



Effect of heating rates on microstructure and superconducting properties of pure MgB_2

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ABSTRACT

The influence of different heating rates, ranging from 5 to 30 K min^{-1} , on the microstructure and superconducting of the MgB_2 bulk was investigated. No obvious variation in the grain size was found for the samples heated from 5 K min^{-1} to 20 K min^{-1} except for the changes in morphologies. Moreover, the grain refinement was obtained under the heating rate of 30 K min^{-1} . The critical current density (J_c) suggested that the 5 K min^{-1} sample had the best performance in high field. Here, the differential thermal analysis (DTA) was employed to analyze the kinetics of MgB_2 phase formation with the different heating rates. The results showed that the large amount of MgB_2 formed at low temperature, which lead to compact structures under the slow heating rate. The fast heating rate would promote the evaporation of Mg at high temperature, which was considered to generate the vacancy and impurities in the sample.

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1. Introduction

The discovery of superconductivity in magnesium diboride (MgB_2) at 39 K [1] has attracted worldwide interest for both fundamental physical study and application [2,3]. The relatively high critical temperature, the low cost and the ‘weak-link’ free grain boundaries make MgB_2 an excellent candidate for the fabrication of wires and tapes [4]. In recent years, significant progress has been made in improving the superconducting properties especially those for enhancing the critical current density (J_c) in magnetic field [5]. Nevertheless, J_c of pure MgB_2 drops rapidly with increasing applied magnetic field because the pinning force is not strong enough [6]. So far, various dopants, such as nano-SiC [7,8], carbon nanotubes (CNTs) [9,10] and nano-diamond [11] have proved to be the most effective way for increasing J_c . Despite many kinds of chemical doping were used to improve the J_c performance in field, the annealing conditions also have strong influence on microstructure and corresponding J_c . In previous studies [12], the relationships between sintering temperature and critical current density had been well investigated. The pure and SiC doped MgB_2 had best J_c values when sintered at low temperature because of strong grain boundary defects. Meanwhile, Matsumoto et al. [13] studied the contribution of connectivity, flux pinning and upper critical field to critical current density. It was concluded that higher reaction temperature en-

hanced the connectivity but degraded H_{c2} and flux pinning, so the better J_c (4.2 K) was obtained for the 600 °C reaction.

Until now, it seems to be little research on the effect of different heating rates on the microstructures and corresponding properties of MgB_2 . Chen et al. [14] had successfully analyzed the influence of heating rates on the J_c of pure MgB_2 , carbon nanotube and nano-SiC-doped in-situ MgB_2/Fe wires. It was found that higher J_c was obtained for pure MgB_2 samples when treated with slower heating rates. The large amount of MgB_2 formed below 650 °C without Mg deficiency was responsible for the highest J_c . However, the influence of heating rates on the microstructures, grain size and superconducting properties has not been clearly clarified.

In this study, therefore, we fabricated the MgB_2 bulk sample by an in-situ reaction process and four different heating rates were selected for comparison. The effect of heating rates on the formation of MgB_2 phase and microstructures was explored in detail. The superconducting parameters such as transition temperature (T_c), resistivity (ρ), active cross-sectional area fraction (A_F), and critical current density (J_c) were also evaluated. The strong correlations between microstructures characteristics and superconducting properties were also investigated.

2. Experimental procedure

Bulk MgB_2 samples were synthesized by traditional in-situ reaction method, described in Ref. [15]. Powders of Magnesium (99%

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purity) and amorphous boron (99% purity) were mixed at the atomic ratio of 1:2 in an agate mortar. Then the well-mixed powders were mechanically pressed into pellets of 4 mm in diameter and 2 mm in thickness. The samples were sintered in the differential thermal analysis (DTA) under the protection of argon atmosphere. Four heating rates, namely, 5, 10, 20 and 30 K min⁻¹, were brought into comparison, and the influence of duration was omitted in this study. The samples were heated to 1073 K at different heating rates, and then directly cooled down to room temperature at a rate of 40 K min⁻¹.

The phase compositions of the samples were characterized by X-ray diffraction (XRD) by Rigaku D/max 2500 X-ray diffractometer with Cu K α radiation. Field-emission gun scanning electron microscopy (FEG-SEM) was employed to analyze the microstructure. The superconducting transition temperature, T_c was performed by the standard four-point probe method. The critical current density, J_c was measured by the standard dc superconducting quantum interference device (SQUID) and calculated from the width of magnetization hysteresis loops based on the extended Bean model. In order to monitor the changes of phases during the sintering, the DTA curves were obtained by DTA measurement using a Netzsch DSC 404C calorimeter.

