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Interplay between cold densification and malic acid addition (C4H6O5) for the fabrication of near-isotropic MgB2 conductors for magnet application

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© 2020 The effect of cold high pressure densification (CHPD) on anisotropy of the critical current density (Jc) in « in situ » single core binary and alloyed MgB2 tapes has been determined as a function of temperatures at 4.2 K, 20 K and 25 K as well as at applied magnetic fields up to 19 T. The study includes binary and C4H6O5 (malic acid) doped MgB2 tapes before and after CHPD. It is remarkable that the CHPD process not only improved the Jc values, in particular at the higher magnetic fields, but also decreased the anisotropy ratio, Γ = Jc///Jc$^{\perp}$. In binary MgB2 tapes, the anisotropy factor Γ increases with higher aspect ratios, even after applying CHPD. In malic acid (C4H6O5) doped tapes, however, the application of CHPD leads only to small enhancements of Γ, even for higher aspect ratios. This is attributed to the higher carbon content in the MgB2 filaments, which in turn is a consequence of the reduced chemical reaction path in the densified filaments. At all applied field values, it was found that CHPD processed C4H6O5 doped tapes exhibit an almost isotropic behavior. This constitutes an advantage in view of industrial magnet applications using wires with square or slightly rectangular configuration.

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Interplay between cold densification and malic acid addition ($C_4H_6O_5$) for the fabrication of near-isotropic MgB_2 conductors for magnet application

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Abstract

The effect of cold high pressure densification (CHPD) on anisotropy of the critical current density (J_c) in « *in situ* » single core binary and alloyed MgB_2 tapes has been determined as a function of temperatures at 4.2 K, 20 K and 25 K as well as at applied magnetic fields up to 19 T. The study includes binary and $C_4H_6O_5$ (malic acid) doped MgB_2 tapes before and after CHPD. It is remarkable that the CHPD process not only improved the J_c values, in particular at the higher magnetic fields, but also decreased the anisotropy ratio, $\Gamma = J_c^{//}/J_c^{\perp}$. In binary MgB_2 tapes, the anisotropy factor Γ increases with higher aspect ratios, even after applying CHPD. In malic acid ($C_4H_6O_5$) doped tapes, however, the application of CHPD leads only to small enhancements of Γ, even for higher aspect ratios. This is attributed to the higher carbon content in the MgB_2 filaments, which in turn is a consequence of the reduced chemical reaction path in the densified filaments. At all applied field values, it was found that CHPD processed $C_4H_6O_5$ doped tapes exhibit an almost isotropic behavior. This constitutes an advantage in view of industrial magnet applications using wires with square or slightly rectangular configuration.

Keywords: Magnesium diboride; Cold high pressure densification; Anisotropy; Tapes; Critical current density.

1. Introduction

The manufacture and commercialization of industrial MgB_2 wires and tapes is currently in progress and a large number of works has been published since its discovery in 2001 [1]. The perspectives of magnet application are not only based on the promising superconducting and electronic

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properties of MgB_2 tapes, but also on their low production costs. There is still much work required to be done in regards to the study and the improvement of the basic material properties, e.g. the grain connectivity and the mass density inside the MgB_2 filaments. At the present day, industrial lengths of MgB_2 wires and tapes have been manufactured by both *ex situ* and *in situ* techniques for large scale magnet applications while promising results have been obtained by a third process, Internal Mg Diffusion (IMD) [2].

