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Optimization of processing conditions towards high trapped fields in MgB_2 bulks

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ABSTRACT

The present investigation focuses on the effects of various sintering temperatures on the critical current densities and the trapped field values of disk-shaped bulk MgB_2 superconductors fabricated with a simple solid state reaction. The samples were prepared by varying the sintering temperature from 750 to 950 °C in pure Ar atmosphere. Scanning electron microscopy (SEM) and X-ray diffraction analyses showed that single phase and homogenous MgB_2 bulks are produced in using sintering temperatures in the range of 750–825 °C. The samples sintered at 775 °C showed the highest critical current density (J_c) values of 250 kA/cm^2 at 10 K and 181 kA/cm^2 at 20 K in self field. We also measured the trapped field values at 20 K for bulk MgB_2 samples 20 mm in diameter and 7 mm in thickness, sintered at temperatures in the range of 700–950 °C with the same sintering duration of 3 h. Almost all the samples exhibited the trapped field values higher than 1 T, which shows the high potential of sintered MgB_2 bulk materials as trapped field magnets. The highest value of 1.51 T at 20 K was achieved in the MgB_2 sample sintered at 775 °C, reflecting its high pinning performance and homogeneous microstructure.

1. Introduction

Since the discovery of superconductivity in MgB_2 at 39 K [1], enormous efforts have been made on processing, characterization, and applications of MgB_2 material [2–6]. The superconducting transition temperature of MgB_2 is significantly lower than that of the $YBa_2Cu_3O_y$ "Y-123", but MgB_2 benefits from BCS-like superconducting features like a large coherence length and thus, a high critical current density (J_c) can be obtained in the polycrystalline state, which makes these materials still promising candidates for several industrial applications [7]. Other advantages of the bulk MgB_2 material are cheap raw materials, light weight, and short processing time [8].

Practical applications of bulk MgB_2 magnets are similar to those of melt-textured Y-123 [9], where the most attractive applications are trapped field magnets which can be employed in nuclear magnetic resonance (NMR) and magnetic resonance imaging (MRI), fault current limiters, non-contact bearings for liquid pumping, and for magnetic shielding screens [9,10]. All these applications require high critical current densities and good quality, large materials.

As reported earlier, flux pinning enhancement in MgB_2 materials could be achieved by using SiC particles as additives [11]. The transport J_c values at liquid helium temperature and 10 T for silicon-doped MgB_2 samples reached the level of 10^5 A/cm^2. Similar improvements were also observed with additions of carbon, boron carbide, and carbon nano-tubes [12,13]. Recent reports have proved that the J_c values at liquid helium temperature in high fields could be also improved with the addition of carbohydrates or hydrocarbons or a tritiation process [14–17]. On the other hand, densification was also found to be effective in enhancing J_c values of MgB_2. A variety of high pressure techniques have been employed to produce highly dense MgB_2 material [18]. Without employing external high pressure, densification is also possible with a reactive Mg-liquid infiltration process [19].

Recent reports have demonstrated that the MgB_2 bulk magnets are suitable for operation at 20 K [20–22]. Several groups have already reported the trapped field performances of bulk MgB_2 materials. High field trapping abilities of MgB_2 bulk superconductors about 1 T at around 20 K was first demonstrated by Yoo and Murakami during the IWCC 2003 [23]. Viznichenko et al. [24] reported trapped field values of around 1.5 T at 20 K and 2.3 T at 6 K for uniform polycrystalline MgB_2 material with a diameter of 28 mm and 11 mm height produced under a high pressure of 2 GPa. Subsequently, Giunchi [25] announced a trapped field of

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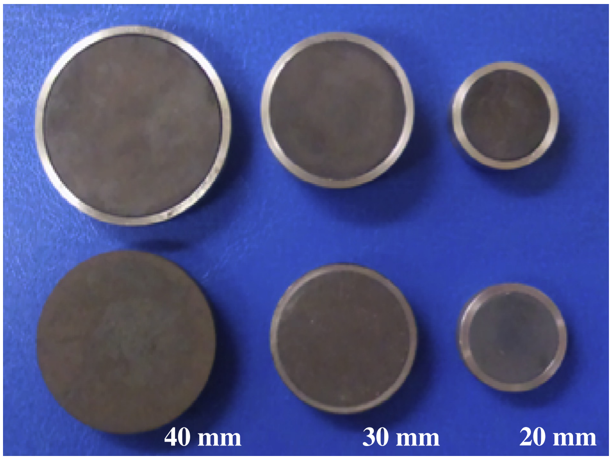

Fig. 1. Photos of bulk MgB_2 samples 20, 30, and 40 mm in diameter, produced by sintering process in Ar atmosphere. The samples are reinforced with stainless steel rings for trapped field measurements. One sample without a ring is in the as-grown state.

around 1.3 T at 15 K in a MgB_2 bulk sample with 55 mm in diameter and 15 mm in height. Yamamoto et al. [26] reported that a MgB_2 bulk of 20 mm diameter and 5 mm thickness made with a simple sintering method exhibited trapped field of around 1.2 T at 20 K. The trapped field values are dramatically improved when the value was measured between two stacked samples, mainly due to the minimization of the demagnetizing effect. A stack of two MgB_2 samples with a diameter of 30 mm showed a trapped magnetic field of 3 T at 17 K [20]. Similar trapped field values were obtained at 17.5 K in a magnetized stack of two disc-shaped, bulk MgB_2 superconductors with a diameter of 25 mm and a thickness of 5.4 mm fabricated by uni-axial hot pressing [21]. More recently, Naito et al. [27] also reported maximum trapped fields at the center of the bulk surfaces of 1.43 T at 13.4 K and 1.5 T at 16.4 K.

