

Effects of Carbon Doping on Trapped Magnetic Field of MgB_2 Bulk Prepared by *in-situ* Hot Isostatic Pressing Method

Tomoyuki Naito , Arata Ogino , Hiroyuki Fujishiro , and Satoshi Awaji 

Abstract—We report the effects of carbon doping on the trapped field properties of MgB_2 bulks doped with graphite (C) and B_4C , which were prepared by an *in-situ* hot isostatic pressing method. The trapped fields, B_T 's were degraded by the graphite and B_4C doping above 15 K, which was predominantly due to the lowered T_c by the C substitution. However, the slope of $B_T(T)$ and the irreversibility line, $B_{\text{irr}}(T)$, determined from the resistive transitions indicated that the B_T of the graphite-doped bulk was expected to exceed that of the pristine bulk below 15 K. Contrary to the literature by other groups, B_4C did not enhance the vortex pinning properties.

Index Terms— MgB_2 , trapped field, carbon doping.

I. INTRODUCTION

THE MgB_2 bulk magnets have been intensively studied since the discovery of superconductivity in MgB_2 [1], especially in the past decade [2]–[8]. A trapped field, B_T , of 5 T class can be obtained by the combination of various processing methods, impurity doping, and so on, to date [5], [7]. Since the filling factor of MgB_2 fabricated via an *in-situ* route under ambient pressure is only about 50%, high critical current density, J_c , has been generally realized for dense MgB_2 prepared by a high pressure synthesis [9]–[13] or by a diffusion (an infiltration) method [8], [14], [15]. The titanium group elements [11], [16]–[19] and carbon [8], [20]–[26] are well known to be effective dopants and additives to enhance the vortex pinning properties of MgB_2 . The titanium created thin TiB_2 lamellae at the surface of the MgB_2 grain, which prevented the grain growth of MgB_2 , and then enhanced the grain boundary pinning [16], [17]. We obtained a B_T of 4.6 T at 14.1 K in center of the stacked Ti-doped MgB_2 bulks fabricated by an *in-situ* hot isostatic pressing (HIP) method [7]. On the other hand, cost-effective carbon doping has been studied mainly for wire production. Carbon substitutes on the boron site, and shortens the coherence length, that is,

enhances the upper critical field B_{c2} . Since simple forms of carbon such as graphite were found to be difficult to substitute on the B-site [21], various carbon-containing compounds have been used as carbon sources. SiC, which was used in the pioneering work [20], offered enhanced vortex pinning properties, however concomitantly created an impurity phase of Mg_2Si preventing the flow of supercurrent. Among various carbides, B_4C is a promising dopant, because it has only the constituent elements of C-doped MgB_2 and a high reactivity, that is, the carbon was efficiently introduced into MgB_2 . The carbon coating to the boron powder by various aromatic compounds such as coronene is quite effective to enhance both J_c and the irreversibility field, B_{irr} , of wires [24]. However, this attractive method is difficult to apply to the fabrication of a centimeter-scale bulk, because it requires quite a large amount of starting materials, compared to the short wires for the feasibility study. Ball milling was reported to promote the impurity diffusion at the surface of MgB_2 grains, in addition to the increase of grain boundaries [27]. Therefore, carbon substitution should also be encouraged by ball milling. In this paper, we have measured the trapped field, electrical resistivity and critical current density of pristine and carbon-doped MgB_2 bulks, in which graphite (for reference) and B_4C were used as the carbon sources. The bulks were prepared from the as hand-ground and as ball-milled precursor powders. Both the effects of carbon doping and grain refining on the trapped field properties are discussed.

