

## Effect of annealing on superconducting properties of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$ thin films by pulsed laser deposition

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### Abstract

Superconducting thin films of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  system were prepared by depositing the film onto silicon (111) substrate by pulsed laser deposition. Annealing treatment and superconducting properties were investigated by XRD and four probe resistivity measurement. The analysis reveals the evolution of the minor phase of the films 2212 phase to 2223 phase, when the film was annealed at 820 °C. Also the films have superconducting behavior with transition temperature  $\geq 90\text{K}$ .

### Key words

PLD,  
HTS thin films.  
BiSrCaCuO

### Article info

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## تأثير التلدين على الخواص الفائقة التوصيل للاغشية الرقيقة $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$ المرسبة بالليزر النبضي

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### الخلاصة

حضرت الاغشية الرقيقة للنظام  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  على ارضية سليكون (111) بطريقة الترسيب بالليزر النبضي . تم استقصاء المعاملة الحرارية والخواص الفائقة التوصيل باستخدام حيود الاشعة السينية وقياسات المقاومة. بينت تحليلات حيود الاشعة السينية حدوث ارتفاع للطور الى الطور العالي عند  $820^\circ\text{C}$ . كما لوحظ ان الاغشية لها سلوك فائق التوصيل وبدرجة حرارة انتقالية  $90\text{K} \leq$

### Introduction

High  $-T_c$  superconducting thin films have many potential applications<sup>[1]</sup>, such as the

superconducting quantum interference device (SQUID), Josephson junctions microwave cavities and signal

transmission. Since the discovery of superconductivity in the Bi-Sr-Ca-Cu-O, many efforts have been focused to fabricate a single-phase high  $T_c$  sample<sup>[2-4]</sup>.

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<sup>[5]</sup> annealing time and cooling rate play an important role in the formation of high  $T_c$  phase.

Jannah et al<sup>[6]</sup> studied the properties of BSCCO thin film deposited by pulse laser deposition. The XRD patterns and  $T_c$  measurement with zero resistivity temperature at about 60 K indicate that the film were mainly grown in 2212 phase, with 2223 phase which is detected in small structures on the film surface.

Thin films of Bi-Sr-Ca-Cu-O deposited on (100) cubic zirconia by PLD from a bulk superconducting target of nominal composition  $\text{BiSrCaCu}_2\text{O}_x$  had investigated by Kim et al<sup>[7]</sup>. They found that the film quality is affected by the substrate temperature and the annealing process. Their results indicate that the films are oriented with the  $c$ -axis perpendicular to the film plane.

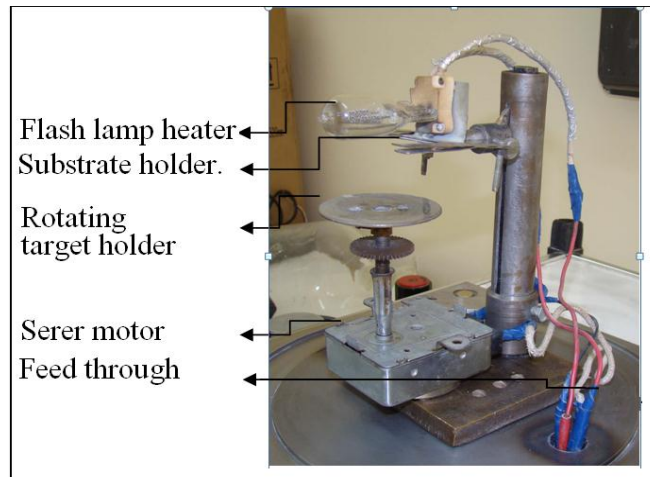
In this search, the structure and electrical properties of the BSCCO thin films at different annealing temperature will be

investigated. These films characterized by XRD and resistivity measurement.

## Experiments

The target used throughout this experiment was prepared from  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  nominal powder by solid state reaction with preparation conditions, calcinations at  $810^\circ\text{C}$  for 24 h, pressed as a pellet at 0.5GPa., diameter of the pellet 13mm and thickness (2-3)mm, the sintering stage at  $860^\circ\text{C}$  for 140h. This pellet was used as a target to deposit a thin film on Si (111) substrate using pulsed laser deposition.