In the present paper, the attention is centered on *in situ* tapes. The volume occupied by the reacted MgB_2 phase is smaller than that of the unreacted Mg + 2B mixture and the *in situ* wires after reaction contain a certain amount of porosities, typically reaching only 50% [3] of the theoretical density of 2.62 g/cm^3 [4,5]. We reported that the CHPD process strongly influences the final mass density of the filaments, resulting in a significant improvement of the transport properties of superconducting MgB_2 wires [6,7]. This is higher than the highest possible density in *ex situ* samples (74%), according to the close packing model [4,5]. The densification approach is very effective and particularly applicable to all kind of filaments fabricated by powder metallurgy. Carbon or carbon based additives have been found to have a beneficial effect on the critical current density (J_c) of *in situ* MgB_2 wires, as reported by Collings et al. [5]. In the most effective additives reported so far, carbon is always present, either as pure C [8,9] or C based compounds, e.g. SiC [10,11], B_4C [12,13], various carbohydrates [14,15] and hydrocarbons [16], and even sugar [17]. Yeoh et al. [18] also reviewed various possibilities of effective carbon based chemical doping on MgB_2. Malic acid ($C_4H_6O_5$) [7,14] is particularly an interesting candidate among the numerous carbon based additives leading to strong improvements reported in [5,18] and this will be the major focus of the present study. Isotropic conductors are important for any superconducting magnet design, i.e., the values of J_c in both parallel and perpendicular orientation should be similar. Higher values for $J_c^{//}$ (parallel field direction) have already been reported for alloyed MgB_2 in the tape forms: Hässler et al. [19] published for a highly textured MgB_2 tape alloyed with carbon with a value of $B(10^4)^{//} = 16$ T, while Gao et al. [20] reported $B(10^4)^{//} = 14$ T, where $B(10^4)$ is the magnetic field, B at which $J_c = 10^4$ A/cm^2. However, there is no full data reported for the perpendicular field direction, $B^{\perp}$, which might be substantially lower. Hässler et al. [21] published a reduction of Γ by pure C substitution. The question arises whether Γ can be reduced by analyzing the behavior of J_c of tapes with field directions parallel and perpendicular to the wire or tape axis, defining an anisotropy ratio $\Gamma = J_c^{//}: J_c^{\perp}$. The average *c* axis grain alignment in the MgB_2 filament is directly reflected by the value of Γ and has been extensively studied by Fujii et al. [22], Kovac et al. [23-26] and Lezza et al. [27] on a variety of *in situ* and *ex situ* tapes. For any *in situ* tape, the anisotropy ratio Γ is influenced by a number of factors: the method of deformation (drawing or swaging or rolling) [23,24], the types of ball milling procedure [22,23,28], the aspect ratio *b/a* of the tape [23,24], the types of sheath material [23,24] and the size and shape of the filament [25]. It is well established fact for both *in situ* and *ex situ* tapes that the anisotropy ratio Γ is the highest for binary MgB_2, but decreases with increasing carbon contents in the MgB_2 lattice [22-24].

The $C_4H_6O_5$ doped MgB_2 tapes treated with the CHPD technique show significantly higher J_c in both parallel and perpendicular directions [7], but the detailed investigation on the J_c anisotropy is critical for the magnet design and is, therefore, worth investigating. In the present work, we have determined the effect of CHPD on the anisotropic behavior of J_c in binary and in $C_4H_6O_5$ alloyed *in situ* monofilamentary MgB_2 tapes at 4.2, 20 and 25 K, at fields up to 19 T. The values of J_c as a function of applied magnetic field in both parallel and perpendicular direction in binary and $C_4H_6O_5$ alloyed MgB_2 tapes at 4.2 and 20 K have been reported in our previous works [6,7]. The major aim of this study was to investigate whether the observed reduction of anisotropy in malic doped tapes compared to binary tapes can uniquely be explained by the higher carbon substitution in the filament after the application of CHPD technique.

2. Experimental details

The round binary and alloyed wires (Ø 0.83 mm) for the present work were manufactured at Hyper Tech Research, Inc., Columbus, OH, USA. The MgB_2 wires doped with 10 wt% $C_4H_6O_5$ were fabricated followed by the route previously described by Hossain et al. [7]. The sheath material was Monel with Nb barrier, while the reaction conditions of MgB_2/Nb/Monel wires were 600 °C for 4 h (heating rate 2.5 °C/min.). All monofilamentary wires did not contain any Cu for electrical stabilization. These round wires were flat rolled to tapes. In this work, CHPD process was applied on short tapes under the same conditions as in Refs. 5 and 6, the sample length being 75 mm, the two-wall pressing tool having a length of 60 mm. In the present work, the transport J_cs of the tapes before and after CHPD have been measured as a function of the applied magnetic field up to 19 T either edge-on (//) or face-on (⊥) to the tape surface, at $T = 4.2$ K, 20 K and 25 K. For each applied magnetic field the J_c anisotropy is defined as the ratio Γ between the critical current density in the direction of parallel magnetic field $J_c^{//}$ and the one in perpendicular magnetic field $J_c^{\perp}$. A four-probe technique was used to measure J_c over the 45 mm lengths of conductor in a cryostat with helium flow and currents up to 250 A. Temperature sensor was placed on a current lead located close to the tape sample. The voltage taps were used on the sample 10 mm apart and 0.1 μV cm^{-1} was the voltage criteria for this transport current measurement. The values of irreversibility fields $B_{irr}^{//}$ and $B_{irr}^{\perp}$ were determined by $J_c = 100$ A/cm^2 as a criterion using an extrapolation of J_c versus B to 100 A cm^{-2}.

