SUPERCONDUCTING YBCO AND BSCCO THIN FILMS PREPARED BY PULSED LASER DEPOSITION AND POST ANNEALING

A.N. Jannah[1] and S.A. Halim[2]

[1]*Faculty of Applied Sciences, Universiti Technologi MARA,*
72000, Kuala Pilah, Negeri Sembilan, MALAYSIA
[2]*Superconductor and Thin Films Laboratory, Department of Physics,*
Faculty of Science & Environmental, Universiiy Putra Malaysia,
43400 Serdang, Selangor, MALAYSIA

nurjannah@ns.uitm.edu.my

Abstract

The YBCO and BSCCO superconducting thin films have been grown on MgO (100) by pulsed laser deposition from a sintered $YBa_2Cu_3O_y$ and $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ target. Post annealing after deposition at 850^OC in 30 minutes resulted in BSCCO superconducting films with zero resistance at 52 K. YBCO film annealed for 2 hours is non-superconducting but it is improved by increasing the time to 4 hours and 5 hours at 920^OC . X-Ray diffraction pattern shows a predominance of 001 peaks for BSCCO film and no improvement of crystallinity after annealed for YBCO film.

Keywords: BSCCO, Pulsed Laser Deposition, superconducting thin films, YBCO.

1. Introduction

Since the discovery of high-temperature superconductivity there have been intense efforts to synthesize thin films suitable for device applications and basic physics study. The most successful techniques at present are high-pressure single-target sputtering and laser ablation (Chen et al, 1992, Aizaki et al, 1988). Films so made can have excellent electrical properties and a high degree of crystalline perfection. To date most ablation work has concentrated on the use of excimer lasers such as ArF, KrF, and XeCl. The Nd-YAG laser has several operational advantages compared to the excimers: it is a solid-state laser and is therefore easier and safer to handle, as it does not use highly hazardous gases (Auge, Hartmut and Heinrich, 1995). Typical applications areas included metrology and electronic instrumentation, radioastronomy and environmental spectroscopy, neurology and medical diagnostics, electronic warfare, nondestructive materials evaluation, telecommunications, ultrafast digital signal and data processing (Awang Kechik et al, 2009).

2. Previous Research

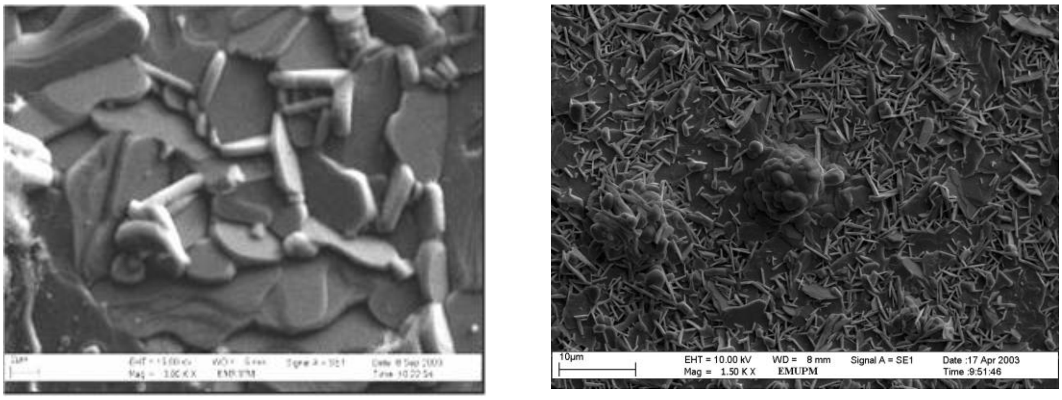
A high-temperature superconducting oxide, Bi-Sr-Ca-Cu-O (BSCCO), was synthesized (Maeda et al, 1988). It contains two prominent phases with $T_{c's}$ in the 110-120 and 70-90 K ranges. Thin films of the BiSrCaCuO family have been grown by single-target pulsed laser deposition by (Viret et al, 1993). The post annealing (*ex situ*) technique normally leads to very granular films. For $Bi_2Sr_2CaCu_2O_{8+\delta}$ (2212) films prepared in this way, the resistivity drops abruptly at temperatures in the range 80-85 K. Post-annealed $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (2223) films could only be obtained in a very narrow annealing temperature window. They all contained the secondary 2212 phase and therefore displayed a two-step resistivity drop at 110 K and 80 K. Superconductive $Bi_2Sr_2CuO_{6+x}$ (Bi-2201) thin films were prepared on MgO (100) substrates by laser ablation using a KrF excimer laser beam and the 4th harmonics of an Nd:YAG pulse laser beam (Uchiyama and Wang, 2000). A stoichiometric non substituted Bi-2201 target was used. In the case of using the Nd:YAG pulse laser, as-deposited Bi-2201 thin films showed high crystallinity, metallic normal-conductivity till a onset of the critical temperature and T_{c0} ~ 4 K. The effect of the growth rate on the $Bi_2Sr_2CaCu_2O_y$ (Bi2212) thin film quality on MgO substrate have been investigated by (Qi et al, 2001) at several growth rate from 0.175 to 3 nm/min. The maximal step height on the film surface is improved from about 100 to 6 nm by the reduction of growth rate to 0.5 nm/min and simultaneously the superconducting critical temperature attaining to a zero resistance T_c (R = 0), is also improved from 50 to 63 K.