Fig (1) shows the general experimental setup that used in present work.  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  target was mounted in vacuum chamber  $10^{-4}$  m bar, and ablated by a double frequency with Q-switched Nd:Yag pulsed laser operated at 532 nm, pulse duration of about 7 nsec and a (0.4 -8)  $\text{J}/\text{cm}^2$  energy density was focused on the target to generate plasma plume. The distance between target and substrate is 5cm. All samples were grown at an optimal Si (111) substrate temperature of  $300^\circ\text{C}$  with an oxygen back ground pressure of  $2 \times 10^{-3}$  mbar; the number of pulses is 150. The sample was thermally treated in the furnace with a heating rate of  $15^\circ\text{C}/\text{min}$ , the films heated typical to the range of  $820^\circ\text{C}$  and  $880^\circ\text{C}$  in an atmosphere of oxygen with flow rate 2 lit/min. Optical interferometric method was used to measure the thickness of the film, its value is 184.2nm. The  $\rho$ -T measurements for films have performed by four-probe resistance measurement and the crystalline structure was examined by X-ray diffraction using a  $\text{CuK}_\alpha$  source.



**Fig. (1) Target and substrate holder**



**Fig. (2) PLD system**

### Results and Discussion

The post- annealing was applied to improve the superconducting properties because the temperature region of the post- annealing for obtaining pure 2223 phase overlaps with that of 2212 phase. From transition temperature for annealed films at 820 and 860 are 102 and 90 K respectively and the behavior of the samples annealed at 840 and 880 °C are

superconductor as shown in Fig(3). The reason of enhancing  $T_c$  value at 820 °C is due to the formation of higher phase. The post- annealing  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  thin films show that transition from normal state resistance to superconducting is not sharp this may be attributed to the existence of small amounts of the secondary phase and/or fluctuation of the oxygen content.

