

MgB₂ superconducting thin films sequentially fabricated using DC magnetron sputtering and thermionic vacuum arc method

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Abstract

In this work, we discuss fabrication and characterization of MgB₂ thin films obtained by sequential deposition and annealing of sandwich like Mg/B/Mg thin films on glass substrates. Mg and B films were prepared using DC magnetron sputtering and thermionic vacuum arc techniques, respectively. The MgB₂ thin films showed superconducting critical transition at 33 K after annealing at 650 °C.

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1. Introduction

The MgB₂ superconductor is of great interest, since it shows the highest superconducting transition temperature T_c of 39 K [1], approximately double compared to the conventional metallic superconductors like Nb₃Sn with a T_c of 23 K. On the other hand, compared with other high temperature superconductors, MgB₂ has a simple hexagonal crystal structure, shows less anisotropy, exhibits a relatively long coherence length and a high current density. Therefore, MgB₂ thin film can be used in many important applications, e.g. in Josephson Junctions. Nevertheless, there are some difficulties in producing MgB₂ thin films. One problem is that Mg is easily oxidized due to the large vapor pressure difference between Mg and B [2].

Several deposition techniques such as magnetron sputtering [3], pulsed-laser deposition [4], melting process [5], two-step annealing [6] and sequential deposition techniques [7] were used to fabricate MgB₂ thin films on various substrates. Among these techniques, thermionic vacuum arc (TVA) has some advantages. During TVA deposition, a buffer gas is not necessary. Furthermore, high ion energies (up to 500 eV) create high adherence, low surface roughness, and compactness between deposited films and substrates. The energy of the ions can be controlled by electrode distance, angle and cathode temperature [8]. Any material, including ceramics, can be deposited on any type of substrate, e.g. plastics. TVA technique has been already successfully used for boron deposition [9].

In this work, we report fabrication and characterization of MgB₂ thin films obtained by sequentially deposition of Mg/B/Mg thin films on glass substrates using DC magnetron sputtering and TVA techniques.

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2. Sample preparation

Glass substrates (Corning Glass) were cleaned first in acetone and then with pure water in an ultrasonic cleaner for 10 min. Initially, a 10 nm thick layer of Cr was evaporated on the glass substrates to improve the adhesion of deposited films on the glass substrate. A Mg layer with a thickness of 500 nm was deposited as a first layer by using a thermal evaporator (LEYBOLD UNIVEX 300). The base pressure was set to 3×10^{-6} Torr with a filament current of 160 A. Then, as a second layer, boron was deposited on the Mg layer by the TVA method [9]. TVA is a relatively new type of vacuum deposition technique for surface coating. In this method, boron deposition was done between a tungsten cathode and a boron anode (with melting point of 2300 °C) under vacuum conditions of 5×10^{-6} Torr as shown in Fig. 1.

The boron anode was bombarded with accelerated electrons emitted from the cathode. Boron plasma occurred between the electrodes when applying an appropriate voltage and density of boron vapor. A plasma current of 200 mA has been used for boron deposition at an electrode distance of 4–5 mm, an applied voltage of 2500 V and a

filament current of 21 A. The thickness of the boron films was measured to approximately 1 μm with a thickness monitoring system (Cressington MTM –10) attached to the deposition system.

As the third layer, Mg was deposited on top of the B layer by using DC magnetron sputtering. For this purpose, the base pressure of the vacuum system was kept below 2.0×10^{-6} Torr using a turbo molecular pump. To create the plasma, Ar gas was used with a purity of 99.99%. The deposition pressure was set to 2.4 mTorr and the Ar gas flow was kept at 20 sccm by a MKS gas flow controller. For pre-sputtering, 30 W DC power and 72 mA current were applied for 30 min to remove any contamination on the target surface. Once the shutter was opened, the DC power and applied current were changed to 15 W and 56 mA, respectively. In one hour, a 500 nm Mg film was deposited. The thickness of the Mg films was measured with a thickness monitor and confirmed with scratching and section analysis techniques using an Atomic Force Microscope (Solver Pro from NTMDT).

