Enhanced critical current density of *in situ* processed MgB$_2$ bulk superconductors with MgB$_4$ additions

S. H. Kim$^{a,b}$, W. N. Kang$^b$, B.-H. Jun$^a$, Y. J. Lee$^{a,b}$, and C.-J. Kim$^{a,*}$

$^a$Korea Atomic Energy Research Institute, Daejeon, Korea
$^b$Sungkyunkwan University, Suwon, Korea

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Abstract

The effects of MgB$_4$ addition on the superconducting properties and the microstructure of *in situ* processed MgB$_2$ bulk superconductors were studied. MgB$_4$ powder of 1-20 wt.% was mixed with (Mg + 2B) powder and then pressed into pellets. The pellets of (Mg + 2B + xMgB$_4$) were heat-treated at 650 °C for 1 h in flowing argon. The powder X-ray diffraction (XRD) analysis for the heat-treated samples showed that the major formed phase in all samples was MgB$_2$ and the minor phases were MgB$_4$ and MgO. The full width at half maximum (FWHM) values showed that the grain size of MgB$_2$ decreased as the amount of MgB$_4$ addition increased. MgB$_4$ particles included in a MgB$_2$ matrix is considered to suppress the grain growth of MgB$_2$. The onset temperatures ($T_{c,onset}$) of MgB$_2$ with MgB$_4$ addition (0-10 wt.%) was between 37-38 K. The 20 wt.% MgB$_4$ addition slightly reduced the $T_{c,onset}$ of MgB$_2$ to 36.5 K. This result indicates that MgB$_4$ addition did not influence the superconducting transition temperature ($T_c$) of MgB$_2$ significantly. On the other hand, the small additions of 1-5 wt.% MgB$_4$ increased the critical current density ($J_c$) of MgB$_2$. The $J_c$ enhancement by MgB$_4$ addition is attributed not only to the grain size refinement but also to the possible flux pinning of MgB$_4$ particles dispersed in a MgB$_2$ matrix.

Keywords: MgB$_2$, MgB$_4$ addition, Superconductor, Critical current density ($J_c$), Grain refinement

1. INTRODUCTION

A MgB$_2$ superconductor has a high superconducting critical temperature ($T_c$) of 39 K [1] and a high critical current density ($J_c$) at magnetic fields [2]. In addition to the excellent superconducting properties, other merits of MgB$_2$ are its long coherent length [3] and the small current anisotropy [4-5]. Moreover, the raw materials (Mg and B) of MgB$_2$ are not expensive and easy to obtain. Owing to these merits, MgB$_2$ is believed to be a promising material that can replace a conventional NbTi superconductor. A superconducting magnetic designed using MgB$_2$ superconducting wires can be operated at 20 K using an electrical cooler instead of liquid helium.

For practical applications of a MgB$_2$ superconductor the $J_c$ under magnetic fields should be enhanced. The presence of defects in superconducting grains or at the grain boundaries of MgB$_2$ leads to the $J_c$ enhancement of MgB$_2$. The defects can be made artificially either through physical [6-13] or chemical [14-16] methods. Particle (neutron, electron or ion) irradiation is a well-known process that can form defects physically through the collision of accelerated particles to the targeting materials. Small defects such as vacancies and cavities can be formed in the superconducting matrix through particle irradiation [15 - 20]. As a result of the defect formation, the $J_c$ at the magnetic fields of the particle-irradiated MgB$_2$ was much higher than that of MgB$_2$ with no irradiation [21,22]. Along with the physical method, the $J_c$ of MgB$_2$ can be increased by the doping of chemical species such as silicon carbide [23], carbon-related materials [24], and other fine impurity phases [25]. The carbon doping to MgB$_2$ not only led to the formation of a lattice strain [26], and lattice substitution [26-31] but also increased the number of grain boundaries [32]. Mechanical milling for boron powder is an alternative way to increase the $J_c$ of MgB$_2$ through the grain size refinement of MgB$_2$ [32].

In this experiment, MgB$_4$ powder was synthesized using Mg and B powder by the powder reaction process. MgB$_4$ is a phase that is compatible with MgB$_2$ in a Mg-B binary phase diagram. MgB$_4$ is often formed with MgB$_2$ in the powder reaction process using boron-enriched compositions. The synthesized MgB$_4$ powder was used as an additive of *in situ* processed MgB$_2$ bulk superconductors. The added amount of MgB$_4$ was changed from 0 to 20 wt.%. The effects of MgB$_4$ addition on the $J_c$, $T_c$ and the microstructure of MgB$_2$ were investigated.

2. EXPERIMENTAL PROCEDURE

MgB$_4$ powder used as an additive for MgB$_2$ superconductors was synthesized by the powder reaction method of (Mg + 4B = MgB$_4$) using Mg and B powders.
The detailed preparation condition of MgB$_2$ was well described in previous reports [33-38]. The impurity phases included in the synthesized MgB$_4$ powder were removed by the HNO$_3$ leaching process [36].

