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Potential ability of 3 T-class trapped field on MgB_2 bulk surface synthesized by the infiltration-capsule method

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Abstract

We successfully synthesized a dense (~90%-filled) MgB_2 bulk with no residual Mg via an infiltration process by overcoming the problems in this process such as the expansion of a B precursor disk under a liquid Mg infiltration and the residuals of unreacted Mg in the bulk using a specially designed capsule. As a result, we have achieved a record-high trapped field to date, B_T, of 2.4 T at the center of the bulk surface at the lowest temperature of 15.9 K among the infiltration-processed MgB_2 bulks. The trapped-fields simulated for a model with the experimental $J_c(\mu_0 H)$ characteristics well reproduced the experimental B_T's and gave a reliable estimated B_T below 15.9 K. The extrapolation of the experimental and simulated B_T curve reached 3 T at 4.2 K. The critical current densities, $J_c(\mu_0 H)$'s, at 20 K were 1.8×10^5 A cm^{-2} under the self-field and 4.5×10^3 A cm^{-2} under the magnetic-field of $\mu_0 H = 3$ T. The connectivity, K, of 16% of the present bulk was comparable with that of the ~50%-filled MgB_2 bulk. The high B_T with low K and the microstructure of the present bulk suggested that the high- and low-J_c regions coexisted because of the wide variation of the MgB_2 grain-size.

Keywords: MgB_2, infiltration process, trapped field, bulk magnet

(Some figures may appear in colour only in the online journal)

1. Introduction

MgB_2 superconducting bulks and wires have been intensively studied to use in a practical superconducting apparatus because of the specific advantages of well-connected grains for supercurrent flowing [1, 2] and a relatively high critical temperature, T_c, of 39K [3]. However, a low filling factor of about 50%–75% of the ideal density and a large degradation of J_c under magnetic-fields are obstacles to producing a strong bulk magnet trapping over 10 T or to replacing the conventional low-T_c materials such as NbTi and Nb_3Sn by MgB_2 for wire applications. High-pressure synthesis including a hot isostatic pressing (HIP) [3, 4], hot pressing (HP) [5], spark plasma sintering (SPS) [6, 7], and so on offers an over 90%-filled MgB_2 with enhanced J_c due to the increase of the supercurrent path and the number density of the grain boundaries as a dominant pinning center. The polycrystalline MgB_2 bulks trapping 1–2 T were produced under high pressure by two groups [8, 9], just after the discovery of the superconductivity in MgB_2. Continuous numerous studies [10–18] confirmed the Tesla-order MgB_2 bulk magnets, in which the B_T has been enhanced up to 4–5 Tesla to date by the improvement of the fabrication process and/or the introduction of the pinning centers [14–16]. The record-high B_T of 5.4 T at 12 K was realized for the MgB_2 bulk (20 mm in diameter and 8 mm in thickness; 20 mmϕ × 8 mm$t$) fabricated by *an ex-situ* uniaxially HP method using ball-milled nanometric MgB_2 powder [14]. The *in-situ* HIP technique also produced a strong MgB_2 bulk magnet (26 mmϕ × 7 mm$t$) trapping 2.5 T at 12.7 K, which was increased by 1.6 times from 1.6 T at the same temperature for the 50%-filled MgB_2 bulk (30 mmϕ × 9 mm$t$) [13]. Moreover, the Ti-doping offered the B_T of 3.6 T at 13.4 K at the surface of a single HIP-processed bulk and 4.6 T at 14.1 K at the center of stacked bulk pair [16]. A homogeneous conical B_T profile [10, 15] due to the polycrystalline form and a highly stable B_T [15, 16] due to the small vortex-creep phenomenon could compensate a shortcoming of relatively low B_T of MgB_2 bulk compared to that of a RE-Ba-Cu-O bulk [19–21], and therefore MgB_2 bulk magnets are good candidates for a practical superconducting application which requires a highly homogenized and stabilized magnetic field.

