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Optimization of the fabrication process for high trapped field MgB_2 bulks

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ABSTRACT

The optimization of the sintering conditions of disk shaped bulk MgB_2 superconductors with respect to the trapped field is described. Series of samples were prepared by varying the sintering temperatures between 700 and 950 °C. The temperature range was divided into three regions, namely the low (<750 °C), medium (<825 °C), and high (>850 °C) temperature region. Scanning electron microscopy and X-ray diffraction indicated that homogenous single phase MgB_2 bulks were produced in the medium sintering temperature range. At this processing temperature range the highest trapped field was also achieved. Samples of 20 mm in diameter and 7 mm thick produced at 775 °C exhibited trapped field of 1.50 T at 20 K.

1. Introduction

Since the discovery of superconductivity in MgB_2 at 39 K [1] an enormous effort has been devoted to processing, characterization, and application of MgB_2 material [2,3]. The superconducting transition temperature of MgB_2 is significantly lower than in $YBa_2Cu_3O_y$ "Y-123". However, MgB_2 possesses several advantages in comparison to Y-123, like much smaller anisotropy, high critical current density (J_c) not hindered by grain boundaries [4], and low cost of the raw material. These features make this material very promising for a number of industrial applications [5]. Further important issues are the shorter processing time and no need of oxygenation. The MgB_2 disks can be used in a similar manner as melt-textured Y-123 bulks [6]. They can be used as a magnetic field source in nuclear magnetic resonance (NMR), magnetic resonance imaging (MRI), fault current limiters, in the pumps for liquid gas pumping, and shielding screens. A number of experiments have been already performed to prepare good quality and high density MgB_2 bulks and wires. High pressure techniques were used to produce a highly dense MgB_2 material [6]. An easy MgB_2 sintering route by the reactive Mg-liquid infiltration (RLI) process was reported. This infiltration process does not require an external pressure to be applied to the reacting material and still produces highly dense final products [7]. It has been shown that MgB_2 bulk magnets could be used at 20 K [8–11]. High critical current density (J_c) and good homogeneity are essential for high trapped field magnets. Superconducting properties of a bulk material depend on the processing conditions, like the synthesis temperature, holding time, annealing atmosphere, additives etc. In order to achieve the high trapped field in MgB_2, optimization of the synthesis temperature is essential.

In this paper, we report on several sets of large bulk MgB_2 blocks prepared by a simple solid state reaction route. To optimize the highest trapped field of the bulk MgB_2, we fixed the initial composition of Mg:B to 1:2, holding time was fixed to 3 h, annealing atmosphere was pure argon and the synthesis temperature was varied from 700 to 950 °C in steps of 25 °C. The phase and microstructure were estimated by X-ray diffraction (XRD) and by scanning electron microscope (SEM). Trapped field measurements performed from 20 K to 40 K showed that the processing temperature was very important for obtaining high trapped fields.

2. Experimental

The Mg and B powders used in the present study were purchased from Furuchi Chemical Corporation, Japan. Polycrystalline MgB_2 samples were made using the in situ solid state reaction. High-purity commercial powders of Mg metal (99.9% purity, 200 meshes) and amorphous B powder (99% purity, 300 meshes) were mixed in a nominal composition of MgB_2. The starting powders were thoroughly ground in a normal glove box in nitrogen atmosphere. The low cost glove box without vacuum facility and nitrogen atmosphere were chosen to reduce the production cost of the MgB_2 material. The powder mixture was pressed into pellets of 20 mm in diameter using a hydraulic press. Eventually, a Ti sheet was wrapped around the tightly pressed pellets, which were then sintered in argon atmosphere in a tubular furnace. The samples were prepared in a single step heat process with the following heat treatment profile: The sintering temperature chosen between

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700 °C and 950 °C was achieved in 5 h after the start from the ambient temperature. The sintering temperature varied in steps of 25 °C. During the sintering in flowing argon gas the temperature was kept constant for 3 h. Then, the temperature was reduced to room temperature with the cooling rate of 100 °C/h. After sintering, the Ti sheets were removed from the MgB_2 bulks.

The microstructure of these samples was studied by means of a scanning electron microscopy (SEM) and chemical composition of the matrix was analyzed by energy dispersive X-ray spectroscopy (EDX). The constituent phases of the samples were determined by means of a high-resolution automated X-ray powder diffractometer RINT2200, using Cu Kα radiation generated at 40 kV and 40 mA. To correlate reliably the sintering temperature with the trapped field of the MgB_2 bulks, several selected samples were always measured. The trapped magnetic field was studied by magnetizing the bulk sample in a 10 T superconducting magnet in temperatures between 20 K and 40 K.