MgB$_2$ bulk superconductors were fabricated through an in situ reaction process using Mg and B powders. Mg and B powders with a ratio of Mg:B=1:2 were mixed for 30 min by hand mixing using a mortar jar and pestle. To compensate the possible Mg loss during heat treatment, 10 wt.% of Mg was intentionally added to a mixture of (Mg + 2B). MgB$_4$ powder of 1-20 wt.% was added to the mixture of (Mg + 2B) powder.

0.3 g of a powder mixture of (Mg + 2B + x wt.% MgB$_4$) was put in a steel mold with a diameter of 10 mm and was uniaxially pressed into a pellet. The pressed pellet was encapsulated with a Ti tube to suppress the possible oxidation of Mg during heat treatment. To determine the optimum heat treatment condition for the formation of MgB$_2$, the heat treatment temperature and time were varied. We determined the heat treatment temperature as 650°C, which is a temperature just above a melting point of Mg and the heat treatment period as 1 h. The short period of 1 h at 650°C is to minimize the grain growth of MgB$_2$. The Ti-encapsulated pellets were located at the center of a tubular furnace, heated to 650°C at a rate of 100°C/h in flowing argon gas, maintained for 1 h and then cooled to room temperature in a furnace.

The heat treated pellets were crushed into a powder form for a phase analysis. The formed phases after heat treatment were analyzed using an X-ray diffraction method (XRD) for the power samples. From the peak intensity of each phase in XRD patterns, volume fractions of the formed phases and the second residual phases were calculated using eq. (1).

$$ F(MgB_2) = \frac{\sum I_{MgB_2}}{\sum I_{MgB_2} + \sum I_{MgB_4} + \sum I_{MgO}} \tag{1} $$

where $F(MgB_2)$ is a volume fraction of MgB$_2$, and $I_{MgB_2}$ and $I_{MgB_4}$ and $I_{MgO}$ are the peak intensities of MgB$_2$, MgB$_4$ and MgO, respectively.

To measure $T_c$ and $J_c$ of MgB$_2$ bulk superconductors, rectangular specimens with approximate dimensions of 3×3×2 mm were cut from the heat-treated pellets using a diamond saw. Susceptibility-temperature curves ($M-T$) and magnetization-magnetic field ($M-H$) curves are obtained using a magnetic properties measurement system (MPMS) with a maximum magnetic field of 5 Tesla. $J_c$ at 5 K and 20 K was calculated using the extended Bean’s critical model [39] of eq. (2).

$$ J_c = 20\Delta M/a(1-a/3b) \tag{2} $$

where $\Delta M$ is the magnetization difference ($M$ decreasing field region-$M$ increasing field region) in a constant magnetic field, and $a$ and $b$ are parameters regarding the sample dimension.

3. RESULTS AND DISCUSSION

Fig. 1 shows the XRD patterns of the samples with (0-20 wt.% ) MgB$_4$ additions, heat-treated at 650°C for 1 h. The major formed phase of all samples is MgB$_2$ regardless of the amount of MgB$_4$ additions. The addition of MgB$_4$ appears to not affect the formation of MgB$_2$. As the amount of MgB$_4$ addition increases, the peaks of MgB$_4$ are intensified (see the XRD patterns with 10 and 20 wt.% MgB$_4$ additions). Another minor phase formed in the heat-treated pellets is MgO. Small MgO peaks observed in all XRD patterns are attributed to the oxidation of Mg during heat treatment.

According to the XRD results of Fig. 1, the formation reaction of MgB$_2$ at the give heat treatment condition can be described by eq. (3).

$$ Mg + 2B + xMgB_4 = MgB_2 + xMgB_4 + yMgO \tag{3} $$

At the heat treatment temperature of 650°C, MgB$_2$ forms through the liquid phase reaction between a Mg melt and a solid B powder, because the heat treatment temperature is high than m. p. (649°C) of Mg. The formation reaction of MgB$_2$ would be conducted by the mass transfer through the Mg melt. Most of the Mg is thought to be consumed to form MgB$_2$, through the reactions of (Mg + 2B = MgB$_2$) and (MgB$_4$ + Mg = MgB$_2$). Some of the Mg might be consumed for the oxidation of Mg or be evaporated during heat treatment. Because 10 wt.% of Mg was intentionally added to the raw powders of (Mg + B) to compensate the Mg loss, the amount of Mg was sufficient for the formation reaction of MgB$_2$. In the case of large MgB$_4$ additions, The peaks of MgB$_4$ were present together with MgB$_2$. The MgB$_4$ is supposed to be present as a discrete particle form within MgB$_2$ grains or at the grain boundaries of MgB$_2$.

