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The microstructures and superconducting properties of MgB_2 bulks prepared by a high-energy milling method

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Abstract

We succeeded in the synthesis of high-J_c MgB_2 bulks via high energy ball-milling of elemental Mg and B powder at ambient temperatures. The mixed powder was ball-milled for 1–10 h and the completed reaction was achieved by subsequent annealing. The correlations among synthesis parameters, microstructures and superconducting properties in MgB_2 bulks were investigated. Samples were characterized by X-ray diffraction and scanning electron microscope, and the magnetization properties were examined by a superconducting quantum interfere device magnetometer. The highest J_c, approximately 2.3×10^5 A/cm^2 (15 K, 3 T), was obtained for samples milled for 5 h and sintered at 750 °C for 1 h. It is even comparable with SiC-doped MgB_2 bulks made by Dou's group, which had exhibited the strongest reported flux pinning and the highest J_c in high field to date.

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Keywords: Bulk MgB_2; High energy ball-milling; High-J_c

1. Introduction

The improvement of the intrinsic properties of MgB_2 was recognized as a decisive goal to enable potential applications [1]. Now, MgB_2 is showing higher H_{c2} than that of conventional NbTi and Nb_3Sn and becoming the first promising metallic superconductor applicable at 20 K. In particular, very high upper critical field, $H_{c2}(0)$, exceeding 30 T has been reported for MgB_2 wires, tapes and bulks. However, it was recognized that the irreversibility field H_{irr} (T) of the samples prepared by standard solid state reaction is apparently lower than H_{c2} due to the lack of sample texture. A typical relationship, $H_{irr}(T) \sim 0.5H_{c2}(T)$, has been observed for the undoped MgB_2 bulks [2,3]. Patnaik et al. [4] showed that the parallel and perpendicular components of the irreversibility field in fact lie very close to the parallel and perpendicular upper critical field by using textured MgB_2 thin film, as would be expected for a strong superconductor with small grains and flux-pinning by grain boundaries. The high energy ball-milling technique facilitates the formation of an optimal microstructure with small particle sizes. Hence, a high grain boundary density is obtained, which is expected to enhance the magnetic flux pinning ability and to improve the critical currents in external magnetic fields.

2. Experimental

Mg (99.8%) and amorphous B (95+%) powders according to a composition of MgB_2 with 5 wt.% Mg surplus were placed under purified Ar-atmosphere in an agate milling

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container and grinding balls. The high energy ball milling was performed on a SPEX 8000M mill for times 1–10 h using a ball-to-powder mass ratio of 3. The mixed powders were cold pressed into pellets under a pressure of 20 MPa and then placed in an alumina crucible inside a tube furnace under ultra-high purity Ar-atmosphere. The synthesis temperatures were particularly chosen to get optimal parameters for in situ MgB_2 bulks prepared by a high energy milling method. The phase compositions of the samples were characterized by the APD1700 X-ray diffraction (XRD) instrument. The surface morphology and microstructures of the samples were characterized by the JSM-6460 and the JSM-6700F scanning electron microscope (SEM). Magnetization versus magnetic field (M–H) curves were measured on rectangular-shaped samples at temperatures of 10–15 K by a superconducting quantum interfere device (SQUID) magnetometer.

3. Results and discussion

The XRD patterns of the samples milled and sintered for 1 h at different temperatures are shown in Fig. 1. Mainly peaks of MgB_2 are visible but some peaks of MgO are present. Element Mg is also present for samples sintered at 650 °C and 700 °C due to the lower reactivity with oxygen compared with other samples.

