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Transport Critical Current Density in Fe-Sheathed Nano-SiC Doped MgB₂ Wires

Shi X. Dou, Joseph Horvat, Saeid Soltanian, Xiao L. Wang, Meng J. Qin, Shi H. Zhou, Hua K. Liu, and Paul G. Munroe

Abstract—The nano-SiC doped MgB₂/Fe wires were fabricated using a powder-in-tube method and an in-situ reaction process. The depression of T_c with increasing SiC doping level remained rather small due to the counterbalanced effect of Si and C co-doping. The high level SiC co-doping allowed creation of the intra-grain defects and nano-inclusions, which act as effective pinning centers, resulting in a substantial enhancement in the $J_c(H)$ performance. The transport J_c for all the wires is comparable to the magnetic J_c at higher fields despite the low density of the samples and percolative nature of current. The transport I_c for the 10wt% SiC doped MgB₂/Fe reached 660A at 5K and 4.5T ($J_c = 133000 \text{A/cm}^2$) and 540A at 20K and 2T ($J_c = 108\,000\text{A/cm}^2$). The transport J_c for the 10wt% SiC doped MgB2 wire is more than an order of magnitude higher than for the state-the-art Fe-sheathed MgB2 wire reported to date at 5K and 10T and 20K and 5T respectively. There is a plenty of room for further improvement in J_c as the density of the current samples is only 50%.

Index Terms—Critical current, magnesium diboride, nano-particle doping, silicon carbide.

I. INTRODUCTION

T HAS BEEN established that Fe sheath is suitable for fabrication of MgB₂ wires using a powder-in-tube method [1], [2]. Extensive research efforts have been made to improve the J_c of Fe-sheathed MgB₂ wires [1]–[5]. However, the J_c performance in high fields and temperatures above 20K remains unsatisfactory for many applications due to the poor pinning ability of this material. Attempts to enhance flux pinning have resulted in an encouraging improvement in irreversibility fields (H_{irr}) and $J_c(H)$ by oxygen alloying in MgB₂ thin films [6] and by proton irradiation of MgB₂ powder [7]. However, these techniques are not readily available for introducing effective pinning centers into MgB₂ wires. Chemical doping has been commonly used to introduce flux pinning centers into a superconductor for enhancing critical current density. Unfortunately, chemical doping of MgB₂ reported so far is limited to addition, rather than sub-

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stitution of the elements. The additives alone appear to be ineffective for improving pinning at high temperatures [8]–[10].

Recently, we found that chemical doping with nano-particle SiC into MgB₂ can significantly enhance J_c in high fields with only slight reduction in T_c up to the doping level of 40% of B [11]. This finding clearly demonstrated that co-substitution of SiC for B in MgB2 induced intra-grain defects and high density of nano-inclusions as effective pinning centers, responsible for the improved performance of $J_c(H)$ in wide range of temperatures [12]. However, all the results reported thus far have been limited to magnetic measurements. As the materials are far from optimum and the sample density was only 50% of theoretical value the current in such a porous material is highly percolative. The major concern is whether the material can carry large transport J_c . In this work, we focus our study on the transport current and its field dependence for the nanometer-size SiC doped MgB₂ wires. Our results reveal that the nanometer size SiC doped MgB₂/Fe wires can carry higher transport I_c and J_c in the magnetic fields ever reported for any form of MgB₂. SiC doped MgB₂ is very promising for many applications, as this chemical doping is a readily achievable and economically viable process to introduce effective flux pinning.

II. EXPERIMENTAL DETAILS

A standard powder-in-tube method was used for the Fe clad MgB₂ tape [2]. Powders of magnesium (99%) and amorphous boron (99%) were well mixed with 0 and 10wt% of SiC nanoparticle powder (size of 10 nm to 20 nm) and thoroughly ground. The pure Fe tube had an outside diameter (OD) of 10 mm, a wall thickness of 1 mm, and was 10 cm long with one end of the tube sealed. The mixed powder was filled in to the tube and the remaining end was crimped by hand. The composite was drawn to a 1.4 mm diameter wire 2 meters long. Several short samples 2 cm in length were cut from the wire. These pieces were then sintered in a tube furnace over a temperature range from 800–850 °C for 10 min to 30 min. This was followed by furnace cooling to room temperature. A high purity argon gas flow was maintained throughout the sintering process.

