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Development of Doped MgB_2 Wires and Tapes for Practical Applications

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***Abstract*—A review of current developments in the study of chemical doping effect on the superconducting properties of MgB_2 wires and tapes is presented, based on the known literature data and our own results. The critical current density of MgB_2 can be improved through various kinds of dopants. Among these dopants, doping with carbon-containing materials seems to be the most effective way to improve the J_c performance. The doping effect of carbon in different forms and carbon-based compounds such as SiC, nano-C, metal carbides, as well as aromatic hydrocarbon and carbohydrate on the J_c-B characteristics of MgB_2 was discussed in detail. The C can be incorporated into the MgB_2 crystal lattice by replacing boron, and thus B_{c2} is significantly enhanced due to selective tuning of impurity scattering of the π and σ bands in the two-band MgB_2. Besides the efforts of increasing B_{c2} by carbon doping, the fine grain size and nano-size inclusions caused by doping would create many flux pinning centres improving the J_c-B property of MgB_2. Based on these considerations, we suggested some principles for the selection of dopants.**

***Index Terms*—MgB_2, critical current, doping, Carbon, tapes and wires.**

I. INTRODUCTION

MgB_2 is a promising superconductor for practical applications in the field of superconducting magnets for MRI, due to its rather high T_c, relatively low material costs and weak-link-free behavior[1]. The development of MgB_2 tapes and wires was much faster than for many others HTS and LTS materials and commercial wires and tapes became available only a few years after the discovery of MgB_2.

From the view point of applications, the critical current density J_c in high magnetic fields is crucially important. However, the pristine MgB_2 always shows lower J_c values because of low upper critical field (B_{c2}) and poor flux pinning. In order to improve J_c–B properties, a number of experimental techniques, including chemical doping [2, 3], irradiation [4], magnetic field annealing [5, 6], and ball-milling methods [7], have been attempted. Compared to other methods attempted, chemical doping with carbon-containing materials was thought as the convenient and effective way to enhance the J_c-B properties of MgB_2. Therefore, effects of such additives are being actively investigated, and some effective dopants have been suggested [2, 3, 8-15]. The additions of SiC, nano-C, carbon nanotubes, B_4C, as well as aromatic hydrocarbon and carbohydrate are effective to improve the J_c-B characteristics of MgB_2 [2, 3, 10-15]. When MgB_2 is doped with these materials, C substitutes for B and introduces electron scattering and impurity scattering which reduce the mean free path and the coherence length ξ, hence increasing B_{c2} [16]. At the same time, they are also effective in the enhancement of pinning strength by introducing lattice distortion and more grain boundaries.

Here we present a review of the recent results in the doping of MgB_2 tapes and wires using different types of carbon sources and some promising processes combined with carbon doping. Based on these results, we suggest some principles for the selection of dopants and give a perspective for the future development of this superconductor.

II. CARBON DOPING

The effects of carbon doping on superconductivity in MgB_2 compound has been extensively studied. The J_c-B performance can be greatly improvement by doping with various different carbon sources, such as nanocarbon [11, 17], diamond [18], graphite [19], and carbon nanotubes [12].

The authors' group has systematically studied the nano-C doping effect on the critical temperature, J_c-B property and B_{c2} of MgB_2 tapes [11, 20-21]. Soon thereafter, our results have been confirmed by many groups worldwide [22-24]. The addition of nano-C causes substitution of boron by carbon, which decreases the critical temperature and increases the upper critical field as well as the current density in high magnetic fields. Fig. 1 shows the transition temperature curves of tapes with different C doping levels determined by susceptibility measurements. The T_c decreases monotonically with increasing nano-C doping level. The depression of T_c is caused by the carbon substitution for B. On the other hand, the carbon substitution for B was found to enhance J_c in magnetic fields. Figure 2 shows the transport J_c at 4.2 K in magnetic fields for Fe-sheathed MgB_2 tapes with various amounts of nano-C doping from 0 to 10 at% that were heat-treated at 650 °C. It can be seen that, in measuring fields of up to 14 T. all the doped tapes exhibited superior J_c values compared to the pure tape. The highest J_c value of the Fe-sheathed tapes was achieved by the 5 % nano-C addition; further increasing C doping ratio caused a reduction of J_c in magnetic fields.