Crystal structures of the samples were characterized by x-ray diffraction (XRD) using Philips PW1730 instrument and lattice parameters (*a* and *c*) were calculated using Rietveld refinement. Microscopic and morphological studies and analysis were carried out using a JEOL field emission gun - scanning

Table 1
Characterization of the monofilamentary binary and alloyed tape with 10 wt.% $C_4H_6O_5$ (Monel sheath, Nb barrier) before and after pressing by 2 GPa.

Sheath Material	Type	*P* (GPa)	Tape cross section (mm x mm)	MgB_2 Section (mm × mm)	Filling factor (%)	Aspect Ratio (*a*/*b*)	Lattice parameters (Å)		C (x) in $Mg(B_{1-x}C_x)_2$	T_c
							a	*c*		
Monel/Nb	Malic doped	0	1.52 × 0.35	0.128	24.4	4.34	3.0749	3.5240	0.2011	23.0
Monel/Nb	Malic doped	2	1.90 × 0.25	0.101	22.7	7.60	3.0721	3.5236	0.0269	32.4
Monel/Nb	Binary	0	1.55 × 0.40	0.130	27.0	3.87	3.8123	3.5256		36.2
Monel/Nb	Binary	2	1.95 × 0.28	0.112	25.2	6.96	3.8144	3.5276		36.5

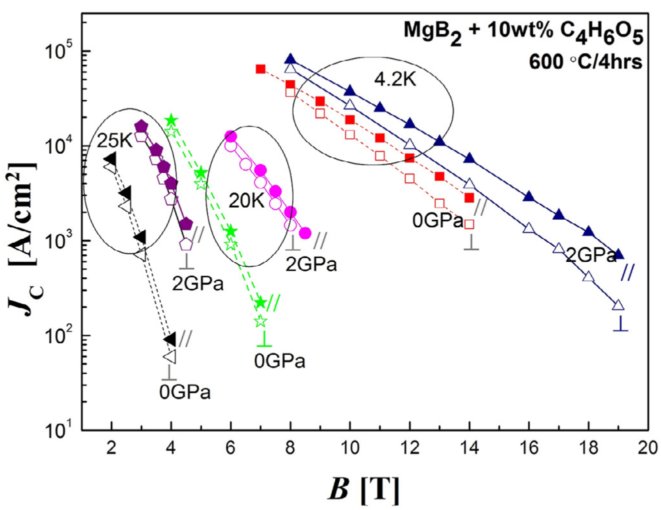

Fig. 1. Variation of the critical current density in parallel and perpendicular direction ($J_c^{//}$ and $J_c^{\perp}$) vs. magnetic field at 4.2, 20 and 25 K for $C_4H_6O_5$ alloyed MgB_2 tapes (Monel sheath, Nb barrier) at 0 and 2 GPa. Reaction conditions: 600 °C/4 h.

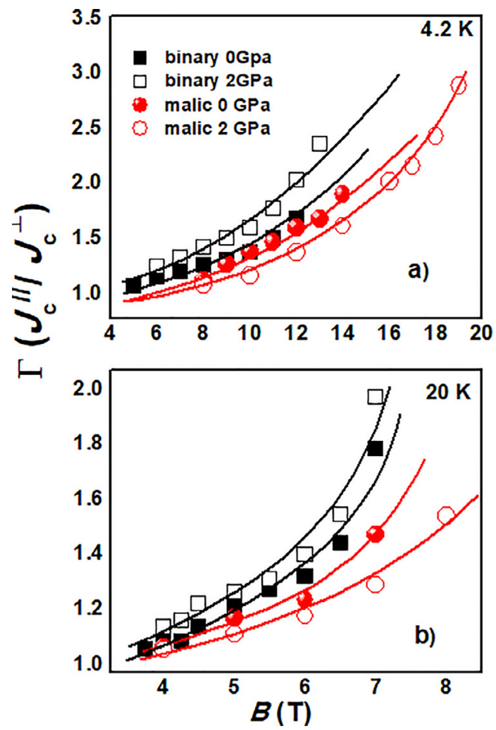

Fig. 2. Variation of the anisotropy, Γ ($J_c^{//}/J_c^{\perp}$) vs. magnetic field at (**a**) 4.2 K and (b) 20 K for binary and $C_4H_6O_5$ alloyed MgB_2 tapes (Monel sheath, Nb barrier) at 0 and 2 GPa. Reaction conditions: 600 °C/4 h.

