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FABRICATION OF SUPERCONDUCTING MgB_2 FROM BORON OXIDE (B_2O_3), AND ITS MICROSTRUCTURAL AND ELECTRICAL CHARACTERIZATION

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The discovery of superconducting MgB_2 (39 K) draws attention to it as a new material for applications based on superconductivity. Many researchers successfully synthesized MgB_2 using commercial boron and magnesium. In this study, elementary boron was obtained via an acid leaching process, after reacting B_2O_3, and Mg in an argon atmosphere at 800 ^oC. Energy Dispersive X-ray Spectroscopy (EDX) results revealed that the powder obtained from the reaction was boron in 92% purity with magnesium as the major impurity. Superconducting MgB_2 was produced from this boron and magnesium, in an argon atmosphere at 900 °C, by a conventional solid-state reaction. Superconducting MgB_2 powders were compressed in a dye to pellets by a hot pressing technique at 500 °C and 1 GPa. The microstructural properties of the MgB_2 were determined by X-ray Diffraction Spectroscopy, EDX, and Scanning Electron Microscopy techniques. The electrical properties of the fabricated MgB_2 were examined by resistivity measurements in a closed-cycle cryopump system, between 20 and 300 K. The critical temperature (T_c) of the MgB_2 pellets was around 32 K.

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Keywords: Superconductivity, MgB_2, Composites

1. Introduction

The discovery, in 2001 of superconductivity at 39 K in the intermetallic compound MgB_2 made this material a new alternative compound for superconductivity applications [1]. The transition temperature of 39 K means a higher operating temperature than commercially available superconductor like Nb_3Sn (18 K) [2]. Mass production of MgB_2 at a low cost is important, due to its great potential in the market for large and small scale applications. MgB_2 has a low fracture toughness, like the Nb based conventional superconductors and high temperature superconductors. Therefore, it is not a suitable material for large-scale applications based on wire production. A commonly used method for manufacturing superconducting wire from a hard and brittle material is the powder in tube method (PIT). In this, the superconducting powder is filled into a ductile metal sheath and swaged into small diameters by a mechanical process for wire applications. The metal matrix composite (MMC) technique can be regarded as an alternative method to the PIT method [3]. Metal matrix composites consist of at least two constituents; the metal matrix and an intermetallic compound. The metal matrix improves the ductility, hardness and elastic modulus of the new compound. The MMC method is a new one for producing bulk superconductors, especially for target production for small-scale applications based on thin film production. This method has been used for the MgB_2 superconductor by using an Al and Mg metal matrix [4,5]. For both the PIT and MMC techniques, production of high quality superconducting MgB_2 powders in large amounts is essential and technologically important, since B_2O_3 is cheaper and easily available in large amounts than elementary boron. Many research groups have successfully fabricated MgB_2 using commercial boron and magnesium [6].

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In this study, elementary boron was obtained using a solid-state reaction of B_2O_3 with Mg, in an argon atmosphere at 800 ^oC. The boron obtained, with 92% purity and with Mg as the major impurity, was used to produce MgB_2. The superconducting and microstructural properties of the MgB_2 were investigated after compressing in a dye to pellets, by a hot pressing technique at various temperatures and a pressure of 1 GPa, by the metal matrix composite method [7].