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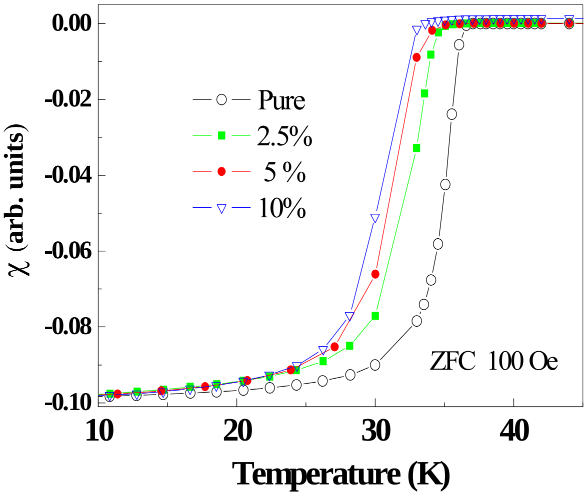

Fig. 1. Normalized magnetic susceptibility versus temperature for the samples with different doping level [11].

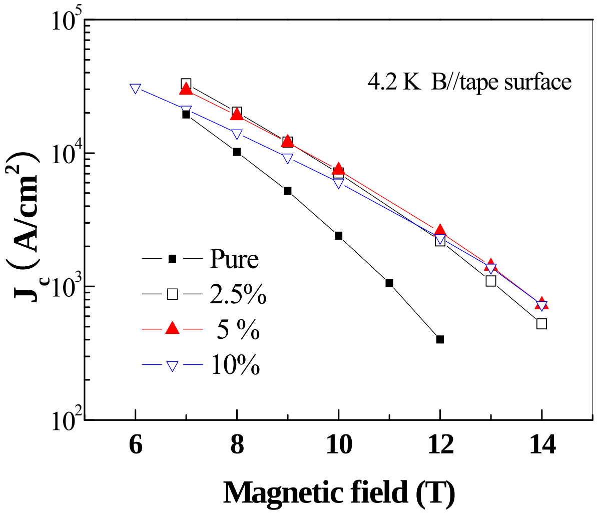

Fig. 2. Critical current densities at 4.2 K versus magnetic field in MgB_2 tapes with nano-C doping level from 0 to 10 at%, which were heat-treated at 650°C, at 4.2 K in magnetic fields.

Fig. 3 shows the J_c at 4.2 K and 10 T for 5% C-doped tapes that were sintered at different temperatures ranging from 600 to 950 °C. From Fig. 3, we immediately notice that the sintering temperature has a significant effect on the J_c-B performance. The J_c values of doped samples increased systematically with increasing sintering temperatures. The tape sintered at 950 °C exhibited the highest J_c values with excellent J_c-B performance compared to all other samples: at 4.2 K, the transport J_c reached 2.11×10^4 A/cm^2 at 10 T. Higher annealing temperatures promote the reaction of C substitution for B [22], thus enhancing flux pinning and improving the high-field J_c.

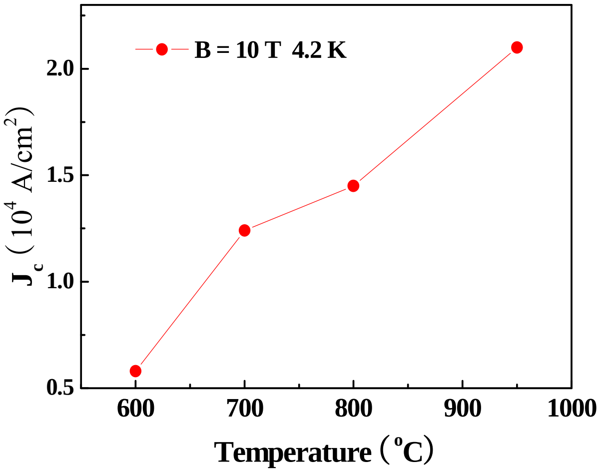

Fig. 3. Transport J_c at 4.2 K in magnetic fields up to 18 T for 5 at.% C-doped MgB_2 tapes sintered at various temperatures.

