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Annealing Treatment of Bi (Pb)-Sr-Ca-Cu-O Thin Films on MgO by Pulsed Laser Deposition

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Abstract: Bi (Pb)-Sr-Ca-Cu-O thin films have been fabricated using Nd-YAG Pulsed Laser Ablation on MgO(100) substrate. The XRD patterns of the as-deposited BSCCO films show broad structures characteristic of a glassy or amorphous material. XRD spectrum exhibit epitaxial growth of BSCCO (2212) after a high temperature oxygen annealing treatment. The possible reasons for such behavior are an improvement of the films morphology, optimization of oxygen content and a change of the phase composition of the thin films. The X-ray diffraction plot shows that a film deposited for 1 h and annealed at 850°C is c-axis oriented and film annealed at 860°C consists of low T_c phase as a major phase. An increase of annealed temperature to 870°C and 880°C led to increasing the intensity of low T_c phase and the appearance of semiconducting phase. The superconducting transition temperature, T_c is highest for sample annealed at 870°C. XRD spectrum exhibit epitaxial growth of BSCCO (2212) after a high temperature oxygen annealing treatment. A high quality thin film can be obtained by not only a suitable target but also a good choice and appropriate post annealing treatment is needed.

Key words: Pulsed laser deposition, MgO, superconducting transition temperature (T_c), thin film

INTRODUCTION

High- T_c superconducting thin films have many potential applications (Bubendorfer *et al.*, 2004) such as the Superconducting Quantum Interference Device (SQUID), Josephson junction, microwave cavities and signal transmission. Since the discovery of superconductivity in the Bi-Sr-Ca-Cu-O and Y-Ba-Cu-O system), many efforts have been focused to fabricate a single-phased high T_c sample (Auge *et al.*, 1995; Giannini *et al.*, 2008; Pavuna *et al.*, 2002). Pulsed Laser Deposition (PLD) has been regarded as a versatile method to grow ceramic thin films. Laser evaporation offers the particular advantages that one may deposit films rapidly and without the need for sophisticated rate control and high vacuum equipment. Many researchers have been working on this technique because the application of high T_c superconductors in microelectronics depends on the availability of high quality superconducting thin films. It has been found that the nominal composition and thermal treatment parameters such as heating-up rate

(Huang *et al.*, 1990) annealing time and cooling rate play an important role in the formation of high T_c phases. In this study, the surface morphology, structure and electrical properties of the BSCCO thin films at different annealing temperature will be investigated. These films will be characterized by SEM, XRD and resistance measurement.

MATERIALS AND METHODS

This research project was conducted in 2004, in Superconductor and Thin Films Laboratory, Department of Physics, Faculty of Science and Environmental, University Putra Malaysia, 43400 Serdang, Selangor, Malaysia. The $\text{Bi}_{1-x}\text{Pb}_x\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ bulk samples were prepared by the conventional solid-state reaction technique. Thin films of Bi (Pb)-Sr-Ca-Cu-O were fabricated using Pulsed Laser Ablation technique. The substrate used in this work was single crystal MgO with the (100) orientation. Green laser with wavelength of 532 nm and 2 Watt had been used. The chamber was

kept in 1×10^{-4} mbar before deposition and oxygen was flow in during deposition process. The typical pressure during the deposition process is 2×10^{-3} mbar and the substrate was heated at 500°C . All the thin films of BiPbSrCaCuO, undergone for heat treatment in oxygen flow for 2 h under different temperature ($850, 860, 870$ and 880°C) with the heating rate of 3°C min^{-1} and cooling rate of 2°C min^{-1} to complete the oxidation. The samples were taken out only when the temperatures reached below 50°C . Morphological and structural analysis of thin films were performed by an SEM and X-ray diffractometer. The electrical properties were measured using a four point probe system.

RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns, measurement using a CuK_α source, for as-deposited and annealed films. X-ray diffraction showed a number of diffraction peaks. The XRD patterns of the as-deposited BSCCO films show broad structures characteristic of a glassy or amorphous material. During the annealing treatment a crystalline order is established. The possible reasons for such behavior are an improvement of the films morphology, optimization of oxygen content and a change of the phase composition of the thin films. Peaks with (*) are from the substrate and the structure at the base of the MgO reflection is due to the absorption edges of MgO (Bowman *et al.*, 1991). The x-ray diffraction plot of Fig. 1 shows that a film deposited for 1 h and annealed at 850°C is c-axis oriented and film annealed at 860°C

consists of low T_c phase as a major phase ($2\theta = 17.3^\circ, 23.2^\circ, 35.0^\circ, 47.3^\circ$ and 60.1°). An increase of annealed temperature to 870 and 880°C led to increasing the intensity of low T_c phase and the appearance of semiconducting phase. We can conclude that good quality of the film deposited for 1 h is at the 850°C annealing temperature.

