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Effects of the post-annealing temperature on the properties of MgB_2 thin films

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가열냉각 온도에 따른 MgB_2 박막의 특성변화

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Abstract

We have fabricated MgB_2 thin films on $(1\ \bar{1}\ 0\ 2)$ Al_2O_3 substrates by using a two-step method. Amorphous B thin films were deposited by a pulsed laser deposition technique and sintered in Mg vapor at various temperatures from 800 to 950 °C. Superconducting properties of the thin films were investigated by temperature dependences of magnetization and critical current density. Structural studies were carried out by an x-ray diffraction and a scanning electron microscope. The films fabricated at 900 °C showed the highest transition temperature of 39 K and critical current density of ~ 10^7 A/cm^2 at 15 K.

Keywords : MgB_2, thin film, superconductor, annealing temperature

I. Introduction

Recently, the superconductivity of the binary metallic MgB_2 with a remarkably high transition temperature T_c = 39 K [1] has brought about great interest of the scientists in applied superconductivity. Strongly linked nature of the inter-grain [2], [3] with a metallic charge density [4] in the polycrystalline samples indicating its potentials for technological applications. Immediately, many groups have synthesized samples of practical use, such as MgB_2 wires [5], [6], tapes [7], and thin films [8] - [13]. The values of critical current density J_c for MgB_2 wires and tapes were reported on the order of 10^5 A/cm^2 and those for MgB_2 thin films J_c ~ 10^6 A/cm^2 at zero field and low temperatures. Especially, the highest value of J_c for these films was reported to be ~ 10^7 A/cm^2 at 15 K [13], [14].

Because of the high volatility of Mg, many groups have fabricated MgB_2 by using the post-annealing process: magnesium diffuses into boron at high temperatures and high Mg pressures. In this process, the fabrication conditions, such as the temperatures and the pressure during deposition of the precursor films and the post-annealing temperature, will decide the sample quality and the superconducting properties. MgB_2 thin films fabricated by many groups have shown different superconducting behaviors. For example, the transition temperature

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was distributed widely from 24 K to 39 K due to some impurity phases. Therefore, we need to study effects of the post-annealing temperatures on the properties of MgB_2 thin films

In this paper, we prepared MgB_2 thin films on sapphire substrate at different annealing temperatures. We found that the MgB_2 film fabricated at the annealing temperature of T_{an} = 900 °C had the best quality with T_c = 39 K.

II. Experiments

We fabricated the MgB_2 thin films using a two-step method [8]. We pressed amorphous B (99.99%) powder into a disk shape with the diameter of 25.4 mm and the height of 5 mm under a pressure of 4 tons. The precursor B film was deposited on a Al_2O_3 (1 $\bar{1}$ 0 2) substrate at room temperature by using pulsed laser deposition technique. The laser energy density was 20 - 30 J/cm^2 at a laser flux of 570 mJ/pulse and a pulse frequency of 8 Hz. The prepared amorphous B film was put into a Nb tube together with a high purity Mg metal (99.9%) and the Nb tube was sealed in a pure Ar atmosphere. The heat treatment was carried out in an evacuated quartz tube to avoid oxidation of the Nb tube. The post-annealing temperature was 800 ~ 950 °C for 10 - 30 minutes, and then the sample was quenched to room temperature

To characterize the MgB_2 thin films prepared at different post-annealing temperatures, we measured the magnetization by a superconducting interference device magnetometer (Quantum Design MPMS). Also, studies on their structure and surface morphology were carried out using an x-ray diffraction (XRD) and a scanning electron microscope (SEM). The film thickness was 4000 Å, which was measured by SEM.

III. Results and Discussion

Fig. 1 shows the zero-field-cooled (ZFC) and the field-cooled (FC) dc magnetization curves of four MgB_2 thin films fabricated at the annealing temperature of 800 - 950 °C. The magnetic field of 4 Oe was applied parallel to the c-axis of each thin film. The onset temperature T_c, of the thin film annealed at 900 °C was 39 K with the transition width ΔT_c = 2 K,

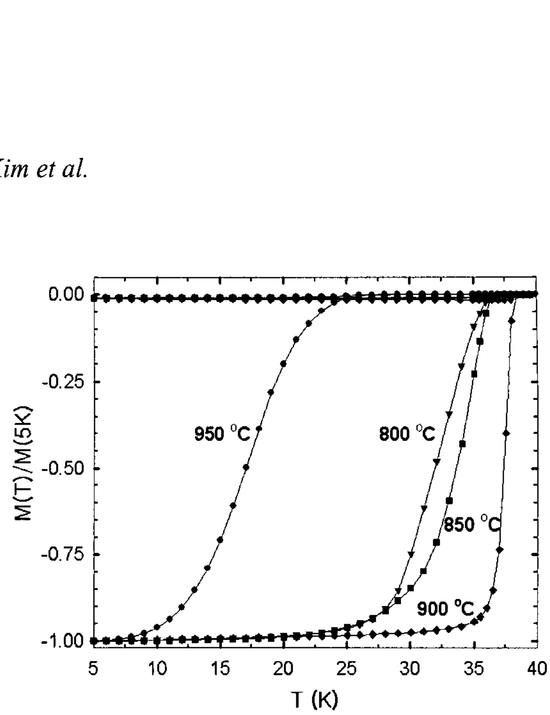

Fig. 1. The temperature dependence of magnetization of the MgB_2 thin films fabricated at different annealing temperatures.