3. Results and discussion

3.1. X-ray diffraction

The sintering temperature was correlated with the phase of the MgB_2 product by means of a powder X-ray diffraction patterns recorded at room temperature. Fig. 1 shows the selected XRD patterns for MgB_2 material sintered at 700, 775, and 950 °C. In all samples the main phase is a pure MgB_2. Amorphous boron powder is not detected but small content of Mg is clearly seen for the sample sintered at 700 °C, which is similar to the earlier reports [12]. In contrast, almost single MgB_2 phase was observed for the sample sintered at 775 °C. When temperature increased to 950 °C, MgB_4 phase formation was observed (see Fig. 1). X-ray diffraction patterns for all samples and their phase analysis indicated that the samples could be divided into three distinguished groups, according to different phase formation. The groups correspond to low (<750 °C), medium (<825 °C), and high sintering temperatures (>850 °C). From the XRD analysis we found that nearly single phase bulk MgB_2 materials could be obtained at around 775 °C.

3.2. Microstructure analysis

Microstructure control is the most complicated but at the same time the most important task in optimization of materials' processing parameters in general. Fig. 2 shows low resolution SEM images

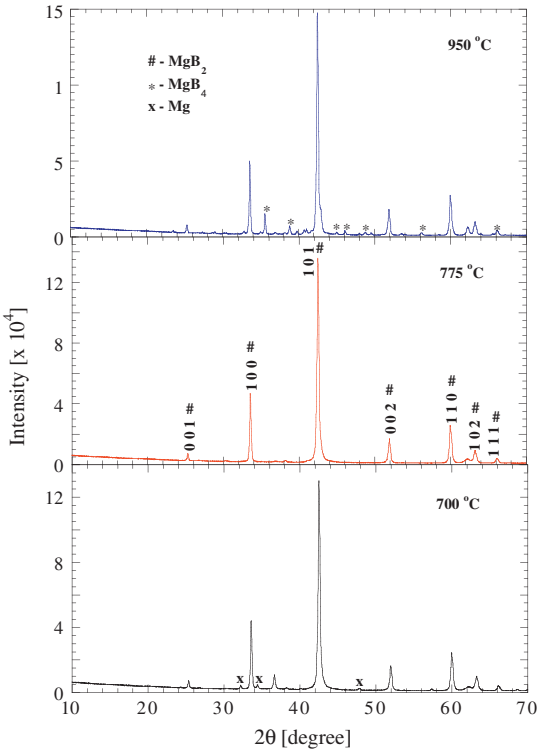

Fig. 1. X-ray diffraction spectra of bulk MgB_2 samples produced by a sintering process in Ar atmosphere: (top) at 950 °C; (middle) at 775 °C; and (bottom) at 700 °C.

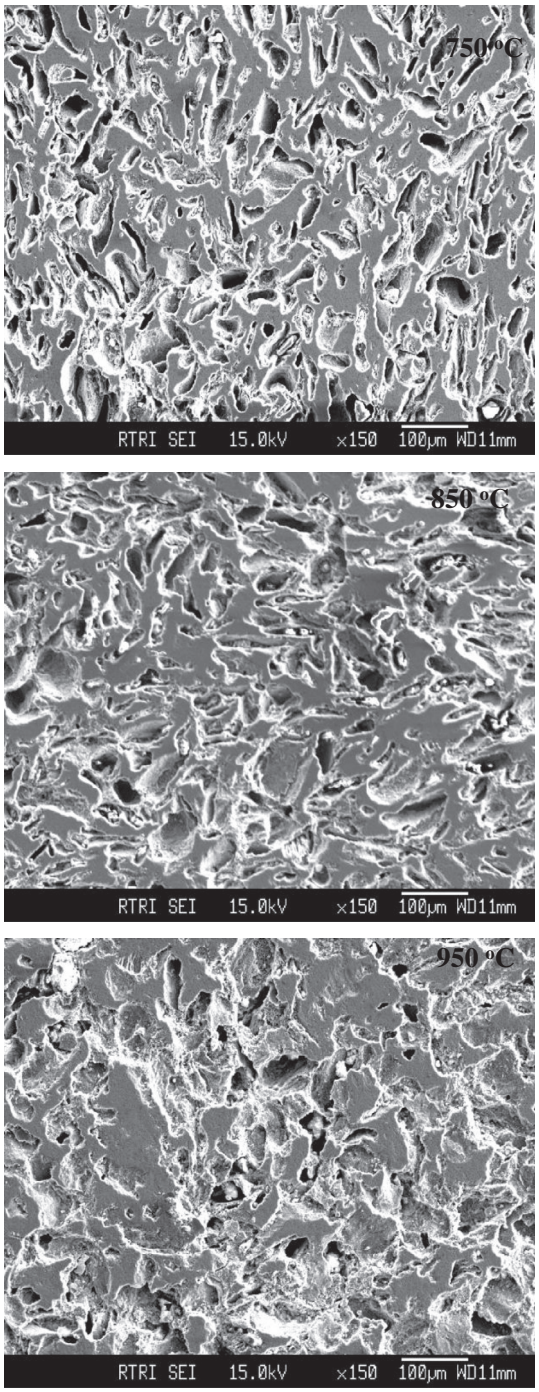

Fig. 2. Low magnification scanning electron microscope (SEM) images of polished cross-sections of sintered bulk MgB_2 samples synthesized in Ar atmosphere: (top) at 750 °C; (middle) at 850 °C; and (bottom) at 950 °C.