Enhanced superconducting properties of rare-earth oxides and graphene oxide added MgB$_2$

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In this paper, the effects of addition of (i) graphene oxide (GO), (ii) a series of rare-earth (RE, RE = La, Sm, Eu, Gd, Tb and Ho) oxides (REO) and (iii) a mixture of GO and rare-earth oxides (GO + REO) on the superconducting properties of MgB$_2$, have been studied with the help of electrical transport and magnetic measurements. All the samples have been prepared following the standard solid-state reaction route. We have used an optimum value of 1 wt% REO and 3 wt% GO for addition on the basis of previous studies. X-ray diffraction studies confirm the formation of hexagonal crystal structure (space group P6/mmm) of MgB$_2$ with small amounts of REB$_x$ ($x = 4$ and 6) and MgO impurity phases in all the synthesized samples. We observe that the critical current density, $J_c$ and upper critical field $H_{c2}(0)$ improve significantly in the REO-added and GO-added samples with no significant change in critical temperature, $T_c$. A substantial enhancement in $J_c(H)$ and $H_{c2}(0)$ is observed with the GO + REO addition in MgB$_2$. The different flux pinning mechanisms in all the samples are studied and it is found that the point pinning is the dominant mechanism in the GO-added samples and grain boundary pinning is the dominant one in the REO added samples. We have seen the combined effect of both types of flux pinning mechanisms in GO + REO added MgB$_2$.

1. Introduction

With its high critical temperature of 39 K [1], MgB$_2$ presents a potential material for technological applications. Its simpler crystal structure, high critical current densities $J_c$, high upper critical magnetic field, $H_{c2}$, transparent grain boundaries to supercurrent and low anisotropy make this material preferable to high-temperature (HTS) as well as conventional low temperature superconductors (LTS) [2]. The lower cost and ease of synthesis are the added advantages of this material. One of the concerns of using polycrystalline MgB$_2$ for technological application is its deteriorating superconducting properties at high-fields due to weak pinning [3,4]. Many groups have reported $J_c$ values as high as $\sim 10^5$ A/cm$^2$ in the absence of an applied magnetic field at 5 K and $\sim 10^4$ A/cm$^2$ at 4.5 T and 5 K [5–7]. Many approaches have been employed to introduce effective pinning sites to improve the $J_c(H)$ behavior of MgB$_2$. It has been well studied that the introduction of grain boundaries, voids, crystal defects, dislocations, strain, impurity phases and nanophase inclusions into the superconductor provides very effective pinning sites. In this regard, many efforts have been made to improve the in-field superconducting properties of MgB$_2$. Many studies have shown that the lattice defects created by substitutions at Mg and B-sites can lead to improved $H_{c2}$ and $J_c(H)$. Among the dopants, boron substitution with carbon in the form of carbon [8–10] and carbon nanostructures [11–15] is reported to improve $J_c$, significantly at high magnetic fields. Also many carbon containing compounds [16–20] are reported to have enhanced $H_{c2}$ and $J_c$. Carbon doping for B induces a crystal lattice distortion, especially in the boron plane, thus giving rise to strong intra-band scattering in the $\sigma$-band [21,22] which improves the pinning properties of MgB$_2$. One problem with C doping for B is the reduction in $T_c$ [9]. However, it has been reported that the effect of C doping on $T_c$ depends on various factors which include processing technique and the precursor materials used [23]. Recently, carbon doping in the form of graphene [24,25] has been reported to improve $J_c$ considerably by increasing mainly the grain connectivity without affecting the value of $T_c$. Furthermore, magnetic elements when doped into MgB$_2$ are reported to suppress superconductivity in MgB$_2$ due the pair-breaking effect [26]. Nevertheless, rare-earth (RE) elements which also possess strong magnetic moments, are found not to suppress superconductivity in MgB$_2$ but rather favoring it by being present in the materials as efficient pinning sites [27,28]. It has been reported that with the introduction of RE...
oxides into the MgB2. \( j_c \) is improved in the range of low and medium magnetic fields. The effect of RE elements addition in MgB2 has been studied intensively, but there is still a requirement to explore the possible approaches to improve \( j_c \) in the entire magnetic field range. In this paper, we have done a thorough study of the effect of combined addition of graphene oxide (GO) and a series of RE oxides (REO, RE = La, Sm, Eu, Gd, Tb and Ho) on the superconducting properties of MgB2. We have used a fixed value of 1 wt% REO for addition and the reported [25] optimum value 3 wt% for GO addition to get best \( j_c(H) \) results. We observed that the critical current density, \( j_c \), and upper critical field \( H_{c2}(0) \) improved significantly with GO + REO addition into MgB2 samples with no significant change in critical temperature, \( T_c \). The pinning mechanisms present in these samples are described and discussed in the present paper.