3. Methods

The $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ and $YBa_2Cu_3O_y$ bulk samples were prepared by the conventional solid-state reaction technique. The powders were mixed in mortar and ground with pestle for 1 hour in order to homogenize the mixtures. The mixtures were calcined at in the tube furnace to drive off CO_2. To obtain a more homogenous mixture, second calcinations step were performed. After a slow cooling to room temperature, the samples were reground again and pressed into a pellet. The pellets were sintered again slowly cooled around 1^0C/min in the tube furnace with air circulating.

The BSCCO and YBCO thin films used in this study were grown on 10mm x 10mm x 10mm (100) MgO substrates by a pulsed laser deposition technique (Chen et al, 1992, Aizaki et al, 1988, Uchiyama and Wang, 2000). First, the target material is attached to the target holder properly using silver paste. The substrate, MgO single crystal (100) is placed at the substrate holder and it is put in the chamber and the motor is connected to the power supply located at the bottom of the chamber. The chamber is pumped to about 1-2 x 10^{-2} mbar. Then the turbo molecular pump is turned. When the chamber is pumped to the pressure of 1 x 10^{-4} mbar, the regulator is opened to let gas (oxygen) flow in. The needle valve is opened to allow oxygen gas to flow into the chamber. The pressure inside the chamber is maintained at 2 x 10^{-2} mbar. The temperature and pressure in the chamber is allowed to become stable before it is ready to deposit any film. During deposition process, the system always should be monitored to make sure everything is functioned properly. After film deposition is completed, HT power of the laser should be switched off. The water is allowed to flow for few minutes to cool down the laser system. The mains power of the laser, power supply for the pressure gauge, motor (target) and heater are switched off. All the thin films undergone for heat treatment in oxygen flow with the heating rate of 3^0C/min and cooling rate of 2^0C/min to complete the oxidation. The samples were taken out only when the temperatures reached below 50^0C. The samples are ready for characterization (SEM, XRD, resistivity, EDAX, and AFM).

4. Results and Discussions



(a) (b)

Figure 1: SEM micrograph of the a) YBCO thin film annealed for 2 hours at 920ºC
b) BSCCO film annealed at 850^{0}C.

The YBCO and BSCCO as-deposited films were very dense with very small grains. The oxidation of the film showed an improvement in the grain size. Figure 1 shows YBCO and BSCCO annealed films. The YBCO film morphology showed that the grains grow as a large interconnected platelets which lie mainly parallel to the sample surface. The upper layer of the BSCCO film consists of small Cu-enriched needles and long crystals. Platelets are observed at some parts of the film. These could be due to the presence of 2212 and 2223 phases (Sharma et al, 1995). In this work, the optimum annealing temperature for BSCCO films is 850^{O}C and it was annealed in oxygen flow for 30 minutes.