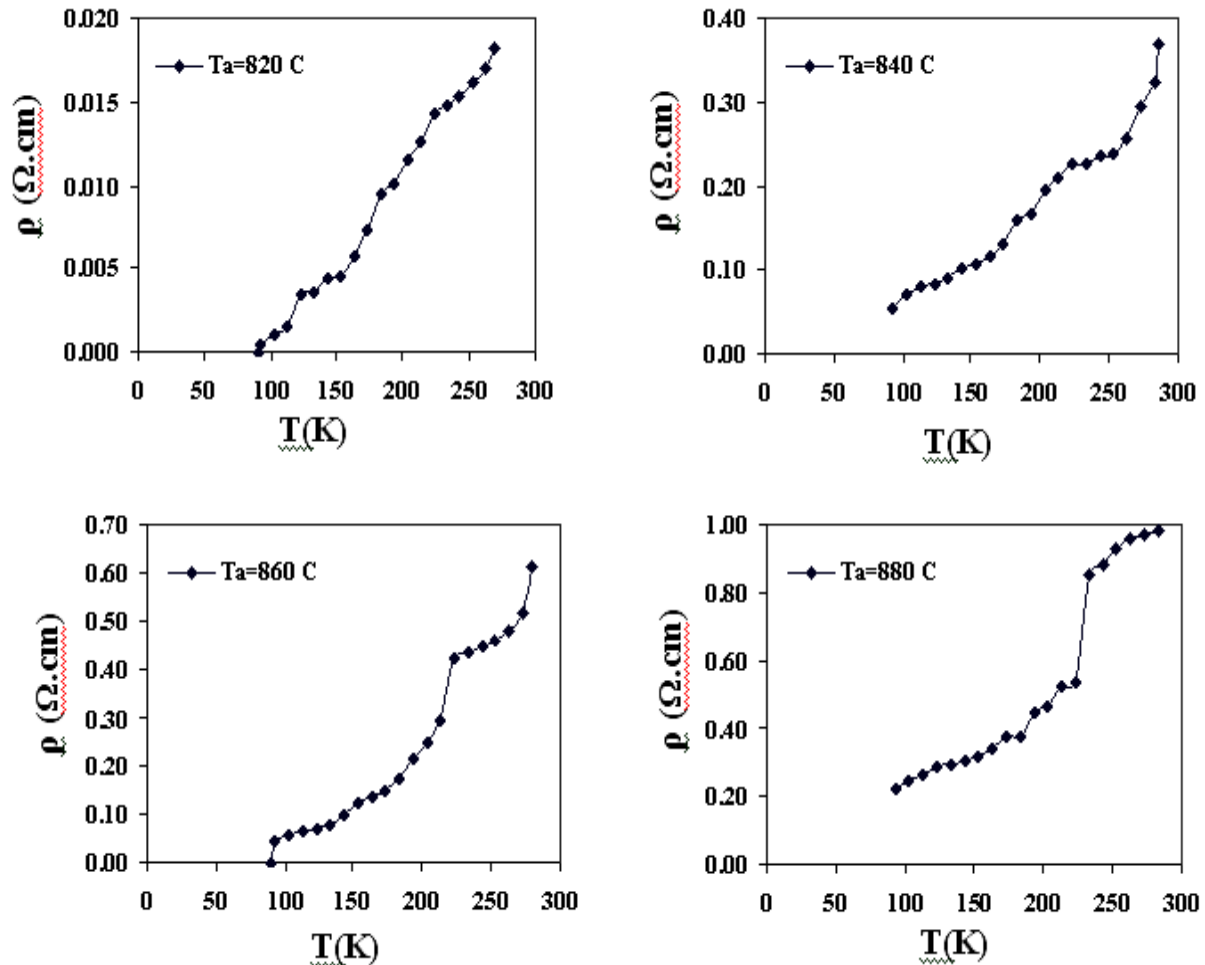


Fig (3)  $\rho$ - $T$  measurements of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  thin film annealed at  $820^\circ\text{C}$ ,  $840^\circ\text{C}$ , and  $860^\circ\text{C}$  and  $880^\circ\text{C}$

The XRD patterns of as-deposited give a broad structures characteristics of amorphous material. During the annealing treatment a crystalline order is established and reveals mixture of phases when heat treated at  $T_a$  from  $820$  to  $880^\circ\text{C}$  as shown in Fig(4).

The prefer orientation of the grown in c-axis, that is observed from the largest value of  $L$  in Miller index, there are increase in the high phase intensity of the peaks at (115), (0012), (0014), (0016), (2014), (0020), (317) and (1023), and the major composition of the films are

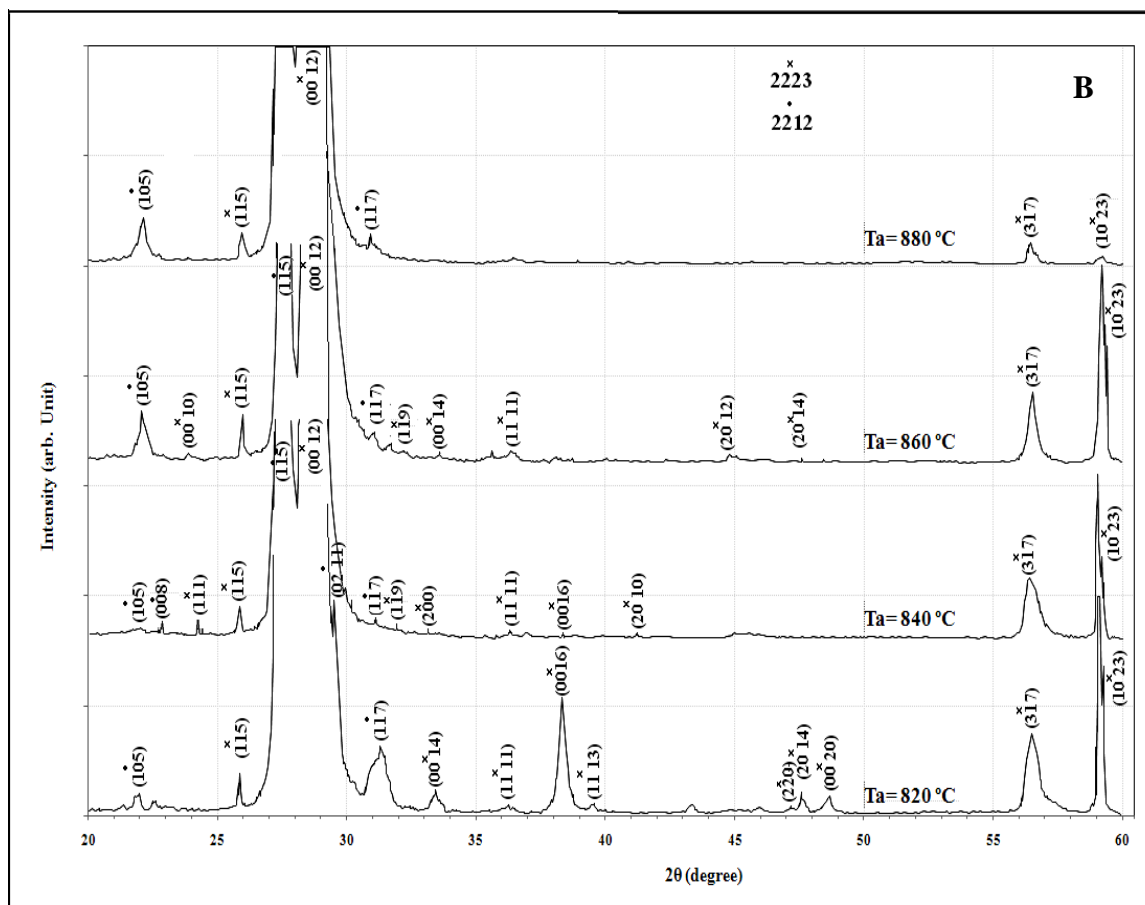
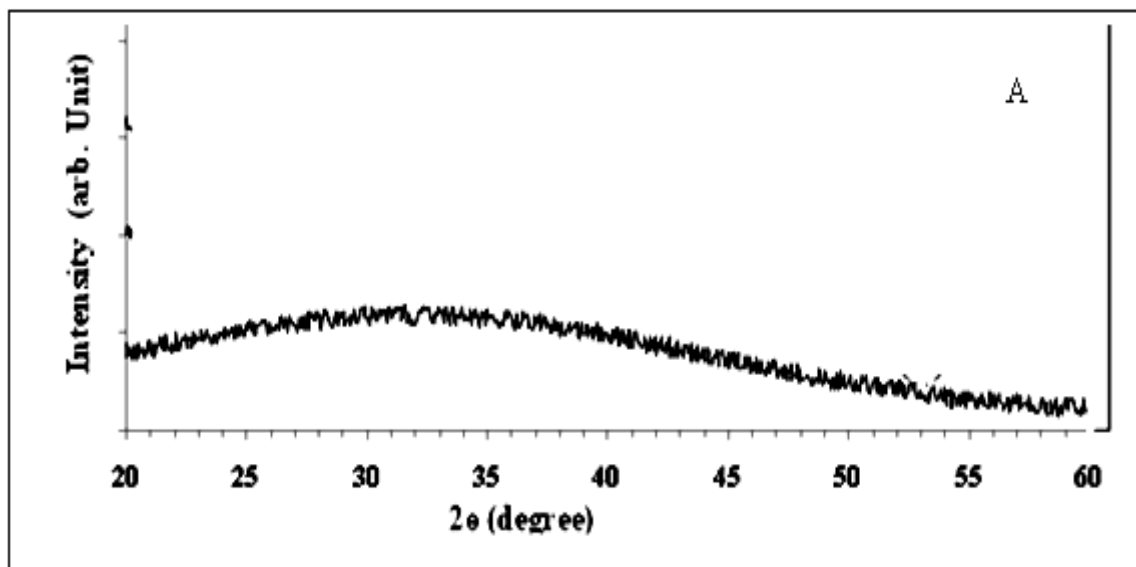
evaluated from 2212 phase to 2223 phase.