2. Experimental

In the present work, we have synthesized (i) pristine MgB2, (ii) graphene oxide (GO, 3 wt%) added MgB2, (iii) a series of rare-earth oxide, REO (1 wt%, RE = La, Sm, Eu, Gd, Tb and Ho) added MgB2 and (iv) a series of GO (3 wt%) + REO (1 wt%) added MgB2 samples using the standard solid state reaction route. The GO used for addition was synthesized in the laboratory using improved Hummer's method [29] as described in previous work [25], and REO used were purchased from Loba Chemie with 99.9% purity and submicron grain size. For the synthesis of samples, appropriate amounts of Mg (Sigma Aldrich, 99.9% pure) and B (Sigma Aldrich, amorphous, 99%) were mixed with required compositions of GO and REO (RE = La, Sm, Eu, Gd, Tb and Ho) in an agate mortar. In order to compensate for the Mg loss during sample synthesis, for all samples excess Mg (–5 wt%) was added [30,31]. The mixing of chemicals was done thoroughly for about an hour and after this the resulting powder was pressed into rectangular pellets. The pellets were placed in an iron tube and then sintered at 850 °C in a reducing atmosphere of Ar/H2 (9/1) for 3 h followed by slow cooling down to 650 °C and subsequently quenching to room temperature. Henceforth, the pristine, GO-added, REO-added and (REO + GO) added MgB2 samples will be represented as MB, MBG, MBR and MBRG (R = La, Sm, Eu, Gd, Tb and Ho), respectively. The crystallographic structure of the samples was studied using X-ray diffractometry with Cu Kα radiation (1.5406 Å) over an angular (2θ) range between 10° and 80°. The morphological study of the samples was done using a field emission scanning electron microscope (FE-SEM) (FEI-QUANTA 200 FEG). The resistance measurement in different applied magnetic fields (0–8 T) was accomplished using the Physical Property Measurement System (PPMS) (Quantum Design-6000) at the University of Fribourg, Switzerland. The irreversibility fields, \( H_{irr} \), and upper critical field, \( H_{c2}(T) \), were calculated using the criteria 10% and 90% of normal state resistivity, \( \rho_n \), for different applied fields, respectively. The DC magnetic measurements were carried out using a SQUID Interference Device (SQUID) magnetometer (Quantum Design MPMS XL).

3. Results and discussion

From the XRD patterns (Fig. 1) the dominant phase in all the samples is MgB2, along with small quantities of MgO and REB (x = 4 and 6, RE = La, Sm, Eu, Gd, Tb and Ho) impurity phases. The peaks corresponding to MgB2 phase are indexed based on the hexagonal AlB2-type (space group – P6/mmm), and the impurity phases MgO, REB and REB are marked by ‘*’, ‘◦’ and ‘#’, respectively, as shown in Fig. 1. The appearance of MgO during the synthesis of MgB2 samples is difficult to avoid due to the entrapped air before the tube is closed. The presence of oxide additives also adds oxygen during the sample preparation. We have calculated the volume percentage of MgO present in the samples using the formula:

\[
X = \frac{\sum \text{peak intensities of impurity phase}}{\sum \text{peak intensities of MgB2}} \times 100
\]