II. EXPERIMENTAL

The pristine and carbon-doped MgB_2 bulks were prepared by the *in-situ* hot isostatic pressing (HIP) method, where graphite and B_4C were used as the carbon source. The precursors were prepared in the following procedure. Raw powders of Mg (99% in purity, $\leq 180 \mu\text{m}$ in particle size, Kojundo Chemical Laboratory Co., Ltd), amorphous B (99% in purity, $\leq 46 \mu\text{m}$ in particle size, Furuuchi Chemical Corp.), C (graphite) (99.9% in purity, $\sim 50 \mu\text{m}$ in particle size, Kojundo Chemical Laboratory Co., Ltd), and B_4C (99% in purity, $\leq 46 \mu\text{m}$ in particle size, Furuuchi Chemical Corp.) were mixed in the molar ratios $\text{Mg} : \text{B} : \text{C} = 1.0 : 1.8 : 0.2$ and $\text{Mg} : \text{B} : \text{B}_4\text{C} = 1.0 : 1.0 : 0.2$, respectively, for the C- and B_4C -doped bulks. These were ground using an agate mortar and pestle, additionally pulverized by a planetary ball milling (BM) device (PM100, Retsch GmbH) with a rotating speed of 250 rpm for 12 h using an agate container

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Tomoyuki Naito, Arata Ogino, and Hiroyuki Fujishiro are with the Faculty of Science and Engineering, Iwate University, Morioka 020-8551, Japan (e-mail: tnaito@iwate-u.ac.jp; cloud3254@gmail.com; fujishiro@iwate-u.ac.jp).

Satoshi Awaji is with the High Field Laboratory for Superconducting Materials, Institute for Materials Research, Tohoku University, Sendai 980-8577, Japan (e-mail: awaji@imr.tohoku.ac.jp).

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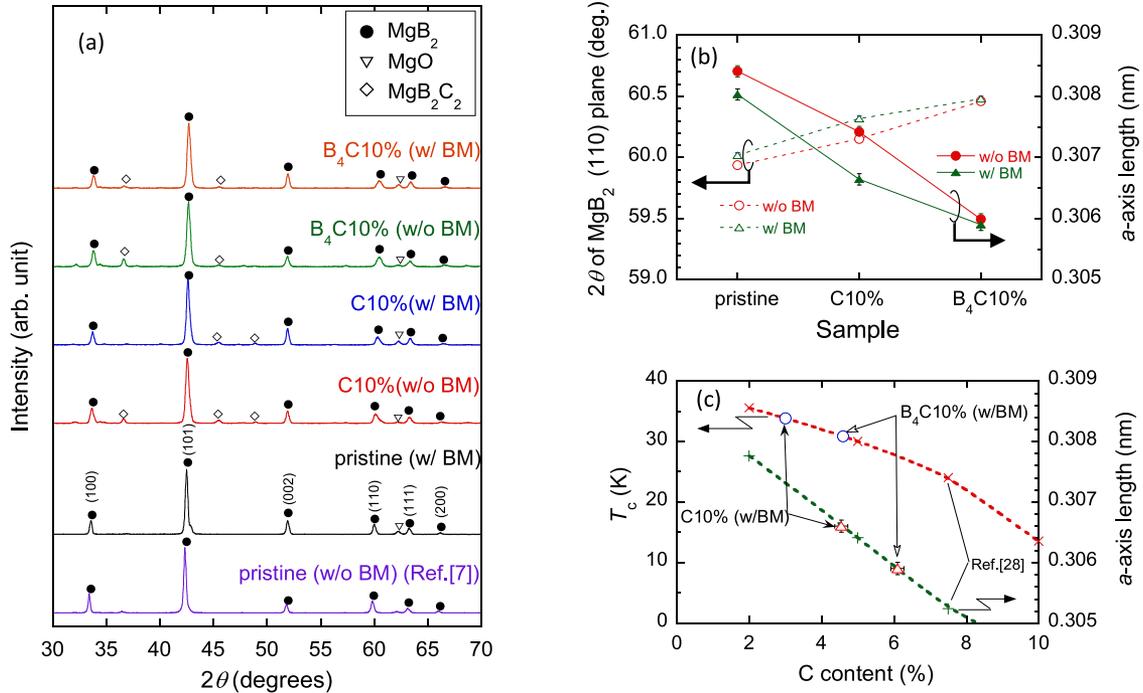