electron microscopy (FEG-SEM). A JEOL JEM-3000F, a 300 keV high-resolution transmission electron microscopy (HR-TEM) has been used to obtain high-magnification images. The filaments were extracted from the MgB_2 tapes treated at 0 and 2 GPa to investigate the grain connectivity and defects in the lattice using FEG-SEM and TEM.

3. Results and discussion

The geometrical changes, the variation of lattice parameters, carbon content and T_c as a function of pressure with and without applying CHPD on *in situ* MgB_2 tapes (binary and doped with 10 wt% $C_4H_6O_5$) are listed in Table 1. After applying CHPD at 2 GPa, the area of ($Mg+B$) and the filling factors decreased in both binary and alloyed tapes (Table 1). The aspect ratio *a/b* of the present malic acid added *in situ* MgB_2 tapes showed a variation from 4.34 to 7.60 after applying CHPD up to 2 GPa. On the other hand, it did not change much for binary tapes under the same conditions. The variation of J_c as a function of B at 4.2, 20 and 25 K of the malic acid doped unpressed tape (aspect ratio $a/b=4.34$) and pressed tape at 2 GPa (aspect ratio $a/b=7.60$) for applied magnetic fields in parallel and perpendicular direction is shown in Fig. 1. A gradual shift of the curves $J_c^{//}$ and $J_c^{\perp}$ as a function of B towards higher values has been observed by the application of pressure. At $p=2$ GPa, the values of $B(10^4)^{//}$ and $B(10^4)^{\perp}$ were increased from 11.3 to 13.2 T, by 1.9 T and 10.6 - 12 T or by 1.4 T, respectively.

The variation of the anisotropy factor, Γ vs. B at 4.2 K and 20 K for binary and malic doped tapes before and after CHPD is shown in Figs. 2(a) and (b). The value of Γ for the binary tape after CHPD increased with increasing aspect ratio from 3.87 to 6.96, in agreement with Kovac et al. [26]. In malic acid doped tapes, we observed that Γ decreased after CHPD, even though the aspect ratio increased from 4.34 to 7.60. However, all these tapes have been fabricated by the same conditions concerning deformation, heat treatment and sheath materials. The reason behind the present differences can be explained by the combination of two effects that influence the J_c anisotropy: carbon substitution and texture. The J_c anisotropy is more pronounced in the case of binary tapes after CHPD, where the texture effect is more visible. In the *in situ* process, the texture is usually introduced by the mechanical deformation of the hexagonal Mg

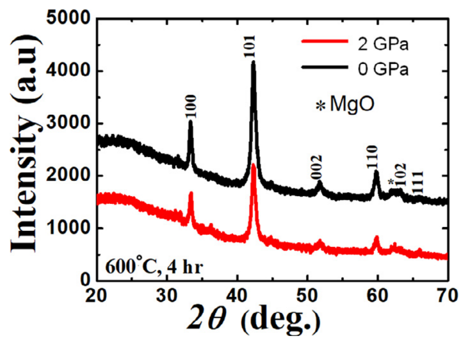

Fig. 3. X-ray diffraction patterns for the $C_4H_6O_5$ alloyed MgB_2 tapes (Monel sheath, Nb barrier) after 2 GPa for observing the *c*-axis grain alignment.