Resistance versus temperature was measured using the four point-probe technique. In the normal state, the films show metallic behavior, with a small but noticeable resistivity drop between 105 and 110 K, suggestive of another superconducting phase with this transition temperature. This behavior is shown in some films in all figures with annealed films. The optimum annealing temperature was found to be 850°C . In most of the films obtained after post-treatments, there is a small drop in resistivity at 110 K, indicating the presence of some of the 2223 phase, although no 2223 peak was found on the X-ray diffraction patterns on some samples. A similar behavior were previously discussed by Rice *et al.* (1988), Viret *et al.* (1993), Mao *et al.* (1990) Fork *et al.* (1988) and Mao *et al.* (1990) proposed that this behavior indicates that there are 2212 and 2223 phases in the film, but the phase formation is not complete, or there is poorly superconducting material at grain boundaries. The superconducting transition temperature, T_c is 62 K for sample annealed at 870°C (Fig. 2).

The SEM photos of fractured surfaces for BSCCO bulk sample is shown in Fig. 3a-d. SEM studies have shown that the surface morphology of the sample comprises platelets of average size $\approx 10 \mu\text{m}$ with uniform and homogenous microstructure. The platelet-type

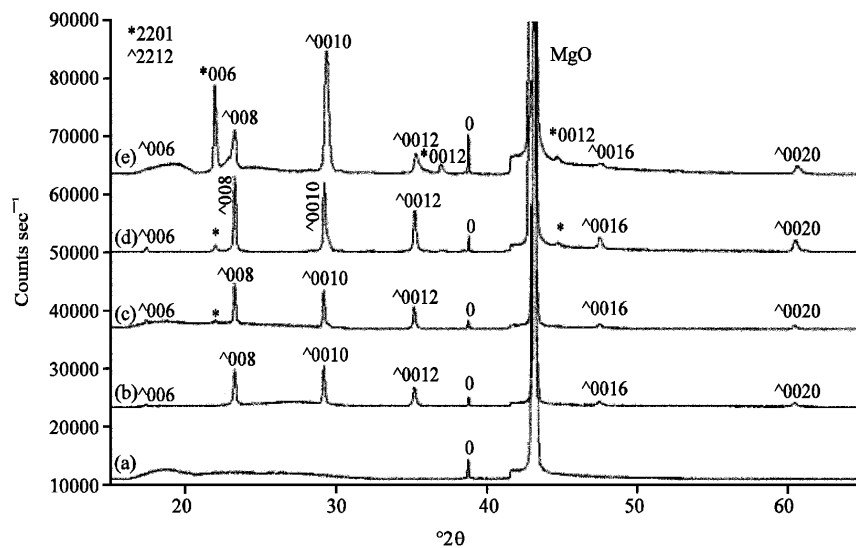


Fig. 1: X-ray diffraction patterns of Bi-Sr-Ca-Cu-O film on (100) MgO: (a) as deposited, (b) annealed at 850°C , (c) annealed at 860°C , (d) annealed at 870°C and (e) annealed at 880°C

features of the morphology are believed to be due to the 110 K phase (Sharma *et al.*, 1995). The typical morphology of the as deposited films is reported in Fig. 3b. In the micrograph of Fig. 3b, besides a continuous phase, there is a granular structure, which spherical particles up to

5 μm in diameter and larger grains are found. The annealed films were black and shiny. By variation of deposition parameters, films showing different kinds of particulates and surface morphology were fabricated. The most prominent types of particulates BSCCO films on MgO are

