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# Trapped magnetic field of dense $MgB_2$ bulks fabricated under high pressure

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**Abstract**

We have measured the trapped magnetic field $B_T$, critical current density $J_c$ and connectivity $K$ between crystal grains for the $MgB_2$ bulks fabricated by a HIP method and a capsule method. The maximum $B_T$ value is 2.51 T at 12.7 K for the bulk fabricated by HIP method. From the SEM images of the bulks prepared by HIP at 98 and 980 MPa, the higher pressure enhances the grain growth. These superconducting characteristic values for the bulks by the HIP method were higher than those for the bulks by the capsule method.


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*Keywords*: $MgB_2$; trapped magnetic field; hot isostatic pressing; density; grain boundary

## 1. Introduction

$MgB_2$ has been researched for the practical applications such as wires and thin films, in general. A tesla-class superconducting bulk magnet has been mainly studied using a single grain of RE-Ba-Cu-O (RE: rare earth elements) bulk [1]. However, the c-axis oriented single grain of RE-Ba-Cu-O bulk must be used, because the intergranular weak links lower the critical current density $J_c$. On the other hand, for the $MgB_2$ bulk, we can ignore the problem of intergranular weak links even in polycrystalline samples because of their long coherence length [2]. Although several groups reported that $MgB_2$ bulk trapped the tesla-order magnetic field [3-6]. We reported the trapped magnetic field $B_T$ of 1.77 T at 15.5 K in a *in-situ* $MgB_2$ bulk with 38 mm in diameter and 9 mm in thickness fabricated by capsule method, in which a precursor pellet was sealed with two commercial stainless steel flanges and copper gasket [3]. Tomita *et al*. suggested the $B_T$ of 2.25 T at 15 K for the *in-situ* $MgB_2$ bulk with 30 mm in diameter and 10 mm in thickness [4]. Viznichenko *et al*. reported that the $B_T$ of about 2.3 T at 6 K was obtained for the $MgB_2$ bulk with 28 mm in diameter and 11 mm in thickness which was sintered under the pressure as high as 2 GPa [5]. Recently, Durrel *et al*. reported the average $J_c$ of $8.0\times10^8$ A/m$^2$ in the self field at 20 K in the *ex-situ* $MgB_2$ bulk with 25 mm in diameter and 5.38 mm in thickness fabricated by uniaxial hot pressing [6].

The *in-situ* $MgB_2$ bulk gives us the good intergranular connection and high $J_c$. However, the density of *in-situ* $MgB_2$ bulk is about 50% value compared with the theoretical density (2.62 g/cm$^3$), because Mg reacts with B in precursor pellet. Therefore it is possible to improve the $J_c$ using high-density bulk. In this study, we fabricated high dense $MgB_2$ bulks using a HIP (Hot Isostatic Pressing) method and compared the trapped magnetic field properties with those fabricated under ambient pressure. We discussed the correlation between the trapped magnetic field, density, and micro structure.

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Table 1. Specification of the $MgB_2$ bulks.

| Sample | Diameter (mm) | Thickness (mm) | Heat treatment | Pressure | Filling fraction |
|---|---|---|---|---|---|
| S-HIP#23 | 23.1 | 24.0 | 900 ℃×3 h | 980 MPa | 93.8% |
| HIP#26 | 26.1 | 6.5 | 900 ℃×3 h | 98 MPa | 95.0% |
| CAP#20 | 20.2 | 5.0 | 800 ℃×6 h | ambient | 59.9% |
| CAP#30 | 30.4 | 9.0 | 800 ℃×6 h | ambient | 52.7% |

## 2. Experimental

### 2.1. Sample preparation

Raw powder of Mg (99% in purity, ≦180 μm in grain size) and B (99% in purity, 300 mesh in grain size) were weighted with 1.05~1.1:2.0 in molar ratio and ground. High dense $MgB_2$ bulks were prepared by the HIP method. The mixture was pressed into pellets by CIP (Cold Isostatic Pressing) method. The precursor pellet was sealed in the stainless steel (SS) container by EBW (Electron Beam Welding) in vacuum. The sealed SS containers were set into the HIP machine and subsequently sintered at 900 ℃ for 3 h under a pressure of 98 or 980 MPa. Ambient pressure bulks were prepared by the capsule method [3]. The precursor pellet was set in the hole of the SS flange, covered by the SS plates, and finally closed by the other SS flange with the copper gasket using the bolts and nuts. The SS plates were inserted to prevent the reaction of Mg with Cu and the expansion of the pellet during the sintering. The precursor pellet was prepared in air and the capsule was sealed in Ar-atomosphere using a glove box. The closed capsule was sintered at 800 ℃ for 6 h in a box furnace and cooled down to room temperature by furnace cooling. The $MgB_2$ bulk prepared by the HIP at 980 MPa with 26.2 mm in diameter and 24.0 mm in thickness was named S-HIP#23. The $MgB_2$ bulk prepared by the HIP at 98 MPa with 26.1 mm in diameter and 6.5 mm in thickness was named HIP#26. The $MgB_2$ bulks fabricated by capsule method with 20.2 mm in diameter and 30.4 mm in diameter, respectively, were named CAP#20 and CAP#30. Sample specifications are listed in Table 1.