powder. On the other hand, it is well established that the malic acid addition improved the critical current densities at 4.2 K and 20 K [7,14], due to effective carbon substitution. The texture of the tape is affected by the impurity scattering due to carbon- doping and hence, the anisotropy in malic acid doped tapes is lower than in binary tapes, in good agreement with other researchers [21,26]. At both operating temperatures, the striking result for the malic doped tape is that there is a further reduction of anisotropy after pressing at 2 GPa. From Table 1, it is clearly seen that the aspect ratio is larger after pressing at 2 GPa in malic acid doped tapes: this should cause stronger texture in the filament and hence the anisotropy factor in these tapes should be higher after CHPD. But the interesting point is that malic acid tapes pressed at 2 GPa exhibit lower T_c values and a higher carbon-substitution (the *a* lattice parameter is slightly decreased) than the unpressed ones, which certainly causes the reduction of anisotropy. This statement is further supported by Fig. 3, showing the XRD patterns of malic acid treated MgB_2 layers obtained from Nb/Monel sheathed tapes, at 0 and at 2 GPa. These XRD patterns indicate that the main phase is well developed hexagonal MgB_2, with small peaks corresponding to MgO impurity. The effect of high non-superconducting MgO impurity is detrimental on the transport current carrying capability of MgB_2 superconductors. Our previous study shows that higher amount of malic or tartaric doping on MgB_2 enhances the possibility of MgO generation and hence, leads to poor superconducting properties [29]. In this work, we have chosen the optimum (10 wt% malic acid) doping level. It is also interesting to notice from XRD peaks that the relative intensity of the (0 0 2) diffraction peaks of malic treated MgB_2 at 0 GPa is higher than that one after 2 GPa. This result clearly indicates that *c*-axis grain alignment of the malic treated MgB_2 layer is obtained by the CHPD process, and that the degree of alignment decreases with increasing pressure and additional carbon substitution. The Γ is influenced by this *c*-axis grain alignment as discussed above.

At 4.2 and 20 K, almost isotropic behavior found at lower fields in both binary and malic doped tapes, regardless of CHPD condition. From Figs. 2(a) and (b), it is interesting that even at *a/b* = 7.60 after pressing at 2 GPa, the anisotropy ratios Γ were quite small at 4.2 and 20 K in malic tapes compared to 0 GPa: for example, at 4.2 K after 2 GPa, it is $\Gamma = J_c^{//}/J_c^{\perp}$ = 1.22 and 1.60 at 10 and 14 T, respectively (from Fig. 1) for malic doped tapes. These values are considerably higher with tapes treated at 0 GPa (*a/b* = 4.34), the values being 1.35 and 1.80 at 10 and 14 T, respectively. The corresponding values for 20 K at 2 GPa were 1.13 and 1.30 at 5 T and 7 T, respectively, the values for 0 GPa being 1.20 and 1.51 at 5 T and 7 T, respectively. For *in situ* MgB_2 tapes, these values of Γ are certainly among the lowest ones ever reported [23–24, 27], which is assumed to be the consequence of various parameters during the low energy ball milling e.g. mass of the balls, rotating speed of the planetary mill and milling duration. More C substitution and reduction of T_c after CHPD played an important role here. From Table 1, it follows that the lattice parameters *a* decreased and hence the C substitution increased with pressure, as documented by the values $a = 3.0749$ Å and $c = 3.5240$ Å at $p = 0$ GPa, and $a = 3.0721$ Å and $c = 3.5236$ Å after pressing at $p = 2$ GPa. This explains the enhancement of J_c and B_{irr} as well as the reduction of anisotropy after CHPD at all fields, in agreement with Kovac et al. [23-26] who described a series of J_c anisotropy results correlated with various additives, sheath materials and mechanical deformations. In CHPD treated tapes, the amount of C substitution from malic acid into boron is enhanced. This is due to a reduced reaction path under the same reaction conditions, i.e. 600 °C/4 h. This can be explained by an improved packing factor of the precursor powders in the densified filaments, resulting in shorter reaction paths. This phenomena makes the C substitution more effective in doped tapes, leading to a reduction of T_c as well as anisotropy due to poor texturing.

This interesting observation can be further supported by the microstructural characterization conducted by SEM and TEM shown in Figs. 4(a), (b) and (c). The observed improvement of J_c after applying CHPD at 2 GPa (Fig. 1) in malic doped wires [7] is due to the appearance of less voids (Fig. 4(a) and (b)) and to additional defects in the lattice (Fig. 4(c)). High resolution scanning transmission electron microscope (STEM) investigations revealed that the subgrain lattice is randomly oriented and that many crystal lattice defects, such as stacking faults and dislocations found in subgrains are due to the typical combination of C doping and cold pressure. In our previous work [30], we investigated this interesting behavior of CHPD in binary and malic acid doped wires, based on the T_c distribution as well as on connectivity and the percolation. In this work, the results arising from the analysis of the T_c distribution and those from resistivity measurements were combined and found that the minimum superconducting volume fraction required for the percolation of a superconducting path is strongly reduced in CHPD treated samples.