In contrast to high-pressure synthesis, highly dense MgB_2 can also be prepared by an infiltration (diffusion) process above the melting point of Mg ($T_{m.p.} = 650\,°C$) without external physical pressure [22, 23]. In this process, the liquid Mg initially infiltrates into a B preform via the capillary effect, which leads to the expansion of the B preform, reacts with B to form MgB_2, shrinking about 25% in volume. The change in volume and the uncontrollable capillary effect result in an inhomogeneous bulk with cracks and a large amount of unreacted Mg as a channel- and/or thread-like shape, as reported previously [18, 22]. Several B_T values have been reported for the infiltration processed (IP) MgB_2 bulks; the MgB_2 bulk (55 mmϕ × 10 mm$t$) fabricated by a reactive liquid-Mg infiltration (Mg-RLI) method offered a B_T of 1.98 T at 19.4 K [17]. This B_T was not necessarily larger than that of a smaller HIP MgB_2 bulk (26 mmϕ × 7 mm$t$) [13], although the filling factor ($f \geqslant 90\%$) and $J_c(\mu_0 H)$ of both bulks were nearly identical. The inhomogeneity in the microstructure of the Mg-RLI bulk probably degraded the B_T, which was supported by the non-uniform B_T distribution obtained by pulsed-field magnetization (PFM) for the same bulk [24]; the vortex pinning properties in PFM is known to be quite sensitive to the inhomogeneity of the superconductivity in the bulk. Recently, a homogeneous MgB_2 bulk (32 mmϕ × 6 mm$t$) with finely dispersed Mg particles was successfully obtained by a modified precursor infiltration and growth (MPIG) method [18]; the highest B_T of 2.1 T at 5 K was achieved, which corresponded to that at 20 K for the smaller HIP bulk [13]. To enhance the trapped field properties of the IP MgB_2 bulk as high as that of high-pressure synthesized bulks, we have to resolve the problems of the volume expansion of the B preform at the initial process and of the residuals of unreacted Mg in the MgB_2 bulk. In the present paper, we report on the trapped magnetic-field properties of a highly dense and homogeneous MgB_2 bulk prepared by the infiltration method using a home-made capsule and discuss the potential ability of the IP MgB_2 bulk as a quasi-permanent magnet.

2. Experimental Procedure

2.1. Fabrication of MgB_2 bulk via an infiltration process

A MgB_2 bulk was fabricated by an *in-situ* infiltration method in a closed home-made capsule. As schematically shown in figure 1, the capsule consisted of a pair of stainless steel (SS) containers with holes (30 mm in diameter and 9 mm in depth) and a soft-iron gasket (1.6 mm in thickness) with 58 through holes (2 mm in diameter); the sealing mechanism was the same as that of a commercial CF flange and gasket. Mg powder (99% in purity, $\leqslant$180 μm in grain size, Kojundo

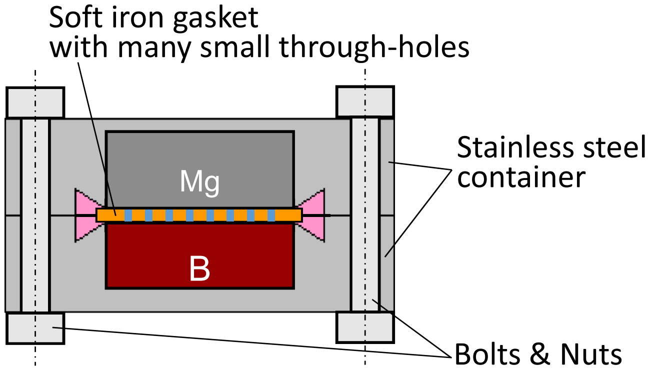

Figure 1. Schematic image of the capsule for the infiltration process.

Chemical Laboratory Co., Ltd) and crystalline B powder (99% in purity, $\leqslant$45 μm in grain size, Kojundo Chemical Laboratory Co., Ltd) were weighed with Mg : B = 1.6 : 2.0 in molar ratio and pelletized individually under a uniaxial pressure of about 30 MPa in air; the excess Mg was expected to compensate for the lack of liquid Mg source. Each Mg and B precursor was set individually in the hole of the stainless steel container, and then sealed with the soft-iron gasket using the bolts and nuts under Ar atmosphere in a glove box. The closed capsule was fired at 900 °C for 9 h in a box furnace and cooled down to room temperature by furnace cooling. Above the melting temperature of Mg, 650 °C, the liquid Mg started to infiltrate into the B pellet through the holes of the gasket, and then MgB_2 was created by a liquid–solid reaction. As-synthesized MgB_2 bulk was machined into a disk with 30 mm in diameter and 6.6 mm in thickness, as shown in figure 2, in which a large amount of the residual unreacted Mg on the bulk surface was fully removed by polishing. Left and right pictures, respectively, are the top surface, which was facing the Mg pellet, and the bottom one (opposite side) of the MgB_2 bulk. The soft-iron gasket could not fully suppress the expansion of the B pellet, resulting in the creation of cracks less than 1 mm in depth on the top surface.

