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Physics Procedia 67 (2015) 958 – 962

Physics Procedia

25th International Cryogenic Engineering Conference and the International Cryogenic Materials Conference in 2014, ICEC 25–ICMC 2014

Trapped magnetic field properties of MgB_2 bulks doped with Ti

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Abstract

We have studied the trapped magnetic field properties on doped MgB_2 bulk with Ti (nominal content was Mg:Ti =0.9:0.1 and 0.8:0.2). The trapped magnetic field, B_T, was enhanced by approximately 1.3 times from 2.6 T of the pristine bulk to 3.5 T of the Ti-doped bulks at 15 K. The extrapolated $B_T(T)$ curve reached 5 T below 4.2 K, indicating that 5 T class bulk magnet can be realized using MgB_2 with Ti-doping. The critical current density, J_c, under the magnetic field was also enhanced by Ti-doping, therefore, an irreversibility field, B_{irr}, of the Ti-doped bulks exceeded 5 T at 20 K. Ti and TiB_2 impurities confirmed by the powder X-ray diffraction acted as the pinning centers and resulted in the enhancement of the trapped field and the critical current density.

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Keywords: MgB_2; superconducting bulk magnet; Ti doping

1. Introduction

A large polycrystalline MgB_2 superconducting bulk with centimeter size in diameter and in thickness has been fabricated to realize a quasi-permanent magnet because of weak-link-free grain boundaries (GBs) (Kambara et al. (2001)). However, since the bulk fabricated by an *in-situ* method under an ambient pressure has intrinsically a low packing density of about 50% (Naito et al. (2012)), the high-pressure sintering is one of the routes to enhance the density and the critical current density, J_c, i.e., the trapped magnetic field, B_T. Just after the discovery of MgB_2, MgB_2 bulk sintered under a pressure of 2 GPa offered the possibility of 2 T class bulk magnets at 6 K (Viznichenko et al. (2003)). We also reported the 2.5 T MgB_2 bulk magnet operated at 12.7 K using dense bulk, which was prepared by a hot isostatic pressing (HIP) method under a pressure of 98 MPa (Sasaki et al. (2013)). Since the GBs are well known to act as pinning centers in MgB_2, the effect of the grain-size refining on the flux pinning has been investigated. Recently, Fuchs *et al.* achieved a trapped field of 5.4 T in hot pressed MgB_2 using ball-milled powder, which demonstrates that simultaneous grain refining and densification are extremely effective to enhance B_T (Fuchs et al. (2013)). Contrary to the mechanical milling, the impurity doping such as Ti was also reported to fine down the MgB_2 grains. Zhao *et al.* argued that the critical current density was improved over three orders of magnitude from $\sim 10^3$ A/cm^2 of the pristine sample to $\sim 10^6$ A/cm^2 of Ti10% doped sample at 20 K (Zhao et al. (2001)). However,

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Peer-review under responsibility of the organizing committee of ICEC 25-ICMC 2014
doi:10.1016/j.phpro.2015.06.162

further Ti-doping suppressed the J_c value and the J_c of the Ti20% sample was about one order of magnitude smaller than that of the Ti10% sample at 20 K. TiB_2 with lamellar structure was created between nanometer-size grains and acted as the flux pinning center, which was observed by TEM (Zhao et al. (2002)). Therefore, fine grains, high packing density and a novel flux pinning center such as TiB_2 are also possible to bring about a higher J_c and higher B_T. In this paper, we report on the fabrication of MgB_2 bulks doped with two Ti contents by the HIP method in order to realize the high density and the introduction of novel pinning center at the same time. We measured their trapped field and critical current density, and finally discuss the effect of Ti-doping.

2. Experimental

MgB_2 bulk samples were fabricated by an *in-situ* HIP method. The precursors were prepared as follows; raw powders of Mg (99% in purity), Ti (99% in purity), and amorphous B (99% in purity) were weighted with $(1-x)$:x:2.0 in molar ratio and ground for 1 h. The mixture was pressed into a pellet 40 mm in diameter and 20 mm in thickness under an uniaxial pressure of about 12 MPa. Subsequently the pellet was further densificated by a cold isostatic pressing method under a pressure of 196 MPa. The precursor pellet was sealed in a stainless steel container under vacuum by an electron beam welding method. The MgB_2 precursor in the container was sintered at 900 °C for 3 h under Ar-gas pressure of 98 MPa in the HIP furnace and cooled down to room temperature by furnace cooling. The disc-shaped MgB_2 bulk with about 38 mm diameter and 6.9 mm thickness was prepared by the dry machining.

