

# Effect of Carbon and SiC Doping on Microstructures of MgB<sub>2</sub> Bulks and Wires by a Two-Step Reaction Method

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**Abstract:** Carbon and SiC doped MgB<sub>2</sub> bulks and wires were fabricated by a two-step reaction method. The MgB<sub>2</sub> samples were sintered at 900 °C for 2 h in closed argon atmosphere, and then an appropriate amount of Mg powder was mixed with the ground MgB<sub>2</sub> powder to get MgB<sub>2</sub> green compacts, which were sintered at 750 °C for 2 h to get bulk samples or at 680 °C for 2 h to get wire samples in closed argon atmosphere. Carbon or SiC doped MgB<sub>2</sub> bulk samples were also prepared by conventional solid state sintering for comparison. The phase evolution and the microstructure of MgB<sub>2</sub> samples were characterized by means of X-ray diffraction (XRD) and scanning electron microscope (SEM). The SEM results indicate that the compactedness and the dopant distribution homogeneity of MgB<sub>2</sub> samples prepared by the two-step reaction method are improved obviously. The carbon doping can more effectively introduce the dopants into MgB<sub>2</sub> crystalline lattice and grain boundaries compared with SiC doping.

**Key words:** MgB<sub>2</sub>; doping; phase evolution; sintering; two-step reaction

MgB<sub>2</sub> has been regarded as one of the most promising superconductors since its discovery in 2001<sup>[1]</sup>. However, the applications of pure MgB<sub>2</sub> superconductor face serious obstacles due to its low upper critical magnetic field ( $H_{c2}$ ) and irreversibility field ( $H_{irr}$ )<sup>[2]</sup>. Chemical doping is an effective way to improve the superconductivity of MgB<sub>2</sub>, and the SiC and C are thought to be the effective dopants<sup>[3-5]</sup>. In general, the MgB<sub>2</sub> samples were prepared by conventional direct sintering of the mixed powders of Mg, B and dopants in previous experiments<sup>[6-11]</sup>. The prepared MgB<sub>2</sub> samples had a high porosity, which decreased the effectively carried current area and restricted the increase of current density of MgB<sub>2</sub> superconductors. The two-step reaction method can effectively introduce the dopants into the MgB<sub>2</sub> crystalline lattice, and therefore improve the upper critical field<sup>[12]</sup>.

However, the phase evolution and the microstructure of MgB<sub>2</sub> samples prepared by the two-step reaction method have

not been studied yet and compared clearly with that prepared by conventional solid state sintering. In the present work, the doping effect of C and SiC on the phase evolution and the microstructure of MgB<sub>2</sub> bulks and wires prepared by the two-step reaction method were studied.

## 1 Experiment

The starting powders for the present investigation were Mg powder, crystalline boron powder, amorphous carbon and nano SiC powders. The powders with a nominal atomic ratio of MgB<sub>1.92</sub>C<sub>0.08</sub> or MgB<sub>1.92</sub>(SiC)<sub>0.08</sub> were prepared by a two-step sintering method. In the first step, magnesium, boron and carbon or nano SiC powders were separately weighed according to the nominal atomic ratio of MgB<sub>3.84</sub>C<sub>0.16</sub> (for sample A) or MgB<sub>3.84</sub>(SiC)<sub>0.16</sub> (for sample A'), and then they were separately mixed and ground in an agate mortar for 30 min in a vacuum glove box in argon atmosphere. The two

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powder mixtures were separately pressed into green disks at a pressure of 10 MPa; afterward they were enveloped in a niobium foil and put into a horizontal tubular furnace. The disks were sintered at 900 °C for 2 h in closed argon atmosphere. After sintering, the samples were cooled naturally to get sintered sample A and sintered sample A'. In the second step, the sintered bulks were separately ground into powders by mechanical grinding and then separately mixed with an appropriate amount of Mg powder to get the  $\text{MgB}_{1.92}\text{C}_{0.08}$  (for sample B) or  $\text{MgB}_{1.92}(\text{SiC})_{0.08}$  (for sample B') powders. A part of the powders were separately pressed into green disks at a pressure of 10 MPa, and then sintered at 750 °C for 2 h in closed argon atmosphere to get bulk sample B and bulk sample B'.  $\text{MgB}_{1.92}\text{C}_{0.08}$  (sample C) or  $\text{MgB}_{1.92}(\text{SiC})_{0.08}$  (sample C') bulks were also prepared by conventional solid state sintering for comparison. Another  $\text{MgB}_{1.92}\text{C}_{0.08}$  (for sample B) or  $\text{MgB}_{1.92}(\text{SiC})_{0.08}$  (for sample B') mixed powders were separately filled in Nb/Cu composite tubes, and then the filled composite tubes were drawn to 1 mm in diameter. There was no any annealing during the fabrication process. After cold deformation, the composite wires were cut and sintered at 680 °C for 2 h in closed argon atmosphere, and then furnace-cooled to room temperature to get wire sample B and wire sample B'.

The phase composition was identified by XRD with Cu-K $\alpha$  radiation. Microstructure was examined on cross section of  $\text{MgB}_2$  bulks and wires by SEM.

