

Preparation and Characterization of Y doped $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+d}$ Films Grown by IR PLD

Jeffrey C. de Vero*, Jaziel D. Vitug, Glaiza Rose S. Blanca,
Wilson O. Garcia, and Roland V. Sarmago

National Institute of Physics, University of the Philippines- Diliman

*jcdevero@up.edu.ph

Abstract

Yttrium doped BSCCO (Bi-22Y2) films on MgO (100) were fabricated by infrared Nd: YAG pulsed laser deposition (IR-PLD) with ex-situ post heat treatments. Chunks or blocks of Bi-22Y2 arrive on the substrate surface as clusters of spheroids. The size of the spheroids decreases with higher doping concentration. Highly c-axis oriented, smooth and homogeneous films were obtained after heat treatment. Partial substitution of yttrium on Bi-2212 exhibits the expected increase in the superconducting critical temperature due to reduction of doping level. This property of the film is due to fact that the material transfer using infrared (1064 nm) Nd: YAG pulsed laser is block-by-block and not atomized.

Key words: Pulsed laser deposition, BSCCO, oxide thin films

Introduction

Pulsed laser deposition (PLD) has been successful in the fabrication of ceramic thin films with complex stoichiometry such as high temperature superconductors [1-5]. Common pulsed laser deposition system uses an ultraviolet (UV) laser excites the target to provide deposition flux, substrate at high temperature and deposition in an oxidizing environment. Excimer and solid state lasers were used for this purpose. Also, Incorporation of oxygen and heat on the arrival site are implemented to resolve inhomogeneities in the composition of the deposited films [2, 5-10]. The resulting films are thin and smooth [1-5]. However, the high UV photon energy can result to undesirable effects such as photochemical reactions in the ablated area that alters the stoichiometry of the target and sputtering of the previously deposited material on the substrate by the energetic plasma plume [1, 2].

Films deposited at infrared (1064 nm) wavelength, green (532 nm) wavelength, and ultraviolet (355 nm) wavelength have been reported to have different morphologies [1,3-4,12]. In UV PLD of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$, the transfer of material is regarded as a result of photofragmentation in which the high photon energy of the laser pulse is strongly absorbed by a small volume of the target material [1, 2]. This allows the ablation plume to consist primarily of atomic, diatomic and other low mass species. In fact for Bi-Sr-Ca-Cu-O it is revealed as a unit-cell by unit-cell material transfer [1-2, 6-7].

The use of the infrared 1064 nm wavelength however, would simply heat the target and produce flux of ejected material due to thermal evaporation of the target species. Hence, splash of molten materials from the target is observed on the substrate area [1-3, 10, 11-12]. Infrared pulsed laser deposited films are characterized by a rough surfaces and thick films.

In this work, we prepared and characterized yttrium (Y) doped $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+d}$ films (Bi-22Y2). The deposition was carried out without substrate heating in 10⁻² mbar pressure similar to un-doped Bi-2212 [13]. The as-deposited films characterized before post deposition heat treatment to better understand the effect of using infrared 1064 nm pulsed laser for deposition. Also, series of post heat treatments were carried out to study the nucleation and growth in the case of film deposited by an infrared 1064 nm Nd:YAG pulsed laser.

Experimental

Bi-22Y2 thin films were deposited on (100) MgO substrate by infrared 1064 nm Nd:YAG pulsed laser at 10 Hz repetition rate and 8 ns pulsed duration. The laser was focused by a 500 mm lens onto a Bi-22Y2 sintered target at a 45° oblique incidence to provide energy fluence of 5.5 J/cm². The deposition is carried out in 10⁻² mbar chamber pressure at 30 mm substrate to target distance for 180 minutes.

The details of the deposition process can be found in following publications [13, 14]. Scanning electron microscopy (SEM), Energy dispersive X-ray analysis (EDX) and x-ray diffraction (XRD) was used to investigate the morphology composition and crystal properties of film. The superconducting property of the film was verified by four point probe resistance measurement.

