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Effects of sintering condition on the trapped magnetic field properties for MgB_2 bulks fabricated by *in-situ* capsule method

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Abstract

We have studied the trapped field properties of MgB_2 bulks which were fabricated by the *in-situ* capsule method under various sintering temperatures of 700-900 °C for 1-24 h. The trapped field, $B_T(T)$, was 1.8-1.9 T for all the bulks approximately at 16 K. The $B_T(T)$ values on the bulks sintered at 900 °C for 6 h and at 800 °C for 24 h were slightly lower than those of other bulks because of the decrease of critical current density, J_c, which originates from both the lower connectivity, K, and the promotion of the grain growth. The high temperature and/or the long periods sintering decrease the $B_T(T)$ value. From the obtained results, the sintering at 700-800 °C for 1-6 h is the optimum condition to fabricate MgB_2 bulks by the capsule method.

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Keywords: Superconducting bulk magnet; MgB_2 superconductors; Trapped field properties

1. Introduction

MgB_2 has the highest superconducting transition temperature T_c=39 K among intermetallic compounds. Since its coherence length is long [1] in comparison with RE-Ba-Cu-O (RE: rare earth elements) system, which allows us to discard the weak-links at grain boundaries, we can produce a superconducting bulk magnet using a polycrystalline MgB_2. However, MgB_2 must be fabricated in vacuum or Ar-atmosphere because Mg and B are oxidized in air. Therefore the complicated fabrication process is needed.

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Several groups reported that the MgB_2 bulks trapped the tesla-order magnetic field [2-5]. We reported the trapped magnetic field B_T of 1.77 T at 15.5 K on the MgB_2 bulk with 38 mm in diameter and 9 mm in thickness, which was fabricated by the *in-situ* capsule method [2]. Viznichenko *et al*. reported the B_T value of about 2.3 T at 6 K for the MgB_2 bulk with 28 mm in diameter and 11 mm in thickness which was sintered under high pressure of 2 GPa [3]. Tomita *et al*. reported the B_T of 2.1 T at 20 K for the *in-situ* MgB_2 bulk with 60 mm in diameter and 10 mm in thickness and 3.5 T at 13.5 K for the disc-pair bulks with 40 mm in diameter and 10 mm in thickness [4]. And also, Durrel *et al*. reported the B_T value of over 3 T at 17.5 K was obtained for stack of the disc-pair MgB_2 bulks of diameter 25 mm and thickness 5.4 mm [5].

The sintering condition of the reports is different from each other. The culminant pinning centers are the grain boundaries in MgB_2, therefore the trapped field properties should be optimized by controlling the grain size and boundaries. In this paper, to optimize the grain condition, we have studied the trapped field and superconducting properties of MgB_2 bulks sintered under various conditions.

2. Experimental

2.1. Sample preparation

MgB_2 bulks were fabricated by the capsule method [2]. Figure 1 shows the schematic image of the capsule. The precursor pellet was prepared from mixed powders of Mg (99.5% in purity, ≦180 μm in grain size) and B (99% in purity, 300 mesh in grain size) with the molar ratio of 1:2. It was set in the stainless steel (SUS) container, and was sealed with both soft iron gasket and SUS cap in Ar atmosphere. The sealed capsule was sintered at the temperature T_{sin} = 700-900 °C for the duration t_{sin} = 1-24 h in a box furnace, and cooled down to room temperature by furnace cooling. The typical size of the bulk is 30 mm in diameter and about 10 mm in thickness. Each bulk was named as listed in Table 1. The packing factor of the bulks was about 50%, which was also shown in Table 1.

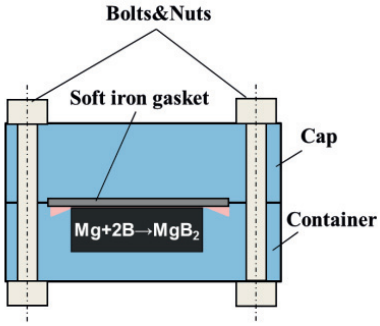

Fig. 1. Schematic illustration of the closed capsule.

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 -0.222 T/min. Trapped field B_T value at the center of the bulk surface was measured by a cryogenic Hall sensor (BHT-921, F.W. BELL). After the FC experiment, we measured the magnetization curve of several small pieces cut from the bulk using a commercial SQUID magnetometer (MPMS-XL, Quantum Design Inc.), and evaluated the critical current density J_c using the extended Bean model. Electrical resistivity ρ was measured by a standard four probe technique. The micro structure of the MgB_2 bulks was observed by a scanning electron microscope (SEM).

