Influence the Number of Laser Pulses on the properties of Bi$_{1.7}$ Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_{2.9}$Ti$_{0.1}$O$_{10+\delta}$. Superconducting Thin Films

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INTRODUCTION

The first technological installation for laser deposition and epitaxy was made in 1980. A few research groups achieved remarkable results on manufacturing thin-film structures using this technique. In 1987, PLD was successfully used to grow high-temperature superconducting films. Chang et al.,1988, prepared BiSrCaCuO, YBaCuO, and TiBaCaCuO films on (100) MgO or (100) ZrO$_2$ substrates used dc-sputtering method with a single target. All films annealed at 850 - 880 °C. Results of four probe resistance measurements gave transition temperature of 80 K, 78 K for YBaCuO and BiSrCaCuO film respectively. Whereas the Tc of TiBaCaCuO film was found to be the best at107 K. Ohkubo et al.,1995, prepared Bi$_2$Sr$_2$Ca$_2$Cu$_2$O$_{x}$ films on (001) MgO substrate by dc- sputtering employing a single target. The films are of almost pure Bi$_2$Sr$_2$Ca$_2$Cu$_2$O$_{x}$ (2223) or intergrowth phases known as random stacking fault structures of two phases. Deviation from the sputtering condition for the (2223) growth induced the phase intergrowth of 2212, 2223 and 2234. It has been observed that the intergrowth depends on the film composition. Guldeste, 1998 studied the properties of rf-sputtered Bi-Sr-Ca-Cu-O thin films. The T$_c$ (zero) around 80K is achievable for (Ca+Sr)/Bi ratio between 1.4 and 1.65, while T$_c$-onset remains above 90K. The authors Hermiz et al., 2013 studied the effect of substrate temperature on growth of Bi$_{1.7}$Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_{2.9}$Zn$_{0.6}$O$_{10+\delta}$ thin films. They found an increase of the transition temperature from 95K to 112K with the enhancement of substrate temperature from 300 to 400 °C.

Mua et al., 2014 investigated the electrical transport properties of Bi$_2$ Sr$_2$ Ca$_2$ Cu$_3$ O$_{10+\delta}$ superconducting thin films fabricated by Pulsed - Laser deposition on SrTiO$_3$ substrate. A critical temperature as high as 110K and critical current density of 6.2x10$^5$ A/m$^2$ at 20 K were obtained at substrate temperature of 760°C with annealing time of 4h and deposition rate of 1.5 nA/s. They also investigated the effect of Li doping on Bi-2223 thin films.

In this research, we study the effect of the number of laser pulses on the properties of superconducting Bi$_{1.7}$ Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_{2.9}$Ti$_{0.1}$O$_{10+\delta}$ thin films samples prepared by the PLD method and the target prepared by the solid –state reaction method.

MATERIAL AND METHOD

The target was prepared by two step solid –state reaction method. Predetermined amounts of high purity 99.999% starting chemicals (Bi$_2$O$_3$, Sr(NO$_3$)$_2$, CaO, CuO, PbO and TiO$_2$), were used the fixed nominal composition of Bi$_{1.7}$ Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_{2.9}$Ti$_{0.1}$O$_{10+\delta}$. These Powders were well mixed and ground by using an agate mortar for 1 hr., the mixture homogenization was calcined in air at 800°C for 24 hours in furnace with rate of 4°C/min. Pellets 13 mm in diameter and 1-2 mm in thicknesses were pressed by using a manually hydraulic press type (SPECAC) under pressure of 0.7 G Pa. These pellets were sintered in two steps, the samples in the first
step were sintered at 835°C for 100 hr, in the second step the pellets regrinding for 30 minutes, repressed then resintered at 835°C for 40hr then cooled to room temperature with the same rate.

The PLD of thin films of $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.9}\text{Ti}_{0.1}\text{O}_{10+\delta}$ carried out by using laser Nd:YAG. The laser having a wavelength of (532 nm). The beam energy was focused onto the target to obtain an energy density 0.6-0.7 J/cm$^2$, the ablated frequency at (6 Hz) by a double frequency with Q-switched Nd:YAG pulse laser.

