Journal of Physical Science, Vol. 29(Supp. 1), 25–31, 2018

Effects of Annealing Treatment and Elemental Composition on $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ Thin Films Grown by Pulsed Laser Deposition

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Published online: 25 February 2018

To cite this article: Azman, N. J., Shaari, A. H. & Abdullah, H. (2018). Effects of annealing treatment and elemental composition on $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ thin films grown by pulsed laser deposition. *J. Phys. Sci.*, 29(Supp. 1), 25–31, https://doi.org/10.21315/jps2018.29.s1.4

To link to this article: https://doi.org/10.21315/jps2018.29.s1.4

ABSTRACT: *In this work, $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ thin films have been fabricated by pulsed laser deposition (PLD) onto substrate of MgO single crystal. Bi(Pb)SrCaCuO thin films with different deposition times were annealed at different annealing temperature. Zero resistances of thin films were registered in the range of 30–68 K after annealing treatment and strongly dependent on the annealed temperature. From scanning electron microscope (SEM) pictures, it is observed that annealing temperature have a pronounced influence on the particle structure. Energy dispersive X-ray (EDX) analyses show the presence of elements Bi, Pb, Sr, Ca and Cu and suggest a strong loss of the Pb component in all annealed thin films. All measurements indicate that the films were mainly dominated by the 2212 phase and results showed that superconducting transitions are affected by the annealing treatment temperature and it can be an effective method to change the oxygen in the Bi(Pb)SrCaCuO system and vary its superconducting properties.*

Keywords: Bi(Pb)SrCaCuO superconductor, pulsed laser deposition, 2212 phase, annealing treatment, elemental composition

1. INTRODUCTION

Studies of superconducting Bi(Pb)SrCaCuO (BSCCO) in a form of thin film are stimulated by the possibilities of the thin film's utilisation of high-current devices such as Superconducting Quantum Interference Devices (SQUIDS) and Josephson junctions.[1–3] Several techniques for producing high quality thin films have been investigated for devices.[4,5] The parameters such as vacuum condition, target distance, mechanical setups, laser parameters, deposition temperature and substrate options can be controlled and the pulsed laser deposition (PLD) is an extremely versatile technique for preparing a wide range of thin films and multi-layered structures for any kind of materials by using appropriate lasers.[6–9] Its advantages include flexibility, fast response and congruent evaporation of energetic evaporates, and it has a cost advantage over many other vacuum techniques.[5] The precursor powder, the mechanical processing parameters and the application of post-annealing heat treatment with oxygen strategy are important factors that affects directly the superconducting properties of final thin films.[10–12] To achieve the best results in electrical properties, it is necessary to optimise several variables such as the starting powder composition and its phase distribution. In this work, we report the effect of annealing treatment and elemental composition on Bi(Pb)SrCaCuO thin films deposited on magnesium oxide, MgO (100) single crystal substrates by PLD.

2. EXPERIMENTAL

2.1 Materials and Preparation

The $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ compound samples were synthesised by using the solid-state reaction technique using.[11] The starting chemical powders of bismuth oxide (Bi_2O_3), strontium carbonate ($SrCO_3$), calcium carbonate ($CaCO_3$), plumbum oxide (PbO) and copper oxide (CuO) with high purity (99.9%) from Sigma-Aldrich were used in this work. The powders were mixed in mortar and ground with pestle for ~1 h in order to homogenise the mixtures. The mixtures were calcined at 800°C for 24 h in the tube furnace to drive off carbon dioxide (CO_2). After a slow cooling process in the tube furnace, the calcined powders were ground again in acetone with a pestle and mortar for ~1 h. To obtain a more homogenous mixture, second calcination step were performed at temperature of 830°C for 14 h. After a slow cooling to room temperature, the samples were reground again and pressed into 2.54 cm diameter pellets under a pressure of about 60 kN cm^{-2} . The pellets were final sintered (2°C min^{-1}) at 850°C for 150 h and again slowly cooled around 1°C min^{-1} in the tube furnace with open atmosphere.

2.2 PLD System

In this work, Handy YAG (neodymium-doped yttrium aluminium garnet; Nd:Y3Al5O12) Lasers (model: HYL 101 E) has been used. It is a high power class 4 solid state (ND: YAG) Q-switched pulsed laser and green laser (532 nm wavelength) has been used to ablate the films. The substrate must be unreactive in the oxygen-rich ambient and thermal expansion match. Magnesium oxide (MgO) single crystal had been chosen in this work and 50 cm focal length lens is used to focus the laser beam to the target. The distance between the substrate and the target is always maintained in 5 cm. Edwards turbo molecular pumps (model: EXT70/NW40) which support by a Edwards rotary pumps (model: E2M8) have been used as the vacuum system.

Table 1: Experimental parameters of samples fabrication for PLD system.