Miller indices in the patterns are after Primo *et al.*<sup>[8]</sup> and Koyama *et al.*<sup>[9]</sup>. Films annealed at  $840^\circ\text{C}$  show decreases in the intensity in some peaks at (115), (0016), (317) and (1023) and disappearing some high peaks such as (0014), (220), (2014), (0020) and decreasing in  $T_c$  value. The same behavior was found for films annealed at  $860$  and  $880^\circ\text{C}$ .

This could be interpreter as the heat treatments change the oxygen content of the films, which causes of the broadening

of some peaks with the increasing of annealing temperature.

This in connection with the angular shift of the peaks will introduce the local strains.



Fig(4) The XRD pattern of  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.2}\text{Zn}_{0.8}\text{O}_{10}$  thin film (A) as deposited (B)annealed at (a) 820 ° C (b) 840 ° C (c) 860 ° C and (d) 880° C

On the other side the lattice mismatches between the substrate and the film, relativity increases at temperature greater than 860°C, this will reduce the grain growth of 2223 phase as indicated by Ishii and Hatano<sup>(10)</sup>.

The lattice constants of thin film samples were calculated using d,h,k and l values of the strong peaks in the XRD patterns according to

$$\frac{1}{d^2} = \left( \frac{h^2 + k^2}{a^2} \right) + \frac{l^2}{c^2}$$

As calculated, the lattice parameters a and c are 4.8 Å and 37.1 Å respectively.

### Conclusions

The physical properties of T<sub>c</sub> are related to the phase's evolution and formation of intergrowth. Besides, grow nearly pure 2223 superconducting films of T<sub>c</sub> ≥ 90 K can be made at annealing temperature T<sub>a</sub> equal to 820 °C and 860 °C. Enhancement of T<sub>a</sub> above 860 °C leads to the increases of low phase and disappear of high phase.

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