So far, the only element well known to increase B_{c2} is carbon. The significant improvements of B_{c2} in carbon doped samples have been reported by several groups [25–28]. Figure 4 shows the temperature dependence of B_{c2} and B_{irr} for the pure MgB_2 tapes and nano-C doped samples, where the B_{c2} and B_{irr} obtained from the 10% and 90% values of the normal-state resistance ρ. Clearly, as a result of nano-C doping, the B_{c2} (B_{irr})-T curve became steeper, indicating an improved B_{c2} and B_{irr}. For instance, at 20 K, the B_{irr} achieved 9 T for C-doped tapes, which was comparable to the B_{c2} at 4.2 K of NbTi conductors [29].The enhancement of B_{c2} can be interpreted by two-band impurity scattering. The C can enter the MgB_2 structure by substituting into boron (B) sites, and thus J_c and B_{c2} are significantly enhanced due to selective tuning of impurity scattering of the π and σ bands in the two-band MgB_2 [16].

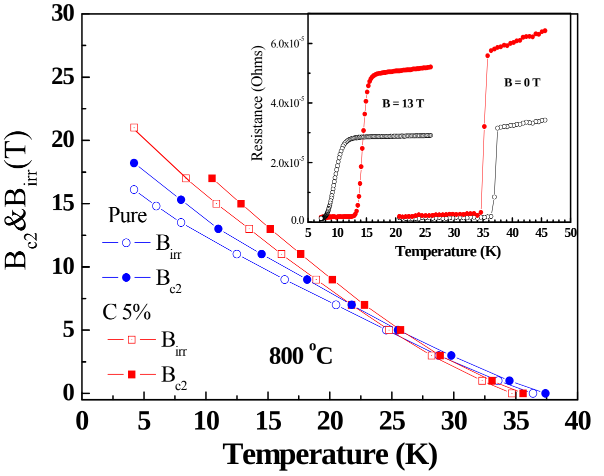

Fig. 4. B_{c2} and B_{irr} values as a function of the temperature for the pure and 5 at.% C-doped MgB_2 tapes.

The type of carbon sources is an important parameter determining the effect of doping on the J_c-B performance.

Figure 5 shows the transport J_c at 4.2 K in magnetic fields for MgB_2 tapes with various carbon sources studied by our group. It should be noted that the J_c value of tapes doped with activated carbon was lower than for samples doped with other carbon sources. The average particle size of activated carbon is over 200 nm, much larger than that of nano-C. So the degree of reacting activated carbon particles with MgB_2 is much lower than in the case of nano-C particles. Thus too few carbon atoms are incorporated into the MgB_2 lattice during its formation. Also, these large particles existing in MgB_2 matrix will block the superconducting current flow, and thus decrease the MgB_2 superconducting connectivity. However, the J_c-B properties for hollow carbon spheres (HCS) and C_{60} doped tapes are better than for nano-C-doped samples. Compared to nano-C dopants, HCS and C_{60} have very special geometrical configuration. They can be broken into nanoflakes and particles during the process. These nanocarbon flakes and particles would be easily incorporated into the crystal lattice of MgB_2 and substituted into B sites via the reaction.

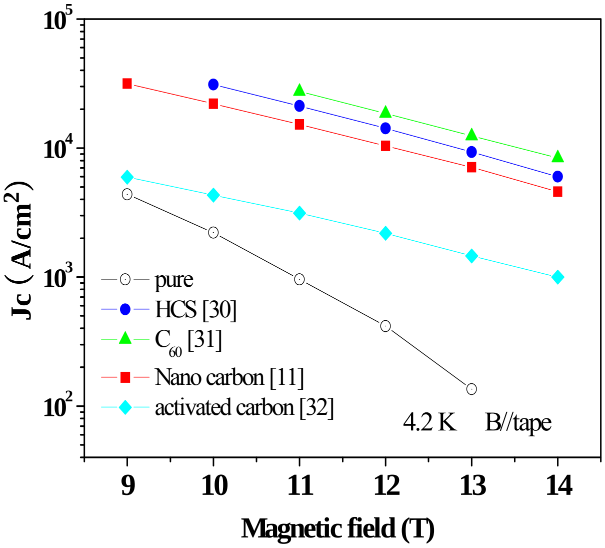

Fig. 5. The transport J_c at 4.2 K in magnetic fields for MgB_2 tapes with various carbon sources studied by our group.

