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The effects of the post-annealing time on the growth mechanism of $Bi_2Sr_2Ca_1Cu_2O_{8+\partial}$ thin films produced on MgO (100) single crystal substrates by pulsed laser deposition (PLD)

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Abstract

$Bi_2Sr_2Ca_1Cu_2O_{8+\partial}$ thin films were deposited on MgO (*100*) substrates by pulsed laser deposition (PLD). The effects of post-annealing time on the phase formation, the structural and superconducting properties of the films have been investigated by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), temperature dependent resistivity (*R–T*), atomic force microscopy (AFM), and DC magnetization measurements. The films deposited at 600 °C were post-annealed in an atmosphere of a gas mixture of Ar (93%) and O_2 (7%), at 860 °C for 10, 30, and 60 min. All films have demonstrated a mainly single phase of 2212 with a high crystallinity (FWHM ≈ 0.159°) and *c*-axis oriented. The critical temperature, T_C, of the films annealed for 10, 30, and 60 min were obtained as 77, 78, and 78 K, respectively. The highest critical current density, J_C, was calculated as 3.34×10^7 A/cm^2 for the film annealed at 860 °C for 30 min at 10 K.

Keywords: C. Superconductivity; Bi-2212 thin film; Pulsed laser deposition (PLD); Post-annealing; Critical current density

1. Introduction

Following the discovery of high temperature superconductors [1], superconductivity was found in the bismuth family of high-T_C ceramic materials [2], and the subsequent work reported that the first cuprate family had a T_C up to 110 K and that it was discovered without rare-earth elements [3]. The bismuth-based superconductors have three members of BSCCO families described by the $Bi_2Sr_2Ca_{n-1}Cu_nO_x$ general formula, where $n=1$, 2 and 3. Moreover, n is related to the number of CuO_2 layers in the crystal structure, producing the Bi-2201, 2212 and 2223 phases with 20, 85, and 110 K critical temperatures, respectively [4–6]. In view of applications, it is highly desirable that the superconducting material has a stable single phase. The Bi-2212 phase has a much better stability compared to other phases of the same compound; hence, it makes it advantageous for applications. After the discovery of the high temperature superconductors [1], the possible applications of these materials have been realized, and several groups have focused on the synthesis of these materials in a specific form like a thin film which is necessary for the applications. Studies in this field are stimulated by the possibilities of the film's utilization of high-T_C devices as SQUIDs and Josephson junctions. Several techniques for producing the thin films have been investigated in various laboratories to advance the qualities of thin films for devices. The parameters of the device can be controlled by the preparation techniques, some of which involving the molecular beam epitaxy (MBE) [7,8], dc sputtering [9], rf sputtering [10], chemical vapor deposition (CVD) [11,12] and pulsed laser deposition (PLD) techniques [13–16].

By controlling all parameters such as target distance, vacuum level, conditions of the ambient gases, mechanical setups, laser parameters, substrate option, and deposition temperature, the pulsed laser deposition (PLD) technique is rather promising regarding the deposition of smooth surface in nanoscale for

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mono or multilayer material with a good electrical property, and highly textured thin films with a complex stoichiometry. Single crystal of MgO is the most preferred material for producing Bi-2212 thin films because of its low-cost, compatible features such as dielectric constant, stable structure and chemical stability [13–18]. Recently, it was reported that a significant improvement in superconducting properties of thin films could be obtained by PLD and the application of post-annealing heat treatment in an atmosphere containing a mixture of argon and oxygen (Ar:93,O_2:7) [15,16,19,20]. Among various factors, the quality of substrate also affects directly the superconducting properties of thin films.

In this study, we have reported the results of effects of post-annealing time on Bi-2212 thin films deposited on MgO (100) single crystal substrates by pulsed laser deposition.