### 2.2. Measurements

The $MgB_2$ bulk was magnetized by field cooling (FC) in a magnetic field of 5 T, and then the applied field was decreased to 0 T at a rate of -0.22 T/min. Trapped field was measured by a cryogenic Hall sensor mounted on the center of the bulk surface, and the temperature of the bulk measured by a Cernox thermometer which was also mounted on the bulk surface. We measured the magnetization curve by using a commercial SQUID magnetometer using several small pieces cut from the bulk after the FC magnetization measurements and evaluated $J_c$ using the extended Bean model. Electrical resistivity $\rho$ was measured by a standard four probe technique. The micro structure of the $MgB_2$ bulks was observed by SEM (Scanning Electron Microscope).

## 3. Results and discussion

Fig. 1 shows the temperature dependence of the trapped field, $B_T(T)$, of the $MgB_2$ bulks. The maximum $B_T(T)$ of S-HIP#23, HIP#26, CAP#20, and CAP#30, respectively, are 2.42 T at 13.4 K, 2.51 T at 12.7 K, 1.43 T at 13.5 K, and 1.52 T at 16.6 K. The $B_T(T)$ values of the HIP bulks were higher than that of the bulks fabricated by the capsule method due to the high density of HIP bulks. Surprisingly, the $B_T(T)$ of the HIP#26 (98 MPa) bulk was higher than that of the S-HIP#23 (980 MPa) bulk. The $B_T$ values of both HIP#26 and S-HIP#23 exceed that of the bulk fabricated under pressure of 2 GPa [5]. The high pressure synthesis over 980 MPa seems to decrease the $B_T$. Durrel *et al.* reported the $B_T$ of 3.14 T at 17.5 K using the stack of two disk-shaped $MgB_2$ bulks. Although there is no data of single disk [6], our bulks also expected to give us the $B_T$ over 3 T in the similar configuration.

Fig. 2 shows the magnetic field dependence of the $J_c$ at 20 K for four $MgB_2$ bulks. The $J_c$ values in the self field at 20 K of S-HIP#23, HIP#26, CAP#20, and CAP#30, respectively, are $3.0\times10^5$, $4.8\times10^5$, $1.4\times10^5$, and $1.1\times10^5$ A/cm$^2$. The $J_c$ values of the HIP bulks were 2-3 times higher than that of the bulks fabricated by the capsule method, because the filling fraction of the HIP bulks was twice as high as that of the bulks fabricated by the capsule method. This result of $J_c$ qualitatively correlated with that of $B_T$.

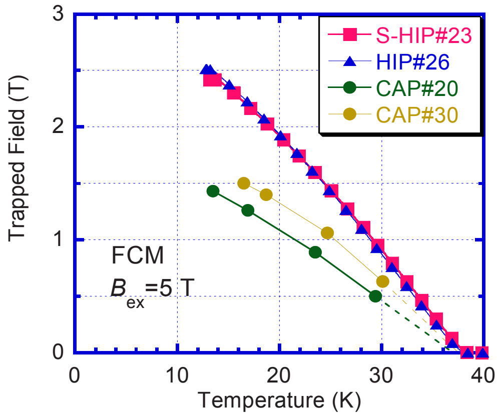

Fig. 1. Temperature dependence of trapped field $B_T$.

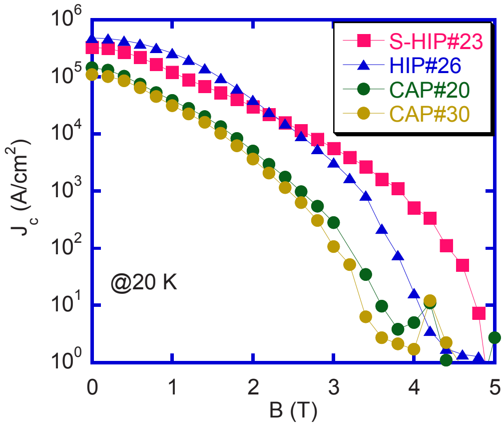

Fig. 2. Temperature dependence of critical current density $J_c$ at 20 K.

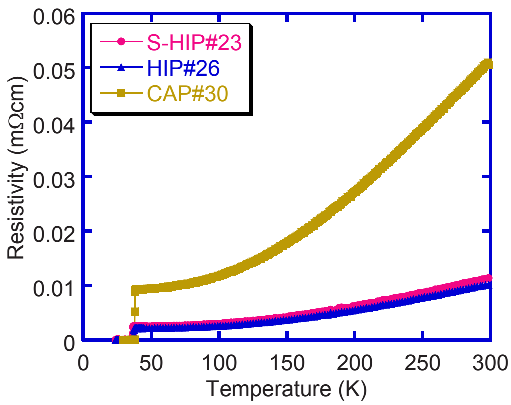

Fig. 3. Temperature dependence of electrical resistivity $\rho(T)$.