Results and Discussion

Figure 1 (a) and (b) show the SEM images of the as-deposited Bi-22Y2 film. Clusters of random distributed spheroidal particulates are observed. These indicate that the material arrives in a molten or plastic state and solidifies on the substrate surface. Also, spheroidal particulates grow on top of each other. All as-deposited films show similar morphology at all doping levels. It can be observed that the particulates diffuse to one another and aggregate on the substrate surface. These aggregations of arriving particulates were also observed in the undoped Bi-2212 films prepared by IR-PLD [12]. The SEM images of Bi-22Y2 films heated at 930°C and annealed in 850°C for 5 hours in ambient air are shown in Fig. 1(c)-Fig. 1e. Relatively flat and smooth film surface morphology was obtained after heat treatment steps. Plate-like feature typical of BSCCO is also evident. Remnant spheroidal particulates inherent to pulsed laser deposition are observed on film surface even after heat treatment. The grain boundaries become more apparent with increasing yttrium content [14]. The average thicknesses of the films are about 2 μm.

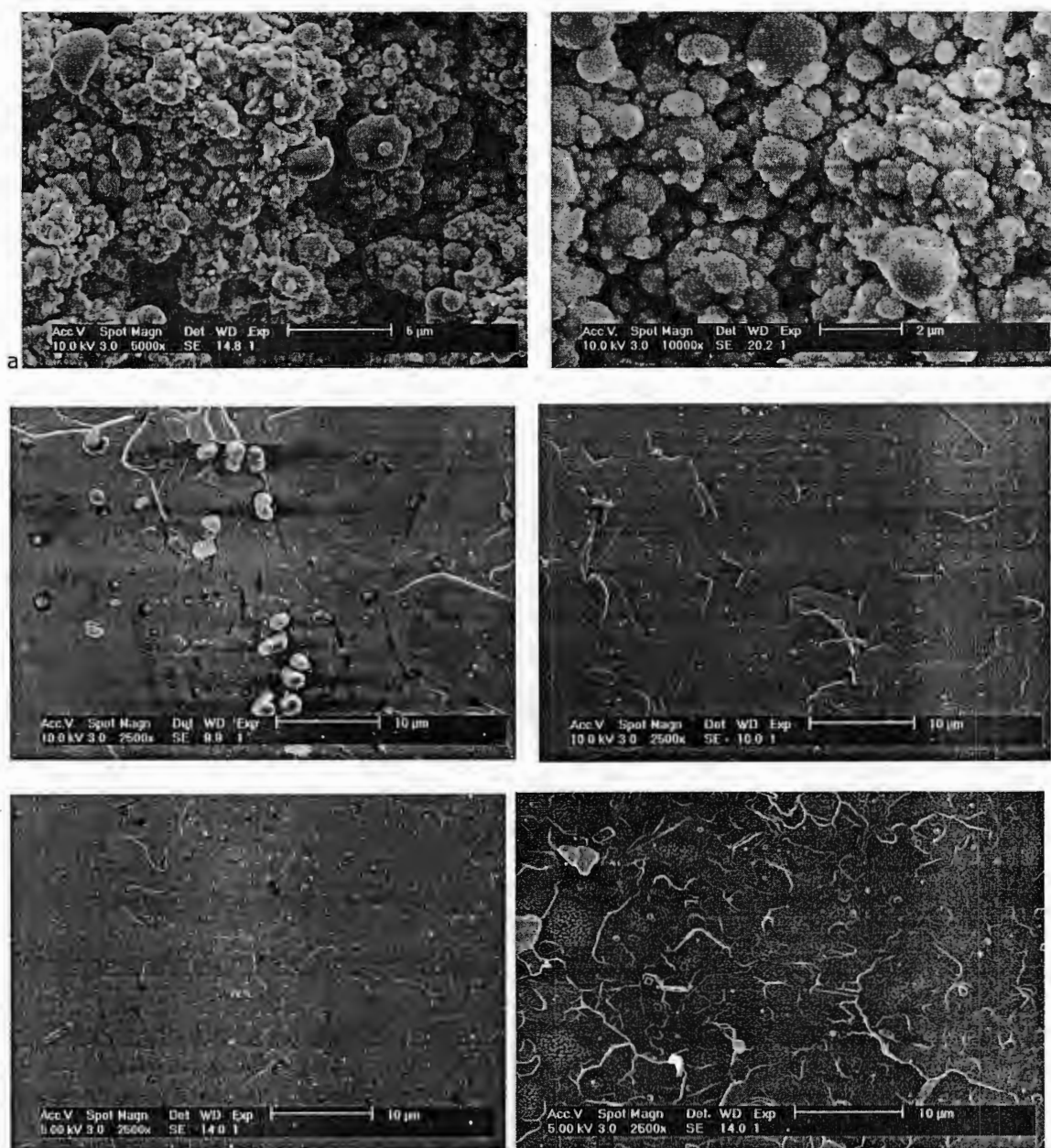
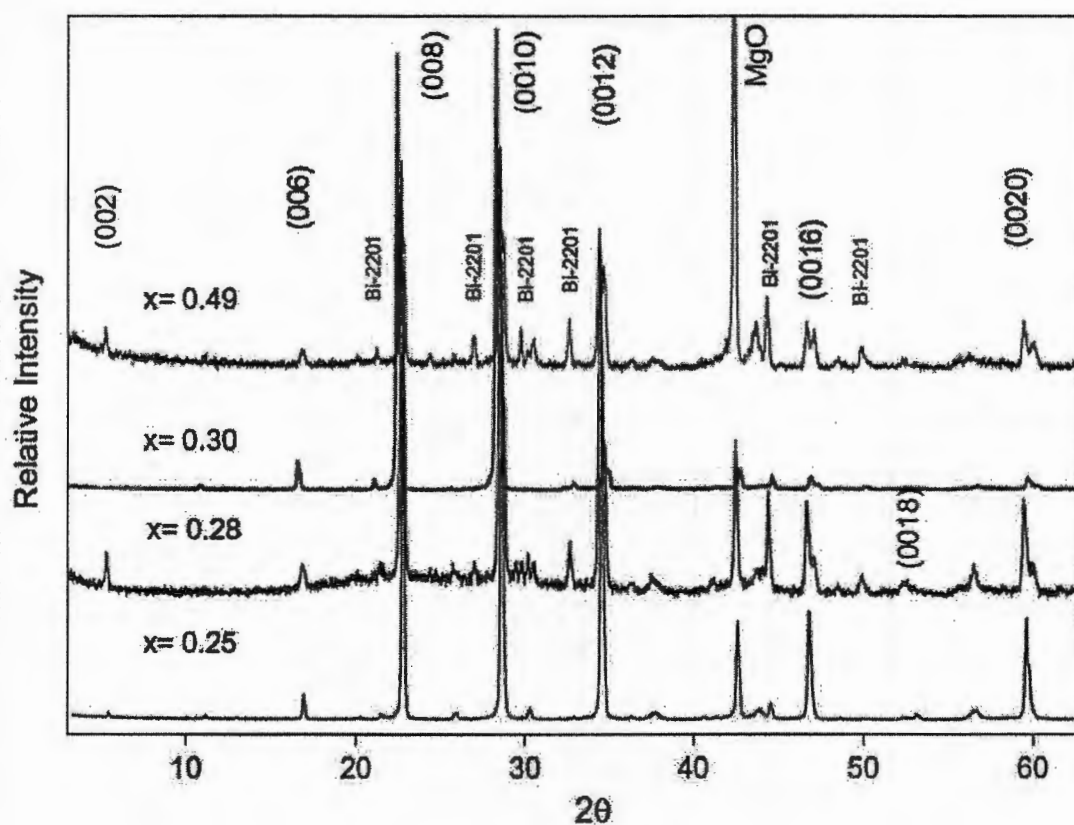