3. Results and discussion

Figure 2(a) presents the temperature dependence of the trapped field, $B_T(T)$, of the MgB_2 bulks with the different sintering temperature T_{sin} and for constant t_{sin} (= 6 h). The maximum $B_T(T)$ values of the CAP1, CAP2 and CAP3 bulks, respectively, were 1.92 T at 16.7 K, 1.95 T at 16.3 K and 1.82 T at 16.3 K. The $B_T(T)$ value of the CAP3 bulk

Table 1. Specification of the MgB_2 bulk.

Sample name	T_{sin} (°C)	t_{sin} (h)	Packing factor (%)	T_c (K)	Connectivity K (%)	Max of B_T
CAP1	700	6	46.6	38.8	16.1	1.92 T at 16.7 K
CAP2	800	6	50.8	38.9	19.0	1.95 T at 16.3 K
CAP3	900	6	48.7	39.0	17.3	1.82 T at 16.3 K
CAP4	800	1	49.5	39.0	17.6	1.42 T at 23.6 K
CAP5	800	24	47.7	38.8	14.0	1.79 T at 15.2 K

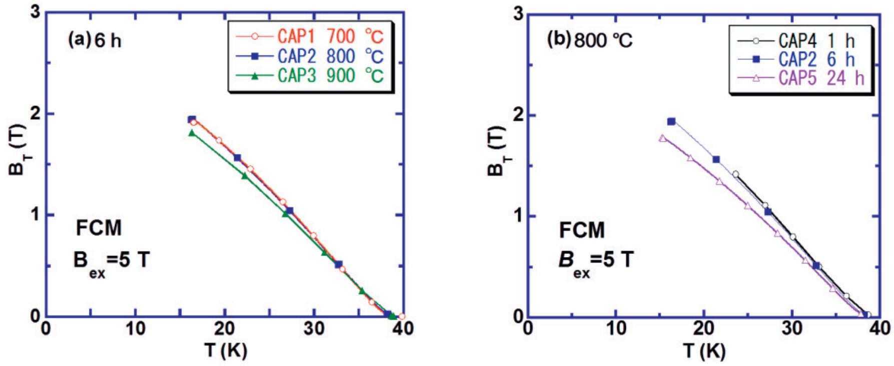

Fig. 2. Temperature dependence of trapped field B_T (T) for the (a) variation of temperature 700-900 °C (t_{sin} = 6 h) and (b) variation of sintering period 1-24 h (T_{sin} = 800 °C).

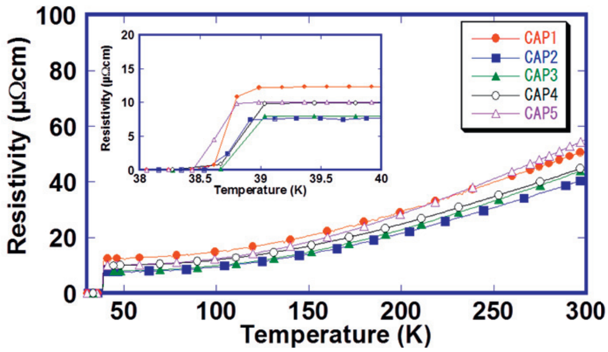

Fig. 3. Temperature dependence of resistivity. Inset shows the expanded view near T_c.

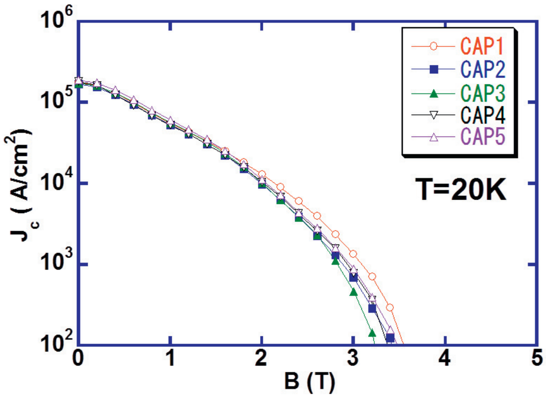

Fig. 4. Magnetic field dependence of the critical current density J_c at 20 K for the MgB_2 bulks.

was slightly lower than those of the other bulks. Figure 2(b) shows the B_T (T) of the MgB_2 bulks with the different sintering time t_{sin} and at constant T_{sin} (= 800 °C). The maximum B_T (T) values of the CAP4 and CAP5 bulks, respectively, were 1.42 T at 23.6 K and 1.79 T at 15.2 K. The B_T (T) value of the CAP5 bulk was lower than those of other bulks. Therefore, these results suggest that the sintering at higher temperature and/or for the long-time, decrease the B_T (T) value. The B_T (T) of the CAP1, CAP2 and CAP4 bulks were comparable.