As a result, a 1 μm thick boron film between two 500 nm thick Mg layers is obtained. The following annealing step provides a chemical reaction between Mg and B to get the superconducting MgB_2 film. The prepared films were annealed in two stages. In the first stage, the films were heated in a tube furnace to 400 °C with a 15 °C/min temperature increase and kept at this temperature for 30 min. In the second stage, the temperature was increased to 650 °C with the same rate, and the samples were kept at this temperature for 15 min. After annealing, the furnace was switched off and the films cooled down to room temperature in the Ar atmosphere. Finally, the MgB_2 films were characterized with XRD and low temperature resistivity was measured using an Oxford cryopump system.

3. Results and discussion

Fig. 2 shows the normalized XRD results of the MgB_2 thin films manufactured at Izmir Institute of Technology

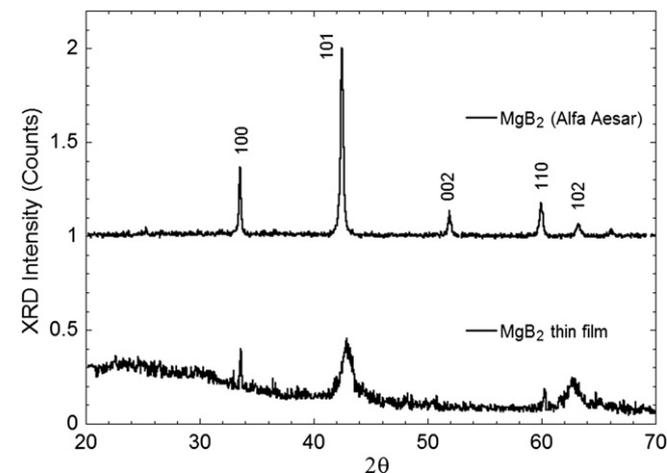


Fig. 2. Normalized XRD data for MgB_2 thin film and commercial MgB_2 powder from Alfa Aesar.

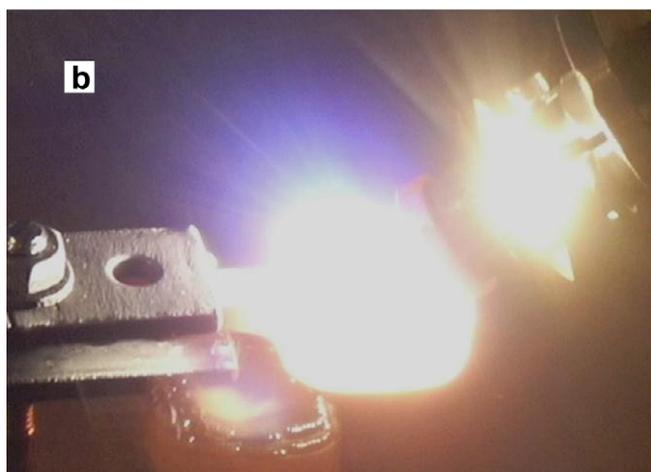
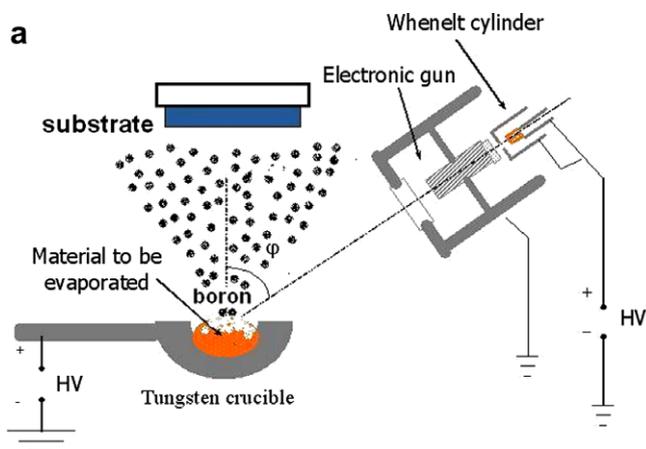


Fig. 1. (a) Schematic diagram of TVA system used for boron deposition. HV indicates the high voltage power supply and (b) boron plasma from deposition process.

and commercial MgB_2 powder from Alfa Aesar. The mirror mode method was used for the MgB_2 films. However, the observed diffraction peaks for the MgB_2 thin film were not as sharp as for the commercial MgB_2 powder. The broadened peaks around 43° (close to plane 101) and 62.5° (close to plane 102) indicate some MgO growth inside the film. This actually implies a poor crystallinity of MgB_2 thin films. Similar results have been reported in other studies [2,6].