2.2. Method of measurements

The MgB_2 bulk was magnetized at 15.9 K by field-cooled magnetization under a magnetic filed of $\mu_0 H = 5$ Tesla. A trapped field on the top surface of the bulk was measured by a cryogenic Hall probe (HP-VVP, AREPOC s.r.o.) with elevating temperature at a rate of 0.1 K/min. The temperature of the bulk was monitored by a Cernox thermometer which was adhered on the bulk surface by a GE7031 varnish. Magnetization, M, was measured as functions of temperature and magnetic-field using some small pieces cut from the bulk by a commercial SQUID magnetometer (MPMS-XL, Quantum Design Inc.). Magnetic-field dependence of the critical current density, $J_c(\mu_0 H)$, was estimated from the magnetic hysteresis curve, $M(\mu_0 H)$, using the extended Bean model [25, 26], $J_c = 20\Delta M/a(1 - a/3b)$, where ΔM is the width of the hysteresis, and a and b ($a < b$) are the cross-sectional dimensions of the sample perpendicular to the applied magnetic-field. Electrical resistivity, ρ, was measured by a

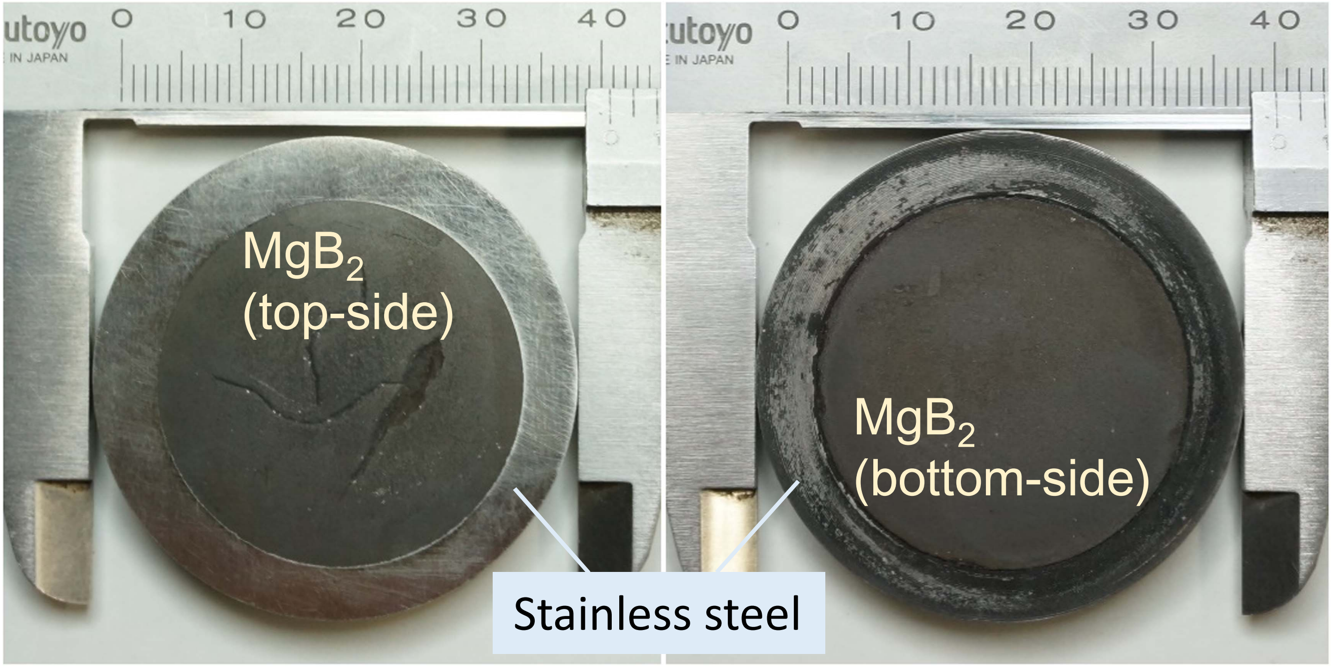

Figure 2. Photographs of the top (left) and bottom (right) surfaces of the infiltration-processed MgB_2 bulk after machining.

standard four-probe method with a typical current density of 1 A cm^{-2}. Microstructure of the bulk surface was observed by a scanning electron microscope (SEM).