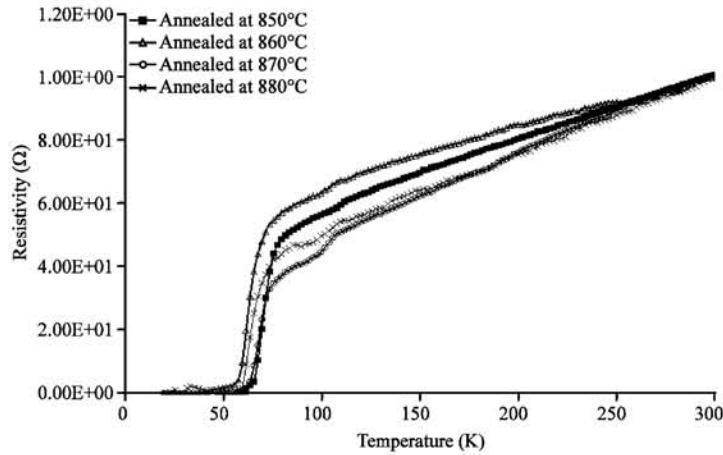


Fig. 2: R-T curve of a Bi (Pb)-Sr-Ca-Cu-O thin film deposited 1 h on (100) MgO annealed at 850, 860, 870 and 880°C in oxygen flow

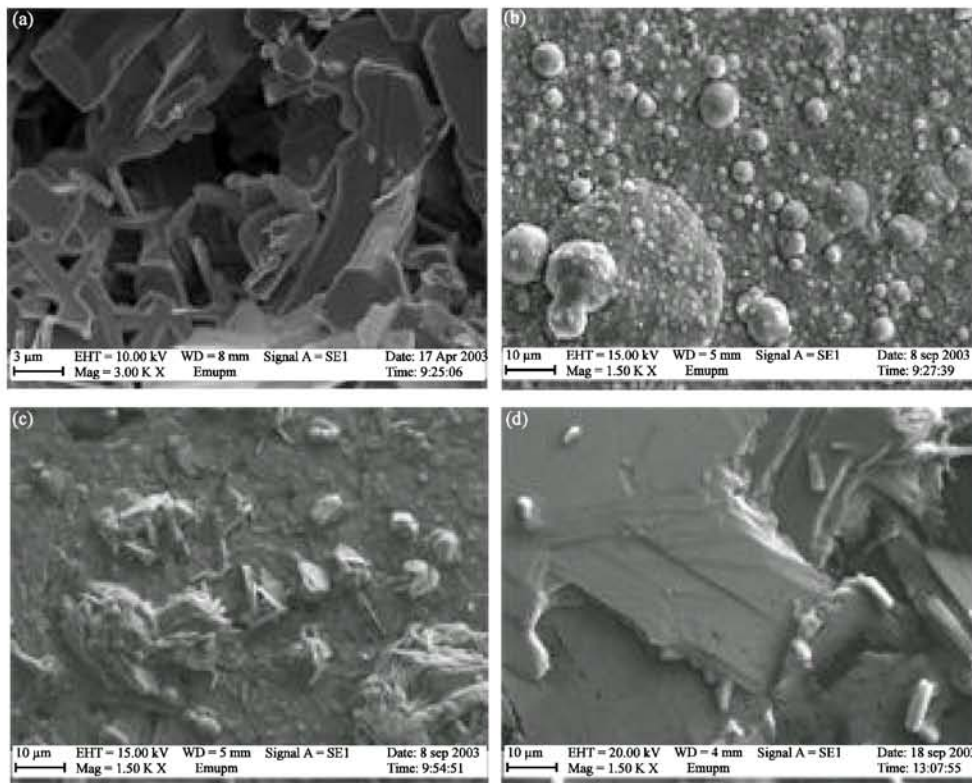


Fig. 3: SEM micrograph of Bi(Pb)SrCaCuO: (a) bulk sample prepared by solid state method, (b) as-deposited thin film, (c) thin film deposited for 1 h and annealed at 850°C and (d) thin film deposited for 1 h and annealed at 870°C

droplets with smooth surface, bigger droplets with granular surface, spherically-shaped features confined by randomly oriented facets, submicron rod-like features, Cu-enriched needles, platelets, irregularly-shaped Cu-rich outgrowths, strongly Cu-enriched tabular outgrowths, big target fragments (Proyer *et al.*, 1996), island growth structure, cubic and rectangular cubic structures. From those pictures, it is observed that annealing temperature have a pronounced influence on the particle size. The size of grains increases with annealing temperature.

CONCLUSION

The X-ray diffraction plot shows that a film deposited for 1 h and annealed at 850°C is c-axis oriented and film annealed at 860°C consists of low T_c phase as a major phase. An increase of annealed temperature to 870 and 880°C led to increasing the intensity of low T_c phase and the appearance of semiconducting phase. The highest superconducting transition temperature, T_c is given by sample annealed at 870°C. SEM micrograph shows that annealing temperature increases the size of grains. To obtain high quality thin films not only a suitable target but also a good choice and appropriate post annealing treatment is needed.

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