The effect of operating temperature on Γ after pressing at 2 GPa (only with malic acid doped tapes) with similar *b/a* ratio is shown in Fig. 5. For all operating temperatures, a sharp and nearly linear increase of Γ has been observed at lower fields, which turns into an exponential one at higher fields. The measured Γ values increased rapidly with temperature e.g. Γ ≈ 1 (8 T) at $T = 4.2$ K, Γ ≈ 1.6 (8 T) at $T = 20$ K and

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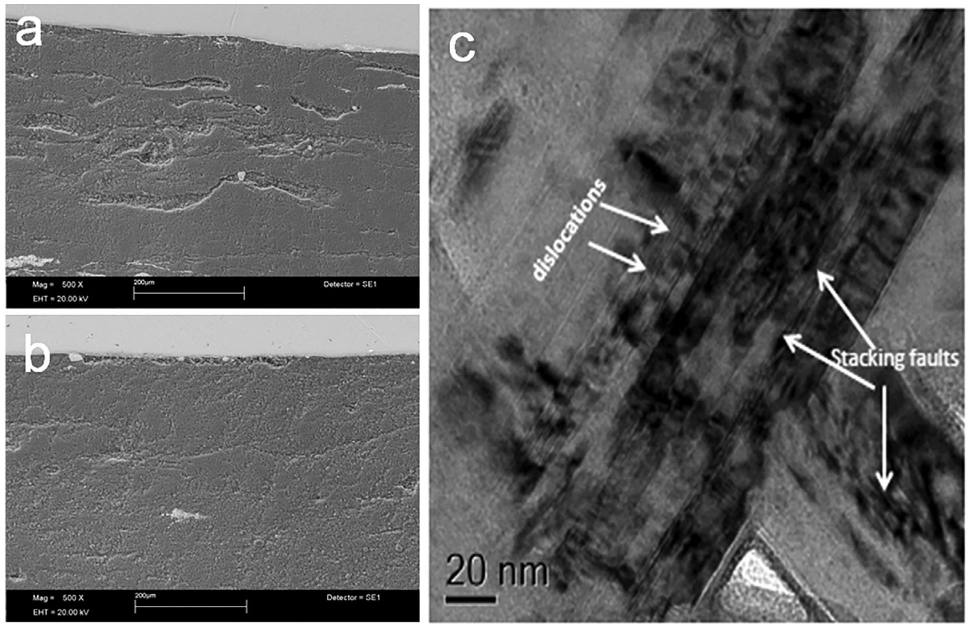

Fig. 4. (a, b) High resolution scanning electron microscopy (SEM). (c) Dark field (DF) scanning transmission electron microscopy (STEM). Investigations revealed a reduced porosity, stacking faults and dislocations, due to the typical combination of C- doping and cold pressure.

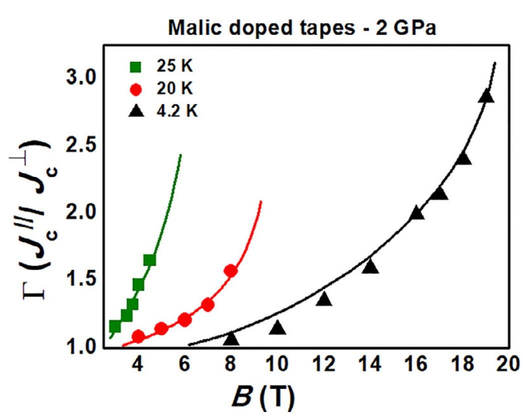

Fig. 5. Variation of the anisotropy, Γ ($J_c^{//}/J_c^{\perp}$) vs. magnetic fields at 4.2, 20 and 25 K for $C_4H_6O_5$ alloyed MgB_2 tapes (Monel sheath, Nb barrier) after 2 GPa. Reaction conditions: 600 °C/4 h.

