Dependence of magnetoelectric properties on sintering temperature for nano-SiC-doped MgB2/Fe wires made by combined in situ/ex situ process

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Dependence of magnetoelastic properties on sintering temperature for nano-SiC-doped MgB$_2$/Fe wires made by combined in situ/ex situ process

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Very fine nano-SiC particles (<15 nm) were doped into a MgB$_2$ superconductor. The influence of self-field supercurrent on the high-field performance of the nano-SiC-doped MgB$_2$/Fe wires is discussed based on comparison of the critical current densities of the in situ processed nano-SiC-doped MgB$_2$ wires and those of the nano-SiC-doped MgB$_2$/Fe wires processed by the combination of in situ/ex situ methods. © 2012 American Institute of Physics. [doi:10.1063/1.3677660]

The critical current density ($J_c$) of MgB$_2$ superconductors has been advanced through many different kinds of dopants or additives, especially different carbon sources. $^2$–$^8$ $J_c$ values as high as $1 \times 10^4$ A cm$^{-2}$ at $\sim$12.5 T, 4.2 K have been reported for 10 wt % SiC-doped wires. However, the self-field critical current density values, $J_c(0)$, of these samples are much lower than those of samples made by hybrid physical–chemical vapor deposition (HPCVD), $^9,10$ 3.5 $\times$ 10$^7$ A/cm$^2$ at 4.2 K and 1.6 $\times$ 10$^8$ A/cm$^2$ at 2 K. The depairing current density, $J_d$, is $\sim$8.7 $\times$ 10$^8$ A/cm$^2$ for pure MgB$_2$. $^9$ The connectivity is poor in the in situ MgB$_2$ samples because the in situ technique involves a solid–liquid reaction process with considerable shrinkage as a result of the high theoretical density of MgB$_2$ compared to the initial mixture of Mg and B. $^{11,13}$ It is critically important to discover how to make a high-density sample to obtain high $J_c(0)$ and then introduce strong flux-pinning force to keep $J_c$ dropping as slowly as possible with increasing magnetic field. High-pressure sintering, especially hot isostatic pressing (HIP) treatment is used in making high-density bulks and wires. $^{14}$ Cold high-pressure densification (CHPD) is also effective in improving the density to as high as 73% in wires. $^{12,13}$ However, the equipment for the high-pressure processes is quite complicated, and the sample densities are still lower than needed for practical application. The ex situ technique is promising for making high $J_c$ MgB$_2$ superconductors. $^{13,18}$

The problem with the ex situ technique is the low-quality connections between the MgB$_2$ grains. In this work, a mixed in situ/ex situ technique was employed to develop nano-SiC-doped MgB$_2$ wires with high connectivity and strong flux-pinning force to increase both the low- and high-field $J_c$ properties. The SiC particle size is another critical issue for introducing strong flux-pinning forces into MgB$_2$. The size of SiC used in this work is smaller than the sizes used in previous research, and the $J_c$ dependence on sintering temperature also shows a very different trend. $^{2,15,16,17}$

The powder-in-tube (PIT) process was employed to make practical MgB$_2$ wires from a ball-milled mixture of Mg (99%), B (99%, amorphous), and SiC (<15 nm). A part of the mixture was made into MgB$_2$/Fe wires with a diameter of 1.4 mm, which were sintered in high-purity Ar at temperatures of 750, 850, 950, and 1050 $^\circ$C for 30 min as standard in situ samples, which were marked as 750in, 850in, 950in, and 1050in, respectively. The other part of the mixture was sintered at 650 $^\circ$C for 30 min in pure argon flow and then ball-milled to yield precursor MgB$_2$ powder. Then in situ processed MgB$_2$/Fe wires were made by the PIT method using this precursor powder and a mixed powder in a 1:3 ratio. All the green wires were annealed in high-purity Ar at temperatures of 750, 850, 950, and 1050 $^\circ$C for 30 min, yielding samples that were marked as 750inex, 850inex, 950inex, and 1050inex, respectively.