Transport current was measured using pulse DC method. A pulse of the current was obtained by discharging a capacitor through the sample, coil of thick copper wire and noninductive resistor connected in series. The current was measured via the voltage drop on the noninductive resistor of 0.01 Ohm. The current reached its maximum value (700A) within 1ms. The voltage developed on the sample was measured simultaneously with the current, using a 2-channel digital oscilloscope. Because both

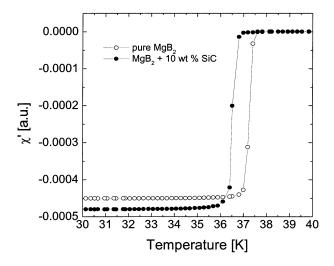


Fig. 1. Critical transition temperature (T_c) measured using magnetic susceptibility versus temperature for pure MgB $_2$ and 10wt% SiC doped MgB $_2$ /Fe wires.

channels of the oscilloscope had the same ground, the signal from the voltage taps was first fed to a transformer preamplifier (SR554). This decoupled the voltage taps from the resistor used for measuring the current, thereby avoiding creation of the ground loops and parasitic voltages in the system, as well as of an additional current path in parallel to the sample. The transformer amplified the voltage 100 times, improving the sensitivity of the experiment. Magnetic field was produced by a 12T superconducting magnet. Sample mounting allowed for orienting the field either perpendicular to the wire, or parallel to it. In the later case, the field was also parallel to the current passing through the sample. The sample was placed into a continuous flow helium cryostat, allowing the control of temperature better than 0.1K.

The magnetization of samples was measured over a temperature range of 5 to 30 K using a Physical Property Measurement System (PPMS, Quantum Design) with a sweep rate of magnetic field of 50 Oe/s and amplitude up to 8.5T. Samples are in the form of bars cut from the pellets which were processed under the same conditions as the wires. All the samples had the same size of $0.56 \times 2.17 \times 3.73$ mm³. A magnetic J_c was derived from the height of the magnetization loop using Bean's model.

III. RESULTS AND DISCUSSION

Fig. 1 shows the transition temperature (T_c) for the doped and undoped samples determined by ac susceptibility measurements. The T_c obtained as the onset of magnetic screening for the undoped sample was 37.6K. For the 10wt% SiC doped sample, the T_c decreased only for 0.7K. In contrast, the T_c was depressed by almost 7K for 10% C substitution for B in MgB₂ [13]. This suggests that the higher tolerance of T_c of MgB₂ to SiC doping is attributable to the counterbalance effect of the co-doping by C and Si. This is because the average size of C (0.077 nm) and Si (0.11 nm) atoms is close to that of B (0.097 nm). As reported previously, the 10wt% SiC doped sample consists of major phase with MgB₂ structure and minority phases: Mg₂Si and MgO which occupied about 10% to 15% volume fraction.

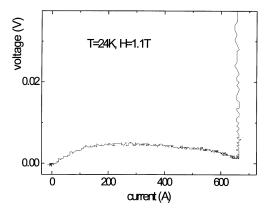


Fig. 2. I-V curve for nondoped MgB₂/Fe wire. $I_c=665\,\mathrm{A}$ at 24K and 1.1T.

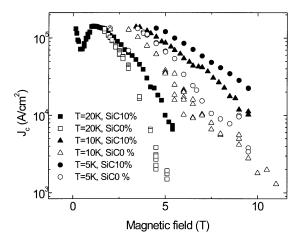


Fig. 3. Transport J_c-H dependence at 5 K, 10 K and 20 K for the pure MgB $_2$ /Fe and 10wt% SiC doped MgB $_2$ /Fe wires.

Fig. 2 shows a typical $V\!-\!I$ characteristic for the MgB $_2$ /Fe wire. It is noted that the self-field of the current pulse induced a voltage in the voltage taps, which gave a background voltage. It was easy to distinguish the voltage created by the superconductor on this background, because the voltage developed very abruptly when the current reached the value of I_c . It is interesting to note that the total current that the wire can carry reached 665A at 24K and 1.1T. The transport I_c for the 10wt% SiC doped MgB $_2$ /Fe reached 660A at 5K and 4.5T and 540A at 20K and 2T. Due to the limitation of our power source all the I_c measurements were limited to the maximum 700A.

Fig. 3 shows the $J_c(H)$ curves for the undoped and the 10wt% SiC-doped MgB₂ samples at 5K, 10K, and 20K. It is noted that all the $J_c(H)$ for 10wt% SiC doped MgB₂/Fe wire are significantly higher than the undoped sample at higher fields. The transport J_c for the 10wt% doped MgB₂/Fe reached 133 000A/cm² at 5K and 4.5T and 108 000A/cm² at 20K and 2T. The transport J_c for the 10wt% SiC doped MgB₂ wire increased by a factor of 6 at 5K and 9T, and 20K and 5T respectively, compared to the undoped wire. These results indicate that SiC doping strongly enhanced the flux pinning of MgB₂ in magnetic fields. The enhancement of pinning by SiC doping is also evident from the pinning force density versus magnetic filed as shown in Fig. 4. The volume pinning force density of 5.5 GN/m³ at 20K is comparable to that of NbTi at 4.2K. Although the maximum pinning force density only has a

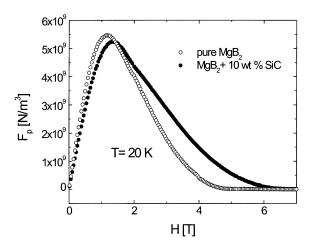


Fig. 4. Pinning force density versus magnetic filed for the undoped and 10wt% SiC doped MgB₂/Fe wires.