Experimental results reported in the literature [10] showed that the effect of the annealing temperature is a critical parameter for the transition temperature. For instance, the MgB_2 films annealed at a temperature higher than 650°C or for a longer time longer than 30 min did not show superconducting transition within the present study. This might be due to the high oxidation tendency of Mg and the large vapor pressure difference between Mg and B.

A SEM surface topography of MgB_2 thin films is given in Fig. 3. EDX results taken from several points on this particular surface are given in Table 1. The oxygen detected by the EDX analysis may be an indication of MgO seen in the XRD plots. Additionally, a signal is coming from the Cr on the surface of the glass substrate which was deposited for a better adhesion. Although it is commonly agreed that for the preparation of high quality MgB_2 films epitaxial growth is the desired method, our sequential deposition results showed that superconducting MgB_2 films can be also obtained, even on amorphous glass substrates.

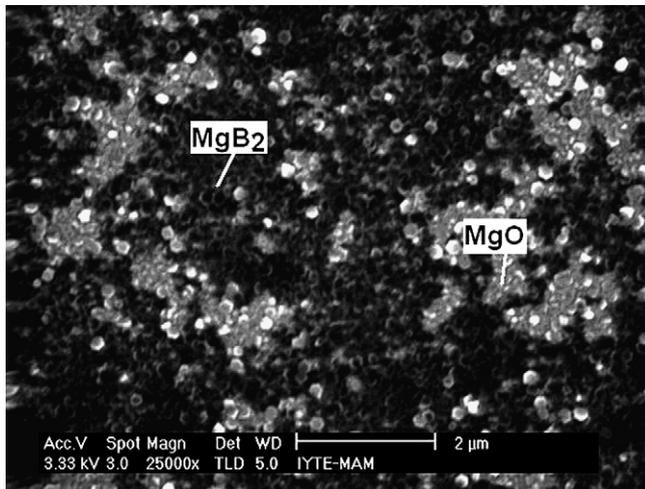


Fig. 3. SEM image of MgB_2 thin film surface grown on a glass substrate. Dark regions are MgB_2 and bright regions are MgO phase.

Table 1
EDX results of MgB_2 thin films

Element	Wt (%)
B	49.41
O	9.63
Mg	37.19
Cr	3.77

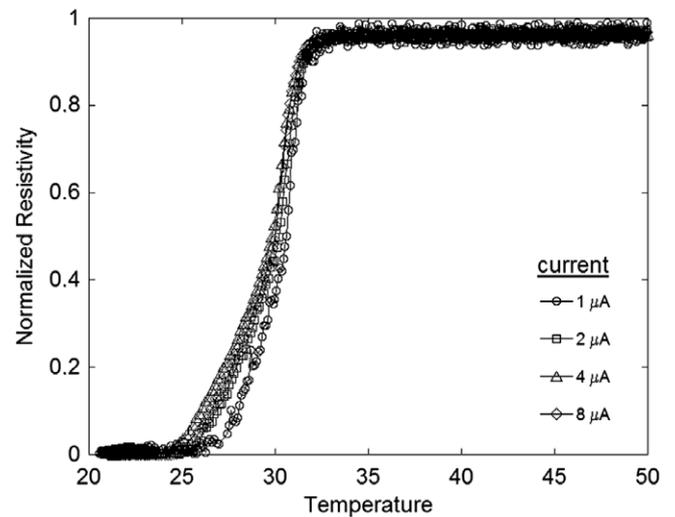


Fig. 4. $R-T$ plot for superconducting MgB_2 thin film on a glass substrate.

The four-point probe method was used for resistance versus temperature measurements. Currents from $1\ \mu\text{A}$ to $8\ \mu\text{A}$ were applied in steps to see the joule heating effect on the films due to metallic impurities on the superconducting transition temperature as shown in Fig. 4. The T_c onset value was obtained to be approximately 33 K with a transition width of 6 K.

4. Conclusions

In this work, MgB_2 superconducting thin films were prepared on glass substrates by sequential deposition of Mg and B using DC magnetron sputtering and thermionic vacuum arc techniques for the first time. We found that the effect of the annealing temperature is a critical parameter for the transition temperature. MgB_2 thin films fabricated using sequential deposition and two-step annealing showed zero resistivity at 27 K with a transition onset temperature of 33 K and a transition width of 6 K.

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