3. Results and discussion

The XRD patterns for the prepared MgB₂ samples with different heating rates are shown in Fig. 1. The samples mainly consist of MgB₂ phase, and only a small amount of MgO was found. However, obvious differences are detected in patterns; residual Mg remained under the heating rates of 20 K min⁻¹ and 30 K min⁻¹, respectively. The peaks of Mg disappeared with decreasing the heating rate, which suggests that the slow heating process could promote the reaction between Mg and B powders, thus the high pure MgB₂ is obtained thoroughly. In addition, the intensities of X-ray diffraction in sample with 30 K min⁻¹ are found to be weaker than the other three samples, the weaker peak corresponds to a finer structure of the grains. In order to investigate the change of grain size with the different heating rates, the full width at half maximum (FWHM) was employed to determine the average grain size by Williamson-Hall analysis. The changes in FWHM of different planes are shown in Fig. 2 and the calculated grain sizes are listed in Table 1. It is notable that the grain size gives large value reaching at 230 nm before 20 K min⁻¹, and no obvious variation was detected for the sample heated from 5 K min⁻¹ to 20 K min⁻¹. In contrast to the lower heating rates, the grain size decreased rapidly to

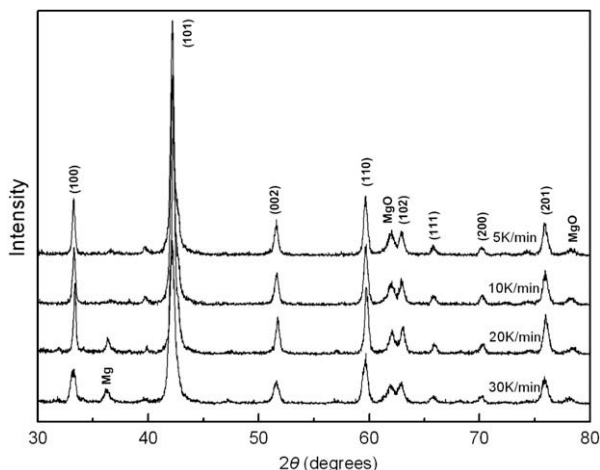


Fig. 1. X-ray diffraction patterns of the sintered MgB₂ samples with different heating rates.

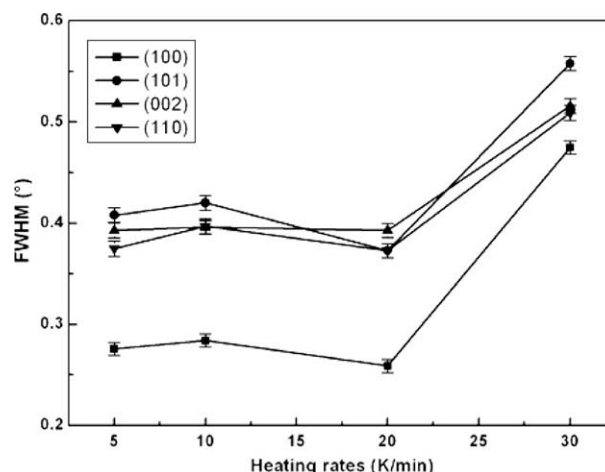


Fig. 2. FWHM of the pure MgB₂ samples prepared at different heating rates. The corresponding planes of MgB₂ are (1 0 0), (1 0 1), (0 0 2) and (1 1 0), respectively.

152 nm for the sample 30 K min⁻¹, which is consistent with the intensity and widths of the XRD peaks seen in Fig. 2. In other words, the heating rate above 30 K min⁻¹ may lead to the grain refinement.

These grain sizes analysis agreed fairly with the observation of the FEG-SEM images in Fig. 3. The MgB₂ grains treated at the rate of 5 K min⁻¹ are in the range of 100–200 nm with irregular crystal structures (shown in Fig. 3a). A large amount of crystal-like grains with regular hexagonal morphology can be detected with the increase of heating rates before 20 K min⁻¹, as shown in Fig. 3b and c. However, the grain sizes of the first three samples seem to be at the same level and just exhibit the different morphologies. As for the sample heated at the rate of 30 K min⁻¹, the grains appear to refine to smaller ones, moreover, no visible hexagonal structure was found in the image. In addition, SEM images also display well-consolidated structures in the 10 K min⁻¹ and 20 K min⁻¹ samples. It is believed that better crystallinity and larger grain size may benefit to the improvement of connectivity in MgB₂ samples. The smaller grain size in Fig. 3d will weaken the intergranular connection between grains.

The different heating rates are considered to be responsible for the formation of different grain sizes and morphologies in the four samples. The process of reaction between Mg and B powders recorded by differential thermal analysis (DTA) is investigated for the further explanation of the results. Fig. 4 shows the obtained DTA curves for the samples. In general, the DTA curve consists of two exothermic peaks, which is associated with the formation of MgB₂ phase at different stages, the only endothermic peak appears at 923 K is related with the Mg melting. Comparing with the four curves, some differences were discovered by the variation of peaks. It is found that the start temperature for the first exothermic peak decreased with the decreasing heating rates. Moreover, the intensities of the second exothermic peak gradually exceed the first peak with the increasing heating rates. For example, the second exothermic peak is lower and narrower than the first exothermic peak in 5 K min⁻¹ sample, while in 30 K min⁻¹ sample, the second peak is higher and broader compared with the first peak. The results show that more MgB₂ phase forms at the first stage with the low heating rate, and the fast heating rates promote most of the MgB₂ phase formed at the second stage. In addition, an obvious difference of the endothermic peaks among four curves could also be detected in Fig. 4 (the arrow is pointing to the minimum of the endothermic peak, which is corresponding to the totally melting of Mg). It is found that the temperature where Mg totally melts moves to high value with the increasing heating rate.

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