MgB_2 thin film fabrication by rf magnetron sputtering

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Abstract

Superconducting MgB_2 thin films on MgO substrate were fabricated by rf magnetron sputtering. Sputtering was preformed in a 50mTorr Ar atmosphere at room temperature using sintered MgB_2 or pure B and a Mg metal target. Sputtering deposition was followed by in-situ annealing at 650°C for 5 minutes in a high vacuum. The film deposited by a pure B and Mg metal had a superconducting transition temperature of 27K. On the other hand, sputtering using a sintered target brought on the oxidation of Mg and obstructed the formation of superconducting thin films.

Key words: MgB_2 thin film; rf magnetron sputtering; oxidation of Mg

1. Introduction

MgB_2 is one of the promising materials being developed for superconducting electronic device applications because of its simple crystal structure, long coherence length and high critical temperature. For this purpose, fabrication of high-quality thin films by using conventional technology is important. To date several approaches have been reported, such as molecular beam epitaxy [1], pulsed laser deposition (PLD) [2], and sputtering [3]. In those processes the oxidization of Mg metal is common obstructions to the formation of a high-quality thin film. To solve these problems a two-step growth technique [4] (the first step is the deposition of a Mg-B precursor film and the second is crystallization by high temperature annealing) is adopted as an effective means. In comparison with PLD or the system of sputtering by multi targets, the single target system has the advantage of easy operation and simple equipment requirements. In this paper, we report on a method for MgB_2 thin film fabrication using single target rf magnetron sputtering.

2. Experiment

MgB_2 thin films were deposited on a polished MgO (100) single crystal. The base pressure of the deposition chamber was 1×10^{-6} Torr. Two types of targets were used: One was MgB_2 pellets, sintered at 900°C under high vacuum, and the other was pure B and Mg metals. B metal was put on pure Mg plate in a 60 mm diameter circle. In the later case, a suitable arrangement of B pellets on the Mg plate was sought to control the composition of deposited thin films. The schematic for this arrangement is shown in fig. 1. The erosion area was 40mm in diameter. The pressure of Ar was 50mTorr during deposition. Before deposition, pre-sputtering for 30 minutes was done to remove the oxidized layer on the target surface. The applied sputtering power was 100 W. The film thickness was 300 nm for a 3-hour deposition. During deposition a substrate heater was not used. After each deposition of thin film, the Ar gas was put under vacuum and samples were subsequently heated to 650°C for 30 minutes. This annealing process is essential for the inducement of superconductivity in thin films. The composition of the thin film was examined by X-ray photoelectron spectroscopy (XPS).

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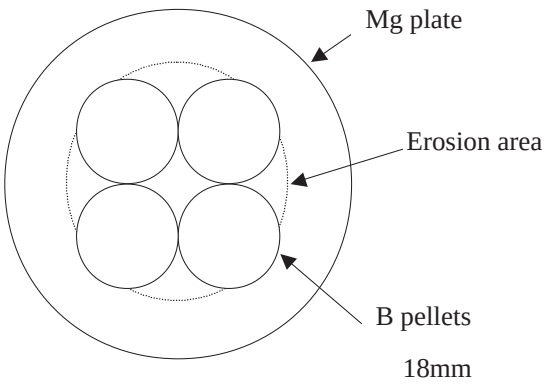

Fig. 1. Schematic of B pellet arrangement on the Mg plate

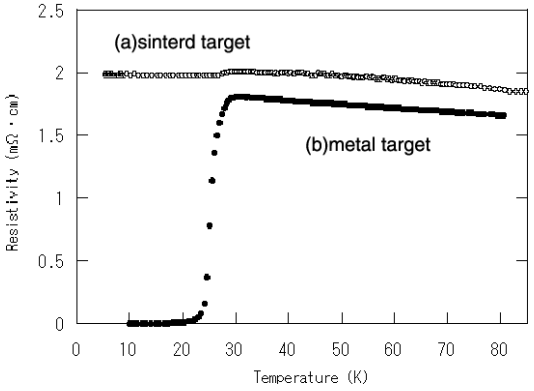

Fig. 2. Comparison of electrical transport of the thin films deposited by a (a) sintered MgB_2 target (scaled down to 1/100) and (b) pure Mg and B metal target.

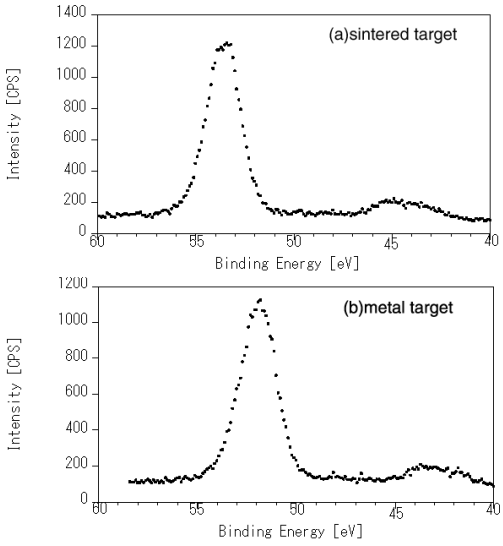

Fig. 3. Comparison of Mg_2p XPS spectra of the thin films deposited by a (a) sintered MgB_2 target and (b) pure Mg and B metal target.

3. Results and discussion

The resistivity vs. temperature (ρ-T) curves for the thin films deposited by different targets are shown in Fig.2. The film deposited by the sintered MgB_2 target shows semiconductor-like properties and a little superconducting transition. The value of resistivity was about 100 times greater than thin films deposited by a B metal target (fig. 2). In the XPS analyses of this sample, a clear peak shift of Mg_2p was observed as shown in fig.3. This peak position of Mg_2p is in agreement with the spectra of Mg in MgO. When the metals are oxidized their XPS peak positions shift over to a higher energy. This suggests that the Mg was oxidized during the deposition process and accounts for the absence of superconductivity in the film. With this material we were not able to fabricate superconducting films by varying substrate temperature, Ar gas pressure or annealing temperature. On the other hand, the film prepared using a pure B and Mg target shows a clear superconducting transition at 35 K and zero resistivity at 27 K. The XPS peak of Mg_2p is close to that of pure Mg metal. However the reason why oxide is formed in the film using the sintered MgB_2 target is that oxygen is contained in the target itself. In the case of the PLD process using a sintered MgB_2 target, it has been reported that low gas pressure during deposition is essential for obtaining superconducting samples [2]. On the other hand, compared to the PLD method, a relatively high Ar gas pressure is essential to maintain discharge during sputtering. Oxygen coming out from a sintered target is relatively difficult to diffuse and is easily incorporated into the deposited film. In the case of MgB_2 thin film fabrication by sputtering, the use of an oxygen-free target is essential for superconductivity. All of these films were formed by aggregate of minute crystals of MgB_2 and could not be detected by X-ray diffraction.

References

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