

Effects of ZrH_2 doping and sintering temperature on the critical current density of MgB_2 wires

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Abstract

ZrH_2 -doped MgB_2 wires, with the composition of $\text{Mg}:\text{ZrH}_2:\text{B} = (1-x):x:2$ ($x = 0\%, 5\%, 8\%, 10\%, 12\%, 15\%$), were fabricated through the in situ powder-in-tube method by using low carbon steel tubes as sheath materials. Samples were sintered at 700 °C, 750 °C, 800 °C and 900 °C for 2 h respectively in a flow of high purity argon. It is found that the amount of ZrB_2 increases with the doping ratio, and the content of Fe_2B increases with the sintering temperature. Critical current density (J_c) of all the doped samples decreases with the increase of sintering temperature, but the optimum doping ratio goes up correspondingly. Among all the doped samples, the samples of $x = 5\%$ and $x = 8\%$ exhibit better J_c properties at the sintering temperature of 700 °C, but the samples of $x = 10\%, 12\%$ and 15% present higher J_c values when sintered at 900 °C.

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1. Introduction

The discovery of superconductivity in MgB_2 with critical temperature of 39 K [1] has stimulated a lot of interests from scientists. Compared with

high temperature superconductors, MgB_2 has no weak-link problem at grain boundaries [2], and the cost of raw materials is low. Therefore, MgB_2 is believed a promising candidate for the engineering applications in the temperature range of 20–30 K.

Many methods have been tried to fabricate MgB_2 wires [3–8], in which powder-in-tube (PIT) technique is an effective one. However, the critical current density (J_c) of MgB_2 usually decreases rapidly with the increase of magnetic field (B), due to the lack of pinning centers. Fortunately, it is reported that chemical doping can improve the J_c and flux pinning of MgB_2 superconductors effectively [9–13]. Ma et al. [13] demonstrated that the J_c of MgB_2/Fe tape had been improved by the 5 at% doping of ZrSi_2 , ZrB_2 and WSi_2 , respectively, but the doping ratios have not been optimized in their investigation. Feng et al. [11] confirmed that all the Zr-doped MgB_2 bulk samples have better performance than the undoped one, and the highest J_c of $2.1 \times 10^6 \text{ A/cm}^2$ in 0.56 T at 5 K and $1.83 \times 10^6 \text{ A/cm}^2$ in self-field at 20 K had been achieved in the $\text{Mg}_{0.9}\text{Zr}_{0.1}\text{B}_2$ sample.

In this paper, MgB_2 wires were fabricated by using ZrH_2 as dopant. As ZrH_2 decomposes to be Zr and H_2 at high temperature, we believe it can act as good as Zr powders for the improvement of MgB_2 performances. Therefore, we have paid our attentions to the optimum doping ratio of ZrH_2 and its relation with the sintering temperatures.

2. Experimental

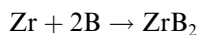
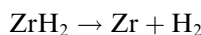
ZrH_2 -doped MgB_2 wires were fabricated by using the in situ PIT method. Mg (100–200 mesh, 99%), ZrH_2 (1250 mesh, 90%) and B (325 mesh, 99%) powders with the composition of $(1-x):x:2$ (doping ratio $x = 5\%, 8\%, 10\%, 12\%, 15\%$) were ground together in agate mortar for 3 h in air. Mixtures were properly filled into low carbon steel tubes (outer diameter: 16 mm, inner diameter: 12 mm). These composite tubes were groove-rolled, with four times of intermediate annealing performed at 400 °C, 450 °C, 590 °C and 580 °C for 1 or 2 h, respectively, to recover the plasticity and ductility of the low carbon steel. Subse-

quently, they were drawn to wires with the diameter of 1.6 mm. In the end, short samples were heated up to 700 °C, 750 °C, 800 °C, 900 °C and sintered for 2 h respectively in a flow of high purity argon. Then furnace-cooled to room temperature. Undoped MgB_2 wire ($x = 0$) was fabricated by using the same method, but only having two times of intermediate annealing at 590 °C and 580 °C, respectively.

Phase identification was performed by using the X-ray diffractometer. Microstructure analysis was carried out by scanning electron microscopy (SEM). Critical current (I_c) was measured by the standard four-probe resistance method in magnetic fields up to 7 T at 4.2 K. The criterion for the I_c definition was 1 $\mu\text{V/cm}$. J_c was obtained by dividing I_c by the cross-sectional area of the superconducting core.

3. Results and discussion

Fig. 1 illustrates X-ray diffraction (XRD) patterns of $x \text{ mol ZrH}_2$ -doped samples. MgB_2 , ZrB_2 , MgO , Fe_2B , $\delta\text{-ZrH}_2$ and Mg can be identified in the samples sintered at 700 °C. The appearance of ZrB_2 is because that ZrH_2 decomposed and reacted with B during the sintering process, as shown following:



As the relative peak intensity of ZrB_2 increases with the increasing of x , we believe the amount of ZrB_2 rises up correspondingly. However, the existence of $\delta\text{-ZrH}_2$ indicates that ZrH_2 did not decompose thoroughly when sintered at 700 °C.

Fe_2B appears mainly as the interface layer between the sheath materials and the superconducting core. It is the formation of Fe_2B that consumed some B atoms and led to the surplus of Mg.

As the sintering temperature increasing, the phase compositions of these samples change slightly. No $\delta\text{-ZrH}_2$ can be found in all samples sintered at 750 °C, such as shown in the sample $x = 8\%$. MgB_4 appears in the samples sintered above 850 °C, especially at 900 °C. The concentration of

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