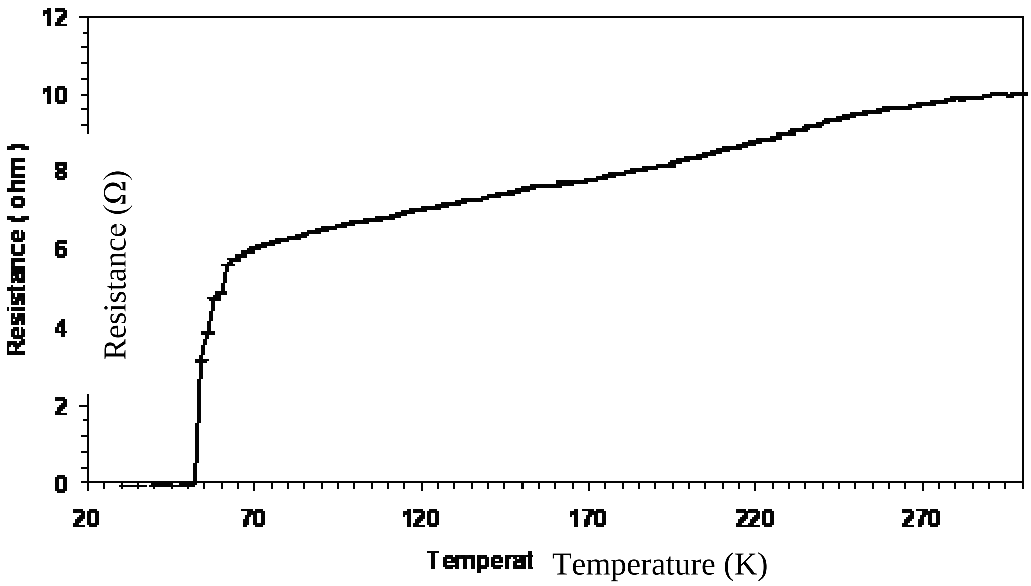


Figure 2: *R*-*T* curve of a BSCCO thin film annealed at 850^{0}C.

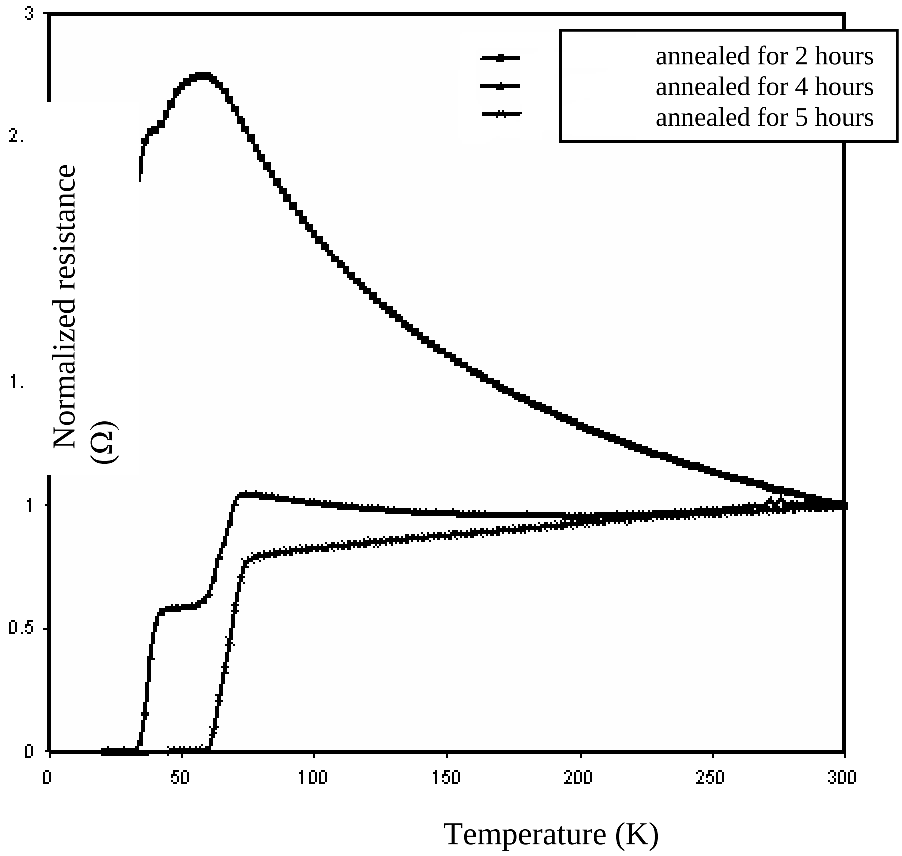


Figure 3: The variations in resistance with temperature of YBCO films.