Fig. 2 shows the volume fraction of the formed phases in the samples with MgB$_4$ additions, calculated using eq. (1).
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![Graph showing volume fractions of MgB$_2$, MgB$_4$, and MgO as a function of MgB$_4$ addition.]

**TABLE I**

| Amount of MgB$_4$ (wt.%) | FWHM (deg.) | Lattice parameter (Å) |
|--------------------------|-------------|-----------------------|
|                          | (110)       | (002)     | a      | c      |
| 0                        | 0.389       | 0.443     | 3.090  | 3.532  |
| 1                        | 0.415       | 0.449     | 3.091  | 3.500  |
| 3                        | 0.405       | 0.413     | 3.087  | 3.534  |
| 5                        | 0.391       | 0.425     | 3.088  | 3.540  |
| 10                       | 0.403       | 0.455     | 3.090  | 3.526  |
| 20                       | 0.396       | 0.486     | 3.087  | 3.497  |

The volume fraction of MgB$_2$ of the sample with no addition is approximately 80%. As is normally expected, the volume fraction of MgB$_2$ is dependent on the added amount of MgB$_4$. The volume fractions of MgB$_2$ for the sample with 10 wt.% and 20 wt.% MgB$_4$ additions are 74% and 66.3%, respectively. Interestingly, the volume fraction of the MgO formed during heat treatment is approximately 18% regardless of the MgB$_4$ addition.

Through a precise analysis of the XRD patterns of Fig. 1, the lattice parameters of MgB$_2$ and the full width at half maximum (FWHM) of the peaks were calculated, the results of which are summarized in TABLE I. As illustrated in TABLE I, the lattice parameters $a$ and $c$ were not influenced by the MgB$_4$ additions. This is because both MgB$_2$ and MgB$_4$ are stoichiometric line compounds and there is no interaction between MgB$_2$ and MgB$_4$ (MgB$_4$ is a high temperature phase). The values of FWHM estimated from the (110) and (002) peaks of MgB$_2$ increase as the amount of MgB$_4$ addition increases, which indicates the change in the grain size of MgB$_2$.

The grain sizes of MgB$_2$ samples with no addition and MgB$_4$ additions were calculated from the formula $t = 0.9\lambda / B \cos \theta$ \cite{40} using the FWHM data. Where $t$ is the grain (crystallite) size, $\lambda$ is the wavelength of the target used, $B$ is the half width of the peak, and $\theta$ is the Bragg angle of the incident beam.

![Graph showing variation of grain size of MgB$_2$ as a function of MgB$_4$ addition.]

Fig. 3 shows the variation of the grain size of MgB$_2$ as a function of the amount of MgB$_4$ addition. The grain size of MgB$_2$ increases with increasing amount of MgB$_4$ addition.

![SEM micrographs of samples with (a) no addition, (b) 5 wt.% MgB$_4$ addition, and (c) 20 wt.% MgB$_4$ addition.]

Fig. 4. SEM micrographs of samples with (a) no addition, (b) 5 wt.% MgB$_4$ addition, and (c) 20 wt.% MgB$_4$ addition.
MgB₂ with no addition is 45 nm. The grain size decreases as the amount of MgB₄ addition increases. For 5 wt.% and 20 wt.% MgB₄ additions, the grain sizes of MgB₂ are 30 nm and 22 nm, respectively. These results indicate that the MgB₄ addition suppressed the grain growth of MgB₂. In other words, a larger grain boundary area exists in the pellet with MgB₄ addition in comparison with no addition.

Fig. 4 shows SEM micrographs of the samples with (a) no addition, (b) 5 wt.% MgB₄ addition and (c) 20 wt.% MgB₄ addition. Many plate-like grains whose thickness is smaller than 1 μm are observed in sample (a). As already confirmed through the XRD analysis of Fig. 1, approximately 80 % of the formed phase of sample (a) was MgB₂. It can thus be said that the plate-like grains are MgB₂. The SEM investigation showed that the grain size of MgB₂ decreased as the amount of MgB₄ addition increased. The MgB₂ grains observed in the sample with 20 wt. % MgB₄ addition are smaller than that of sample (a) (see Fig. 4c)). The SEM observation agrees with the XRD FWHM of Fig. 2.

The reason why the grain growth of MgB₂ was suppressed by the addition of MgB₄ can be explained in terms of the thermal stability of MgB₂ at the heat treatment temperatures. There is a large difference in the melting temperature between Mg and B. The m. p. of B is as high as 2300℃ [41], whereas the m. p. of Mg is very low at 649℃ [42]. MgB₂, which has more boron than MgB₄, would be more stable at high temperature. The MgB₄ particles can be present at the grain boundaries of MgB₂ or trapped within MgB₂ grains (see the schematic illustration in Fig. 5). The MgB₄ particle present at the grain boundaries of MgB₂ can suppress the grain growth of MgB₂ during heat treatment.