## 2 Results and Discussion

Fig.1 shows the XRD patterns of (a)  $\text{MgB}_{3.84}\text{C}_{0.16}$  (sintered sample A), (b)  $\text{MgB}_{1.92}\text{C}_{0.08}$  (bulk sample B) prepared by two-step reaction method and (c)  $\text{MgB}_{1.92}\text{C}_{0.08}$  (bulk sample C) prepared by conventional solid state sintering. It can be seen from pattern (a) in Fig.1 that the  $\text{MgB}_2$  is the main phase, and a great amount of  $\text{MgB}_4$  is formed in the first sintering reaction step. It can be seen from pattern (b) in Fig.1 that  $\text{MgB}_4$  phase fully disappears because the reaction between  $\text{MgB}_4$  and Mg converts into  $\text{MgB}_2$  during the second sintering reaction step. Some residual magnesium can be found in the sample B and C.

Fig.2 shows the SEM images of bulk sample B and bulk sample C. It can be seen that the micro-scale pores are formed in the bulk sample C due to the vacancy and evaporation of magnesium, and the porosity of bulk sample B is evidently lower than that of bulk sample C. The density of sample C is  $1.05 \text{ g}\cdot\text{cm}^{-3}$ , while the density of sample B is  $1.36 \text{ g}\cdot\text{cm}^{-3}$ . Nardelli et al calculated the theoretical density of  $\text{MgB}_2$  prepared by the two-step reaction method, which was  $2.25 \text{ g}\cdot\text{cm}^{-3}$ , while the theoretical density of  $\text{MgB}_2$  prepared by the conventional solid state sintering method was  $2.01 \text{ g}\cdot\text{cm}^{-3}$  [13]. An increase of the density due to the change of reaction route likely contributes to the increase of  $J_c$ , which has been proved by our previous work [14]. The carbon doping could effectively introduce the dopants into the  $\text{MgB}_2$  crystalline lattice and

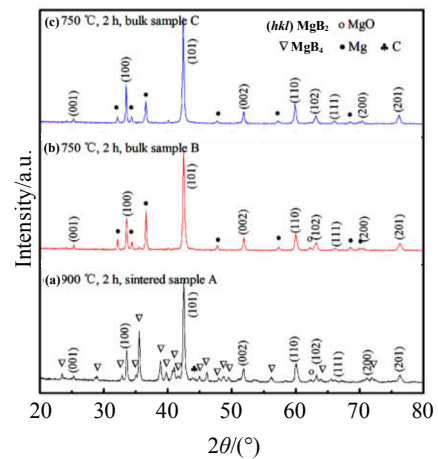


Fig.1 XRD patterns for sample A, B and C

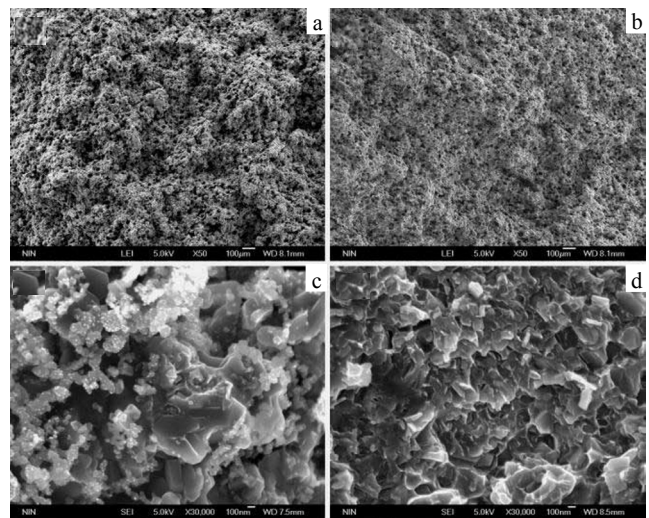


Fig.2 Typical SEM images for bulk sample B (a, c) and bulk sample C (b, d)

grain boundaries. Yan et al proved that the carbon atoms were uniformly distributed in the  $\text{MgB}_2$  bulk prepared by the two-step reaction method [15]. According to the above-mentioned results the following finding can be drawn: the two-step reaction method can improve the compactedness and the dopant distribution homogeneity of  $\text{MgB}_2$  sample compared with the conventional solid state sintering method.

Fig.3 shows XRD patterns of (a)  $\text{MgB}_{3.84}(\text{SiC})_{0.16}$  (sintered sample A'), (b)  $\text{MgB}_{1.92}(\text{SiC})_{0.08}$  (bulk sample B') prepared by two-step reaction method and (c)  $\text{MgB}_{1.92}(\text{SiC})_{0.08}$  (bulk sample C') prepared by conventional solid state sintering. It can be seen from pattern (a) in Fig.3 that  $\text{MgB}_2$  is the main phase, and a great amount of  $\text{MgB}_4$ , some  $\text{Mg}_2\text{Si}$  and  $\text{MgO}$  are formed in the first sintering reaction step. It can be seen from pattern (b) and (c) in Fig.3 that  $\text{MgB}_4$  phase also fully disappears. Some residual magnesium and  $\text{Mg}_2\text{Si}$  still can be found in bulk samples B' and C'.

Fig.4 shows SEM images of bulk sample B' and bulk

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