The obtained values are listed in Table 1. We observe that the amount of MgO in the samples added with GO and REO is larger than the pristine MgB2 sample. The lattice parameters, \( a \) and \( c \) for all samples have been obtained from the refinement of XRD data using the X’pert HighScore software. The obtained values of \( a \) and \( c \) are shown in Table 1. We observe no significant change in the lattice parameters of MBG, MBR and MBRG samples as compared to pure MgB2. This indicates that neither RE (La, Sm, Eu, Gd, Tb and Ho) nor carbon from GO enter into the MgB2 crystal lattice. From the XRD patterns, we see that for the MBG, MBR and MBRG samples the full width at half maxima (FWHM) of the MgB2 peaks is enhanced as compared to the corresponding peaks of pristine MgB2. The FWHM values of the (1 1 0) peak are listed in Table 1, and it can be seen that FWHM for the MBRG samples is larger than for the MBR samples. Since the FWHM values are influenced by the crystal lattice strain and size in the lattice, this result is indicative of an increased lattice strain in the MBRG samples [32]. The grain connectivity in the samples with the introduction of GO is also seen in the FESEM micrographs (see Fig. 2). Fig. 2 compares the FESEM micrographs of (a) MB, (b) MBG (inset shows the FESEM micrograph of GO powder), (c) MBR (R = Ho) and (d) MBRG (R = Ho). It reveals that the grain size decreases with REO-addition in the samples. This result is in conformity with the XRD result discussed above. The EDX analysis reveals the presence of all elements in the samples. We annealed the GO powder separately at 850 °C in Ar/H2 (9/1) atmosphere and from XRD result we found that GO reduces to reduced-graphene oxide (rGO). It has been studied and found that the presence of rGO in the samples creates micro-strain which helps in improving the superconducting properties of the MgB2 sample [14].

Fig. 3(a) and (b) shows the resistivity versus temperature plots for the MB, MBR, MBG and MBRG samples in the temperature range 30 K–300 K. To estimate the effect of introducing REO and GO additives on the sample quality and carrier scattering, we have calculated the value of the residual resistivity ratio, \( RRR = (\rho(300 \text{ K})/\rho(40 \text{ K})) \). The latter provides information about the sample quality in terms of the presence of impurities and lattice defects. Samples with relatively high RRR values are known to be of high quality [33]. In the present case, the RRR values for MB samples have decreased as compared to pristine MgB2. This is due to the presence of MgO, impurity phases which cause increased electron scattering and thus decrease the value of RRR. However, among the MBR samples, the RRR value remains almost unchanged; this is in line with the similar amounts of impurities that are present in these samples. In the case of the MBRG samples, the RRR values are even lower than that of the MBR samples. From the temperature dependence of the resistivity, we have also calculated the superconducting area fraction, \( A_t = \Delta P_{\text{ideal}}/\Delta P \), \( \Delta P = (\rho(300 \text{ K}) – \rho(40 \text{ K})) \) and \( A_{t, \text{ideal}} = 7.3 \mu\Omega \text{cm}^{-1} \) [34], which has been proposed by Rowell [35] as a parameter to describe the strength of the impurity scattering. Here \( \Delta P_{\text{ideal}} \) is the ideal value of \( \Delta P \) for a fully connected system. The calculated values of \( A_t \) are listed in Table 1. According to Rowell’s analysis, fully connected MgB2 shows a well defined temperature dependent scattering which gives a characteristic change in \( \rho \) from 300 K to 40 K. We observe that \( A_t \) has increased with RE addition in the MgB2 samples which is consistent with the earlier studies done with rare-earth oxide doping [27]. With addition of GO + REO in the MgB2 samples, we see a further enhancement in the \( A_t \) values. This indicates that the connectivity is improved by the GO addition. From this result, an improvement in the \( J_c-H \) behavior can be expected.
The transition temperature, \( T_c \), for all the samples was determined from the onset of the superconducting transition in the resistivity versus temperature (\( \rho - T \)) curve in zero applied magnetic field. The values of \( T_c \) are shown in Table 1. It shows that with respect to the MB sample there is no significant drop in \( T_c \) of MBG, MBR and MBRG samples. This result is consistent with the unchanged lattice parameters of all the GO, REO and GO + REO added samples. From the magneto-resistivity measurements in applied fields in the range 0–8 T, we have calculated the values of \( T_c(0) \) for (a) MBR (MgB\(_2\) + 1 wt% REO) and (b) MBRG (MgB\(_2\) + 1 wt% REO + 3 wt% GO) samples.