Fig. 6 shows normalized susceptibility-temperature (M-T) curves of the samples with various MgB₄ additions. The onset temperature of superconducting MgB₂, Tc,onset, and the midpoint of the superconducting range, Tc,mid, of the sample with no addition are 37 K and 35.9 K, respectively, whereas Tc,onset and Tc,mid of the sample with 10 wt.% MgB₄ addition are 37 K and 36.1 K, respectively. In the case of a large MgB₂ addition of 20 wt.%, Tc,onset and Tc,mid are 36.5 K and 35.8 K, which are slightly lower than the other samples. The superconducting critical temperatures seem to not be significantly influenced by the MgB₄ addition. The M-T result confirms again that MgB₄ is a chemically stable and thermally compatible additive with MgB₂, which does not react with a superconducting MgB₂. The superconducting critical temperatures of the samples with various MgB₄ additions are summarized in Table II.

| MgB₄ addition (wt.%) | Superconducting critical temperatures of MgB₂ (K) |
|---------------------|-----------------------------------------------|
|                     | Tc,onset | Tc,mid |
| 0                   | 37       | 35.9   |
| 1                   | 37       | 36.0   |
| 3                   | 37.5     | 36.3   |
| 5                   | 38       | 37.3   |
| 10                  | 37       | 36.1   |
| 20                  | 36.5     | 35.8   |

Table II: Superconducting transition temperatures of the samples with MgB₄ additions.

Fig. 7 shows Jc-B curves at 5 K and 20 K of samples with MgB₄ additions, heat-treated at 650℃ for 1 h. The Jc values of samples with 1-5 wt.% MgB₄ additions are slightly higher than that of the sample with no addition. Moreover, Jc at 5 K of the sample with 10 wt.% MgB₄ is similar to that of the sample with no addition, in spite of the low volume fraction of MgB₂. The enhanced Jc of the samples with small MgB₄ additions is attributed to the grain refinement and the presence of MgB₄ particles in the superconducting MgB₂ matrix, which were already observed by FWHM and SEM investigations (Figs. 3 and 4). These two factors appear to act as flux pinning centers which pin the magnetic flux. When the amount of MgB₄ addition is more than 20 wt.%, Jc decreases because the superconducting volume fraction in the sample decreases. Although not shown here, in our study, the addition of MgB₄ up to 70% resulted in a decrease in Jc, in proportion to the amount added. The Jc-B characteristics at 20 K of samples with MgB₄ additions show similar trend to that at 5 K: 1-5 wt.% MgB₄ additions increases the Jc of MgB₂.

Fig. 8 shows the Jc-B curves at 5 K and the magnetic fields of 3.5-5.0 T. With the exception of the 20 wt.% MgB₄ addition, the Jc values at 5 K and the given applied magnetic fields of the samples are higher than that of the sample with no addition. Jc at 5 K, 4 T of the sample with no addition is 50,850 A/cm², whereas Jc at 5 K, 4 T of the samples with 1 wt.% and 3 wt.% MgB₄ additions are 59,920 A/cm² and 60,760 A/cm², respectively.
Fig. 7. $J_c$-$B$ curves at 5 K and 20 K of the samples with 0-20 wt.% MgB$_4$ additions.

Fig. 8. $J_c$-$B$ curves at 5 K and magnetic fields of 3.5 T-5.0 T of the samples with 0-20 wt.% MgB$_4$ additions.

4. CONCLUSIONS

We studied the effects of MgB$_4$ additions (1-20 wt.%) on the superconducting properties of MgB$_2$ bulk superconductors prepared using the powder reaction process. The onset temperature ($T_{c, onset}$) of MgB$_2$ with MgB$_4$ addition (0-10 wt.%) was between 37-38 K. Even for a large MgB$_4$ addition of 20 wt.%, the $T_{c, onset}$ was 36.5 K. This result indicates that the MgB$_2$ addition did not significantly influence the superconducting transition temperature ($T_c$) of MgB$_2$. On the other hand, the $J_c$ level of MgB$_2$ with 10 wt.% MgB$_4$ addition was similar to that of MgB$_2$ with no addition in spite of the reduced superconducting volume. The FWHM and SEM investigation showed that the grain size of MgB$_2$ decreased as the amount of MgB$_4$ addition increased. The presence of MgB$_4$ in a MgB$_2$ matrix is likely to suppress the grain growth of MgB$_2$ at the heat treatment temperature. The $J_c$ enhancement of MgB$_2$ by the MgB$_4$ addition appears to be attributed to the grain size refinement of MgB$_2$ and the possible flux pinning of MgB$_4$ particles dispersed within MgB$_2$ grains.

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