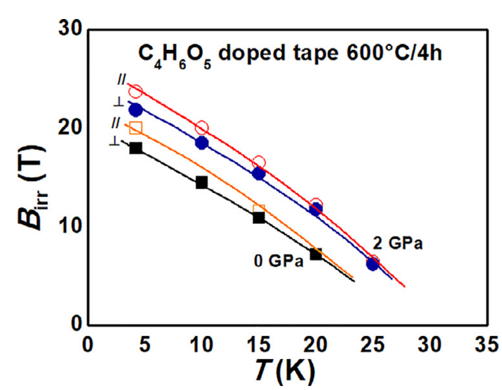

Fig. 6. Variation of the irreversibility field (B_{irr}) vs. operating temperatures (T) for $C_4H_6O_5$ alloyed MgB_2 tapes (Monel sheath, Nb barrier) at 0 and after 2 GPa. Reaction conditions: 600 °C/4 h.

$\Gamma \approx 1.65$ (4.5 T) at $T = 25$ K. High J_c anisotropy at elevated temperatures can lead to serious problems in designing superconducting magnets for various applications, such as in the case of Bi-2223/Ag tapes or YBCO thin films at 77 K, where the total transport current of the coil winding has strongly been reduced by the high radial field component [31].

The variation of the irreversible fields (B_{irr}) with operating temperature at 0 and 2 GPa of malic acid doped tapes has been plotted in Fig. 6. It has been shown that the expected convergence of $B_{irr}^{//}$ and $B_{irr}^{\perp}$ when approaching near T_c. At 20 K, $B_{irr}^{//}$ and $B_{irr}^{\perp}$ are almost equal at around 12.2 T for 2 GPa and at 7.7 T for 0 GPa. The difference between $B_{irr}^{//}$ and $B_{irr}^{\perp}$ increases at lower temperatures in tapes treated with both 0 and 2 GPa pressures. The extrapolation to $T = 0$ yields $B_{irr}^{//}(0) = 22$ T and $B_{irr}^{\perp}(0) = 20$ T for the tape with 0 GPa, the corresponding values for the tape treated at 2 GPa being 25 and 23 T, respectively.

It is known [19,32] that up to 30% of the volume are transformed to MgB_2 after mechanical alloying ($Mg + B$ mixtures treated with high energy ball milling for long time). After reaction, large values of the anisotropy factor Γ are observed, in particular at higher fields, due to a strong texturing of the MgB_2 phase in the tape filaments. In this work, no trace of reacted MgB_2 was observed by X-rays in the mixtures of $Mg + B$ after low energy ball milling. However, a degree of texturing was still present in the reacted binary tapes after 2 GPa, especially at 4.2 K, as evidenced by the different Γ in Fig. 2. The anisotropy in tapes based on low energy ball milled powders is originated due to the particular conditions at the interface between MgB_2 filament and Nb barrier during the reaction process [23,24]. As shown by Lezza et al. [27], the degree of texture is indeed maximum at the MgB_2/Nb interface and decreases towards the center of the tape. In our previous articles [6,7,31], we reported that CHPD induces a strong improvement of J_c and B_{irr} in binary and alloyed MgB_2 wires due to better grain connectivity.

4. Conclusion

In this article, it has been shown that the effects of CHPD on anisotropy in binary tapes as well as in malic acid doped MgB_2 tapes are correlated. For the pressed binary tape at 4.2 and 20 K, the anisotropy ratios Γ were larger compared to unpressed tapes due to a certain rolling induced degree of texturing in the tape filaments. The Γ values were reduced for the CHPD treated malic doped tapes densified at 2 GPa at 4.2 and 20 K compared to binary and unpressed malic acid doped tapes. Thus, CHPD contributes to the significant anisotropy reduction in MgB_2 tapes doped with malic acid by substituting a higher amount of C into the lattice due to the shorter reaction path in densified filaments. This reduction of anisotropy may be caused from the random oriented subgrain defects in the lattice. This observation is further supported by the reduction of the *c*-axis grain alignment in the MgB_2 layer obtained by both the applied pressure and the additional carbon substitution. The value of Γ is influenced by this *c*-axis grain alignment, as discussed above. In summary, a significant reduction of the anisotropy Γ has been found in MgB_2 tapes doped with malic acid after the CHPD process, which may be advantageous for superconducting magnet design and application.

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