2.3. Numerical simulation procedure

Trapped fields of the present bulk were estimated for 10, 20 and 30 K from the numerically simulated electromagnetic fields using a commercial finite-element-method (FEM) software, Photo-Eddy (PHOTON., Ltd., Japan). A small temperature rise of about 0.1 K on the bulk surface during the FCM allowed the simulation under a constant temperature. A simulation model with two-dimensional axisymmetric coordinates was constructed based on the experimental set-up, which was described elsewhere [27]. The power-*n* value model, $E = E_c(J/J_c)^n$, described the nonlinear $E - J$ characteristics, where E the electric field, $E_c = 1\,\mu\text{Vm}^{-1}$ the criterion and $n = 100$ [27]. The experimental $J_c(\mu_0 H)$ was estimated by the following relation,

$$J_c(\mu_0 H) = \alpha\left\{1 - \left(\frac{T}{T_c}\right)^2\right\}^{\frac{3}{2}} \exp\left\{-\left(\frac{\mu_0 H}{\mu_0 H_0}\right)^{\beta}\right\}. \qquad (1)$$

Here, the used parameters were $\alpha(=J_c$ at 0 K), $\mu_0 H_0$ (characteristic magnetic-field) and β.

3. Results and discussion

Figure 3 shows the x-ray diffraction patterns taken at room temperature using the Cu-Kα radiation for both the top and bottom surfaces of the bulk. The dominant MgB_2 phase with a small amount of impurity peaks of Mg_2B_{25} was confirmed, in which the amount of Mg_2B_{25} impurity phase at both the top

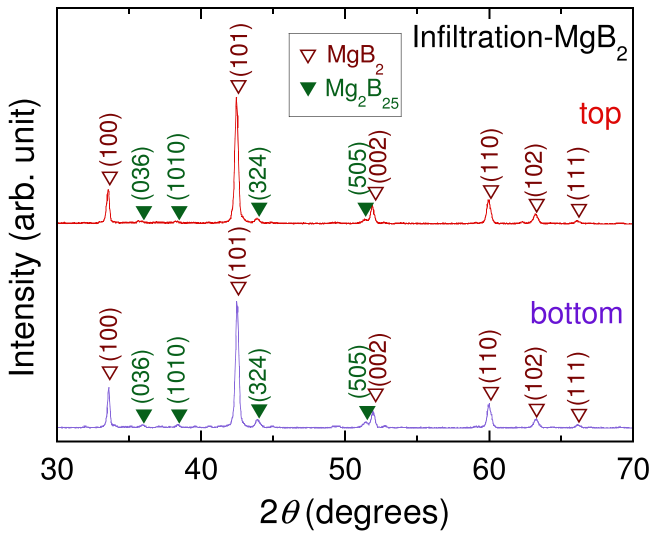

Figure 3. X-ray diffraction patterns of the top and bottom surfaces of the infiltration-processed MgB_2 bulk.

and bottom surfaces are nearly comparable. Giunchi *et al* argued that Mg_2B_{25} was created at the initial stage of the infiltration process and did not prevent the supercurrent flowing [28]. No diffraction peaks of Mg means no residual unreacted Mg in the present bulk. The inset of figure 4 shows the temperature dependence of the normalized magnetization for three positions (top, middle, bottom) of the bulk, which was measured at $\mu_0 H = 0.4$ mT after zero-field cooling. Three pieces were cut from the top and bottom surfaces and the center of the bulk (middle), respectively. Almost the same critical temperatures of about 38.6 K were observed; T_c was defined at the mid-point of the transition. The filling factors, *f*s, evaluated by the Archimedes method, was typically