A typical resistivity curve measured on the BSCCO film annealed at 850^0 C as shown in Figure 2, displays a tail that lead to zero resistivities at temperature around 52 K. The effect of annealing in oxygen on transport T_c is also shown in Figure 3 for YBCO films. From the figure, we can see a very strong T_c depression and the superconducting transitions are sensitively affected by annealing condition. At 920ºC, YBCO film annealed in oxygen flow for 2 hours, the normal state exhibits a semiconducting behavior that is improved by increasing the time to 4 hours and 5 hours. The film annealed in oxygen for 5 hours exhibit T_c values near 60 K. Therefore the optimum duration annealing is 5 hours

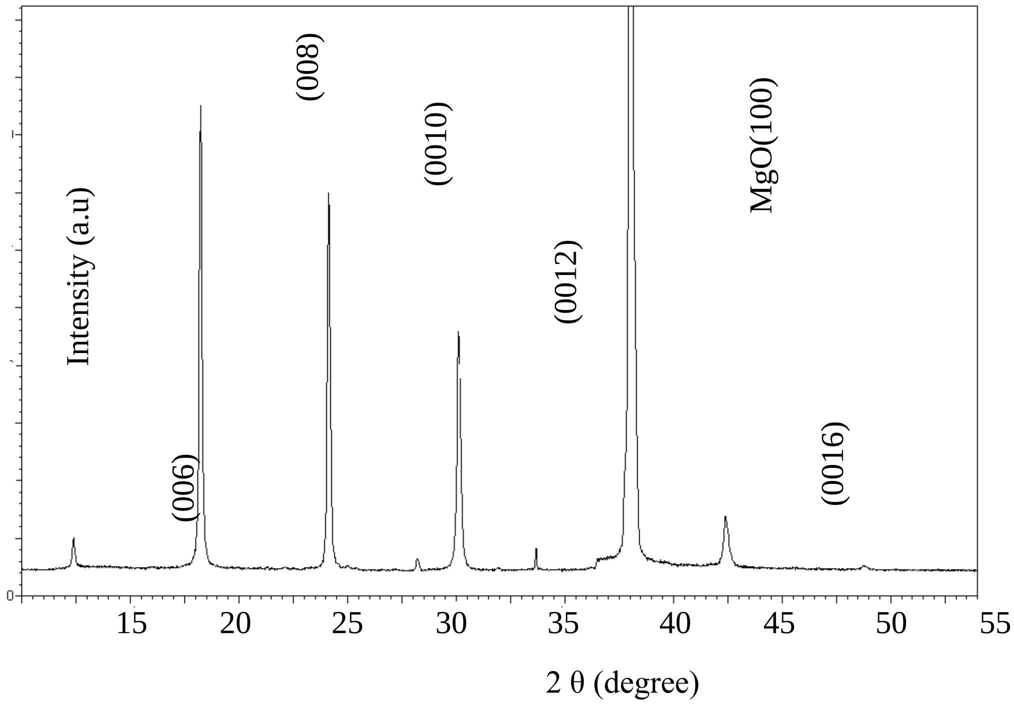
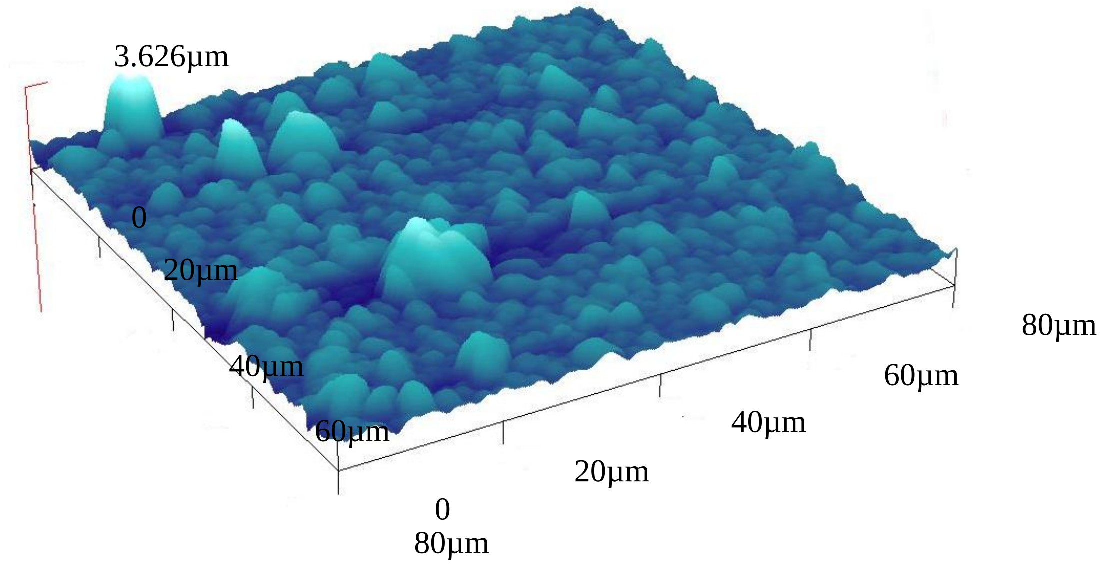
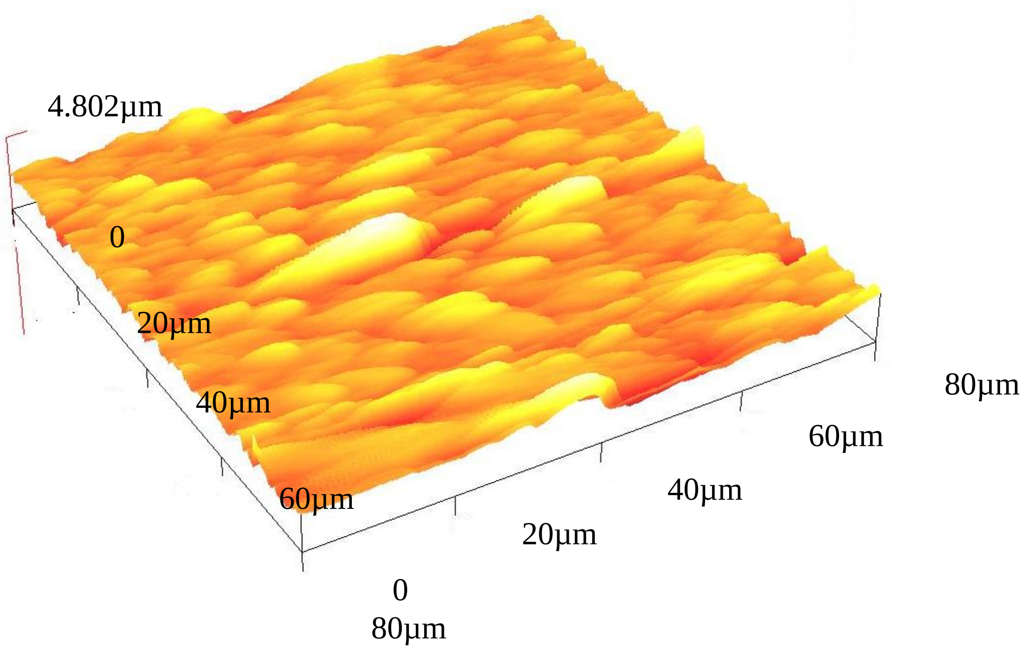