Laser power (ND-YAG laser)	2 Watt
Substrate	MgO single crystal (100)
Substrate temperature	500°C
Background chamber pressure	1×10^{-4} mbar
Oxygen atmosphere pressure during deposition	2×10^{-3} mbar

The properties of superconductivity are very sensitive to the heat treatment parameter and the superconductivity phases are formed in a narrow temperature range, just below the melting point. All the thin films of Bi(Pb)SrCaCuO, undergone for heat treatment using tube furnace in oxygen flow (99% purity) for 2 h under different temperature (850°C, 860°C, 870°C, 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 submitted to the four-probe standard test for the critical temperature and microstructure was evaluated by scanning electron microscopy, SEM (model Philips XL-30) and the element composition of the sample were analysed by using a Philips energy dispersive X-ray (EDX) analyser model PV99.

3. RESULTS AND DISCUSSION

Figure 1 shows the X-ray diffraction patterns, measurement using a CuKα (0.15418 nm) source, for as deposited and thin films annealed at 850°C, 860°C, 870°C and 880°C. "O" symbol refers to absorption edges in MgO. XRD results reveal that the samples were grown with Bi-2201, Bi-2212 and Bi-2223 phases.[1] An increase of annealed temperature to 870°C and 880°C led to increasing the intensity of low T_c phase (2201 phase). This can be possible with longer annealing process resulting in re-crystallisation. The annealed thin films have a high crystal homogeneity resulting from formation of good oriented grains aligned to the *c*-axis on MgO (001) surface.[9]

SEM analysis in Figure 2 showed the annealed thin films with a rough surface. By varying annealing temperature, films showing different kinds of particulates and surface morphology were fabricated. The most prominent types of particulates Bi(Pb)SrCaCuO annealed thin films on MgO are droplets, submicron rod-like features, needles, platelets, irregularly-shaped outgrowths and big target fragments. From SEM pictures, following the processing conditions, it is observed that annealing temperature have a pronounced influence on the particle structure.

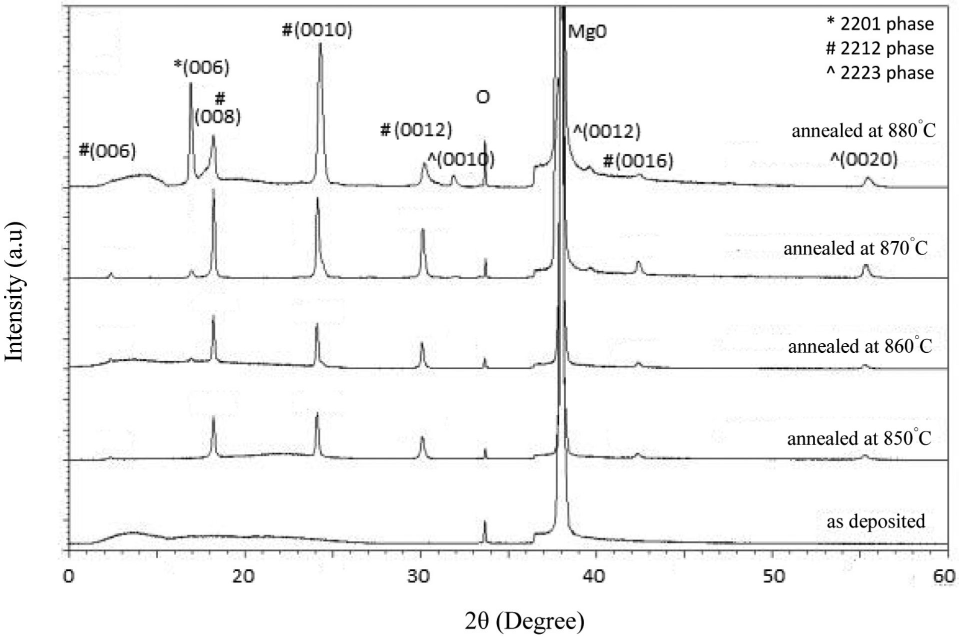

Figure 1: XRD patterns of as deposited and annealed thin films at 850C, 860°C, 870°C and 880°C.

Percentages of the element in the films annealed at 870°C are shown at EDX analyses in Table 2. The spectrum shows the presence of element of Bi, Pb, Sr, Ca, and Cu and suggest a strong loss of the Pb component in all annealed thin films. Lead (Pb) losses are apparently more dramatic for films since the exposed surface area-to-volume ratio is higher for a thin film.[8]

Resistance-Temperature (RT) curve was measured using the four point-probe technique and zero resistances were registered in the range of 34–68 K, strongly dependent on the annealed temperature and the best conditions leading to the highest critical temperature corresponding to the range ~870°C. Too low temperature of the annealing treatment gives rise to the growth of semiconducting or insulating layers in addition to incomplete crystallisation of the films, while higher temperatures (close to the melting point of the material) lead to insulating films, mainly owing to the loss of some element.