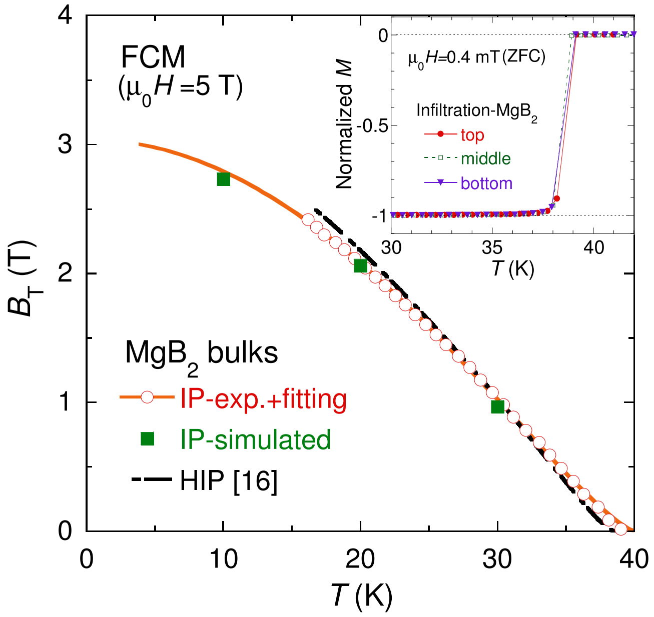

Figure 4. Temperature dependence of the trapped magnetic-field at the top-surface of the infiltration-processed MgB_2 bulk. Open circles and closed squares are the experimental and simulated (see text) B_T values. The thick solid line is a fitting curve as $B_T(T) = B_0\{1 - (T/T_c)^2\}^m$ with the fitting parameters of $B_0 = 3.01$ T and $m = 1.25$. The thick dashed-dotted line is the $B_T(T)$ of the HIP processed MgB_2 bulk (HIP#38 [16]). The inset shows the temperature dependence of the normalized magnetization at $\mu_0H = 0.4$ mT after zero-field cooling for the indicated three positions of infiltration-processed MgB_2 bulk.

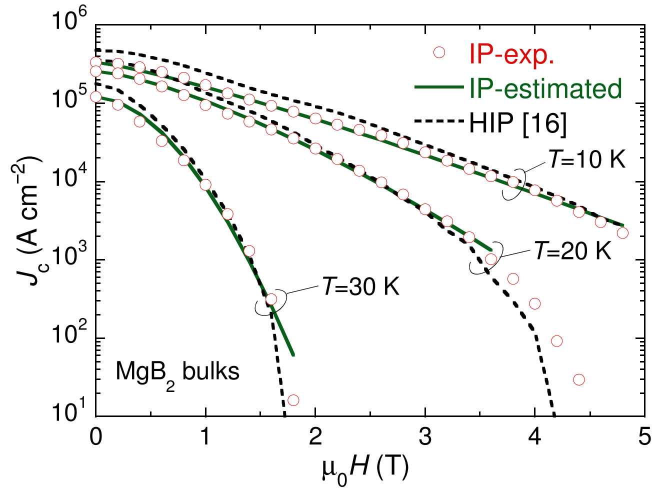

Figure 5. Magnetic-field dependence of the critical current density, $J_c(\mu_0H)$, at 10, 20, 30 K for the infiltration-processed MgB_2 bulk. The thick solid lines are the $J_c(\mu_0H)$'s estimated by the equation (1) with the appropriate parameters (see text), which were used to obtain the simulated B_T in figure 4. The dotted lines are the $J_c(\mu_0H)$ of the HIP processed MgB_2 bulk (HIP#38 [16]).

~90%. These results demonstrate that a highly dense MgB_2 bulk with homogeneous composition and superconductivity was obtained.

The main panel of figure 4 shows the trapped magnetic-field, as a function of temperature, $B_T(T)$, for the present IP bulk. The $B_T(T)$ curve of the HIP MgB_2 bulk ($f \sim 95\%$, 38 mmϕ × 7 mm$t$), named as HIP#38, is also shown for comparison [16]. The B_T of 2.4 T was achieved at 15.9 K and B_T decreased monotonically with increasing temperature due to the J_c-decrease and reached zero at 39.6 K due to the temperature-gradient in the bulk. The $B_T(T)$ value of the present IP bulk was comparable to that of the HIP#38 bulk, regardless of the 20% smaller diameter and smaller f of ~90%. Moreover, comparing at the same temperature of 19.4 K, the B_T of 2.12 T for the present IP bulk (30 mmϕ × 6.6 mm$t$) evidently breaks the record-high B_T of 1.98 T for the Mg-RLI bulk (55 mmϕ × 10 mm$t$) [17]. The home-made capsule with soft-iron gasket almost suppressed the expansion of the bulk along both the radial and thickness directions, resulting in the highest B_T among the IP MgB_2 bulks to date.