2. Experiment

$Bi_2Sr_2Ca_1Cu_2O_y$ target has been prepared by using a polymer matrix route described in detail [21–23]. $Bi(CH_3COO)_3$ ($\geq$ 99.99%, Aldrich), $Sr(CH_3COO)_2 \cdot 0.5H_2O$ (99%, Panreac), $Ca(CH_3COO)_2 \cdot 2H_2O$ (98%, Alfa Aesar) and $Cu(CH_3COO)_2 \cdot H_2O$ (98%, Panreac) commercial powders were used as starting materials. They were weighed in stoichiometric amounts and dissolved in a mixture of glacial acetic acid (CH_3COOH) (Panreac PA) and distilled water. The use of a mixture of glacial acetic acid and water is due to the fact that in one side Sr acetate is insoluble in concentrated acetic acid and, on the other side, Bi acetate is not soluble in water [24]. Once a clear blue solution is obtained, polyethyleneimine (PEI) (Aldrich, 50 wt% water) was added. The mixture becomes dark blue immediately reflecting the formation of Cu–N coordination bonds. The solution was then introduced into a rotary evaporator to reduce its volume (in ~80%), followed by heating on a hot plate at about 100 °C for total solvent evaporation, producing a thermoplastic dark blue paste. Further heating at around 350 °C produces a decomposition step (self-combustion), as described schematically elsewhere [25], which produces the organic material decomposition. The resulting powder was then milled in an agate mortar and calcined twice at 750 and 800 °C for 12 h, with an intermediate manual milling, in order to decompose the alkaline-earth carbonates.

Finally, the pretreated homogeneous powders were pressed into 1 in. diameter pellets and thermally treated in order to produce the Bi-2212 superconducting phase. This process was performed under air, and consisted in two steps: 60 h at 860 °C, followed by 12 h at 800 °C and, finally, quenched in air to room temperature.

The thin films of $Bi_2Sr_2Ca_1Cu_2O_{8+\partial}$ were deposited onto MgO (100) substrates by pulsed laser deposition (PLD), using an excimer laser (248 nm) focused on the target surface at an angle of 45°. Before deposition (at base pressure 1.0×10^{-6} Torr), substrates were first heated up to 1000 °C at a rate of 30 °C/min, soaked during 15 min to clean remaining impurities from their surfaces, and then cooled down to 600 °C. This, we believe, helps also to relieve any stresses build-up on the substrate. During the deposition, the substrates were kept at 600 °C while keeping O_2 gas pressure in the chamber fixed to 250 mTorr. The distance between the substrate and the target was 45 mm. The laser fluence was kept at ~2.39 J/cm^2 and with a pulse repetition rate of 5 Hz. After deposition, the substrates were cooled down to room temperature at a rate 20 °C/min, while maintaining the pressure of O_2 gas at 70 Torr. After deposition, post-annealing heat treatments were performed in a quartz tube having a mixture of (Ar/O_2:93/7) inside, and the tube was placed inside of a tubular furnace heated at 860 °C during 10, 30, and 60 min.

After the heat treatment process, the crystal structure and phase formation of the films were analyzed by using X-ray diffraction patterns with a Rigaku MiniFlex diffractometer with CuKα radiation and a scan rate 1°/min between $2\theta = 10$–70°. The surface morphology and compositional analyses of thin films were also investigated by SEM and EDX measurements by using a FEG/QUANTA 250 Scanning Electron Microscopy SEM. The surface morphology and roughness were observed by atomic force microscopy (scanning area:

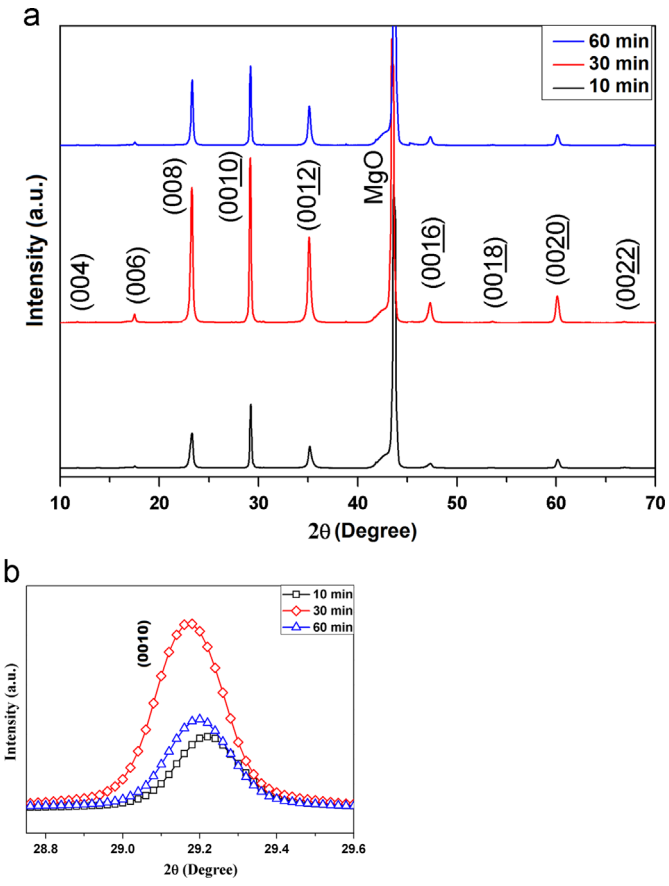

Fig. 1. XRD patterns of films annealed at 860 °C for 10, 30, and 60 min.

Table 1
Lattice parameter c and crystal size L versus post-annealing temperature.

Annealing time at 860 °C	c (Å)	Crystal size L (Å)
10 min	**30.9609**	**560.29**
30 min	**30.8608**	**537.16**
60 min	**30.8864**	**570.30**