The target mounted in vacuum spherical quartz chamber, rotary pump to pump down the chamber to ($10^{-5}-10^{-4}$) mbar levels, a substrate without heater, a target assembly used for rotating the targets during deposition, the distance between substrate and target was 2 cm. pulse duration of about 7 nanosecond for (300,400 and 500 pulses). The samples were grown on silicon wafers (Si) substrate (111) size 10 mm×10 mm at room temperature. The substrate Si were cleaned in HF aced of concentration 0.1% in order to remove any traces of impurities and then washed in distill water. Thin films was heated in the furnace tubs under oxygen flow of 1 lit/min for 2 hr at 800°C with a heating rate of 5°C/min.

The structure of the prepared samples was obtained by using x-ray diffraction (XRD) type (Philips) with Cu$K_\alpha$ source. The four-point probe method was used to measure resistivity and to determine the critical temperature ($T_c$).

RESULTS AND DISCUSSION

Fig (1) shows XRD patterns for films deposited at variable number of shots ($n = 300, 400, \text{ and } 500$) for $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{2.9}\text{Ti}_{0.1}\text{O}_{10+\delta}$ film grown on Si(111) which passed through annealing cycles 800°C in atmosphere of 100% oxygen for 2 hour and heating rates 15 ºC/min. From this figure it has been observed that the intergrowth mixture of high $T_c$ phase Bi (2223) and low $T_c$ phase Bi (2212) are obtained with the variation of the number of shots.

![XRD pattern of Bi$_{1.7}$Pb$_{0.3}$Sr$_2$Ca$_2$Cu$_{2.9}$Ti$_{0.1}$O$_{10+\delta}$ thin film deposited at different number of shots (300,400 and 500).](image-url)

There is an increase in the high phase intensity of peaks at H (119) and new peaks were appearance attributable to the Bi-2223 phase belongs to H (1111) and H (0018) and H (220) with increase the number of 500. Moreover, it can be shown a decrease in the intensity in some low phase peak at L (115) and L (117) and disappearing some peaks that belong to low $T_c$ phase such as L (113) and L (0211). This could be interpreted as the increase the number of shots from the 300 to 500 shots lead to the number of...
X-ray diffraction patterns and Miller indices for specimens exhibit orthorhombic structure of all the samples and most of them have two multiphase high-Tc phase (Bi-2223), low-Tc phase (Bi-2212), beside of these phases there were impurity phases. The effect of number of laser pulses on lattice parameters values are shown in Table (1).

<table>
<thead>
<tr>
<th>N pulse</th>
<th>Tc (K)</th>
<th>a (Å)</th>
<th>b (Å)</th>
<th>c (Å)</th>
<th>c/a</th>
<th>V (Å³)</th>
<th>dm (gm/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300</td>
<td>108</td>
<td>5.432</td>
<td>5.463</td>
<td>37.152</td>
<td>6.8392</td>
<td>1102.53</td>
<td>1.5413</td>
</tr>
<tr>
<td>400</td>
<td>103</td>
<td>5.4138</td>
<td>5.47110</td>
<td>37.0161</td>
<td>6.8373</td>
<td>1144.42</td>
<td>1.4923</td>
</tr>
<tr>
<td>500</td>
<td>106</td>
<td>5.4256</td>
<td>5.4022</td>
<td>37.0084</td>
<td>6.8210</td>
<td>1084.72</td>
<td>1.5820</td>
</tr>
</tbody>
</table>

In fact, it is obvious that bombing the target more than 500 pulse i.e. (600-700) will deterioration the properties of the films and we do not get a good films. This may attributed to the lack (destroyed) adhesion atoms on the cold substrate.

**Conclusions:**

Our conclusion from the result can be summarized as follow:

1. The critical temperature for the films is increased to 106 K for the numbers of shots are increased to 500 shots.
2. XRD analyses have shown an orthorhombic structure of all the samples and have at least two superconducting phases.

**REFERENCES**