Figure 5 shows the magnetic-field dependence of the critical current density, $J_c(\mu_0H)$, at $T = 10$, 20 and 30 K for the present bulk. The $J_c(\mu_0H)$'s of the HIP#38 bulk at the same temperatures are also depicted as a reference [16]. All the $J_c(\mu_0H)$ curves decreased monotonically with increasing magnetic-field, which is commonly observed in MgB_2. The magnitude of $J_c(\mu_0H)$ of the present IP bulk was comparable with that of the HIP#38 bulk, except for the low fields. The irreversibility fields, μ_0H_{irr}'s, of 4.5 T at 20 K and of 1.8 T at 30 K obviously exceeds those of the HIP#38 bulk, where the μ_0H_{irr} was defined as the magnetic-field at $J_c = 10$ A cm^{-2}. The $J_c(\mu_0H)$ behaviors support the excellent B_T characteristics of the present bulk. As shown in the main panel of figure 4, the B_T's at 20 and 30 K simulated using the experimental $J_c(\mu_0H)$ characteristics well reproduced the experimental B_T's, and the simulated B_T was 2.7 T at 10 K. The $J_c(\mu_0H)$'s used in the simulation are depicted in figure 5, which were estimated using the following parameters; $\alpha = 4.0 \times 10^5$ A cm^{-2}, $T_c = 39.6$ K and the combinations of μ_0H_0 and β were 1.3 T and 1.2 for 10 K, 1.1 T and 1.4 for 20 K and 0.5 T and 1.2 for 30 K. The simulation results indicate strongly the good agreement between the microscopic J_c of the millimeter-size specimen and macroscopic J_c of the centimeter-size bulk, and then the homogeneous structure and composition of the present bulk in these scales. The reproducibility of B_T in the simulation using the experimental $J_c(\mu_0H)$ were reported previously for the other MgB_2 bulks [27, 29]. Assuming the relationship between the trapped field and irreversibility field, $\mu_0H_{irr} \propto \{1 - (T/T_c)^2\}^m$ [30], both experimental and simulated $B_T(T)$ was well fitted by $B_T(T) = B_0\{1 - (T/T_c)^2\}^m$ with a B_T at 0 K, $B_0 = 3.01$ T and a characteristic exponent, $m = 1.25$, as shown in figure 4. The estimated $B_T(T)$ of 3 T at 4.2 K confirms evidently the potential ability of the present IP bulk trapping 3 T class magnetic-field on the bulk surface.

Figure 6 shows the temperature dependence of the resistivity, $\rho(T)$, for the present bulk. The $\rho(T)$ of the HIP#38 bulk is also shown as a reference [31]. The zero-resistivity transition temperature of 38.6 K, defined at the mid-point of the transition, agreed well with that of the magnetic transition. More than five times larger $\rho(T)$ is observed for the present IP bulk compared to the HIP#38

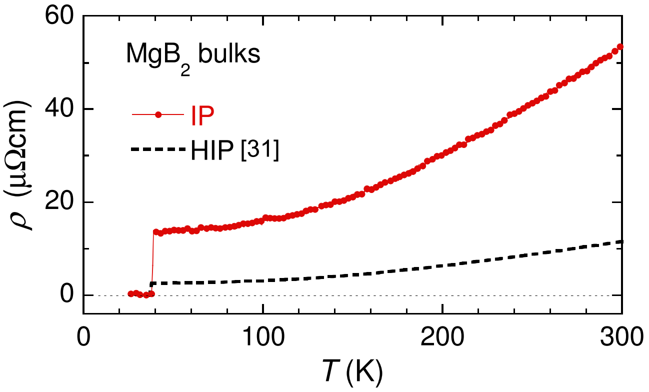

Figure 6. Temperature dependence of the resistivity for the infiltration-processed MgB_2 bulk. The dotted line is the $\rho(T)$ of the HIP processed MgB_2 bulk (HIP#38 [31]).