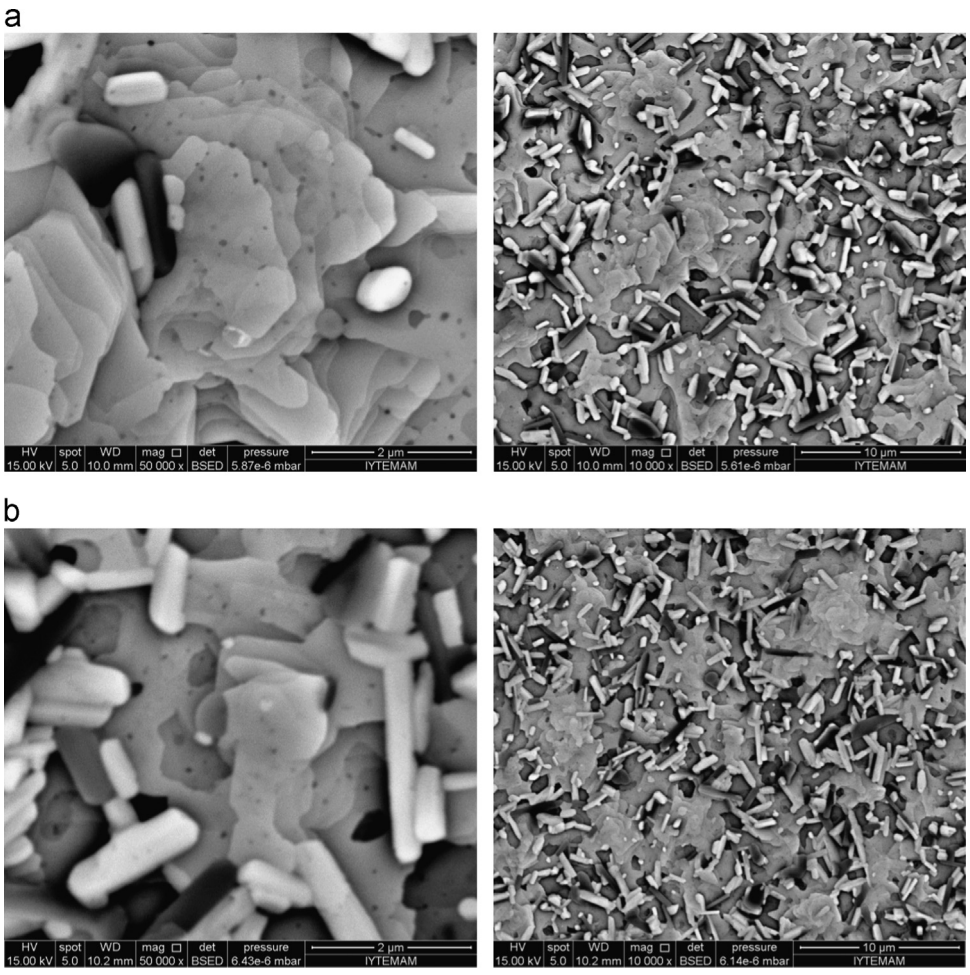

Fig. 2. a. SEM images of the film annealed 860 °C for 30 min. b. SEM images of the film annealed 860 °C for 60 min.

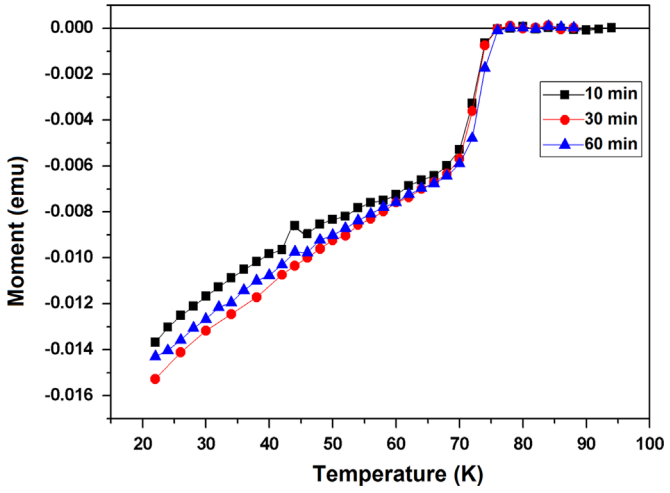

Fig. 3. DC magnetic moments versus temperature for annealed films at 860 °C for 10, 30, and 60 min.

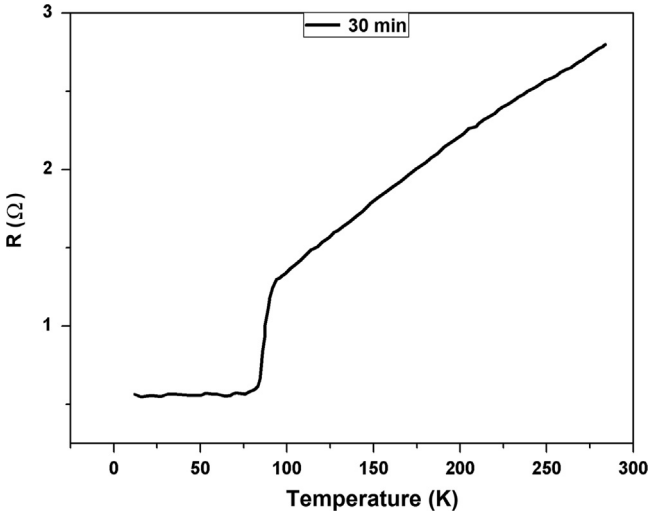

Fig. 4. *R–T* measurements of the film annealed 860 °C for 30 min.

$10 \times 10\ \mu m^2$). The thickness was measured with a profilometer as 500 nm. In order to determine the critical superconducting temperatures, magnetic measurements were also carried out in a model 7304 Lake Shore Vibrating Sample Magnetometer (VSM) system under ZFC mode, under a magnetic field of 50 Oe applied perpendicular to the film surface. In order to determine the zero resistivity critical temperature, we have used a home-made conventional four probe method with a closed cycled liquid He refrigerator and temperature controller. The J_Cs were estimated from the *M–H* loops as measured