The MgB_2 bulk was magnetized by field cooling (FC) in a magnetic field of 5 T parallel to the thickness direction using a 10 T cryogen-free superconducting magnet (JMTD-10T100, Japan Superconductor Technology (JASTEC), Inc.) and then the applied magnetic field was decreased to 0 T at a rate of 0.022-0.22 T/min. The bulk was placed on the cold-stage of a helium refrigerator by inserting a thin indium sheet to obtain good thermal contact. The trapped field was measured by a cryogenic Hall sensor (BHT-921, F.W. Bell Inc.) mounted on the center of the bulk surface. The temperature of the bulk was monitored by a Cernox thermometer which was adhered beside the Hall sensor. After the FC magnetization experiments, we evaluated the critical current density J_c, using a small piece cut from the bulk. J_c was estimated from the magnetic hysteresis using the extended Bean model (Bean (1962)) following $J_c = 20\Delta M/a(1 - a/3b)$, 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. The magnetic hysteresis was measured using a commercial SQUID magnetometer (MPMS-XL, Quantum Design Inc.).

3. Results and discussion

Fig. 1 shows the temperature dependence of the trapped magnetic field $B_T(T)$of the pristine and 10% and 20% Ti doped bulk pieces. The B_T value of the pristine bulk is about 2.46 T at the lowest temperature of 17 K, decreases with increasing temperature, and finally reaches zero at around 39 K, which originates from the decrease of J_c with increasing T. The highest B_T of 3.45 T at 15 K was achieved for the Ti10% bulk; which was approximately 1.3 times lager than the extrapolated value of 2.6 T for the pristine bulk at 15 K. The Ti20% bulk offers almost the same B_T profile as that of the Ti10% bulk. The $B_T(T)$ curve extrapolated to lower temperature reaches 5 T around 4.2 K for Ti10% bulk, which allowed us to realize a 5 T class superconducting bulk magnet. It is noteworthy that the critical temperature T_c, defined by the temperature at B_T=0, is independent of the Ti content. The results suggest that Ti is not a substitute for the Mg-site, because the site substitution usually deteriorates the superconducting properties.

Fig. 2 shows the magnetic field dependence of the critical current density $J_c(B)$of the pristine and Ti-doped bulks at 20 K. The $J_c(B)$ curves of both Ti-doped bulks are nearly the same, which is consistent with the $B_T(T)$ behavior. The J_c value of the pristine bulk decreases monotonically with increasing magnetic field, especially a strong decay is observed under the applied field higher than 4 T. Although the J_c in self-field is not so enhanced by Ti-doping, but in magnetic field it is rather improved. For instance, the J_c=4.1×10^3 A/cm^2 of the pristine sample is increased to 2.0×10^4 A/cm^2 under the applied field of 3 T by 10% Ti-doping. An irreversibility field B_{irr}, defined by the magnetic field at J_c=10 A/cm^2, obviously exceeds 5 T in the Ti10% sample. Assuming the uniform J_c distribution in the bulk, the average macroscopic J_c, which creates the trapped field, can be estimated by the following analytical equation

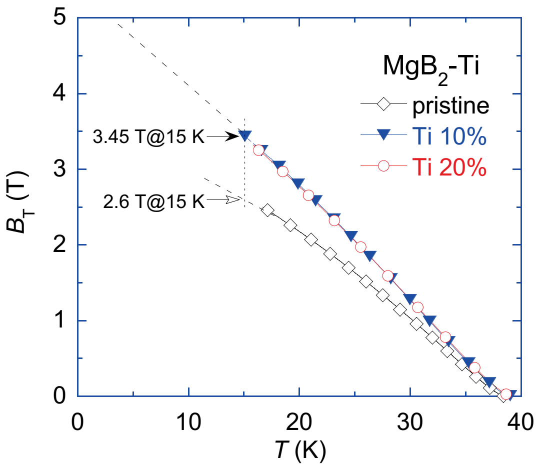

Fig. 1: Temperature dependence of the trapped magnetic field of the pristine and Ti-doped MgB_2 bulks. The dotted lines represent the extrapolation lines.

