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The effect of doping level and sintering temperature on $J_c(H)$ performance in nano-SiC doped and pure MgB_2 wires

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Nanoscale SiC doped Fe/MgB_2 wire samples were prepared by an *in situ* reaction technique using SiC doping levels of 0, 5, 10, and 15 wt %. Samples were heat treated at different temperatures using different temperature profiles. The effects of doping level and sintering temperature on superconducting properties of wire samples were investigated. The important finding of this study was that the enhancement in $J_c(H)$ by nano-SiC doping can be achieved at different field regions by appropriate compromising of the doping level and sintering temperature. *© 2006 American Institute of Physics*. [DOI: 10.1063/1.2173639]

INTRODUCTION

Since the discovery of the MgB_2, a number of additives have been used to improve the critical current density (J_c), irreversibility field (H_{irr}), and upper critical field (H_{c2}).[1–9] Among these, both SiC and C have become well established additives which can result in the enhancement of J_c, H_{irr}, and H_{c2}. The authors' group found that doping of MgB_2 with nanoparticle SiC can significantly enhance J_c in high fields, with only slight reductions in T_c up to a doping level as high as 30% of B.[10] Compared to the undoped sample, the J_c for the 10 wt % SiC doped sample increased by more than an order of magnitude at all temperatures and in the high field region. The exceptional properties of SiC as a dopant have been verified by a number of groups over the past few years.[7–18] However SiC doping was found to have some negative effect on J_c in the low field region. The J_c for SiC doped MgB_2 was lower than that for undoped MgB_2 below 4 T at 5 K and below 2.5 T at 20 K.[2,13,17] There are many applications in the low field region such as in open magnetic resonance imaging (MRI) transformers and electric cables which normally operate at around 1–3 T. Thus it is important that the enhancement of J_c by SiC doping can be extended to include all the field regions. In this Letter we report the results of the improved J_c of SiC doped MgB_2 wires in all fields and temperature ranges.

EXPERIMENTAL DETAILS

Fe clad MgB_2 wires were prepared *in situ* using the standard powder-in-tube technique.[2,12] Powders of magnesium (99%) and amorphous boron (99%) were well mixed for the fabrication of a pure MgB_2 wire. SiC doped MgB_2 wires were prepared from powders with atomic ratios of Mg:2B plus 5, 10, or 15 wt % of SiC additions (SiC powder size of 10–100 nm). The mixed powder was filled into Fe tube and than drawn or groove rolled to 1.4 mm in diameter. Short samples of these composite wires were sintered in a tube furnace at 600, 650, 825, and 1000 °C for 30 min or 3 h and finally furnace cooled to room temperature. A high purity argon gas flow was maintained throughout the sintering process. The description of studied samples is presented in Table I.

Transport J_c of wire samples were measured at the Ohio State University using a dc method for the high magnetic field range from 5 to 15 T. Magnetic J_c was derived from the height of the magnetization loop using Bean's model for a superconducting core after the Fe sheath was mechanically removed. The magnetization of samples was measured over a temperature range of 5–20 K using a Physical Property Measurement System (PPMS). Since there is a large sample size effect on the magnetic J_c for MgB_2 fabricated with an *in situ* reaction process[19] all the samples for measurement were made to the same size of 0.7 mm in diameter and 2.7 mm in length for comparison. The transmission electron microscopy (TEM) images were obtained using a Joel 2011 transmission electron microscope. Powders were scraped from the sintered composite, dispersed in ethanol, and then allowed to settle onto holey-carbon coated support grids.

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TABLE I. Some parameters of undoped and SiC doped MgB_2 wires.

Sample number	SiC doping level (wt %)	Sintering profile (°C × min)	T_c (K)	J_c (20 K, 0 T) (K A/cm^2)	H_{irr} at 20 K (T)
1	0	600 × 180	36.3	315	4.3
2	0	650 × 30	⋯	240	4.2
3	0	825 × 30	37.7	400	3.9
4	0	1000 × 30	38.7	90	3.6
5	5	650 × 30	⋯	310	5.4[a]
6	5	825 × 30	36.4	400	5.2
7	5	1000 × 30	36.5	220	4.8[a]
8	10	600 × 180	34.5	208	4.8
9	10	650 × 30	⋯	170	4.8
10	10	825 × 30	35.5	280	4.8
11	10	1000 × 30	36.5	320	5.4
12	15	650 × 30	⋯	70	4.9
13	15	825 × 30	35.4	170	4.5
14	15	1000 × 30	36	270	5.2

[a]Estimated values.