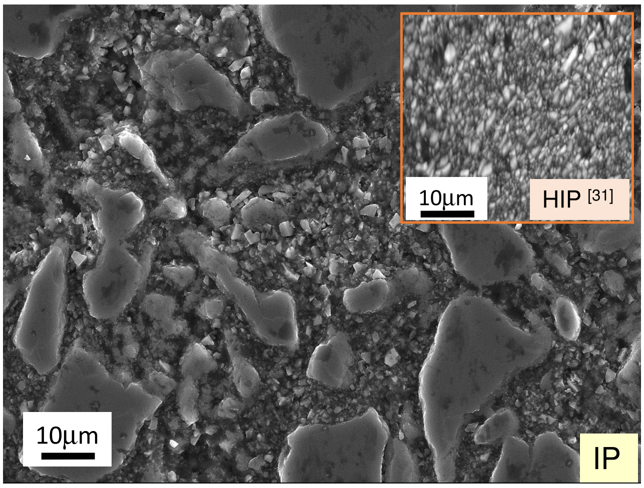

Figure 7. SEM images with the same scales of the top surfaces of the infiltration-processed (main panel) and of the HIP-processed (inset; HIP#38 [31]) MgB_2 bulks.

bulk, although there is a small difference in the filling factor, less than 10%, between them. We evaluated the effective cross-section for the supercurrent-path in the bulk by estimating the connectivity, K, using the Rowell's relation, $K = (\Delta\rho_s/\Delta\rho_p) \times 100$ (%) [32]. Here, $\Delta\rho_s$ and $\Delta\rho_p$ are the difference in the resistivity between 300 K and 40 K for the single crystal and the polycrystal, respectively. In this relation, the connectivity was assumed to be 100% for the single crystal, and we used $\Delta\rho_s = 6.32\ \mu\Omega$cm [33]. The K value was estimated to be 16%, which is rather smaller than 75% of the HIP#38 bulk [31]. The main panel of figure 7 shows a typical SEM image of the top surface of the present bulk; note that almost the same microstructure was also observed at the middle and bottom positions. In contrast to the microstructure consisting of nanometric grains for the HIP#38 bulk [31], shown in the inset of figure 7, the large island-shaped MgB_2 grains with a few tens of microns in size and the assembly of small grains with micron and sub-micron in size were observed in the present bulk. It is noteworthy that no unreacted Mg particle was observed, which is consistent with the XRD result. The wide variation of the MgB_2 grain-size probably originated from the similar distribution of grain size of the as-purchased B powder, revealed by the SEM image (not shown here), as reported previously [34]. These grains do not seem to connect well with each other, which is qualitatively consistent with the estimated small K value of 16% and the high resistivity; in particular, the island-shaped grains prevent the supercurrent flowing. To explain the high trapped field (high-J_c) with low connectivity for the present IP bulk, we suggest the possibility of the coexistence of high- and low-J_c regions owing to the different grain sizes. A MgB_2 wire fabricated by the internal Mg diffusion (IMD) method, which is a kind of infiltration process, is well known to offer a high-J_c of about 10^5 A cm^{-2} under 3 T at 20 K [35]. This J_c is about two orders of magnitude larger than that of the present bulk at the same condition. If the similar high-J_c MgB_2 regions exist locally in the present IP bulk, the high-B_T with low-K can be explained. A direct observation of the supercurrent profiles in the bulk by a scanning Hall probe microscopy [36] probably confirms this scenario.

4. Summary

In summary, we have studied the trapped magnetic-field, B_T, properties of a MgB_2 bulk fabricated by the infiltration method in a home-made capsule. The highly dense MgB_2 bulk free from the residual unreacted Mg (filling factor, f, of ~90%, 30 mm in diameter and 6.6 mm in thickness) was successfully obtained. This bulk offered a record-high B_T of 2.4 T at 15.9 K among the infiltration processed MgB_2 bulks, which was comparable with those of the high-pressure synthesized bulks. The critical current densities at 20 K were about 1.8×10^5 A cm^{-2} in the self-field and 4.5×10^3 A cm^{-2} under $\mu_0 H = 3$ T. The irreversibility field was 4.5 T at 20 K. A good consistency was found for the experimental and simulated B_T's, and the extrapolation of which reached 3 T at 4.2 K, suggesting the potential ability of bulk trapping 3 T on the bulk surface. The effective cross-sectional ratio, K, proposed by Rowell, for the supercurrent path was estimated to be as low as 16%, which was probably caused by the island-shaped large MgB_2 grains a few tens of microns in size. The inconsistency among the high B_T, high J_c and low K remains an unsolved problem.

Acknowledgments

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