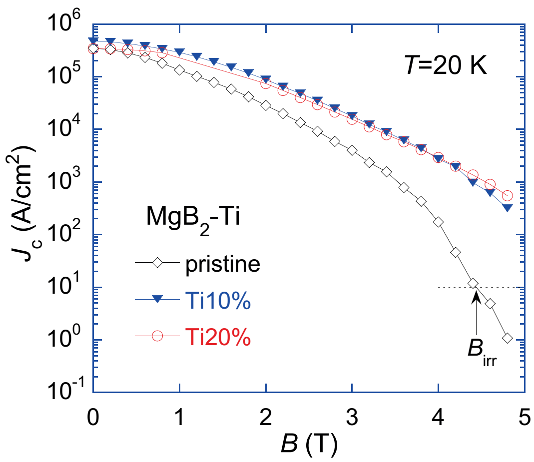

Fig. 2: Magnetic field dependence of the critical current density at 20 K for the pristine and Ti-doped MgB_2 bulks. An irreversibility field, B_{irr}, is defined by a criterion of J_c=10 A/cm^2.

based on the Bio-Savart law,

$$B_T(z) = \frac{\mu_0 J_c}{2}\left((z+t)\ln\frac{(D/2)+\sqrt{(D/2)^2+(z+t)^2}}{z+t} - z\ln\frac{(D/2)+\sqrt{(D/2)^2+z^2}}{z}\right), \tag{1}$$

where, D and t are the diameter and thickness of the disc and z is the distance from the center of the top surface of the bulk. The trapped field of 2.17 T for the pristine bulk is enhanced to 2.79 T by the Ti10% doping at 20 K, as shown in Fig. 1. Using Eq. (1) and the measured B_T values, the average macroscopic J_c is estimated to be 2.9×10^4 A/cm^2 for the pristine and 3.7×10^4 A/cm^2 for the Ti10% bulk, which correspond to the measured J_c under the applied field of 2.0 T and 2.6 T, respectively. These results suggest that the experimentally obtained B_T values can be roughly reproduced by the Bio-Savart law.

Fig. 3 shows the powder X-ray diffraction (XRD) pattern taken at room temperature for the pristine and Ti-doped bulks. The XRD peaks of the MgB_2 phase are labeled by "MgB_2", and an impurity phase is not found for the pristine

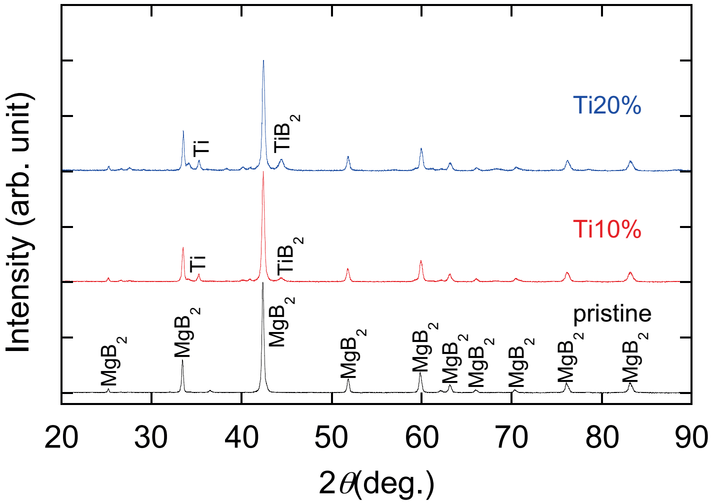

Fig. 3: Powder X-ray diffraction patterns taken at room temperature for the pristine and Ti-doped MgB_2 bulks.