Fig. 1. (a) X-ray diffraction patterns of the pristine and the C10%- and B₄C10%-doped MgB₂ bulks. BM means the ball milling. The data of the pristine MgB₂ without BM are from Ref. [7]. (b) The 2θ value of the MgB₂ (110) plane and the lattice constant a for the indicated MgB₂ bulks. (c) The critical temperature, T_c , and the lattice constant a as a function of the carbon content for the C10%- and B₄C10%-doped MgB₂ bulks and C-doped single crystalline MgB₂ [28]. The dashed lines are guide to the eyes.

and balls. The mixed powder was pelletized to a disc 40 mm in diameter and 20 mm in thickness under a uniaxial pressure of about 12 MPa, and subsequently the pellet was further densified by a cold isostatic pressing method under a pressure of 196 MPa. The precursor, which was enclosed in a stainless steel container under vacuum by electron beam welding, was sintered at 900 °C for 3 h under an Ar gas pressure of 98 MPa in the HIP furnace and cooled down to room temperature by furnace cooling. Disc-shaped MgB₂ bulks of about 38 mm diameter and 7 mm thickness were prepared by machining under dry conditions. The constituent phases and structure were examined by an X-ray diffraction pattern taken at the flat surface of each bulk using a Cu-K α radiation (wave length was 0.15406 nm).

The MgB₂ bulks were magnetized by field cooling (FC) in a magnetic field, parallel to the thickness direction using a commercial 10-T cryogen-free superconducting magnet (JMTD-10T100, JASTEC, Inc.). 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 on the bulk surface. The resistivity and magnetization were measured using a small piece cut from the bulk. The resistivity was measured by a conventional dc four probe method with a typical current density of 10 A cm⁻² under magnetic fields up to 16 T using the 18-T superconducting magnet (JASTEC Inc.) at the High Field Laboratory for Superconducting Materials, Institute for Materials Research, Tohoku University. The magnetic field dependent magnetization was measured using a vibrating sample magnetometer (VSM)

coupled with the 15-T cryogen-free superconducting magnet (JASTEC Inc.) at the HFL-SM, IMR, Tohoku Univ. The critical current density, $J_c(B)$, was estimated from the magnetic hysteresis using the extended Bean model [29], [30].

III. RESULTS AND DISCUSSION

Fig. 1(a) shows the X-ray diffraction patterns of the indicated MgB₂ bulks. The observed major peaks were almost confirmed as the MgB₂ phase for all the present MgB₂ bulks. As shown in the left axis of Fig. 1(b), the peak of the MgB₂ (110) plane at around $2\theta = 60^\circ$ was shifted toward higher angles for both C10%- and B₄C10%-doped bulks, which suggests the shrinkage of the a -axis of MgB₂ due to the carbon substituted for B. The ball-milled precursors offered a further peak shift for both C10%- and B₄C10%-doped bulks. The amount of peak shift for the B₄C10%-doped bulk was somewhat larger than that for a C10%-doped one, regardless of ball milling. Note that a similar peak shift was confirmed for the a -axis related crystallographic planes such as (100) and (101) planes. As shown in the right axis of Fig. 1(b), the a -axis was shortened by the carbon doping. The lattice constants of the a -axis were estimated to be 0.3080, 0.3066 and 0.3059 nm for the pristine, C10%- and B₄C10%-doped MgB₂ bulks respectively after the ball milling process. Note that ball milling also shortened the a -axis for both C10%- and B₄C10%-doped MgB₂ bulks. Fig. 1(c) shows the critical temperature, T_c , (left axis) and the lattice constant a (right axis) as a function of the C content. The actual amounts of carbon substitution to the boron site were estimated to be about 3–4.5%

