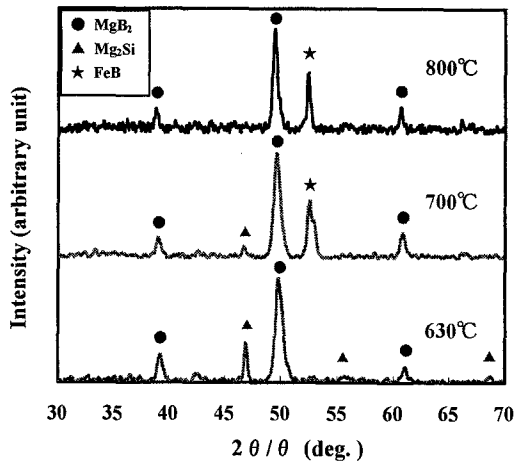


Fig. 4 XRD patterns of the MgB_2 core powder for the 5 mass% SiC addition tapes hot- pressed at 630~800℃ for 10 h under 100 MPa.

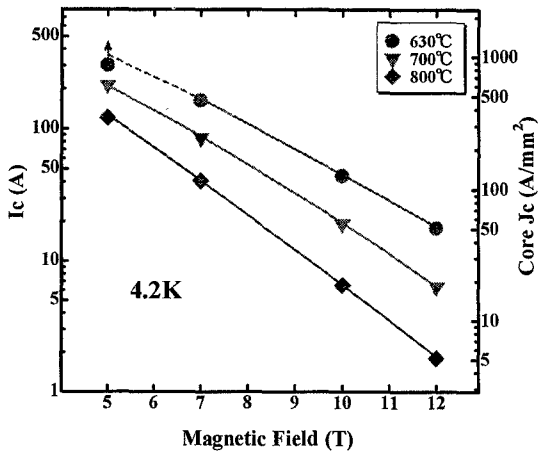


Fig. 5 Magnetic field dependence of Ic and core Jc at 4.2 K in 5% SiC addition tapes hot-pressed at 630-800℃ for 10 h under 100 MPa.

hot-pressed at 630℃. An arrow in the figure indicates that the transport current exceeds the capacity limit of 300 A for the specimen probe and power source. The Ic decreases with elevated temperature for hot-pressing time of 10 h. According to [10], since SiC is strong FeB former, the degradation in Ic for the hot-pressed tapes at relatively high temperatures of 700-800℃ results from the FeB formation in MgB_2 core reacted with steel sheath as shown in Fig. 4. The hot-pressing at 630℃ for 1 h was too short to synthesize MgB_2 in the tape.

Fig. 6 indicates XRD patterns of the MgB_2 core powder and core surface removed from the steel sheath for 5% SiC addition tapes hot pressed at 630℃ for 10 h. The XRD patterns are almost no difference between powder and core surface. The intensity ratio of I(002)/I(101) for core surface is slightly larger than that for core powder. Therefore, a c-axis oriented texture in MgB_2 core was hardly introduced by a hot pressing process.

Magnetic field dependence of core Jc at 4.2 K for 5% SiC addition tapes hot pressed and heat treated at 630℃ for 10 h is shown in Fig. 7. Magnetic field was

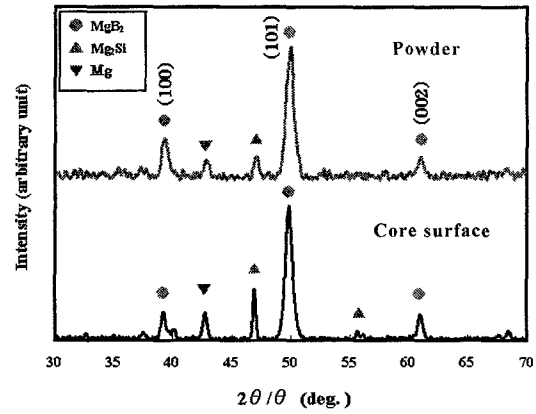


Fig. 6 XRD patterns of the MgB_2 core powder and core surface removed from steel sheath for 5% SiC addition tapes hot pressed at 630℃ for 10 h under 100 MPa.

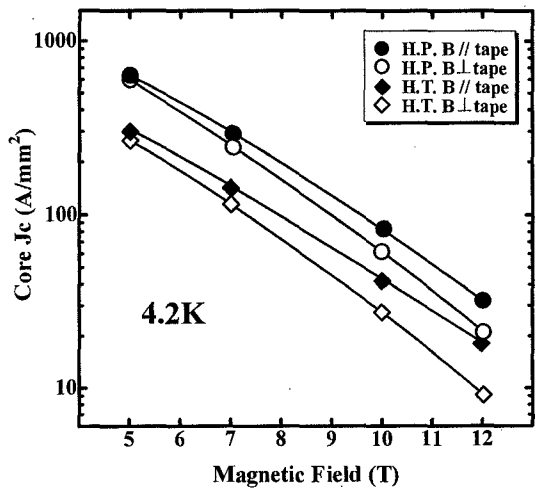


Fig. 7 Magnetic field dependence of core Jc at 4.2 K for 5% SiC addition MgB_2 tapes hot pressed under 100 MPa and heat treated at 630℃ for 10 h. Magnetic field was applied perpendicular and parallel to the tape surface.