Haßler *et al.* [33] have reported the highest J_c (10^4A/cm^2 at 16.4 T and 4.2 K) at high magnetic fields by high-energy ball milling technique with nanocarbon alloying. This excellent performance can be attributed to the nanocrystalline grain size with a high amount of grain boundaries, and the effectivity of carbon substitution by mechanical alloying [34, 35]. The grain boundaries contribute to better flux pinning and the carbon doping greatly improves B_{c2} by impurity scattering. The results demonstrated that nano-C doping combined with high energy ball milling techniques is an effective method for further enhancing the current capacity of MgB_2.

III. SILICON CARBIDE DOPING

The significant enhancement of J_c, B_{irr}, and B_{c2} in MgB_2 by nano-SiC doping was first shown by Dou's group [2]. A high B_{irr} of 29 T and B_{c2} of 37 T have been achieved for nano-SiC doped MgB_2 wires [3, 36]. Fig. 6 shows a comparison of transport J_c at 4.2 K for SiC doped samples by the authors' group [37], Dou *et al.* [38], Matsumoto *et al.* [39], and M. Tomsic *et al.* [40]. The in-field J_c for the nano-SiC doped samples increased by more than one order of magnitude, compared with the undoped samples. Dou *et al.* have proposed a dual reaction model to explain the enhancement in electromagnetic properties by SiC doping [38]. In this model, fresh reactive C is released from the SiC at low temperature (600 °C), when SiC reacts with Mg to form Mg_2Si. This reactive C can effectively substitute into B sites, which leads to the enhancement of the B_{c2}. In addition, the by-products, such as highly dispersed fine particles, including Mg_2Si, and the excess nanosize C can be embedded into the MgB_2 matrix and act as flux pinning centers.

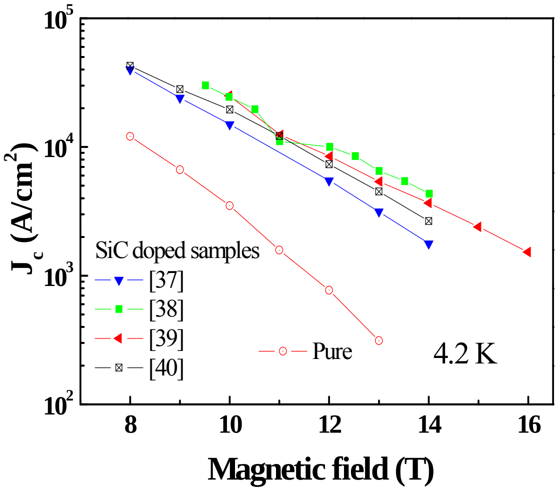

Fig. 6. A comparison of J_c for SiC doped samples from various groups.

IV. ORGANIC COMPOUNDS DOPING

Yamada *et al.* have reported that aromatic hydrocarbon addition to MgB_2 can enhance the flux pinning in MgB_2 tapes [13]. However, this organic solvent is very volatile at ambient pressure and it is difficult to control the doping level. Later, Kim *et al.* have reported that carbohydrate-doped MgB_2 bulks exhibited a positive effect in improving the J_c values at high magnetic field [14]. Our group's recent results [15, 41-42] show that the J_c-B performance of MgB_2 tapes with carbohydrate dopant can be as good as for nano-SiC and nano-C dopants [2, 11]. Organic material can decompose at relatively low temperature, and generate reactive carbon atoms before the MgB_2 phase formation. The reactive carbon atoms and small sized impurities are favorable for good superconducting properties of MgB_2. Compared to nano-sized C or SiC, organic material doping can achieve a more uniform dispersion within the MgB_2 matrix [43].