The microstructure of these samples are shown in Fig. 2. Scanning electron microscope images mainly show fine spherical grains for the samples treated at 700 °C and 750 °C. Apparently it would be favorable for the optimum pinning of magnetic flux lines. The B-rich phase in black is detected by EDX for the sample sintered at 650 °C (see Fig. 3), which is due to the stability of the higher borides at lower temperature. The (Mg–O) compounds in light grey is detected by EDX for the sample sintered at 800 °C, as shown in Fig. 4. One of the crucial points during preparation of MgB_2 is to avoid weak-link-like behavior at grain

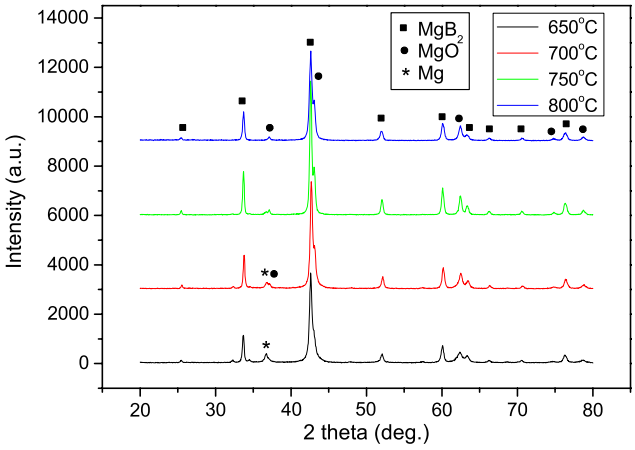

Fig. 1. The XRD patterns of samples milled and sintered for 1 h at different temperatures.

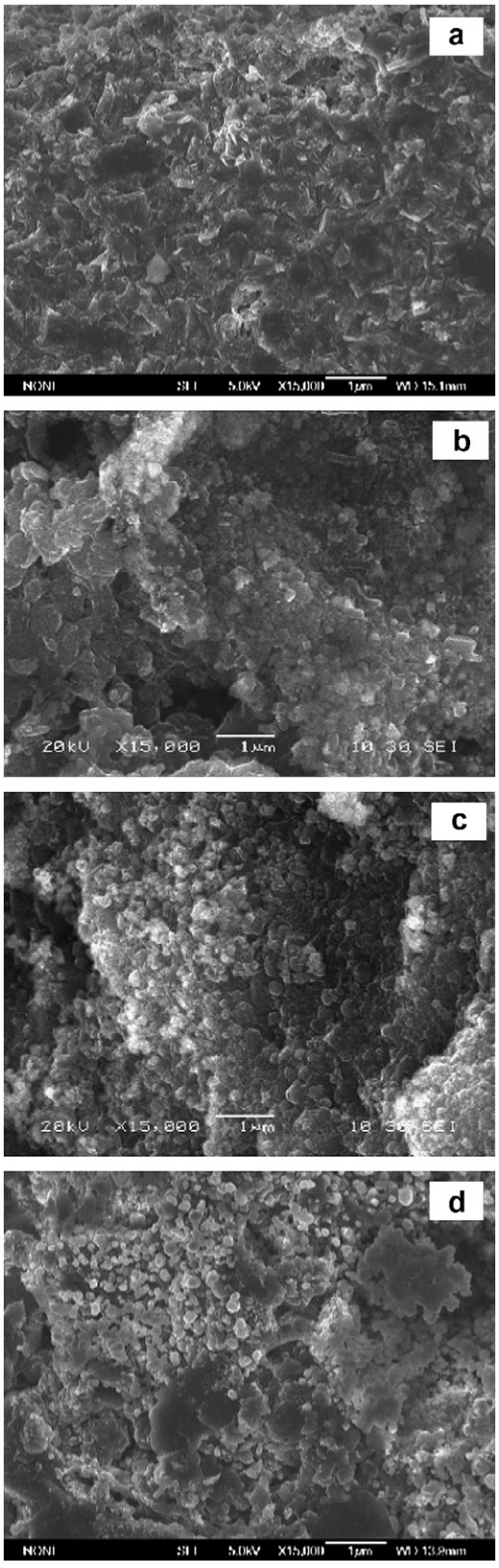

Fig. 2. SEM photograph of the MgB_2 bulks milled and sintered for 1 h at different temperatures: (a) 650 °C, (b) 700 °C, (c) 750 °C, and (d) 800 °C.