The observed XRD peaks for all the bulks were mainly indexed by the MgB_2 phase, but a small amount of impurity such as Mg was observed. Both CAP3 and CAP5 bulks with lower B_T (T) value did not necessarily contain many impurities compared with other bulks.

Figure 3 shows the temperature dependence of the resistivity $\rho(T)$ for each bulk. Inset shows the expanded view near the critical temperature, T_c. The onset T_c of each bulk was 38.8-39.0 K. The electrical connectivity K, which is the effective cross section of the bulk for the supercurrent path, was evaluated from the following expression [6], $K = \Delta\rho_g / \Delta\rho \times 100$, where $\Delta\rho_g = \rho_g(300\text{ K}) - \rho_g(40\text{ K}) \equiv 6.32$ μΩcm [7], and $\Delta\rho = \rho(300\text{ K}) - \rho(40\text{ K})$, i.e., the ratio of the resistivity difference between resistivity of the ideal MgB_2 grains and that of the present sample. The connectivity value of each bulk is shown in Table 1. The K value of CAP5 was 14%, which is lower than those of other bulks and is consistent with the lower B_T (T) of the CAP5 bulk.

Figure 4 shows the magnetic field dependence of the critical current density J_c at 20 K for each bulk. The J_c value in the zero field of each bulk was 1.2-1.6 $\times$ 10^5 A/cm^2. J_c and irreversibility fields, B_{irr}, of the CAP1 bulk are somewhat higher, and those of the CAP3 bulk are lower among these bulks. Although both packing factor and connectivity values of the CAP1 bulk were slightly lower than other bulks, the higher J_c and B_{irr} give rise to the high B_T (T) similarly to that of the CAP2 bulk. On the other hands, for the CAP3 and CAP5 bulks, the B_T (T) values were

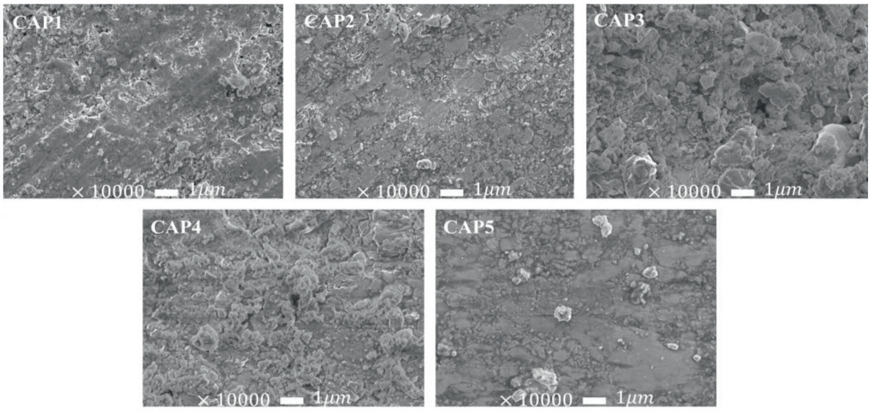

Fig. 5. SEM images of the MgB_2 bulks

low relatively, which originate from the low P, K, J_c and B_{irr} values for the CAP3 and from the low P and K values for the CAP5, respectively.

Figure 5 shows the SEM images of the bulk surface. The sizes of the grains were smaller than several microns for all the bulks. However, some of the grains in the CAP3 and CAP5 bulks became large by gain growth, which was promoted for the bulks fabricated at higher temperature and for the long-time sintering. As a result, the grain boundaries, which act as pinning centers in MgB_2 bulk, were decreased by the grain growth and the B_T (T) value decreased in both CAP3 and CAP5 bulks.

4. Conclusion

We have investigated the trapped field B_T, the critical current density J_c, connectivity K and microstructure of the MgB_2 bulks fabricated by the capsule method. The important obtained results are described in the followings.

(1) The MgB_2 bulks fabricated at 700-900 °C for 1-24 h show nearly the single phase, and the B_T (T) was 1.8-1.9 T for all the bulks approximately at 16 K.

(2) The $B_T(T)$ values on the bulk sintered at 900 °C-6 h and at 800 °C-24 h bulk were slightly lower than those of other bulks because of the low K, and J_c values and the enhancement of the grain growth. The sintering at the high temperature and/or for the long period, decrease the B_T (T) value.

(3) The J_c value in the zero field of each bulk was 1.2-1.6 $\times$ 10^5 A/cm^2. J_c and B_{irr} of the 700 °C-6 h bulk are somewhat higher, and those of the CAP3 bulk are lower among these bulks.

(4) The obtained results suggest that the optimum fabricating condition in the capsule method is the sintering temperature of 700-800 °C and the sintering period of 1-6 h.

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