Figure 1: SEM images of the Bi-22Y2 films grown with energy fluence of 5.5 J/cm² at 10⁻² mbar chamber pressure for 180 minutes. (a) At 5000X magnification with 5μm scale, (b) at 10000X magnification with 2μm scale. Post deposition heat treated Bi-22Y2 films at (a) 25%, (b) 28%, (c) 30% and (49%) yttrium doping concentration. Images taken from [14].

The appearance of spheroidal grains and crystallize upon annealed films is a strong proof that the material arrives on the film surface as a stoichiometric block that are indication that heat treatment steps are necessary for the material to reconfigure and re-orient on the substrate [13,14] Figure 2 shows the XRD spectra of Bi-22Y2 films grown by IR PLD. The XRD measurements confirmed that the films are highly c-

axis oriented without minimal Bi-2201 impurity [14]. The initial Immobile and clustered stoichiometric molten spheroids of the as-deposited films grow and nucleate on the substrate upon heat treatment. And heating outside the chamber also ensures that the already deposited material on the substrate is unaffected by the incoming deposition flux. Hence, smooth films of Bi-22Y2 that reproduce the chemical structure and composition of the starting material were deposited by this method [14].



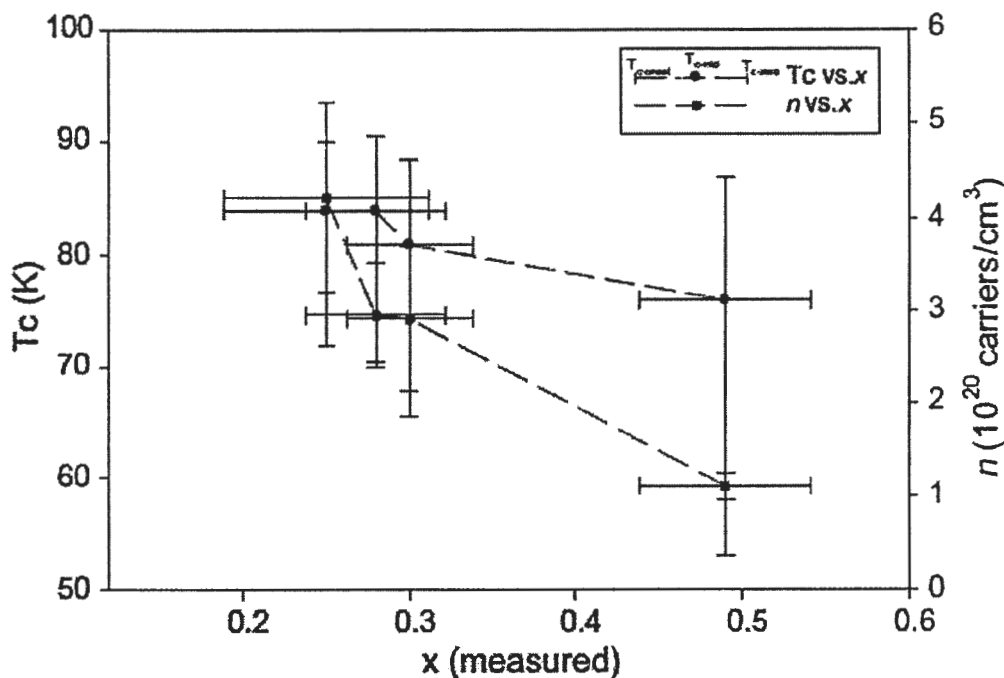


Fig. 3 XRD and transport measurement performed on pattern of yttrium doped Bi-2212 film grown by IR-PLD. After heat treatments highly c-axis oriented films and superconducting films were obtained. This shows the viability of using IR Nd:YAG laser to grow superconducting films. Images taken from [14].

Figure 2b shows transport measurement performed on Bi-22Y2 films. The superconducting transition temperature T_c increases from 85 K to 90.5 K but drop with additional Y until 87 K. The highest critical current density obtained is 723.14 A/cm² at 70 K for 28% Y-doped sample under zero magnetic field with carrier concentration of 4.2×10^{20} carriers/cm³ [14]. These results prove the ability of IR PLD to produced high quality films of complicated stoichiometry.

Conclusion

Stoichiometric molten blocks Bi-22Y2 were observed to arrive on films surface with infrared 1064 nm Nd:YAG pulsed laser as excitation source. Reconfiguration and re-orientation of the transferred material on the substrate by application of heat treatment outside the chamber results into flat, smooth and homogeneous stoichiometric film. Hence, the final film morphology is a result of combined effects of infrared pulsed laser deposition and heat treatments. The infrared 1064 nm Nd:YAG pulsed laser deposition also offers itself as a deposition technique capable of forming stoichiometric films of any material overcoming the disadvantage of high photon ionization of UV PLD. Oxygen control during heat treatments outside the vacuum chamber can also be investigated to improve the electrical property of the film.

Acknowledgement

The authors acknowledge the funding support from the Office of the Chancellor in collaboration with the Office of the Vice Chancellor for Research and Development, of the University of the Philippines Diliman, and the National Research Council of the Philippines (1-89 NRCP Project), as well as the Philippine Commission on Higher Education for the scholarship.

Notes

¹R. Eason. (2007). *"Pulsed Laser Deposition of Thin Films: Applications- Led Growth of Functional Materials"* (Hoboken, New Jersey: John Wiley & Sons, Inc.).

²D.B. Chrisey, et al., (2004). *Pulsed Laser Deposition of Thin Films*. (New Jersey: John Wiley & Sons, Inc., 1994.

³ M. Branescu, et al., (2008) "Scanning electron microscopy and resistive transition of in-situ grown YBCO films by pulsed laser deposition," *Journal of Physics: Conference Series* 94, 012007.

⁴ M. Branescu, et al. (2007). "AFM and complementary XRD measurements of in situ grown YBCO films obtained pulsed laser deposition," *Applied Surface Science*, 253. 8179- 8183.