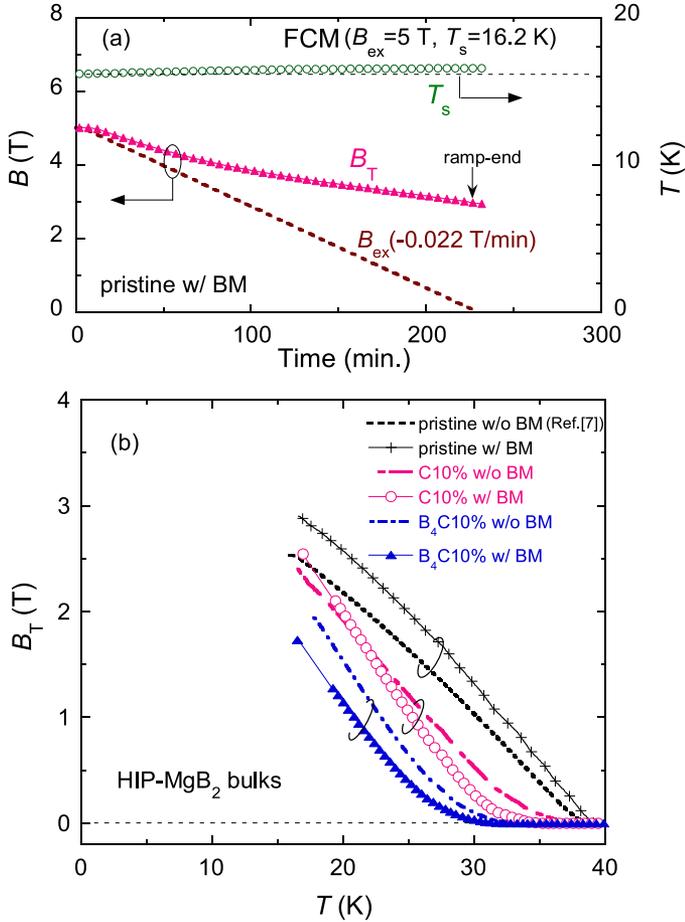


Fig. 2. (a) Time dependences of the trapped field, applied field and temperature of the pristine MgB₂ bulk with ball milling during FCM from the applied field of 5 T at the initial bulk temperature of 16.2 K. The left- and right-hand axes are the magnetic field and temperature, respectively. (b) Temperature dependence of the trapped field, $B_T(T)$, of the pristine and the C10%- and B₄C10%-doped MgB₂ bulks. The data of the pristine without BM are from Ref. [7]. BM means ball milling.

and 4.5–6% for the C10%- and B₄C10%-doping, respectively, considering the relationship among the nominal C content, the length of a -axis and the critical temperature for C-substituted single crystalline MgB₂ [28]; the T_c 's of the present MgB₂ bulks were determined by the temperature dependences of the trapped field and the resistivity, as will be shown later. The C contents estimated from the lattice constant a were about 1.5% larger than those from the T_c for both carbon-doped MgB₂ bulks. This difference corresponded to a 2θ of about 0.15°, which might originate from the shift of the machine origin of the XRD device. The MgO impurity was observed for all the bulks, regardless of ball milling. The MgB₂C₂ impurity phase also appeared for both C10%- and B₄C10%-doped bulks, which originated from the excess carbon. The peak intensity of the MgB₂C₂ phase was reduced by the ball milling for both C10%- and B₄C10%-doped bulks, while on the contrary that of the MgO phase was increased. These results also suggest that the ball milling promoted carbon substitution on the B site.

Fig. 2(a) shows the typical magnetizing process of the pristine MgB₂ bulk with ball milling during field-cooled magnetization

(FCM) from the initial applied field of 5 T at the initial bulk temperature of 16.2 K. The trapped field, B_T , decreased moderately with withdrawing the applied magnetic field, B_{ex} , at a rate of -0.022 T/min and finally reached 2.98 T at the end of ramp ($B_{ex} = 0$ T). After that, the B_T continued to decrease slightly, which originated from vortex creep. The obtained B_T value of 2.98 T at 16.2 K is about 20% higher than the value of 2.5 T at 16.2 K reported for the HIP-processed MgB₂ bulk (38 mm in diameter and 6.9 mm in thickness) without ball milling [7]. This is believed to originate from the increase of grain boundaries, which are well known as the dominant pinning center of MgB₂, by ball milling [5], [27], [31], [32]. Thus, we estimated the crystallite size, τ , of both the pristine bulks with and without ball milling by the following Scherrer's relation [33],