Figure 4. X-ray diffraction pattern of an annealed BSCCO film on (100) MgO.

Figure 4 shows X-ray diffraction patterns of Bi-Sr-Ca-Cu-O film on MgO annealed at 850^0C. It shows the coexistence of two superconducting phases, 2212 phase and 2223 phase. The diffraction patterns show a series of strong peaks at 2θ = 23.2^0, 29.1^0, 35.1^0, and 47.3^0. X-ray diffraction clearly shows a predominance of 001 peaks. There is also a small peaks belongs to 2223 phase. The XRD patterns of the as-deposited YBCO film show broad structures characteristic of a glassy or amorphous material with no improvement of crystallinity after annealed.



(a)



(b)

Figure 5: AFM image of the a) YBCO thin film annealed at 920ºC for 2 hours and b) BSCCO thin film annealed at 850ºC.

Table 1: The mean surface roughness, R_a, and RMS values of thin films.

Sample	Roughness Measurements	
	Mean Deviation (R_a)	RMS Deviation (Variance)
YBCO (annealed for 2 hours)	221.0 nm	329.3 nm
BSCCO (annealed at 850^0C)	263.5 nm	372.4 nm

The surface morphology of YBCO film annealed for 2 hours at 920ºC and BSCCO film annealed at 850ºC was carried out by using AFM to provide a clearer insight and their images are shown in Figure 5. The mean surface roughness, R_a, of the samples were measured with scanning areas of 80 x 80µm^2. Table 1 shows R_a values and the root mean square deviation value (RMS) from AFM images. For YBCO film, there are also some boulders found besides rounded grains.

5. Conclusions

BSCCO films require a much simpler and faster thermal cycle than YBCO compounds. Attempts will be made to obtain high T_c phases in order to enhance the T_c value and the stability by adjusting the doping of Pb in BSCCO target. It is known that the proportion of the high T_c phase closely depends on the heat and annealing treatment conditions so that more detailed optimization is needed to synthesize a high T_c single phase.

Acknowledgements

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