We have investigated the effect of doping by several organic compounds on the superconducting properties of MgB_2 tapes [15, 41-42]. Our results demonstrated that both the J_c and flux pinning ability of MgB_2 tapes are significantly improved through these dopants. Compared to the undoped samples, all doped tapes showed an enhancement of J_c values in high-field region by more than an order of magnitude, as shown in Fig. 7. For example, at 4.2 K and in a field of 12 T, the J_c value of the 10 wt% maleic anhydride doped sample reached 9.2×10^3 A/cm^2, more than 23-fold improvement compared to the pure samples. These J_c values of the tapes investigated in this work are comparable to the best nano SiC doped tapes reported so far [38], and highlight the importance of organic compounds doping for enhancing the J_c of MgB_2 superconductors.

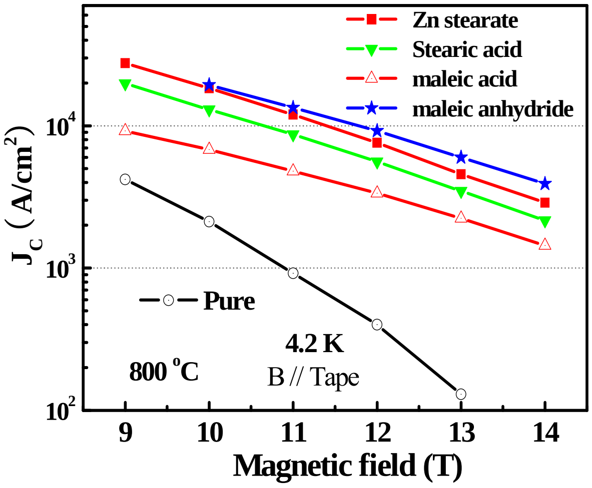

Fig. 7. J_c-B properties of tapes doped by pure and organic compounds.

Fig. 8 shows the typical TEM micrographs for the Zn stearate and stearic acid doped tapes. When an organic compound is heated at above the decomposition temperature, the highly reactive C released from decomposition can effectively substitute into B sites. Substitution of C for B induces disorder on the lattice sites, which leads to the enhancement of the B_{c2}. At the same time, C substitution causes reduction in the grain size, and hence enhances the grain boundary pinning. Moreover, the TEM examination revealed that there are a number of impurity phases in the form of nano-size inclusions in doped samples. Therefore, a combination of C substitution-induced B_{c2} enhancement as well as the strong flux pinning centers are responsible for the superior J_c-B performance of organic compounds doped samples.

The authors' group also found that the oxygen amount in carbohydrate additives strongly affect J_c-B performance of MgB_2 [42]. The transport J_c and the connectivity were observed to decrease as oxygen content in dopants increased. The higher MgO content and porosity formed during the reaction in high oxygen doped samples is responsible for their low J_c values. As a result, the decrease of oxygen content in dopants would be a key point to further improve the transport J_c of MgB_2.

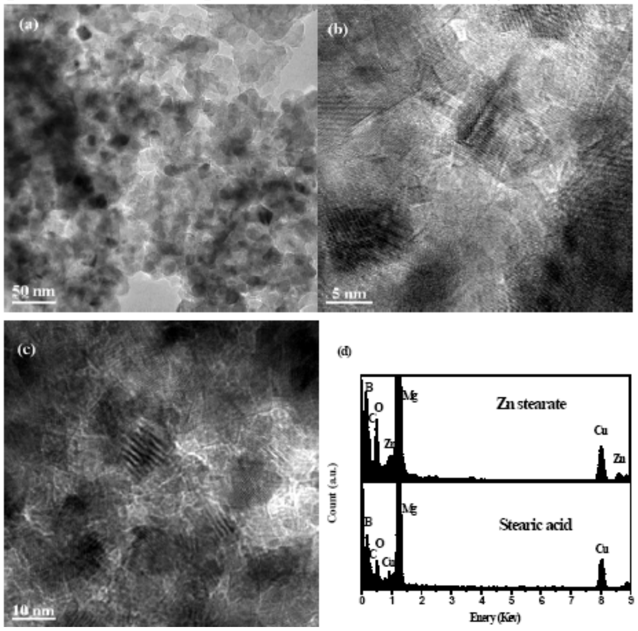

Fig. 8. The TEM images of the Zn stearate and stearic acid doped samples.