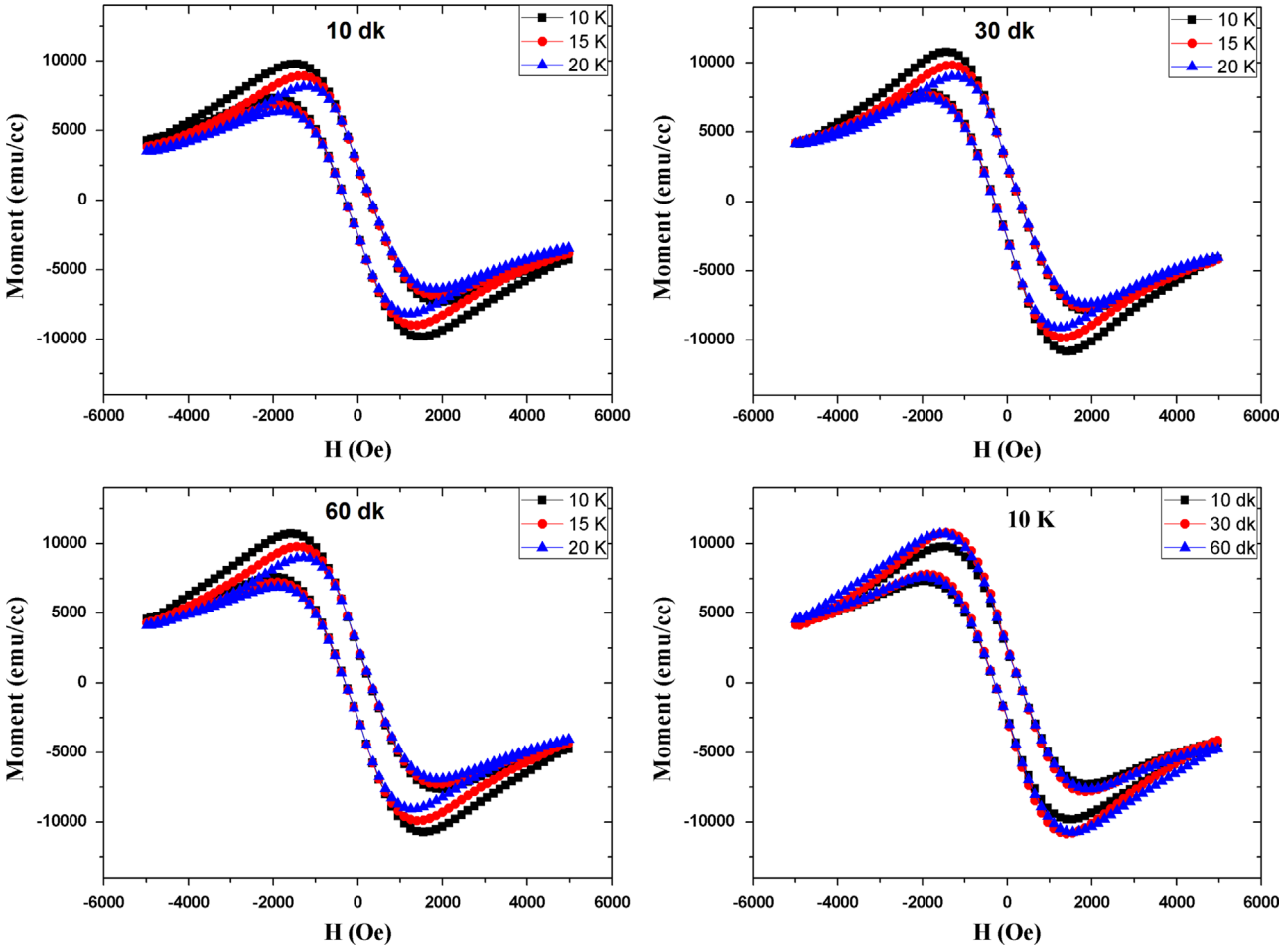

Fig. 5. $M(H)$ Hysteresis loops for annealed films at 860 °C for 10, 30, and 60 min.

between −0.5 and 0.5 T magnetic field applied perpendicular to the film surface, which exhibits a rectangular shape.