2. Experimental details

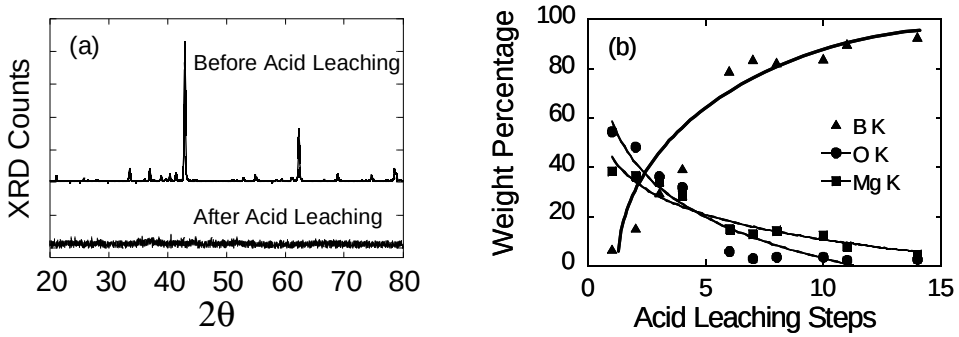

Fig. 1. (a) XRD patterns, before and after acid leaching of the powders obtained after the reaction of B_2O_3 and Mg at 800 °C (b) Variation of the B purity with respect to the number of acid leaching steps.

B_2O_3, reduced from H_3BO_3 with 98.5 % purity (Marmara Research Center, Turkey), and commercial Mg (99.999 % purity and - 44 μm mesh size, Alfa Aesar) were used in this study. The B_2O_3 and Mg were subjected to a solid-state reaction at 800°C in an Ar atmosphere for 1 hour. The particle size of the resulted powder at the end of the reaction was reduced in a mortar. Powder XRD measurements revealed that the resulted powder consisted of MgO and B, as shown in Fig. 1. Acid leaching with 0.5 M hydrochloric acid was applied to the powder, to remove the MgO. High purity (93.05 %) B was obtained after such a repetitive process of acid leaching. This method is known as the simplest available [8], but less pure boron can be obtained. On the other hand, the major impurity is Mg, which is a constituent of MgB_2. Although there are a number of methods to reduce B from B_2O_3, these do not provide as high a purity as in the acid leaching method. Superconducting MgB_2 powder was obtained after the produced elementary B and Mg were subjected to a solid-state reaction in an Ar atmosphere at 900°C for 2 hours. Pellets with diameter of 10 mm were produced by placing the MgB_2 powder in a cylindrical metal die and heating at 500 °C at 1 GPa in air, to investigate the superconducting and microstructural properties. X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX) techniques were used for the microstructural analysis and phase determination. The electrical properties of rectangular samples with dimensions 10 × 2 × 0.9 mm were analyzed by resistivity measurements, in a closed-cycle cryopump system.

3. Results and discussion

XRD patterns of the B + MgO powders after the reaction of B_2O_3 and Mg at 800°C for 1 h in Ar atmosphere, before and after acid leaching, are shown in Fig. 1a. The peaks before acid leaching were reduced to the noise level, showing only amorphous boron after the acid leaching process. This shows that all phases except MgO and B in the sediment disappeared. EDX analysis (Fig. 1b) was carried out in order to detect the amount and purity of the boron in the amorphous powder. This increased with the number of acid leaching steps, as shown. The amount of boron in the initial output product was 6.97 %. It increased to 16 % after the 1st acid leaching and was above 80 % after 6th step. Finally, after the 13th step, a purity of 93% was reached,and the Mg impurity was around

5 %. The 5% Mg impurity is not important, since the final goal was to obtain superconducting MgB_2 using another solid-state reaction of the amorphous boron powder with high purity commercial Mg. Reducing the amount of Mg by 5% in the solid-state reaction compensated for the impurity.

For the production of MgB_2, elemental boron was reacted with pure Mg of 325 mesh at 900 °C in an Ar atmosphere, for 2 hours. Since Mg vaporizes at this temperature, mixing Mg and B in a stoichiometric ratio would cause the amount of Mg in resulting MgB_2 to be less than its stoichiometric value. This situation may degrade the superconducting properties [9]. For this reason, 10% of excess Mg was added to conserve the stoichiometry after the reaction.