According to the indexed XRD patterns as shown in Fig. 1, all samples show quite high purity of MgB$_2$ with small amounts of MgO and un-reacted Mg and SiC. The un-reacted Mg can be detected because of the high content of Mg$_2$Si. The critical transition temperatures ($T_c$) of the two batches of samples are compared in Fig. 3. It is found that the $T_c$ values of the in situ-sintered samples are always slightly lower than those for the samples from the other batch, except for 1050in, and the $T_c$ dependence on sintering temperature of the two batches of samples is exactly the same, which means that the $T_c$ depends greatly on the sintering temperature, but not on the different techniques. However, the
transition widths from the normal state to the superconducting state are quite different for the two batches of samples, as shown in the inset of Fig. 3. The transition widths of the in situ sintered samples are quite broad. The transition width is about 4 K for 750 in and becomes 3 K in the 850 in and 950 in samples sintered at higher temperature because of the high crystallinity. It should be noted that the transition of 1050 in shows a two-step behavior, which may be attributed to the inhomogeneous carbon substitution effect or the inhomogeneous SiC distribution in the raw materials. The transition widths of all the samples made by the in situ/ex situ combined technique are 2.5 K. This means that the crystallinity is increased through the in situ/ex situ combined technique because the precursor MgB2 powder is a source of high-quality nucleating centers for newly formed MgB2 during the solid–liquid reaction between magnesium and boron.

The $J_c$ dependence on the applied field is shown in Fig. 4 for typical samples, which were measured at 5 K and 20 K, respectively. It should be noted that the $J_c$ dependence on sintering temperature in this work is totally different from previously reported results, because the solid–liquid reaction dynamics is different because of the small SiC particle size, less than 15 nm, which is much smaller than particle sizes used before. It is concluded that the in situ/ex situ combined technique only requires a lower sintering temperature to achieve high-quality MgB2 wires, which is very important for industrial application in terms of the energy saving and equipment simplification. To avoid the influence of the flux jumping effect, the low-field performances were compared at 20 K in detail, as shown in the inset of Fig. 4. 750 in and 750 inex show quite low $J_c$ values in lower magnetic field. 1050 in and 850 inex show competitive self-field $J_c$. The ball-milling process used to produce the ex situ MgB2 powder destroyed the porous structure and enhanced the density of MgB2 fabricated by the in situ/ex situ combined process. It is because of the small particle size of SiC used in this work, which induces different reaction dynamics during the in situ or in situ/ex situ processing.2,12,16,17 that the present $J_c$ dependence on sintering temperature is quite different from what has been previously reported. It is proposed that the liquid Mg reacts with SiC first to form Mg2Si and release free C at low sintering temperature, and then the Mg2Si reacts with B to form MgB2 and release free Si at high sintering temperature. Both C and Si are very small and cannot be detected by XRD. The coherence length of MgB2 is anisotropic. $\xi_{ab}(0) = 3.7–12$ nm, and $\xi_c(0) = 1.6–3.6$ nm,19 which is shorter than the particle size of Mg2Si. The Mg2Si particles cannot be effective flux-pinning centers, but are useless impurities in the MgB2 matrix, which decrease the density of current carriers. However, the free C and Si can be very strong flux-pinning centers because of their small sizes, which is responsible for the high $J_c$ performance in high magnetic fields. According to the collective pinning model,21 the density of current carriers is responsible for the $J_c$ performance in the single-vortex pinning regime because of its weak field dependence, while the flux-pinning force is responsible for the $J_c$ performance in the small-bundle regime because of the increased high $H_{c2}$ and $H_{irr}$. The approximate $H_{sb}$ values are also indicated on the $J_c$ curves estimated at 20 K, as shown in the inset of Fig. 4, where $H_{sb}$ is the crossover field from single-vortex to small-bundle

![FIG. 2. SEM photos of (a) 850 in, and (b) 850 inex.](image-url)
pinning. However, $H_{c2}$ has not been detected at 5 K because of the relatively high supercurrents. 22

The strength of the pinning force can be reflected by the dependence of $H_{c2}$ and $H_{irr}$ on normalized temperature, as shown in Fig. 5. Carbon substitution is one of the most effective methods to improve the $H_{c2}$ and $H_{irr}$ because of the increased scattering by C doping, and the increased scattering can also contribute to decreased $T_c$ and merging of the two gaps. 20 It should be noted that the $H_{c2}$ and $H_{irr}$ for 750in, 850in, and 750inex are highest at low temperature, which means a strong flux-pinning force. Their poor $J_c$ is attributed to the lower density of current carriers. Both 1050in and 1050inex show the lowest $H_{c2}$ and the lowest $H_{irr}$ among all the samples. 850in, 950in, 850inex, and 950inex show competitive $H_{c2}$ and $H_{irr}$ performances, which are responsible for their high $J_c$ values under high magnetic field.

In conclusion, high sintering temperature can improve the critical current density of small-particle size SiC-doped MgB$_2$. The two-step reactions between Mg, SiC, and B release free C and Si to form strong flux-pinning centers. The current carrier density and flux-pinning force are important factors in improvement of the $J_c$ performance of nano-SiC-doped MgB$_2$.

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