Table 1: Lattice parameters; \( a \) and \( c \), FWHM, MgO content, critical temperature (\( T_c \)), RRR, \( A_t \) and \( H_{c2}(0) \) for (a) MBR (MgB\(_2\) + 1 wt% REO) and (b) MBRG (MgB\(_2\) + 1 wt% REO + 3 wt% GO) samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>( a ) (Å)</th>
<th>( c ) (Å)</th>
<th>FWHM (1 1 0)</th>
<th>MgO (%)</th>
<th>( T_c ) (K)</th>
<th>RRR</th>
<th>( A_t )</th>
<th>( H_{c2}(0) ) (T)</th>
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<tr>
<td>MB</td>
<td>3.0889</td>
<td>3.5339</td>
<td>0.137</td>
<td>2.24</td>
<td>38.76</td>
<td>3.14</td>
<td>0.19</td>
<td>17.0</td>
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<td>MBG</td>
<td>3.0814</td>
<td>3.5277</td>
<td>0.144</td>
<td>3.33</td>
<td>38.75</td>
<td>2.73</td>
<td>0.23</td>
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<tr>
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<td>3.5271</td>
<td>0.148</td>
<td>4.21</td>
<td>38.80</td>
<td>2.77</td>
<td>0.20</td>
<td>19.6</td>
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<tr>
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<td>3.5259</td>
<td>0.140</td>
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<td>38.82</td>
<td>2.74</td>
<td>0.22</td>
<td>19.5</td>
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<tr>
<td>MBEu</td>
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<td>3.5239</td>
<td>0.144</td>
<td>4.48</td>
<td>38.77</td>
<td>2.86</td>
<td>0.19</td>
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<td>MBGd</td>
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<td>3.5251</td>
<td>0.145</td>
<td>4.39</td>
<td>38.81</td>
<td>2.75</td>
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<td>3.5240</td>
<td>0.138</td>
<td>4.60</td>
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<td>2.68</td>
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<td>0.156</td>
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<td>38.81</td>
<td>2.77</td>
<td>0.23</td>
<td>19.5</td>
</tr>
<tr>
<td>MBLaG</td>
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<td>3.5256</td>
<td>0.152</td>
<td>5.10</td>
<td>39.00</td>
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<td>19.9</td>
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<td>3.5257</td>
<td>0.152</td>
<td>4.80</td>
<td>39.12</td>
<td>2.78</td>
<td>0.29</td>
<td>20.8</td>
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<td>3.5253</td>
<td>0.159</td>
<td>3.78</td>
<td>38.82</td>
<td>2.82</td>
<td>0.29</td>
<td>21.4</td>
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<td>MBGdG</td>
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<td>3.5261</td>
<td>0.156</td>
<td>4.29</td>
<td>38.82</td>
<td>2.71</td>
<td>0.24</td>
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<td>0.151</td>
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<td>38.82</td>
<td>2.84</td>
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<td>22.4</td>
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<td>0.174</td>
<td>4.95</td>
<td>38.61</td>
<td>2.60</td>
<td>0.31</td>
<td>20.5</td>
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The transition temperature, \( T_c \), for all the samples was determined from the onset of the superconducting transition in the resistivity versus temperature (\( \rho - T \)) curve in zero applied magnetic field. The values of \( T_c \) are shown in Table 1. It shows that with respect to the MB sample there is no significant drop in \( T_c \) of MBG, MBR and MBRG samples. This result is consistent with the unchanged lattice parameters of all the GO, REO and GO + REO added samples. From the magneto-resistivity measurements in applied fields in the range 0–8 T, we have calculated the values of the upper critical fields, \( H_{c2} \), using the criteria of \( \rho(T, H_{c2}) \) = 0.9–\( \rho_0(T, H) \), where \( \rho_0 \) is the value of resistivity in the normal state just before the superconductivity transition. The experimental data are plotted in Fig. 4(a) for samples MB, MBG and MBR; in Fig. 4(b) for samples MB, MBG and MBRG. Using the Ginzburg–Landau model [36], \( H_{c2}(T) = H_{c2}(0)[1 - (T/T_c)^2]^{1/2} \) we have calculated the upper critical fields at 0 K. The values of \( H_{c2}(0) \) are listed in Table 1. The value of \( H_{c2}(0) \) for the pure sample is 16.95 T in