3. Results and discussion

3.1. XRD characterization

X-ray diffraction patterns of the films annealed at 860 °C for 10, 30, and 60 min are shown in Fig. 1a. Peak positions and intensities indicate that all main peaks were constructed on (*00l*) reflections of Bi-2212 phase. Additionally, the grown films have a high crystal homogeneity resulting from formation of good oriented grains aligned to the *c*-axis on MgO (*001*) surface.

However, intensities of main peaks decrease with a slight angular shift when the annealing time is exceeded 30 min at 860 °C. This can be possible with longer annealing time resulting in re-crystallization [15]. It can be seen clearly that a pure Bi-2212 main phase is obtained for the all films annealed at 860 °C for different times, since there is no evidence for the reflections of another BSCCO phase or impurity. The full widths at half-maximum values (FWHM) of the (*0010*) peak of the films annealed for 30 min and 60 min at 860 °C were determined as 0.169° and 0.159°, respectively. The lattice parameter c and crystal size L of films were calculated from XRD diffraction patterns as given in [26], and tabulated in Table 1. The c value is calculated around 30.9 Å for all films. This value is in good agreement with the previous publications for Bi-2212 superconductors [27].

3.2. SEM analysis

Fig. 2a and b shows the SEM images of the thin films annealed at 860 °C for 30 and 60 min, respectively. It is seen that a well-stacked and terrace-like typical structures of Bi-2212 grains [28] have formed in the matrix. The appearance of surface morphology of thin films exhibits that the *a–b* plane of large grains of the Bi-2212 films mostly oriented along *c* axis with layered growth on *a–b* plane of MgO (*100*) single crystal substrate. Moreover, the voids among the grains on the film surface annealed at 860 °C for 60 min appear reduced compared to the film annealed at 860 °C for 30 min, since the crystallization process results from further annealing time.