bulk. On the other hand, the impurity phases such as Ti and TiB_2 appear for the Ti-doped bulks, especially the peak intensity due to TiB_2 increases obviously with the increase in the Ti contents. Ti contained impurities act as the pinning center, and enhance both the J_c and B_T. If the grain of MgB_2 is fined down to nanometer size by Ti-doping, as pointed out by Zhao *et al.* (Zhao et al. (2001, 2002)), the trapped field is expected to be over 5 T at around 12 K as observed in the hot-pressed bulk using ball-milled powder Fuchs et al. (2013). However, the highest B_T of 3.45 T at 15 K in the Ti-doped bulks does not reach the expected value. Although the Ti-doping successfully certainly introduced the flux pinning centers, the refining effect of the grain size was not be confirmed at the present stage. Microscopic observation to clarify the grain size is now in progress.

4. Summary

We have studied the effects of Ti-doping on the trapped magnetic field and critical current density of the MgB_2 bulks. Two bulk samples with the nominal composition of Mg:Ti=0.9:0.1 and 0.8:0.2 have been measured. The trapped magnetic field was considerably increased by Ti-doping; the highest value of 3.45 T achieved for both Ti-doped bulks at 15 K was about 1.3 times larger than that of the pristine bulk. The trapped field line extrapolated to lower temperature reached 5 T below 4.2 K. The critical current density was also enhanced by Ti-doping and the irreversibility field, which was defined by the criterion of J_c=10 A/cm^2, exceeded 5 T at 20 K. Ti and TiB_2 phases confirmed by the X-ray diffraction acted as the flux pinning centers. It is noteworthy to point out that both trapped field and critical current density obtained for the Ti-doped bulk pieces hardly depended on the Ti contents. Further studies such as microstructure analysis and further Ti-doping must be considered.

Acknowledgments

We would like to acknowledge Dr. H. Teshima of Nippon Steel & Sumitomo Metal Corporation for his assistance in the production of the MgB_2 bulks using the HIP furnace. This work was partly supported by JST A-STEP (AS242Z02673L), Hitachi Metals · Materials Science Foundation, and JSPS KAKENHI Grant Number 23560002.

References

Bean, C. P., 1962. Magnetization of hard superconductors. Phys. Rev. Lett. 8, 250–253.

Fuchs, G., Häβler, W., Nenkov, K., Scheiter, J., Perner, O., Handstein, A., Kanai, T., Schultz, L., Holzapfel, B., 2013. High trapped fields in bulk MgB_2 prepared by hot-pressing of ball-milled precursor powder. Supercond. Sci. Technol. 26, 122002 (5pp).

Kambara, M., Babu, N. H., Sadki, E. S., Cooper, J. R., Minami, H., Cardwell, D. A., Campbell, A. M., Inoue, I. H., 2001. High intergranular critical currents in metallic MgB_2 superconductor. Supercond. Sci. Technol. 14, L5–L7.

Naito, T., Sasaki, T., Fujishiro, H., 2012. Trapped magnetic field and vortex pinning properties of MgB_2 superconducting bulk fabricated by a capsule method. Supercond. Sci. Technol. 25, 095012 (6pp).

Sasaki, T., Naito, T., Fujishiro, H., 2013. Trapped magnetic field of dense MgB_2 bulks fabricated under high pressure. Physics Procedia 45, 93 – 96.

Viznichenko, R. V., Kordyuk, A. A., Fuchs, G., Nenkov, K., Müller, K.-H., Prikhna, T. A., Gawalek, W., 2003. Temperature dependence of the trapped magnetic field in MgB_2 bulk superconductors. Appl. Phys. Lett. 83, 4360–4362.

Zhao, Y., Feng, Y., Cheng, C. H., Zhou, L., Wu, Y., Machi, T., Fudamoto, Y., Koshizuka, N., Murakami, M., 2001. High critical current density of MgB_2 bulk superconductor doped with ti and sintered at ambient pressure. Appl. Phys. Lett. 79, 1154–1156.

Zhao, Y., Huang, D. X., Feng, Y., Cheng, C. H., Machi, T., Koshizuka, N., Murakami, M., 2002. Nanoparticle structure of MgB_2 with ultrathin TiB_2 grain boundaries. Appl. Phys. Lett. 80, 1640–1642.