A Proposal of New Fabricating Technique of Large MgB_2 Bulk by a Capsule Method

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***Abstract*—We proposed a new fabricating technique of a large MgB_2 superconducting bulk (20–30 mm in diameter) by a capsule method and measured the trapped field distribution for the MgB_2 bulks. The capsule consists of a couple of commercial stainless steel flanges, a stainless steel plate and a copper gasket. The MgB_2 bulks showed a superconducting transition at 38.3–38.6 K with a transition width of 0.6–1.1 K. The trapped magnetic field of the center of the bulk surface reached 1.65 T at 14 K for the bulk 30 mm in diameter. These results indicate that the capsule method is useful to produce the tesla-order large MgB_2 bulk magnet.**

***Index Terms*—Capsule method, MgB_2, superconducting bulk magnet, trapped magnetic field.**

I. INTRODUCTION

A BULK superconductor which traps the magnetic field can be used as a strong permanent magnet. A single grain of RE-Ba-Cu-O (RE:rare earth elements) superconducting bulk fabricated by a top seeded melt-growth method [1]–[3] is one of the promising materials for a bulk magnet because of their high superconducting transition temperature and high critical current density below 77 K. Actually, a Y-Ba-Cu-O bulk reinforced by an epoxy resin trapped the magnetic field of 17 T at 29 K by field cooling [4], which is one or two orders of magnitude larger than that of a permanent magnet. In view of the Bean's critical state model [5] the trapped magnetic field is proportional to the diameter and critical current density, J_c, therefore a larger bulk with a large J_c is desirable. To obtain the single grain RE-Ba-Cu-O bulk with diameter over 100 mm, we have to use a special technique [6]–[8].

Because of the developments of the fabrication techniques, the polycrystalline MgB_2 samples showed a high J_c over 10^5 A/cm^2 [9], [10], which is as high as that of the single grain RE-Ba-Cu-O bulks [11]. Since MgB_2 has a long coherence length, the problem of the weak link can be ignored even for use of polycrystals [12].

Several groups have already developed the MgB_2 bulk magnet [13]–[15]; the MgB_2 bulk (28 mm in diameter and 11 mm in thickness) fabricated under high pressure, trapped a magnetic field of about 2.3 T at 6 K [13]. The 1.2 T MgB_2 bulk magnet (20 mm in diameter) at 17 K was also reported [14]. Therefore, the MgB_2 bulk is a promising material to produce a large superconducting bulk magnet.We have developed a capsule method for fabricating a large MgB_2 bulk. In this paper, the superconducting properties and the trapped magnetic field of MgB_2 bulks fabricated by a capsule method, are evaluated, and the possibility of the capsule method are discussed.

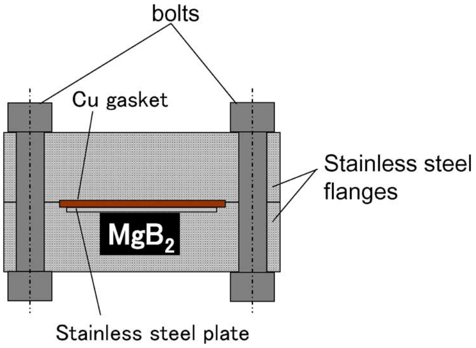

Fig. 1. Schematic image of the capsule.

II. EXPERIMENTAL

A. Preparation of MgB_2 Bulks

MgB_2 bulks were prepared by the capsule method. Fig. 1 shows the schematic image of the capsule. The capsule consists of a couple of commercial stainless steel flanges (70 mm in diameter, 10 mm in thickness; ICF flange), a stainless steel plate and a copper gasket. Raw powders of Mg and B were weighted with 1.1:2.0 in molar ratio and ground. The mixture was pressed into pellets 20–30 mm in diameter and 5–9 mm in thickness under an uniaxial pressure. The precursor pellet was prepared in air, and the preparation of the capsule was performed in Ar-atmosphere using a glove box. The precursor pellet was set in the hole of the stainless steel flange, covered by the stainless steel plate, and finally closed by the other flange with the copper gasket. The closed capsule was sintered at 600–800 °C for 6–48 h in a box furnace and cooled down to room temperature by furnace cooling. Sample specifications are listed in Table I. Fig. 2 shows the photograph of the #20 bulk 20 mm in diameter. The circle at the center is the MgB_2 bulk. Superconducting transition temperature, T_c, at the mid-point of the transition and the transition width, ΔT_c, were obtained by the magnetization measurements. Powder X-ray diffraction pattern (not shown here) was taken at the room temperature and the almost single-phase

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TABLE I
SPECIFICATIONS OF THE MgB_2 BULKS