$$\tau = \frac{K\lambda}{\beta \cos \theta}, \quad (1)$$

where, K is the shape factor, generally ~ 0.9 , λ the wave length of the Cu-K α radiation, β the full width at the half maximum (FWHM) of the diffraction peak, and θ the Bragg angle. Using the main peak of MgB₂ (101) plane, the τ value of 32.8 nm estimated for the pristine bulk without ball milling reduced slightly to 31.6 nm by ball milling; note that this reduction corresponded to the difference in β of only 0.01°, which was smaller than the interval of angle of 0.02°. Therefore, the increase of grain boundaries by ball milling should be confirmed by an additional microstructural analysis such as an electron backscattered diffraction [32], [34]. We found almost the same XRD patterns between the pristine bulks with and without ball milling, although the peak of the MgO impurity of the bulk with ball milling was slightly larger than that of without ball milling. This suggested that the quality of both bulks were almost the same and that the increase of grain boundaries was the most plausible origin for the enhancement of B_T .

During FCM, the temperature of the bulk increased quite moderately from 16.2 to 16.6 K, which was caused by heat generation due to the vortex dynamics. The magnetizing processes of other the MgB₂ bulks (not shown here) were similar to that of the pristine one.

After the magnetizing process, each MgB₂ bulk was heated up beyond the critical temperature. Fig. 2(b) shows the temperature dependence of the trapped field, $B_T(T)$, of the indicated MgB₂ bulks. The $B_T(T)$ decreased monotonically with increasing temperature, because of the decrease in J_c with temperature. The $B_T(T)$ relationships for the pristine MgB₂ bulks with and without BM were just as the trapped fields at the end of ramp. The T_c , defined as the temperature at $B_T = 0$ T, was about 39 K for both pristine bulks. For the C10%-doped MgB₂ bulks, the $B_T(T)$ of the bulk with BM, which was somewhat higher than that without BM at the magnetizing temperature, decreased steeply with increasing temperature, went below the $B_T(T)$ of the bulk without BM around 20 K, and finally reached zero at $T_c = 35$ K. On the other hand, the C10%-doped bulk without BM kept a non-zero B_T up to $T_c = 38$ K. The $B_T(T)$ was strongly suppressed, and simultaneously the curvature of the $B_T(T)$ line was changed from convex to concave by the C10%-doping. A rather small $B_T(T)$ with concave curvature