⁵ M. Yavuz, et al. (2003). "Preparation and Characterization of BSCCO 2212 Thin Films," *IEEE Transaction on Applied Superconductivity*, 13, (2).

⁶ C. Marechal, et al. (1998). "Growth mechanisms of laser deposited BiSrCaCuO films on MgO substrates," *Physica C* 294, 23-32.

⁷ N. Asano, et al. (1999). "Block-by- block deposition of BSCCO thin films by PLD method," *Superconductivity Science Technology*. 12: 203- 209.

⁸ N T Mua, et al. (2008). "High critical current density in Ag- doped Bi-2212 thin films," *Superconductivity Sci. Technology*. 21: 105002 (5pp).

⁹ C.R. Guarnieri, et al. (1988). "Thin film Bi-Sr-Ca-Cu-O high temperature superconductors using pulsed laser evaporation from sintered disk," *Appl. Phys. Lett.* 53 (6).

¹⁰ S. Kaneko, et al. (2002), " Bi2Sr2Ca1Cu2Ox film prepared using 266 nm YAG laser," *Physica C* 378-381, 1270- 1273.

¹¹ L. Torrisi and D. Margarone, "Investigations on pulsed laser ablation of Sn at 1064 nm wavelength," *Plasma Sources Sci, Technology*, 15: 635-641.

¹² C. Andeouli, et al. (2004). "Influence of irradiated target modification on the quality of pulsed laser deposited YBa2Cu3O7-x thin films", *Journal of the European Ceramic Society*, 24 3623-3634.

¹³ J.C. de Vero, et al. (2010). "Growth evolution of Bi2Sr2CaCu2O8+δ thin films deposited by infrared (1064 nm) pulsed laser deposition," *Physica C* 470, 149-154.

¹⁴ J.C. de Vero, et al. (2011). "Stoichiometric transfer of material in the infrared pulsed laser deposition of yttrium doped Bi-2212 films," *Physica C* 471, 378-383.

References

- Andeouli, C. *et al.* (2004) "Influence of irradiated target modification on the quality of pulsed laser deposited YBa₂Cu₃O_{7-x} thin films." *Journal of the European Ceramic Society* 24, 3623-3634.
- Asano, N. *et al.* (1999) "Block-by- block deposition of BSCCO thin films by PLD method." *Superconductivity Science Technology*. 12, 203- 209.
- Branescu, M. *et al.* (2008). "Scanning electron microscopy and resistive transition of in-situ grown YBCO films by pulsed laser deposition", *Journal of Physics: Conference Series* 94, 012007.
- _____. (2007). "AFM and complementary XRD measurements of in situ grown YBCO films obtained pulsed laser deposition", *Applied Surface Science*, 253, 8179- 8183.
- Chrissey, D.B. (1994) *et al.* "Pulsed Laser Deposition of Thin Films", John Wiley & Sons, Inc.
- De Vero, J.C. *et al.* (2010). "Growth evolution of Bi₂Sr₂CaCu₂O_{8+δ} thin films deposited by infrared (1064 nm) pulsed laser deposition." *Physica C* 470, 149-154.
- _____. ". *Physica C* 471 (2011) 378-383.
- Eason, R. (2007). "*Pulsed Laser Deposition of Thin Films: Applications- Led Growth of Functional Materials*." Hoboken, New Jersey: John Wiley & Sons, Inc.
- Guarnieri, CV.R. *et al.* (1988). "Thin film Bi-Sr-Ca-Cu-O high temperature superconductors using pulsed laser evaporation from sintered disk." *Appl. Phys. Lett.* 53 (6).
- Kaneko, S. *et al.* (2002). " Bi₂Sr₂Ca₁Cu₂O_x film prepared using 266 nm YAG laser." *Physica C* 378- 381 1270- 1273.
- Marechal, *et al.* (1998). "Growth mechanisms of laser deposited BiSrCaCuO films on MgO substrates." *Physica C* 294, 23-32.
- Mua, N.T. *et al.* (2008). "High critical current density in Ag- doped Bi-2212 thin films." *Superconductivity Sci. Technology*. 21, 105002 (5pp).
- Torrisi, L. and D. Margarone, "Investigations on pulsed laser ablation of Sn at 1064 nm wavelength." *Plasma Sources Sci, Technology*, 15 : 635-641.
- Yavuz, M. *et al.* (2003). "Preparation and Characterization of BSCCO 2212 Thin Films." *IEEE Transaction on Applied Superconductivity*. 13 (2).