Sample name	diameter (mm)	thickness (mm)	sintering condition	T_c (K)	ΔT_c (K)
#20	20.2	5	800 °C, 6 h	—	—
#30-1	32.0	9	800 °C, 6 h	38.3	0.8
#30-2	30.2	9	600 °C, 48 h	38.4	1.1

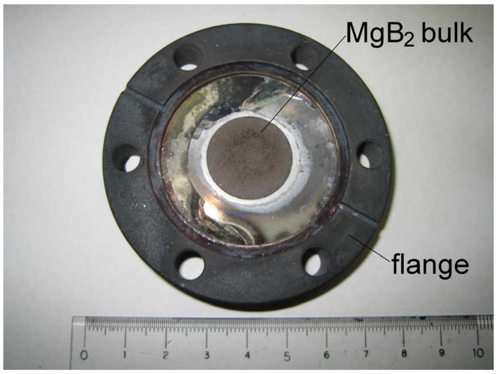

Fig. 2. Photograph of the #20 MgB_2 bulk (center circle) with the stainless steel flange.

structure was confirmed with small amount impurities such as MgO and $MgCu_2$.

B. Measurements

To measure the trapped magnetic field, the MgB_2 bulk was set on a cold head of a Gifford-McMahon cycle helium refrigerator by inserting a thin indium plate to obtain good thermal contact, and cooled down under a magnetic field of 5 T using a 10 T cryogen-free superconducting magnet. The temperature of the bulk was measured by a Cernox thermometer mounted on its top surface. Trapped magnetic field was measured by a cryogenic Hall sensor (BHT-921, F.W. Bell Inc.) adhered on the center of the bulk surface. We also measured the profile of the trapped magnetic field at 4 mm up from the bulk surface by scanning the axial-type Hall sensor. The superconducting properties were evaluated by measuring the resistivity and the magnetization using a small piece cut from the bulk after the measurement of the trapped magnetic field. Resistivity was measured by a standard four probe technique. Magnetization was measured under the magnetic field of 0.4 mT after zero-field cooling using a commercial superconducting interference device (SQUID) magnetometer (MPMS-XL, Quantum Design Inc.).

III. RESULTS AND DISCUSSION

A. Trapped Magnetic Field

Fig. 3 shows the temperature dependence of the trapped magnetic field, $B_{\mathrm{T}}(T)$, of the MgB_2 bulks. The $B_{\mathrm{T}}(T)$ lines in the previous reports [13], [14] are also shown. The B_{T} value of #20 bulk is 1.3 T at 17 K, which is slightly larger than the reported one of 1.2 T at the same temperature for the MgB_2 bulk with 20 mm in diameter [14]. The quality of our bulk seems to be comparable to that of the reported one. The $B_{\mathrm{T}}(T)$ lines of #30-1 and -2 bulks coincide with each other and increase with decreasing temperature. These results suggest that the critical current density, J_c, increases with decreasing temperature. The highest B_{T} value is 1.65 T at 14 K for #30-2 bulk. This value is somewhat smaller than the reported one which was achieved for the bulk with 28 mm in diameter [13]. This probably come from the fact that the MgB_2 bulk in Ref. [13] was fabricated under high pressure, i.e., their bulk is denser than ours. The $B_{\mathrm{T}}(T)$ curves of #30-1 and -2 bulks suggest that the superconducting characteristics do not depend on the sintering condition. The magnitude of B_{T} at 17 K for #30-1 and -2 bulks is approximately 1.5 T, which is estimated by the interpolation of the $B_{\mathrm{T}}(T)$ curve. This B_{T} value is about 1.2 times larger than that of #20 bulk (20 mm in diameter), the value of which is less than 1.5 expected from the ratio of the diameter. Because Bean's critical state model gives the fact that the trapped magnetic field is proportional to the diameter of a disk-shaped bulk, if the J_c is the same. Thus, this might originate from the difference in $J_c(B,T)$ properties between 20 mmϕ and 30 mmϕ bulks. Fig. 4 shows the B_{T} distribution at 30 K for #30-2 bulk. The symmetric conical-shaped

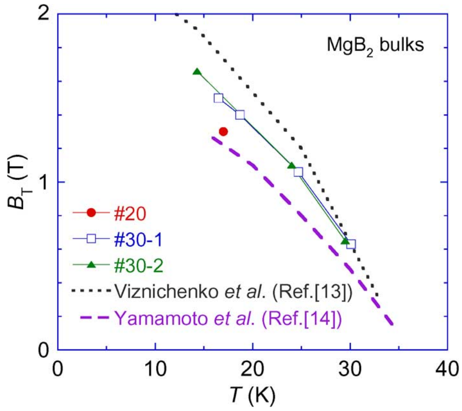

Fig. 3. Temperature dependence of the trapped magnetic field of the MgB_2 bulks.