RESULTS AND DISCUSSION

Critical temperatures T_c of the samples investigated are presented in Table I. As can be seen, for each group of samples with the same SiC doping level, T_c increased with increasing sintering temperature. For instance, the T_c of samples sintered at 600 °C for 3 h was 36.3 K for the pure MgB_2 and 34.5 K for the 10 wt % SiC and reached 38.7 and 36.5 K, respectively, at a sintering temperature of 1000 °C.

Figure 1 shows J_c vs $H[J_c(H)]$ curves at 5 and 20 K for the undoped and SiC doped MgB_2 wires sintered at 825 °C for 30 min. For the undoped MgB_2 wires J_c values reached 400 000 A/cm^2 at 20 K and self-field and 134 000 A/cm^2 at 5 K and 3 T, which are consistent with the results published by a number of groups.[2,13,14] J_c for the 5 wt % SiC doped MgB_2 wire reached the same value in self-field and 20 K and was slightly higher at 3 T and 5 K, compared to the undoped MgB_2 wire. At low fields the J_c values for the 10 and 15 wt % SiC doped wires are lower than those for the undoped

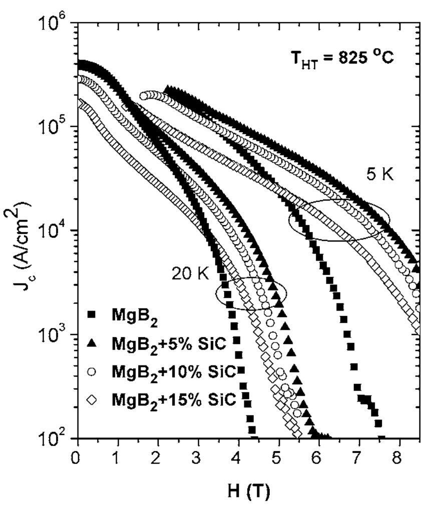

FIG. 1. The $J_c(H)$ curves at 5 and 20 K for the undoped and SiC doped MgB_2 wires sintered at 825 °C for 30 min.

samples at both 5 and 20 K. As it was shown in previous studies, SiC doping deteriorated J_c values at low field regions.[2,13,17] According to x-ray diffraction (XRD) results (not shown; see, e.g., Ref. 12), the main impurity in studied SiC doped samples was Mg_2Si, which increased with increasing doping level, but decreased with increasing sintering temperature. In addition to this, samples could contain some large SiC particles (up to 100 nm) that did not react with MgB_2 and do not act as effective pinning centers because of their large size.[7] The starting material consisted of a Mg:B molar ratio of 1:2, and therefore the amount of the superconducting MgB_2 phase decreases in samples sintered at low temperatures and in samples with higher SiC doping levels as a result of the Mg_2Si impurity formation. Thus for samples reacted at low temperatures there are a considerable amount of impurity phases that tend to reside inside and in between grains,[7] and the latter may inhibit the intergranular current flow. Increasing the SiC level results in a greater amount of these phases and therefore further degrades low field J_c values.

It is important to note that in the measurement range up to 8.5 T the J_c values for 5 wt % SiC doped wires are higher than the 0, 10 and 15 wt % SiC doped ones. The J_c behavior for all these samples sintered at 650 °C showed the same trend as that for samples sintered at 825 °C, but the J_c values in self-field are lower than those obtained for the samples sintered at 825 °C (see Table I). It is evident that for low field applications a 5 wt % SiC doping gives the best performance in $J_c(H)$, without any degradation even in self-field.

Figure 2(a) shows transport J_c measured at 4.2 K in the field range of 5–15 T for the doped and undoped wires sintered at 650 °C. The J_c value for the doped wires at all three doping levels are significantly higher than that for the undoped ones. It is also noted that at 4.2 K the drop rate of J_c with increasing field follows the sequence: undoped $>$5 wt % $>$15 wt % $>$10 wt % doped sample. Furthermore, there is a crossover of the best $J_c(H)$ from the 5 wt % SiC doped wire in the low field range (0–8 T) to the 10 wt % SiC doped wire in the high field range (8–15 T). Figure 2(b) shows the effect of sintering temperature on the transport $J_c(H)$ in the field range from 8 to 15 T for the 10 wt % SiC doped wires. It is evident that better $J_c(H)$ performance in this field region come from samples sintered at lower temperature.