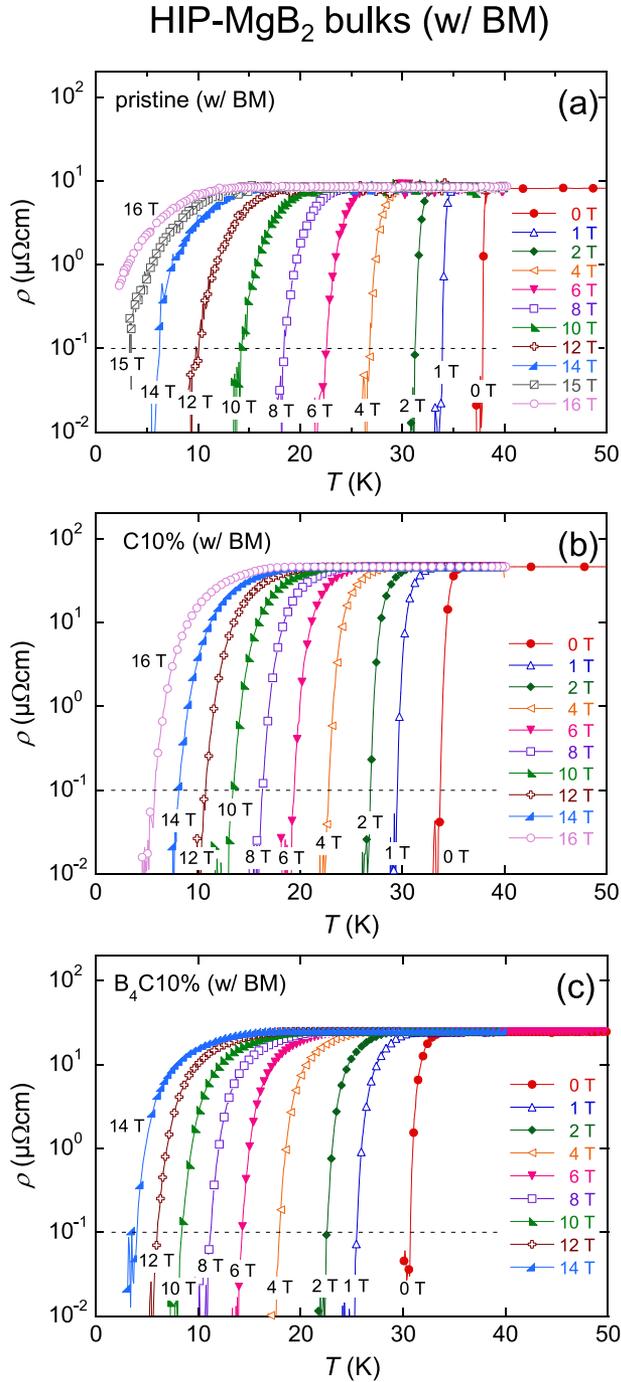


Fig. 3. Temperature dependence of resistivity of (a) the pristine and (b) the C10%- and (c) $B_4C10\%$ -doped MgB_2 bulks with ball milling (BM) under magnetic fields up to 16 T.

was observed for the $B_4C10\%$ -doped MgB_2 bulks, compared with the pristine bulks. Ball milling resulted in the further degradation of $B_T(T)$ also for the $B_4C10\%$ -doped MgB_2 bulks. The T_c 's at $B_T = 0$ T were 32 and 33 K, respectively, for the $B_4C10\%$ -doped bulks with and without BM. The low $B_T(T)$ values observed for the present carbon-doped bulks originated from the suppression of T_c , which is well known as a disadvantage of the carbon doping. For the present carbon-doped

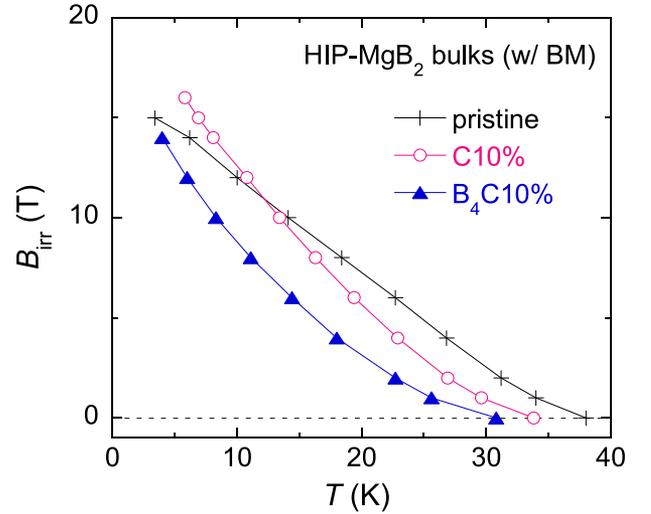


Fig. 4. The temperature dependence of the irreversibility field, $B_{irr}(T)$, of the the pristine and the C10%- and $B_4C10\%$ -doped MgB_2 bulks with ball milling (BM).

MgB_2 bulks, the promotion of the reaction of C with B is believed to exceed the effect of grain refinement by ball milling. However, it is noteworthy that extrapolating all the $B_T(T)$'s toward low temperatures suggested that the B_T of the C10%-doped bulk with BM is expected to exceed the B_T of the pristine bulk with BM below 15 K.

Fig. 3 shows the temperature dependence of the resistivity, $\rho(T)$, under the magnetic fields up to 16 T for the pristine and the C10%- and $B_4C10\%$ -doped MgB_2 bulks with ball milling process. The T_c , defined as the temperature at which the resistivity went to zero, was 38.0, 33.8, and 30.8 K for the pristine, C10%- and $B_4C10\%$ -doped bulks, respectively. Although these resistive T_c values were about 1 K lower than those estimated from the $B_T(T)$ lines for the present three bulks, the magnitude of T_c degradation by carbon doping was almost the same as for the magnetic T_c . The irreversibility temperature, T_{irr} , was defined as the temperature at which the resistivity was $0.1 \mu\Omega\text{cm}$. The estimated T_{irr} was plotted on the magnetic field vs. temperature diagram, as shown in Fig. 4. The irreversibility line, $B_{irr}(T)$, of the C10%-doped bulk exceeded that of the pristine bulk below approximately 13 K. On the other hand, the $B_{irr}(T)$ of the $B_4C10\%$ -doped MgB_2 bulk was smaller than that of the pristine one in the whole measured temperature range. The behaviors of $B_{irr}(T)$ lines support strongly the expectation that a higher trapped field can be obtained for the C10%-doped bulk with BM at low temperatures below 15 K.