good agreement with the reported value [36]. The values of \( H_{c2}(0) \) of the MBR samples are enhanced by ~2 T in comparison to MB. This improvement in \( H_{c2}(0) \) is possibly due to decreased grain size of the MB samples which arises due to the presence of REB (\( x = 4 \) and 6) precipitates at the grain boundaries. In case of the MB sample, an improvement of ~1 T was observed for \( H_{c2}(0) \). In the case of MBRG samples, we observed enhancement in \( H_{c2}(0) \) of 4–5 T over the pristine MB sample. This enhancement of \( H_{c2}(0) \) in MBRG samples is due to the lattice strain in the samples that is caused by the different thermal expansion coefficients of the MgB\(_2\) matrix and rGO [14,24].
values are further enhanced (as shown in Fig. 6(b)) with addition of REO + GO in MgB$_2$. From the XRD results we have seen the presence of REB$_x$ impurity phase in the REO + GO added MgB$_2$ samples (MBRG), which may act as strong pinning centres in the samples. Furthermore, in these samples we have seen improved grain connectivity due to addition of GO. Thus these two effects, i.e., enhanced flux pinning and improved grain connectivity, may be responsible for the improved $J_c(H)$ behavior of MBRG samples over the other studied samples. The values of $J_c$ at low (1.5 T) and high field (5 T) corresponding to 10 K and 20 K are listed in Table 2. In case of the GO added sample (MBG), a significant improvement (6.7 times) over the pure MgB$_2$ sample has been observed as was reported earlier [25]. In the MBR samples also, we see improvements as large as ~12 times (for RE = Ho) at 1.5 T (10 K) in comparison to the pure sample. In case of the MBR samples we believe that the REB$_x$ impurities give rise to the improved $J_c$ values. On the other hand, in case of the MBG sample the improvement in $J_c$ value is due to the improved grain connectivity and the micro-strain introduced into the MgB$_2$ matrix, as discussed above, which enhances the flux-pinning in the sample. Among the MBR samples it also appears that the enhancement of $J_c(H)$ scales with the magnitude of the magnetic moment of the added rare-earth elements. From the $J_c(H)$ plots of MBR samples, we see that the oxides of rare-earth elements of larger magnetic moment, like Tb, Ho, Gd, provide more efficient flux pinning centers. Fig. 6 and Table 2, show that in case of the MBRG samples there is a considerable improvement of $J_c(H)$ over that of the MBR and MBG samples (see Fig. 6). The maximum $J_c$ values at 10 K are $9.59 \times 10^5$ A/cm$^2$ at 1.5 T and $4.66 \times 10^4$ A/cm$^2$ at 5 T of the MBRG ($R$ = Tb) sample. This corresponds to a 23.3-fold improvement of $J_c$ at 10 K and 5 T as compared to the pure MgB$_2$ sample. At higher temperature (20 K) we observe the highest $J_c$ value of $5.02 \times 10^5$ A/cm$^2$ at 1.5 T for the MBRG ($R$ = Tb) sample. These $J_c$ values of the MBRG samples are comparable to the highest reported $J_c$ results in the literature for SiC-doped MgB$_2$ samples [5,39].
In order to understand the flux pinning mechanism in both series (MBR and MBRG) of samples, we have calculated the volume-pinning force density, $F_p$ ($F_p = J_c(H)/C^2_H$), from the $J_c(H)$ curves at 10 K. In Fig. 7(a) and (b), we have shown $f_p = (F_p/F_p^{\text{max}})$ versus the normalized applied magnetic field, $h (= H/H^{\text{max}})$ for the MBR and MBRG samples, respectively. Here, $F_p^{\text{max}}$ is the maximum value of $F_p$ at a particular temperature and $H^{\text{max}}$ is the value of field corresponding to $F_p^{\text{max}}$. The values of $F_p^{\text{max}}$ and $H^{\text{max}}$ have been determined from the average shape of the $f_p$ versus $h$ plots, obtained by ignoring some flux jump points. The signatures of flux jumps at low fields are also observed here. In high temperature superconductors the normalized volume pinning force, $f_p$, often scales with $H/H^{\text{irr}}$. However, it is difficult to calculate the accurate value of $H^{\text{irr}}$ from the dc magnetization measurements. In order to avoid this problem, the data are often scaled with $H/H^{\text{max}}$ instead of $H/H^{\text{irr}}$ [40], as is done in the present case. The scaling for $f(h)$ is often done with the help of following equations [40–42] for the different flux pinning mechanisms:

$$f_p = 3h^3 \left( 1 - \frac{2h}{3} \right), \quad \delta k\text{-pinning}$$  \tag{1}

$$f_p = \frac{9}{4} h \left( 1 - \frac{h}{3} \right)^2, \quad \delta T_c\text{-pinning}$$  \tag{2}

$$f_p = \frac{25}{16} \sqrt{h} \left( 1 - \frac{h}{5} \right)^2, \quad \text{surface-pinning}$$  \tag{3}

![Fig. 4.](http://doc.rero.ch) (a) and (b) presents the $H_{c2}(T)$ versus $T/T_{c0}$ ($T_{c0}$ is the temperature where the value of $\rho$ is 0.9$\rho_n$, $\rho_n$ is the normal state resistivity) plots for the MB, MBG and MBR samples; and MB, MBG and MBRG samples, respectively. (c) and (d) presents the $H_{c2}(T)$ versus $T/T_{c0}$ ($T_{c0}$ is the temperature where the value of $\rho$ is 0.1$\rho_n$, $\rho_n$ is the normal state resistivity) plots for the MB, MBG and MBR samples; and MB, MBG and MBRG samples, respectively.

![Fig. 5.](http://doc.rero.ch) Magnetic hysteresis loop for the RE-added (0–7 T) and RE and GO added (–7 to 0 T) samples at 10 K (a) and 20 K (b).
In both series of samples, MBR and MBRG, the scaling suggests that the dominant pinning mechanism in the pure and MBR samples is related to surface pinning or to grain boundaries, while in the GO added sample we observe that $\delta T_c$ or point-pinning is the dominant one. However, in the MBRG samples we see that the $f_p$ curve shift towards the point-pinning. This suggests that the GO-added MgB$_2$ sample has enhanced point pinning mechanism.

In comparison to the previous studies done with co-doping of rare-earth oxide and carbon sources, e.g. graphite [16] and SiC [6], it is clear from the present study that GO is a better source of carbon to be added along with rare-earth oxides in MgB$_2$. In the previous studies, the $J_c$ has improved in the region of high field, while in the present case there is improvement in $J_c$ in the entire magnetic field range (0–7 T) without affecting the critical temperature, $T_c$. At the synthesis temperature, although GO does not dissociate to give C for lattice substitution, but it helps in improving the grain connectivity, i.e. provide easy path for the flow of superconducting current. We have also observed from the $J_c(H)$ behaviors of MBR and MBRG samples that the additions of oxides of rare earth elements with higher magnetic moment are more effective in improving flux pinning and thus $J_c(H)$.

### 4. Conclusion

In the present work, we have systematically investigated the effect of addition of (i) graphene oxide (GO), (ii) a series of...
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Acknowledgments

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References