Figure 3 shows TEM images illustrating the effect of sintering temperature on the nanostructure of SiC doped MgB_2 wires processed at (a) 600 °C for 3 h and (b) 825 °C for 30 min. The grain sizes in the SiC doped MgB_2 sintered at 600 °C were very small (less than 50 nm) with limited crystallinity, as evidenced by the typical spotty ring shape of the selected area electron diffraction pattern [inset in Fig. 3(a)]. In comparison, the sample sintered at 825 °C [Fig. 3(b)] contained many larger grains ($>$100 nm) with better defined crystallinity, [see inset of Fig. 3(b)]. The impurity particle size showed the same trend, with the lower temperature sintering resulting in a smaller size of inclusions and the higher temperature sintering promoting the growth of crystallites of both MgB_2 and impurities. The TEM observations are consistent with the $J_c(H)$ performance. The small MgB_2

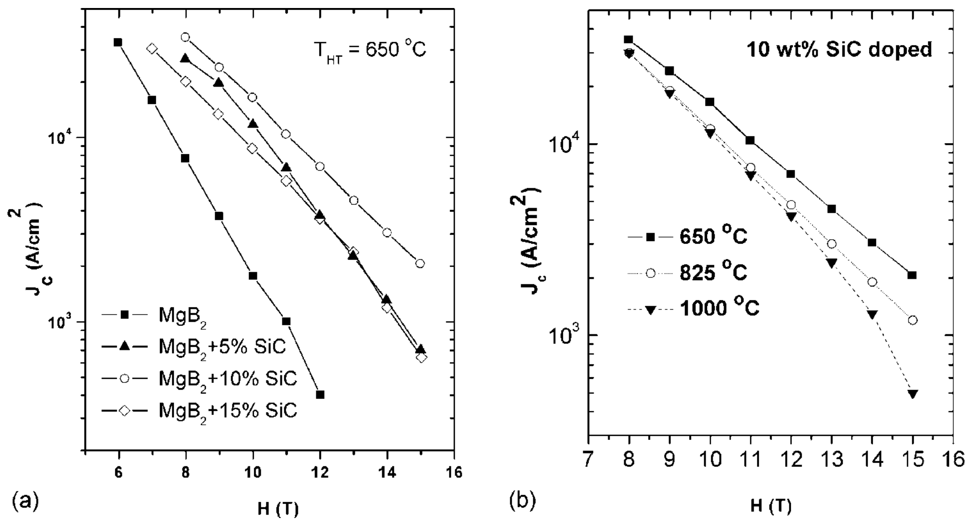

FIG. 2. (a) The transport $J_c(H)$ measured at 4.2 K for the doped and undoped wires sintered at 650 °C. (b) The effect of sintering temperature on the transport $J_c(H)$ for the 10 wt % SiC doped wires.

crystallites and impurity inclusions are beneficial to flux pinning and hence enhance the $J_c(H)$ behavior in the high field region. On the other hand, higher sintering temperatures result in a better crystallinity and may result in the formation of grain boundaries that are more transparent to the current flow, thereby giving a better low field J_c performance.

In Table I the irreversibility field H_{irr} at 20 K, defined by the J_c criterion of 100 A/cm^2 is presented. For all the SiC doped MgB_2 samples H_{irr} values ranged from 4.8 to 5.4 T and are higher than those for undoped MgB_2 wires (from 3.6 to 4.2 T). Although the SiC doped samples sintered at 1000 °C have a lower J_c at 20 K their H_{irr} values are higher than those of samples processed at lower temperatures. This suggests that at 1000 °C, higher amount of C substitutes for B in the lattice, resulting in higher H_{c2} and hence higher H_{irr}.

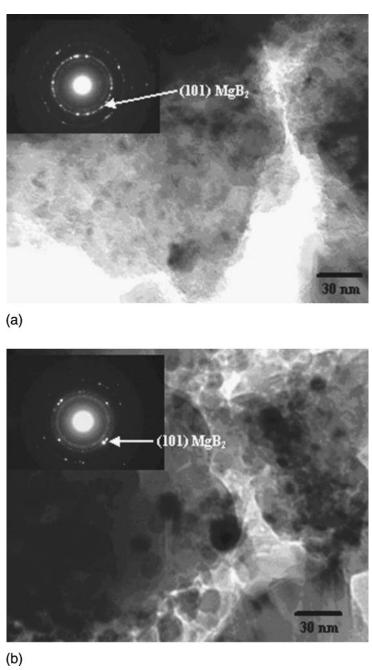

FIG. 3. TEM images and selected area diffraction patterns obtained from the (a) 10 wt % SiC doped MgB_2 samples sintered at 600 °C and (b) 5 wt % SiC doped MgB_2 samples sintered at 825 °C.

SUMMARY

In summary, the enhancement of J_c in MgB_2 samples can be achieved at different field regions by varying the SiC doping level, sintering temperature, and time. To achieve a high critical current in low field regions, a low doping level of the 5 wt % SiC and a sintering temperature of 825 °C should be used. For field regions of 8–15 T a doping level of the 10 wt % SiC and a sintering temperature of 650 °C resulted in the best J_c value. In addition, higher SiC doping levels (10–15 wt %) and a higher sintering temperature (1000 °C) results in increasing of H_{c2} and hence H_{irr}.

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