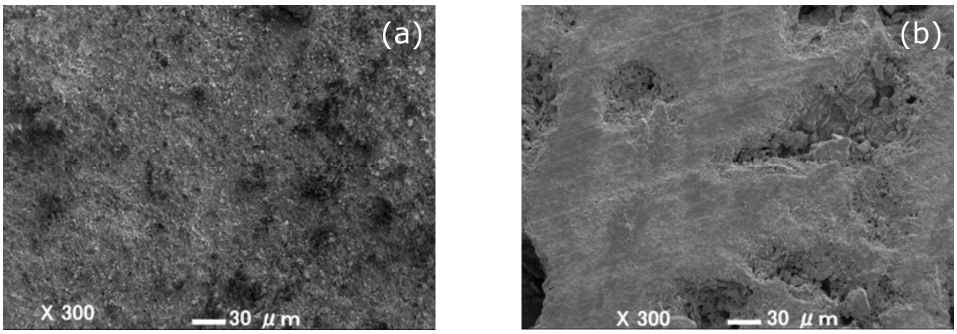

Fig. 4. SEM images of the bulks by the (a) HIP method (S-HIP#23) and by the (b) capsule method (CAP#30).

Fig. 3 shows the temperature dependence of resistivity $\rho(T)$. The connectivity $K$ was evaluated from the following expression [7],

$$K = \frac{\Delta\rho_{crystal}}{\Delta\rho_{\exp}} \times 100 \qquad (1)$$

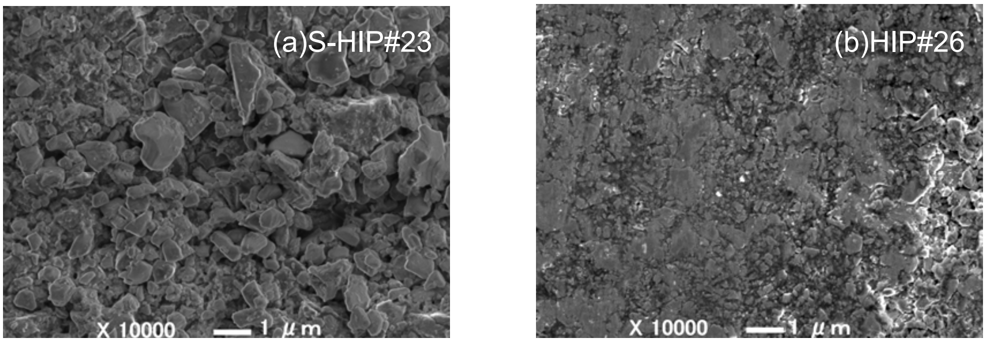


Fig. 5. High magnification SEM images for the (a) S-HIP bulk (S-HIP#23) and the (b) HIP bulk (HIP#26).

where $\Delta\rho_{crystal}$ = 6.32 μΩcm [8], and $\Delta\rho_{\exp} = \rho$ (300 K)- $\rho$ (40 K). The connectivity values of S-HIP#23, HIP#26, and CAP#30 are 70.2%, 81.0%, and 15.3%, respectively, which demonstrate that the good grain connection for the HIP bulks enhances the superconducting circular current flowing in the bulks and enhances the $B_T$ and $J_c$.

Fig. 4 shows SEM images of the HIP bulk (S-HIP#23) and the bulk by the capsule method (CAP#30). Many large voids can be observed in the micro structure of the bulk by the capsule method. However, no large void was found in the HIP bulk. The increase of the volume fraction of superconductivity was confirmed for the HIP bulk by the SEM observation and was correlated with the $B_T$ and $J_c$ values.

Fig. 5 shows the high magnification SEM images of the S-HIP#23 and HIP#26 bulks. The grain size of S-HIP#23 is about 0.2~2.0 μm and that of HIP#26 is about 0.2~1.5 μm. This suggests that the grain growth is enhanced by the higher pressure sintering. Therefore, the number of the grain boundaries which seems to act as the pinning center in the $MgB_2$ decreases in the S-HIP#23 and the $B_T$ and $J_c$ values are lower than those for HIP#26.

## 4. Conclusion

We measured the trapped magnetic field $B_T$, critical current density $J_c$, and connectivity $K$, for the $MgB_2$ bulks fabricated by both HIP and capsule methods. The important obtained results are described in the followings.

(1) The highest $B_T$ of 2.51 T at 12.7 K was achieved for the HIP#26 (98 MPa). The $B_T$ and $J_c$ of the HIP bulks were higher than that of the bulks fabricated by the capsule method, and these results were qualitatively correlated with the result of $K$.

(2) The SEM images of $MgB_2$ bulks prepared by the HIP at 98 and 980 MPa suggest that the higher pressure enhances the grain growth. From the results, the difference of the $B_T$ between S-HIP#23 and HIP#26 is explained.

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