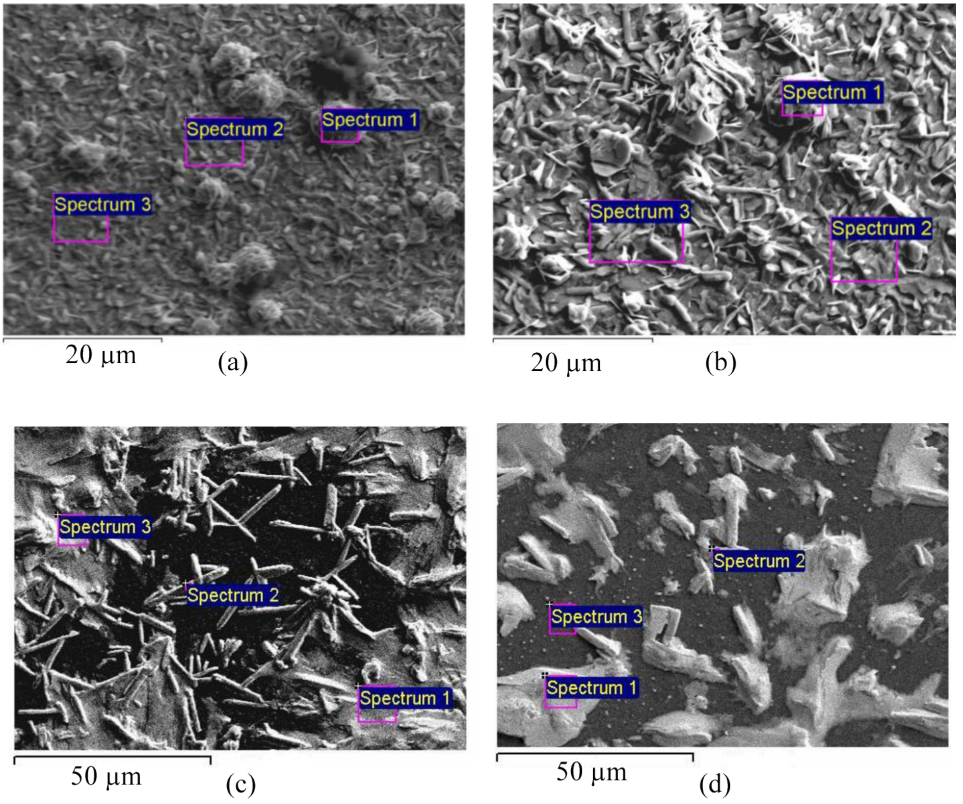

Figure 2: SEM images for $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ deposited 3 h and annealed at (a) 850°C, (b) 860°C, (c) 870°C and (d) 880°C.

Table 2: The percentage of the element in the thin film annealed at 870°C.

Materials	O	Ca	Cu	Sr	Pb	Bi
Spectrum 1	16.40	5.99	13.96	17.82	−3.87	30.99
Spectrum 2	30.23	9.50	44.74	13.93	−0.51	34.52
Spectrum 3	16.19	6.10	14.28	17.18	−3.98	36.83

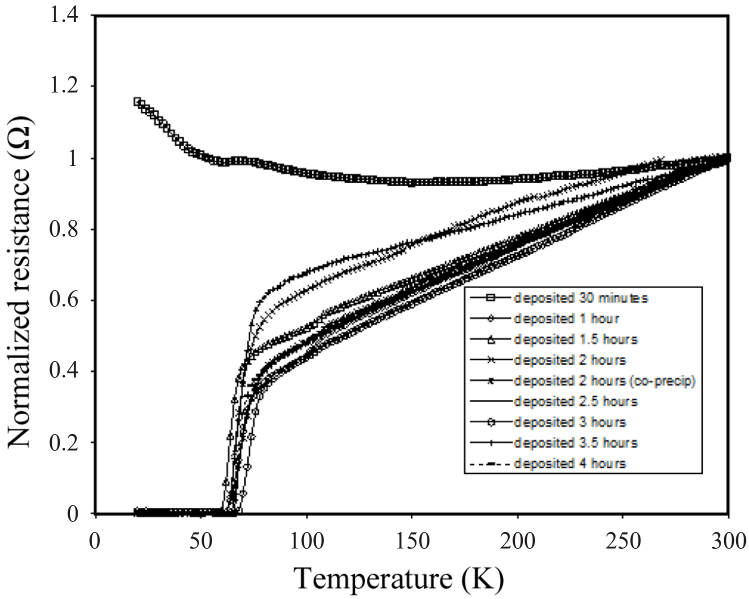

Figure 3: Temperature dependence of resistance for all samples annealed at 870°C.

4. CONCLUSION

Superconducting transitions are affected by the annealing treatment temperature and it can be an effective method to change the oxygen in the Bi(Pb)SrCaCuO system and vary its superconducting properties. Although the superconducting properties of the films described are not as good as those prepared using an excimer laser, they can be improved further by optimising various deposition parameters.

5. ACKNOWLEDGEMENTS

This work is supported by Universiti Teknologi MARA (UiTM), the Ministry of Science and Technology, Universiti Kebangsaan Malaysia (UKM) (grant UKM-OUP-NBT-26-117/2008), and staff of PLD Laboratory (Universiti Putra Malaysia, UPM).

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