applied parallel (//) and perpendicular(⊥) to the tape surface. The core Jc with the magnetic field parallel to the tapes surface is larger than that with perpendicular magnetic field in both hot pressed and heat treated with no pressing MgB_2 tapes. The anisotropy of Jc is defined as the ratio between the values measured with the magnetic field parallel and perpendicular to the tape surface [11].

$$\Gamma = \frac{Jc(B//)}{Jc(B\perp)} \quad (1)$$

The anisotropy ratio Γ increases with increasing magnetic field, being 1.47 for hot pressed tapes and 1.97 for heat treated with no pressing tapes at 12 T, respectively. The low anisotropy ratio below two in the MgB_2 tapes may be useful for practical application.

The magnetic field dependence of core Jc at 4.2 K for 5% SiC and no addition tapes hot-pressed at 100 MPa and heat-treated with no pressing at 630℃ for 10 h are summarized in Fig. 8. The addition of SiC nanopowder enhances the core Jc, especially the Jc at

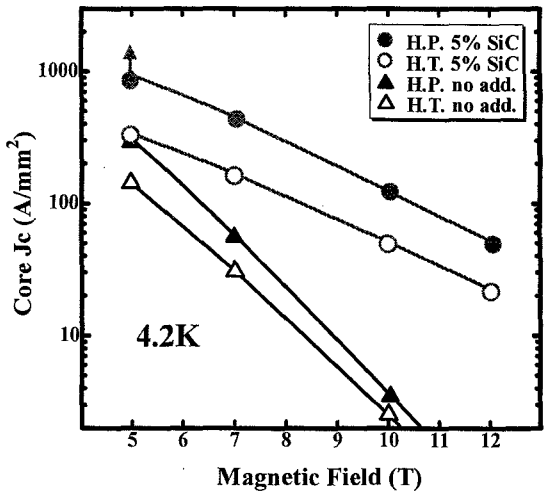


Fig. 8 Core Jc at 4.2 K versus magnetic field for 5% SiC and no addition tapes hot pressed at 100 MPa and heat treated with no pressing at 630℃ for 10 h.

high magnetic field due to the improvement of Hc_2 and refinement of the MgB_2 grains. In the previous study for the SiC addition tapes [10], the resistivity just above the Tc, ρ_n increases by a factor of two, while Tc slightly decreases by SiC addition due to the carbon substitution for Boron site in MgB_2 crystal. Since the upper critical field Hc_2 is proportional to the product of ρ_n and Tc, considerable increase of ρ_n enhances the Hc_2 of the MgB_2 core with SiC addition. Therefore, the improvement of core Jc at high magnetic field may result from the enhancement of Hc_2 due to the incorporation of SiC nano-sized powder. Furthermore, hot pressing during heat treatment appreciably enhances the core Jc for the tapes. The core Jc for 5 mass% SiC addition tape is 125 A/mm^2 at 10 T, 500 A/mm^2 at 7 T and close to 1,000 A/mm^2 at 5 T, respectively. The difference in core Jc between hot-pressed and heat-treated with no pressing tapes becomes larger under lower magnetic field than under high magnetic field. The enhancement of the core Jc in low magnetic field results from the improvement of MgB_2 core structure, that is to say, denser structure without voids and strong linkage among MgB_2 grains. Thus, the hot pressing during heat treatment and addition of SiC nanopowder performed higher core Jc at 4.2 K by a factor of 5 at 5 T and by a factor of 40 at 10 T in comparison with no pressing during heat treatment and no addition of effective nanopowders.

4. CONCLUSIONS

The combination of hot pressing during the heat treatment and the addition of SiC nanopowder appreciably enhanced the core Jc for the in-situ PIT processed MgB_2 tapes with steel sheath. The core Jc at 4.2 K for the 5 mass% SiC addition tape hot pressed at 100 MPa at 630℃ for 10 h was 125 A/mm^2 at 10 T, 500 A/mm^2 at 7 T and close to 1,000 A/mm^2 at 5 T, respectively. The core Jc with the magnetic field parallel to the tapes surface was larger than that with perpendicular magnetic field, and the anisotropy ratio of core Jc increased with increasing magnetic field, being below two up to 12 T. Hot pressing during heat treatment prevented voids due to the volume reduction in MgB_2 synthesis reaction, and strengthened the linkage among MgB_2 grains. The addition of SiC nanopowder enhanced the core Jc, especially the Jc at high magnetic field due to the improvement of Hc_2 and refinement of the MgB_2 grains.

ACKNOWLEDGEMENT

The authors wish to thank Mr. Kyuna and Mr. Miyamoto of Future Science and Technology Joint Research Center of Tokai University for their SEM image observations.

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(Recieved September 6, 2007 ; Accepted December 30, 2007)