Recently, Flukiger's goup found that the J_c and B_{irr} in $C_4H_6O_5$ alloyed MgB_2 wires were significantly enhanced by cold high pressure densification (CHPD) [44]. $C_4H_6O_5$ alloyed MgB_2 square wires after conventional processing were submitted to cold high pressure densification (CHPD), resulting in the highest J_c values reported so far for nearly isotropic *in situ* wires: $B(10^4)^{\perp} = 12.5$ T and $B(10^4)^{\parallel} = 12.7$ T at 4.2 K. Here, $B(10^4)$ means the field at which $J_c = 1\times10^4$ A/cm^2. The results demonstrate that organic material doping combined with CHPD techniques is one of promising methods for the enhancement of current capacity of MgB_2.

V. CARBIDE DOPING

The effect of metal carbides like ZrC [45], NbC [46], TaC and MoC_2 [47] has been studied in our group. We found that these carbides show little effect on J_c, because they are stable in contact with Mg and B even at high temperature; thus no fresh reactive C is available for incorporating into the MgB_2 lattice during its formation.

VI. SIMULTANEOUS ADDITIVES

Yamada *et al.* found that the additive combination of ethyltoluene + SiC were very effective for the improvement of J_c-B properties of MgB_2 tapes [48]. The highest J_c values at 4.2 K reached 1.4×10^4 A/cm^2 in 12 T for tapes with added ethyltoluene and SiC. This can be attributed to a cumulative effect on J_c - one comes from the addition of ethyltoluene and the other comes from the carbon substitution for boron by the SiC addition.

The substitution of boron by carbon is known to enhance the impurity scattering and thus of the critical field. In addition, the pinning behavior is expected to be improved by nanosize precipitations. Since the two mechanisms are independent, their effect on J_c is expected to be cumulative [49, 50].

Flukiger's group has reported on the effects of additive combinations B_4C+SiC and B_4C+LaB_6. For both additive combinations, J_c(20 K) at high fields is markedly higher than for SiC additives, in contrast to the data at 4.2 K. Mikheenko *et al.* [51] used a combination of Dy_2O_3 for pinning, together with B_4C for doping and successfully improved the J_c-B performance of MgB_2 in the intermediate field regime (2–5 T) at 20 K. The optimum amount of Dy_2O_3 and B_4C additions is 0.5 wt % of Dy_2O_3 and 0.04 wt % of B_4C, yielding a J_c (20 K) of 10^5 A/cm^2 at 2.7 T. These results demonstrate that simultaneous additives are promising for applications at 20 K.

Recently, our group realized the simultaneous introduction of "scattering + pinning" using a single dopant of organic rare earth salt. Figure 9 shows the XRD results. Rare earth borides and MgO were detected as impurity phases in doped samples. It can be seen that the position of (110) peak slightly shifts to higher angles due to the organic rare-earth salt addition, meaning a decrease in the a-axis lattice parameter. It indicates that carbon has been doped into the B sites in the crystal lattice.

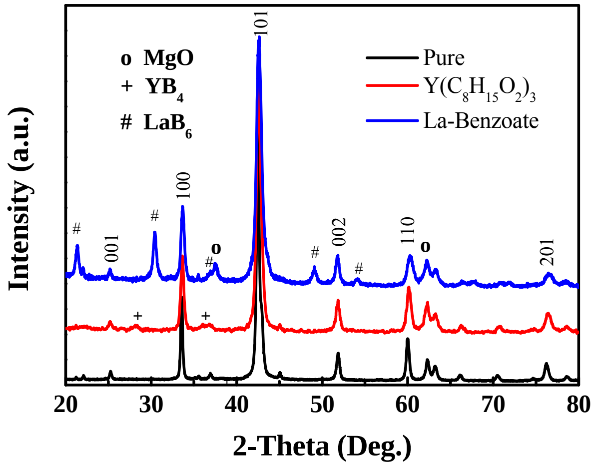

Fig. 9. XRD results for the samples doped with organic rare-earth salts.

Fig. 10 shows the transport J_c at 4.2 K in magnetic fields for two organic rare earth salt doped samples. It is evident that both doped samples exhibited superior J_c values compared to the pure one. For example, at 4.2 K and in a field of 12 T, the J_c value for La-Benzoate doped tape reached 1.84×10^4A/cm^2, 23 times higher than that of undoped tape.