For future engineering applications, a simple processing route is attractive for mass production of bulk MgB_2 materials that can then be used for a wide range of commercial applications. Hence, it is desirable if one can achieve high trapped fields in the samples fabricated with a simple solid state reaction route like sintering.

In this paper, we report on the superconducting performances and trapped field values for MgB_2 materials fabricated with a simple sintering method, having the future mass production in mind. We fixed the initial composition of the samples to Mg:B = 1:2 and sintered them at various temperatures ranging from 700 to 950 °C for 3 h. Trapped field measurements showed that the processing temperature was the key to obtaining high trapped field values in the bulk MgB_2 materials. We also confirmed that the improvement in the field trapping ability was possible even for sintered MgB_2 bulk superconductors when the processing conditions were optimized.

2. Experimental

Polycrystalline MgB_2 samples were prepared by using in situ solid state reaction. High-purity commercial powders (Furu-uchi Chemical Corporation, Japan) of Mg metal (99.9% purity, 200 meshes) and amorphous B powder (99% purity, 300 meshes) were mixed in a nominal ratio of Mg:B = 1:2. The starting powders were thoroughly ground in a glove box under nitrogen atmosphere. The powder mixture was pressed into pellets 40 mm, 30 mm, and 20 mm in diameter and 7 mm in thickness using a uni-axial pressing machine. The consolidated pellets were then wrapped in tantalum foils and subjected to the heat treatment in argon atmosphere in a tube furnace.

The samples were heated to the target sintering temperature (from 700 °C to 950 °C in steps of 25 °C) in 5 h and kept for 3 h there in flowing argon gas. Finally, the temperature was lowered to room temperature at a cooling rate of 100 °C/h. All the samples are reinforced with stainless steel rings for trapped field measurements. Fig. 1 shows photos of MgB_2 bulks in the as-sintered state and some with encapsulation. No appreciable defects like cracks are present.

The microstructure of these samples was studied with a scanning electron microscope (SEM). Chemical compositions were analyzed by energy dispersive X-ray spectroscopy (EDX). The constituent phases of the samples were identified with a high-resolution automated X-ray powder diffractometer (RINT2200), using Cu Kα radiation generated at 40 kV and 40 mA. Small specimens with dimensions of $1.5 \times 1.5 \times 0.5$ mm^3 were cut from the bulk MgB_2 samples and subjected to the measurements of the critical temperature (T_c) and magnetization hysteresis loops (M-H loops) in fields from -1 to +5 T at 10–35 K using a SQUID magnetometer (Quantum Design, model MPMS5). J_c values were estimated using the extended Bean critical state model for a rectangular sample [28].

Fig. 2 shows schematic illustration and a photo of the cryostat along with a photo of an activation superconducting magnet for trapped field measurements. The trapped field was measured by magnetizing the bulk sample with a superconducting magnet in the temperature range between 20 K and 40 K. The cryostat consists of a cryo-cooler with the power enough to cool an MgB_2 disk down to 20 K. A standard Pt sensor was mounted inside the top and bottom copper plates. A transverse Hall sensor was directly glued to the top and bottom surfaces of the MgB_2 bulk, for which the distance between the probe and the sample surface was only 0.5 mm. The bulk MgB_2 superconductor was magnetized using the field-cooling process in a hybrid superconducting magnet composed of NbTi and Nb_3Sn coils cooled by a cryo-cooler. The hybrid magnet has the power to generate a magnetic field of 10 T within a room temperature bore of 100 mm diameter. The cryostat was inserted into the bore of this superconducting hybrid magnet. Bulk MgB_2 sam-

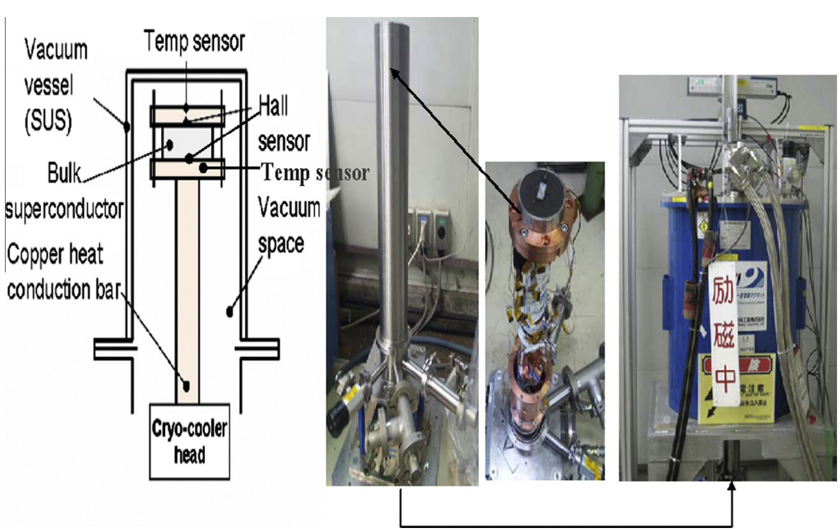

Fig. 2. Schematic illustration of the cryostat for the trapped field measurements at 20–40 K. The right figure presents a hybrid superconducting magnet composed of NbTi and Nb_3Sn coils that can generate a maximum field of 10 T at room temperature bore of 100 mm diameter.