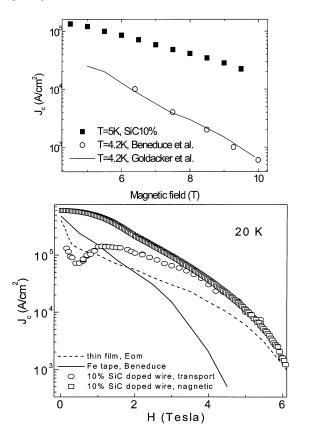


Fig. 5. Comparison of the transport J_c with magnetic J_c for the 10wt% SiC doped MgB₂/Fe wire, including the best transport J_c of a strongly pinned thin film by Eom *et al.*, [8] and Fe-sheathed MgB₂ tape by Goldacher *et al.*, [3] and Beneduce *et al.*, [14].

little shift to higher field, the pinning force density for the SiC doped MgB₂/Fe wire is clearly greater than for the undoped wire at field above 1.5T.

Fig. 5 shows the comparison of the transport J_c with magnetic J_c . Although there is quite different voltage standard for measuring the transport J_c and magnetic J_c , due to steep V-I characteristics, J_c is expected to be similar for both methods. It is noted that the transport J_c is lower than magnetic J_c in low fields as the transport J_c showed some type of "peak effect." The peak effect originates from the interaction between the Fe

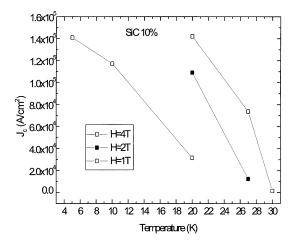


Fig. 6. $J_c(H)$ versus temperature for the 10wt% SiC doped MgB $_2$ wire at 1T, 2T and 4T.

sheath and superconductor. However, the transport J_c for the wires is comparable to or higher than the magnetic J_c at higher fields despite the low density of the samples and percolative nature of current. Fig. 5 also shows a comparison of the transport $J_c(H)$ for 10wt% SiC doped MgB₂/Fe wire with the thin film [6] and the Fe-sheathed MgB₂ tape at 5K and 20K [3], [14] reported previously. We see the J_c for the 10wt% SiC doped wire is more than an order of magnitude higher than the best transport J_c reported in Fe–MgB₂ tape at 5K and 8T and 20K and 4T respectively. It is even comparable to the strongly pinned thin film (magnetic J_c for the thin film) at 20K. This is the best transport $J_c - H$ performance ever reported for MgB₂ in any form.

Fig. 6 shows the $J_c(H)$ versus temperature for 10wt% SiC doped wire at 1T, 2T and 4T. With SiC doping, we can achieve J_c values from 50 000A/cm² to 150 000A/cm² in temperature range between 15K and 25K and field range of 2T to 5T. These results demonstrate that the nano-SiC doping into MgB₂/Fe wire makes a number of applications practical, including MRI, moderate magnets, magnetic windings for energy storage, magnetic separators, transformers, levitation, motors and generators. The SiC substituted MgB₂/Fe wire is attractive from the economic point of view. The main cost for making MgB₂ conductors will be the high purity B. Furthermore, the SiC doping has already shown a significant benefit by enhancing flux pinning. It is evident that the future MgB₂ conductors will be made using a formula of MgB_xSi_yC_z instead of pure MgB₂.

Fig. 7 is TEM image of Sic-doped MgB_2 showing a very high density of dislocations and massive nano-meter size inclusions inside the grains 7(a)). These inclusions are most likely the Mg_2Si as this is the major impurity phase picked up by the XRD analysis. The EDS analyses showed that the grains consisted of Mg, B, C, Si and O. The presence of oxygen within the grain is consistent with the results obtained from a thin film with strong pinning where the ratio of Mg:B:O reached 1.0:0.9:07 [6]. All the intra-grain defects and the inclusions within the grains act as effective pinning centers, responsible for the enhanced flux pinning.

The present study for optimization is only limited by the processing conditions, the density of the Fe-sheathed MgB₂ wires is still very low, only about 1.2 to 1.3 g/cm³. Thus, a higher J_c

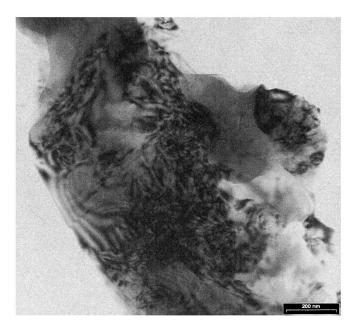


Fig. 7. TEM image for the undoped and 10wt% SiC doped MgB₂/Fe wires.

and better flux pinning enhancement can be achieved if the density of the samples is further improved.

IV. CONCLUSION

In summary, we have further demonstrated that very high transport critical current and current density of Fe-sheathed MgB_2 wires can be achieved by a readily achievable and economically viable chemical doping with nano-SiC. J_c of over $100\,000\,\text{A/cm}^2$ at 5K and 5T and 20K and 2T were obtained, comparable to NbTi and HTS respectively. The high performance SiC doped MgB_2 wires will have a great potential to replace the current market leader, Nb–Ti and HTS for many practical applications at 5K to 25K up to 5T.

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