When organic rare-earth salt is decomposed at low temperature, the highly reactive C can effectively substitute into B sites. Simultaneously, the rare-earth boride inclusions with nano-sizes resulting from the reaction also can act as effective flux pinning centers and hence improve the J_c performance of organic rare-earth salt doped samples.

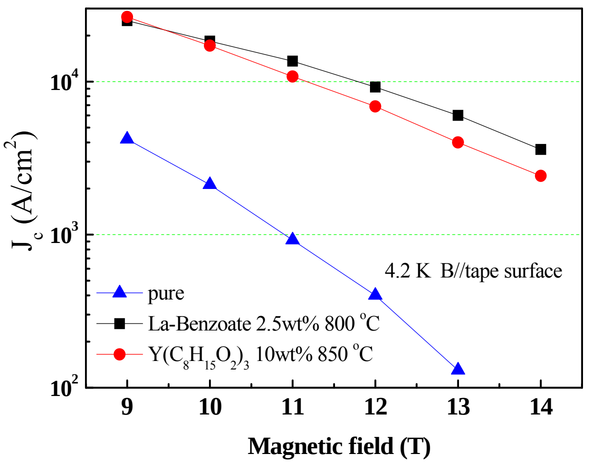

Fig. 10. J_c-B properties of pure and organic rare-earth salts doped tapes.

VII. DISCUSSION

In the past several years, various kinds of dopants have been introduced into MgB_2 [8, 52-53]. Fig. 11 shows the doping effects on the J_c-B properties of MgB_2 with various kinds of dopants studied in our group. Based on the results mentioned above, we suggest some guiding principles for the selection of dopants.

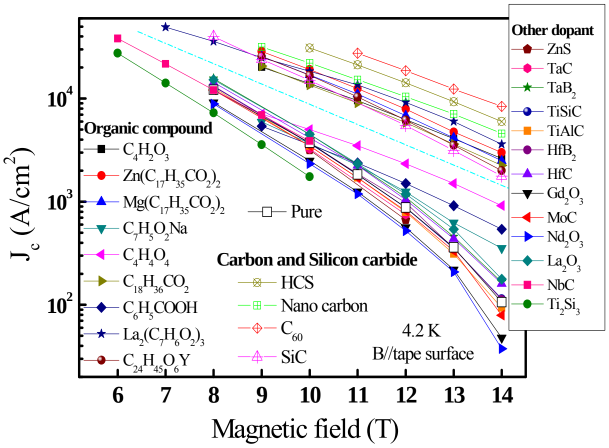

Fig. 11. The transport J_c at 4.2 K in magnetic fields for MgB_2 tapes with various kinds of dopants studied in our group [47].

1. Dopants should contain carbon. Carbon is the only element confirmed to be able to remarkably enhance B_{c2} of MgB_2.
2. In selecting dopants, reaction chemistry is an important consideration. Carbon-containing dopants with high reactivity decompose or react at low temperatures, releasing fresh C which can substitute for B efficiently, resulting in small grains.
3. Proper amount of secondary phases induced by carbon sources may enhance the vortex pinning, but high amount of impurities strongly degrade the connectivity if present between MgB_2 grains, as seen in O-contaminated MgB_2 [42].

The doping directly influences the B_{c2}, flux pinning and connectivity of MgB_2. We should carefully tune the balance between them and optimize the doping effects.

Many dopants can significantly enhance the B_{c2} and J_c of MgB_2, but often at the expense of homogeneity and connectivity. Furthermore, the porosity of *in situ* made MgB_2 greatly reduces the super current path. More research and new techniques are needed to increase homogeneity and connectivity of the MgB_2.

VIII. CONCLUSION

The experimental results show that both the B_{c2} and J_c of MgB_2 wires and tapes doped with carbon sources are significantly enhanced. The performance of MgB_2 is expected to further increase in the coming years through improved flux pinning, homogeneity and connectivity. In particular, the excellent J_c-B performance of MgB_2 wires and tapes was achieved through C-based doping combined with CHPD or high energy ball milling techniques. This result demonstrates that MgB_2 has potential for use in high magnetic fields and as a strong competitor for the currently used NbTi and Nb_3Sn conductors.

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