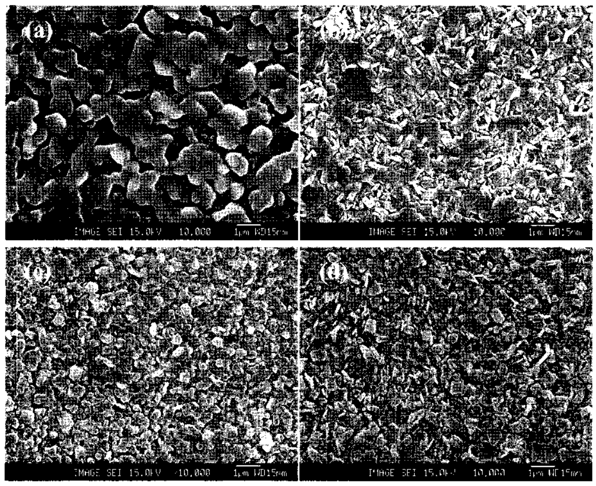

Fig. 2. SEM pictures of MgB_2 thin films annealed at (a) 950 °C, (b) 900 °C, (c) 850 °C, and (d) 800 °C.

which was determined from the 90%-to-10% drop-off of the normal-state magnetization in the ZFC curves. For the samples fabricated at higher temperature, we observed T_c of 28 K with ΔT_c = 10 K. This is in contrast with the report by C. Ferdeghini *et al* [12], in which the MgB_2 thin films annealed at over 900 °C showed semiconducting behavior with no hints of superconductivity. The samples annealed at lower temperatures of 800 - 850 °C had T_c = 37 K with ΔT_c = 7 K. From these results, we found that the optimum annealing temperature for fabrication of the MgB_2 thin films is about 900 °C.

To evaluate the structural properties of the samples, we carried out XRD measurements. The diffraction patterns show that the each film is highly oriented along the c-axis with some impurity phases.

The surface morphology of each thin film was characterized by SEM and are illustrated Fig. 2. We observed crystal-like grains on their surfaces with the dimensions of about several hundred nm in width and about 1 μm in length for the thin film annealed at 900 °C. The grain shapes on the surface of the samples treated at 900 °C appeared plate-like whereas those of the thin films annealed at lower temperatures of 800 - 850 °C appeared slightly tilted as shown in Figs. 2 (c) and (d). We observed that the grains were strongly linked without weak connections. These observations indicate that the critical current density J_c would be very high. [14]. However, the grains on the surface of the thin film annealed at 950 °C appeared a little melted as shown in Fig. 2(a). This seems due to very high annealing temperature so that its superconducting properties may be reduced.

The magnetic hysteresis curves of each film were obtained at 5 K in perpendicular fields up to 5 T. To estimate the critical current density J_c, we used the Bean's model $J_c = 30 \times \Delta M/r$, where ΔM is the width of the hysteresis loop and r is 1/2 of the sample width. The field dependence of J_c at 5 K for the whole samples are shown in Fig. 3. At zero field, the highest J_c value was ~ 1.2 × 10^7 A/cm^2 at 5 K for the film annealed at 900 °C. For the samples annealed at lower temperature than 850 °C, J_c was ~ 5 × 10^6 A/cm^2 at H = 0 Oe and T = 5 K. The slopes of the critical current density per field (dJ_c/dH) for these samples annealed at 800 °C ~ 900 °C show similar behaviors with the very high J_c of ~ 10^5 A/cm^2 at H = 4 T, suggesting that MgB_2 thin film is promising for practical applications.

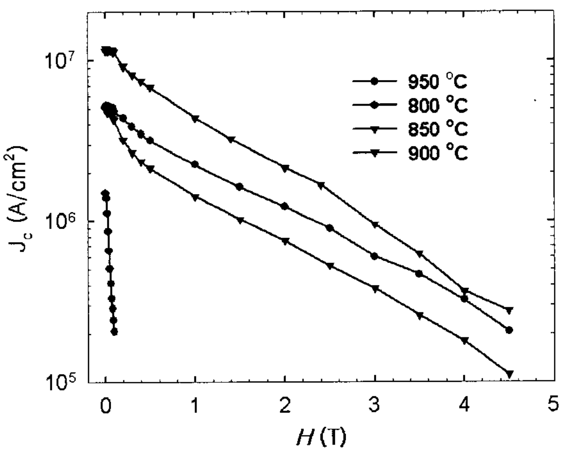

Fig. 3. The field dependence of critical current density at 5 K for the thin films annealed at 800 – 950 °C. The sample annealed at 900 °C shows the highest J_c ~ 10^7 A/cm^2

IV. Conclusion

We fabricated MgB_2 thin films at different post-annealing temperatures of 800 - 950 °C. In contrary with the report of other groups, the sample annealed at 950 °C still showed the superconducting behavior. Thin films annealed at lower temperature also showed strongly linked nature, suggesting that MgB_2 thin film is promising for practical applications. Especially, the MgB_2 films annealed at 900 °C showed the highest transition temperature of about 39 K with J_c ~ 10^7 A/cm^2 at 5 K.

Acknowledgments

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