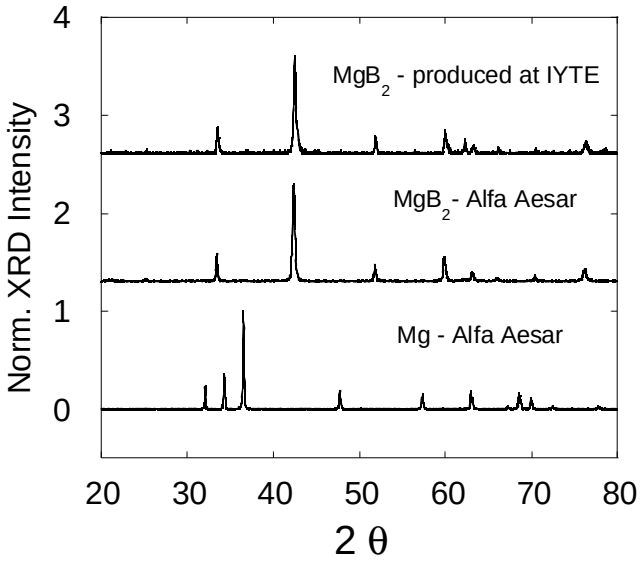

Fig. 2. Normalized XRD patterns of the produced MgB_2.

The normalized XRD patterns of commercial Mg, MgB_2 and the produced MgB_2 are shown in Fig. 2. The patterns for the commercial MgB_2 and the produced MgB_2 are normalized to 1 and shifted vertically. The produced and commercial MgB_2 peaks match exactly, except for a Mg peak present at $2\theta = 63.6$ as an impurity.

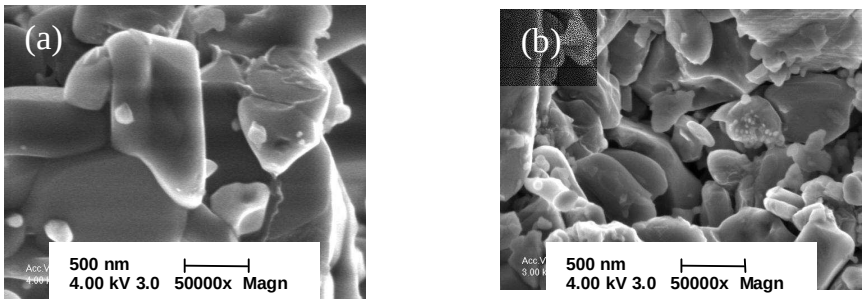

Fig. 3. SEM images of the produced MgB_2 (a) as a powder, (b) as a pellet.

SEM images of the produced MgB_2 in powder form and in a pellet compressed at 1 GPa at 500 °C for 2 hours are shown in Figs. 3a and 3b respectively. The average particle size was smaller for the pellet material, due to pressure and annealing. The small particle size increases the number of grain boundaries, to increase the critical current density. This is also an important superconducting parameter of the material, to improve the pinning properties [10].

Rectangular samples were cut from the pellets, to measure the resistivity between 20 and 300 K. A resistivity versus temperature plot is given in Fig. 4, with the resistivity value normalized to that at 50 K. The superconducting transition occurs at 32 K. The measured density of the superconducting MgB_2 pellet was 1.99 g/cm^3, which is smaller than the theoretical value of 2.62 g/cm^3. This shows that a large amount of voids is present in the material. This high porosity ratio causes a low connectivity between the MgB_2 grains. The low transition temperature and broadened transition interval indicate the low grain connectivity due to the porosity.

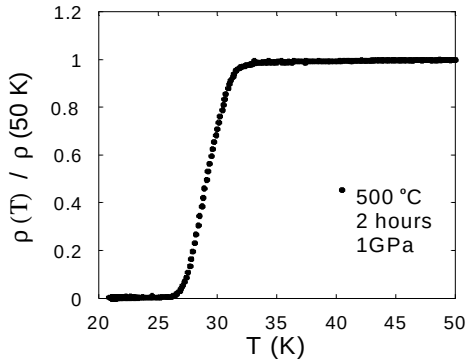

Fig. 4. Temperature dependence of the resistivity of the produced MgB_2.

4. Conclusions

The XRD, EDX, SEM and temperature dependent resistivity results revealed that superconducting MgB_2 is successfully produced from the reaction of Mg and B reduced from B_2O_3. The produced MgB_2 pellet showed a superconducting transition at about 32 K.

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