Fig. 5 shows the magnetic field dependence of the critical current density, $J_c(B)$, at temperatures of 10 and 20 K for the pristine and C10%-doped MgB_2 bulks with ball milling. The $J_c(B)$ at 20 K of the C10%-doped bulk was smaller than that of the pristine one, and furthermore the irreversibility field of 3.8 T, defined as the magnetic field at which the J_c was 100 A cm^{-2} , was also low compared to $B_{irr} = 4.7$ T of the pristine one. At 10 K, a comparable magnitude of $J_c(B)$ above about 5 T and almost the same $B_{irr} = 8.6$ T were found for both pristine and C10%-doped bulks. The somewhat smaller B_{irr} compared to the

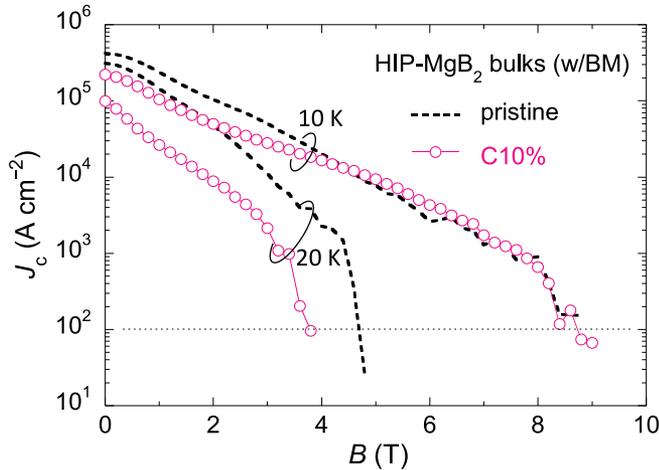


Fig. 5. Magnetic field dependence of critical current density, J_c , of the pristine and C10%-doped MgB₂ bulks with ball milling (BM) at 10 and 20 K.

resistive B_{irr} is due to the difference in the criterion of J_c , as is well known. These results also suggest that carbon doping is effective at low temperatures, that is, the operation of the MgB₂ bulk magnets doped with the carbon is suitable below 15 K.

IV. SUMMARY

We have studied carbon doping effects on the trapped field properties of MgB₂ bulks doped with C (graphite) and B₄C, which were prepared by the *in-situ* HIP method using as hand-ground and as ball-milled starting mixtures. Although the nominal content of carbon was 10% with respect to boron for both dopants, the actual contents were estimated to be approximately 3–4.5% and 4.5–6% for C and B₄C, respectively, from the lattice constant of *a*-axis and the critical temperature, T_c . The ball milling offered a slightly shorter *a*-axis and lower T_c . These results indicated that the reactivity of B₄C was higher than that of C (graphite) and ball milling promoted the reaction between C and B in addition to grain refinement. The carbon doping degraded the trapped field, B_T , especially for the B₄C-doping, above 15 K, which originated mainly from the decrease in T_c . The ball milling did not offer an enhancement in B_T , suggesting that the effect of the T_c reduction was believed to exceed that of grain refinement. However, the temperature dependent B_T and the irreversibility line, $B_{irr}(T)$, suggested that the B_T of the C10%-doped MgB₂ bulk should exceed that of the pristine one below 15 K. Therefore, graphite-doped MgB₂ bulk magnets are expected to fulfill their potential below 15 K. Contrary to previous reports by other groups, we found that a simple form of carbon (graphite) could substitute on the boron site and enhance the vortex pinning. Furthermore, an actual substitution amount of carbon less than 2% was preferable to avert the substantial T_c reduction.

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