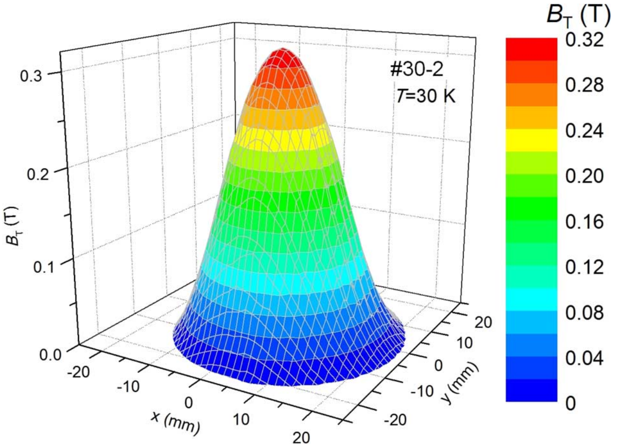

Fig. 4. Profile of the trapped magnetic field of the #30-2 bulk at 30 K.

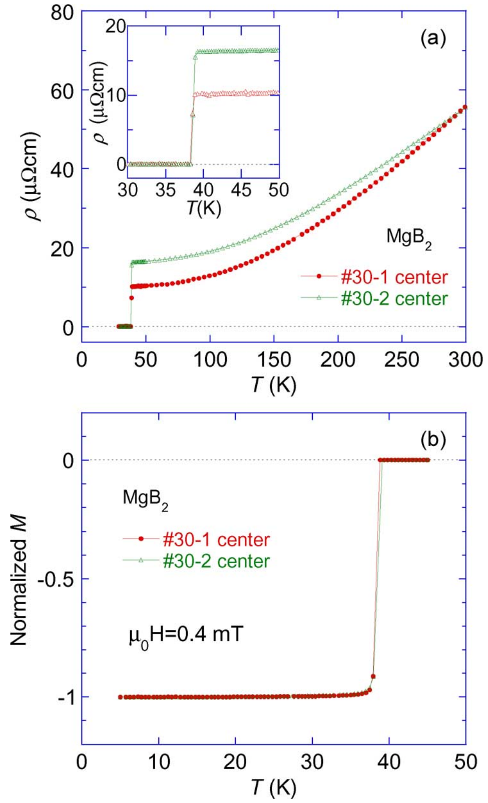

Fig. 5. Temperature dependence of (a) the resistivity and (b) the normalized magnetization of the small pieces cut from the center of the #30-1 and −2 bulks.

B_T profile indicates that superconducting characteristics of the MgB_2 fabricated by the capsule method is homogeneous. To study deeply the mechanism of the trapping magnetic field in the MgB_2 bulks, the evaluation of J_c distribution using small pieces cut from the bulk is now in progress.

B. Superconducting Properties

Fig. 5(a) and (b), respectively, show the temperature dependence of the resistivity and the normalized magnetization of the small pieces cut from the center of the #30-1 and -2 bulks. Resistivity decreases monotonically with decreasing temperature and shows the sharp superconducting transition. Inset of Fig. 5(a) is the enlarged figure around transition region. The superconducting transition temperatures, T_c's, defined at the midpoint of the transition, are approximately 38.5 K for #30-1 bulk and 38.6 K for #30-2 one. The transition widths, ΔT_c's, are about 0.6 K for #30-1 bulk and 1.0 K for #30-2 one. In Fig. 5(b), the reduced magnetization also demonstrates the sharp superconducting transition, and the T_c's at the midpoint and ΔT_c's are approximately 38.3 K and 0.8 K for #30-1 bulk and 38.5 K and 1.1 K for #30-2 one. These results indicate that almost the stoichiometric MgB_2 bulks are fabricated by the capsule method.

IV. SUMMARY

We have fabricated MgB_2 bulks (20–30 mm in diameter and 5–9 mm in thickness) by a capsule method using commercial stainless flanges, a stainless steel plate and a copper gasket and evaluated their superconducting properties and ability to trap the magnetic field. 1.65 T bulk magnet was realized at 14 K using a bulk with 30 mm in diameter, of which magnetic field was somewhat less than the MgB_2 bulk magnet (28 mm in diameter) synthesized under high pressure. The superconducting transition temperature and width of the transition were about 38.3–38.6 K and 0.6–1.1 K, respectively. These results indicate the MgB_2 bulks fabricated by the capsule method have good superconductivity and high ability to trap the magnetic field. Our capsule method is found to be useful to produce larger MgB_2 bulk magnets than 50 mm in diameter.

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