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Fabrication of MgB_2 thin film by rf magnetron sputtering

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Abstract

Fabrication of superconducting MgB_2 thin film on $SrTiO_3$ and sapphire substrates by rf magnetron sputtering has been studied. We have tried both single target sputtering method using an Mg-excessive MgB_2 target and co-sputtering of Mg and B. Argon sputtering pressure was 20 mTorr and 5% of hydrogen gas was added to trap the remanant oxygen gas in the deposition chamber. Films made by co-sputtering at room temperature followed by in situ annealing at 600 °C showed transition temperatures of about 24 K. However, those by single target sputtering or in situ co-sputtering showed either no superconducting transition or low T_c.

Keywords: MgB_2 thin film; Superconducting; Sputtering

The discovery of superconductivity in MgB_2 [1] has both fundamental and practical importance. MgB_2 is known an isotropic BCS superconductor with the highest transition temperature of 39 K among intermetallic compounds. In a practical point of view, the relatively long coherence length and the high critical current density are favorable to the thin film electronic device applications.

MgB_2 thin films have been fabricated mostly by pulsed laser deposition [2–6], and also by e-beam evaporation [7] or molecular beam epitaxy [8,9]. Due to the extremely different vapor pressures of Mg and B, an in situ deposition process has not produced high quality superconducting films except the complicated MBE process. Most of the films were made either by diffusion of Mg in the B precursor film in capsules [2,7] or by in situ annealing [3–6]. However, these processes are not appropriate for a large area deposition which is necessary for the microwave device fabrication.

We have studied fabrication of MgB_2 films by rf magnetron sputtering which is advantageous for producing large area films. We have studied both an MgB_2 single target sputtering and Mg + B dual target co-sputtering. The films were either in situ deposited or in situ annealed after deposition. X-ray diffraction (XRD) spectra and resistive transition of the films were measured.

For the single target sputtering, we prepared an MgB_2 target by compacting the mixture of Mg and B powder with 5–30% Mg excess from the stoichiometric ratio and sintering it at 30 Mbar and 1040 °C in Ar environment for 4 h. XRD showed mostly MgB_2 signals with minor secondary peaks. We used a cylindrical vapor shield to reduce the escape of the Mg vapor during deposition. MgB_2 films were deposited in situ on $SrTiO_3$ (1 0 0) substrates. The deposition temperature was 450–650 °C, the Ar pressure was 10 mTorr, and the rf power was 200 W. To trap any remanant oxygen gas in the chamber, argon gas was balanced by 5% of hydrogen gas. After deposition, the samples were chamber cooled by turning off the substrate heater with maintaining the argon pressure. All the in situ samples showed metallic

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behavior in the resistance versus temperature (R–T) measurements. Although a few of them showed resistance anomalies at 30–40 K, they did not show a distinct superconducting transition. In XRD spectra MgB_2 signals were not identified. We also made films without the vapor shield. The film deposition rate was lower compared with the shielded case. In addition, the R–T curve was semiconducting or barely metallic at best. In the single target sputtering study, it is believed that the stoichiometric MgB_2 phase was difficult to achieve due to the extreme disparity of vapor pressure between Mg and B sputtering, and the relatively slow deposition rate.

Co-sputtering by using Mg and B targets was much more advantageous in terms of the film stoichiometry. Since the ratio of the molar densities of Mg (0.0716 mol/cm^3) and B (0.217 mol/cm^3) is 1:3, the deposition ratio (thickness/unit time) of Mg : B = 3 : 2 will guarantee the MgB_2 stoichiometry. However, to compensate the loss of Mg during the in situ annealing, the deposition rate of Mg was adjusted at higher values than the stoichiometric ratio. rf power used was 250 W for B target and 70 W for Mg target. The deposition rates at those powers were 9 nm/min for B and 20 nm/min for Mg. Films were made by co-deposition of Mg and B on Al_2O_3(1 1 0 2) substrates at 20 mTorr of Ar at room temperature for 30 min, followed by in situ annealing. After deposition the film was heated to 600 °C at the rate of 60 °C/min, annealed at 600 °C for 20 min, and finally cooled down by turning off the substrate heater. 20 mTorr argon pressure was maintained during the annealing process.

Figs. 1 and 2 are the XRD spectra and the R–T curve of the MgB_2 film made by co-deposition on an Al_2O_3(1 1 0 2) substrate with in situ annealing at 600 °C. XRD data show MgB_2 peaks along with Mg signals. The R–T curve shows a typical transition behavior of the MgB_2 superconductor with the zero-resistance transition temperature of 24 K.

In summary, we have studied fabrication of superconducting MgB_2 thin film by rf magnetron sputtering of a single MgB_2 target and co-sputtering of Mg and B targets. Best result was obtained by co-sputtering at room temperature followed by in situ annealing at 600 °C. XRD data confirmed the formation of the superconducting MgB_2 phase and the R–T showed a zero-resistance transition temperature of 24 K.

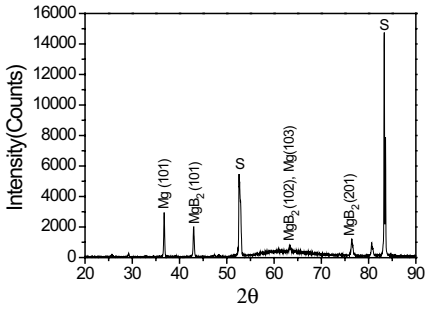

Fig. 1. XRD spectra of the MgB_2 film made on Al_2O_3(1 1 0 2) by co-deposition with the in situ annealing. The peaks denoted by 'S' are the substrate peaks.

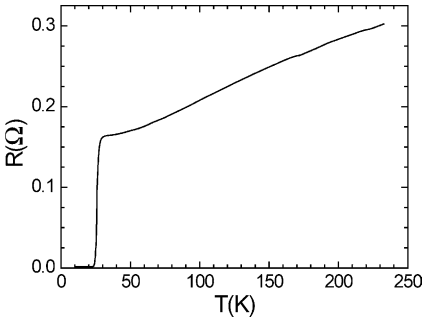

Fig. 2. R–T curve of the same sample shown in Fig. 1. The zero-resistance transition temperature is 24 K.

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