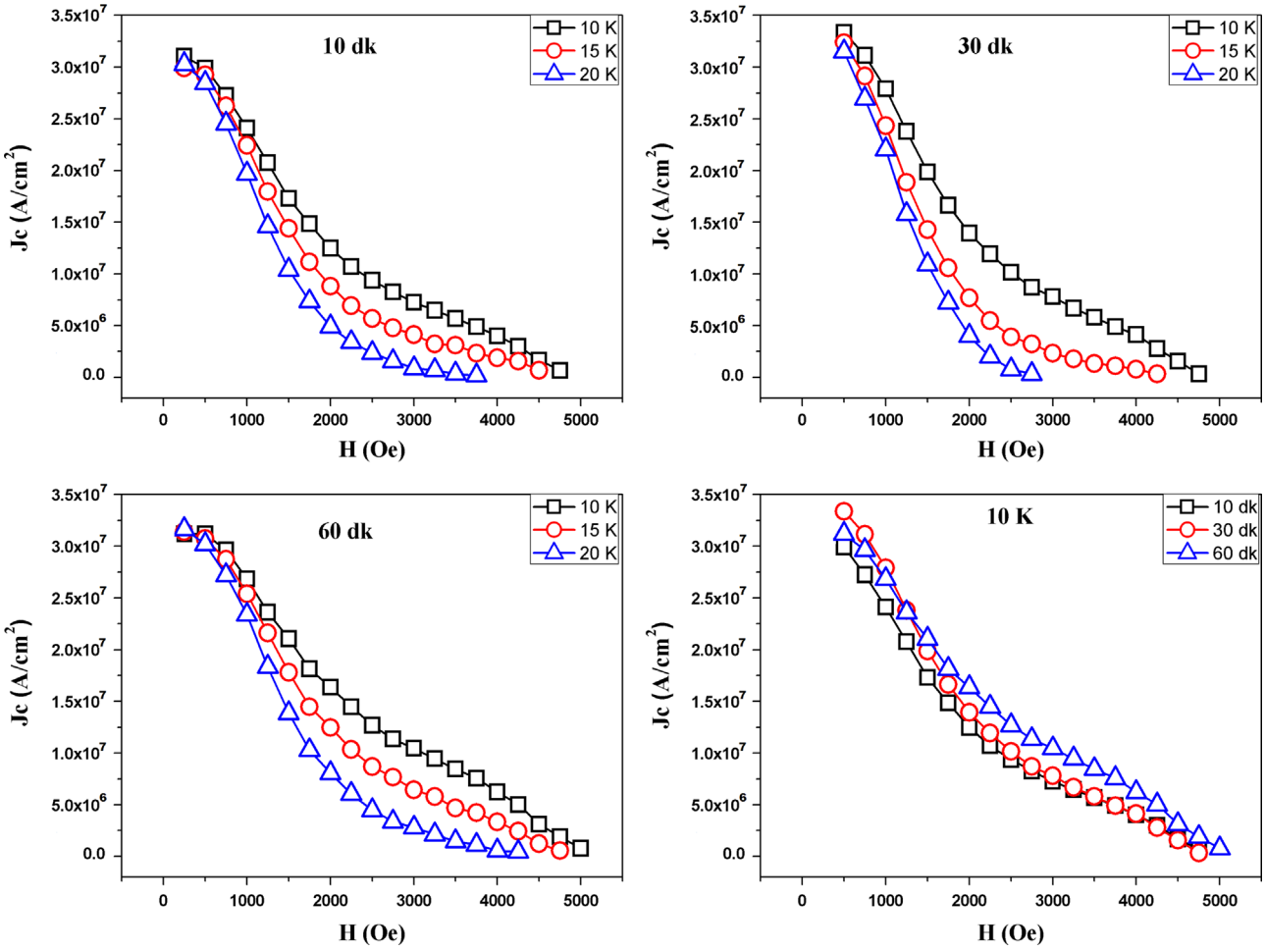

Fig. 6. Critical current densities J_C calculated from *M–H* loops for annealed films at 860 °C for 10, 30, and 60 min.

3.3. Magnetic properties

Fig. 3 shows the results of the DC magnetization measurement of Bi-2212 thin films produced. *M–T* measurement was performed on the samples under a magnetic field of 50 Oe. The field was applied parallel to the *c*-axis of the film surface. All films exhibited a sharp diamagnetic transition around the critical temperature. The T_C*s* of the films annealed at 860 °C for 10, 30, and 60 min were determined as 77, 78, and 78 K, respectively. Additionally, Fig. 3 indicates that while annealing time increases, the transition temperatures of the samples show no significant change.

3.4. Electrical measurement

The electrical resistance versus temperature (*T*) curve obtained from room temperature down to 10 K for the film annealed at 860 °C for 30 min is displayed in Fig. 4. The sample shows a metallic behavior at normal state region and then a superconducting transition starts while the temperature is decreasing down to lower values. The zero-resistance temperature value was obtained around 0.57 Ω arising from copper wires. In addition the zero resistance critical temperature, T_{C0}, and superconducting transition widths, ΔT, of the film annealed for 30 min were evaluated as 82 K, and 6 K, respectively. It may be stated that the film exhibits typical characteristics of single phase Bi-2212 superconductors.

3.5. Magnetic hysteresis

Fig. 5 shows the hysteresis curves of the films annealed at 860 °C for 10, 30, and 60 min. The *M–H* measurement was performed at 10, 15, and 20 K, while the magnetic field was applied perpendicular to the surface of the films. It is easily seen that, while the annealing time increases, the area of hysteresis loops remarkably becomes larger. It is attributed to the fact that an increase of the annealing time leads to a larger grain structure with better intrinsic properties for the SC, stemming from a better crystallization process taking place. The graph of *M–H* measurement obtained at 10 K also reveals that the symmetric peaks of magnetization arising from pinning on *a–b* plane may be caused by intrinsic pinning of the CuO planes [29]. The increasing of deficiencies in intragranular space seems to improve flux pinning mechanism. Hysteresis curves have been slightly enlarged by larger grain structure due to the increase of annealing time.

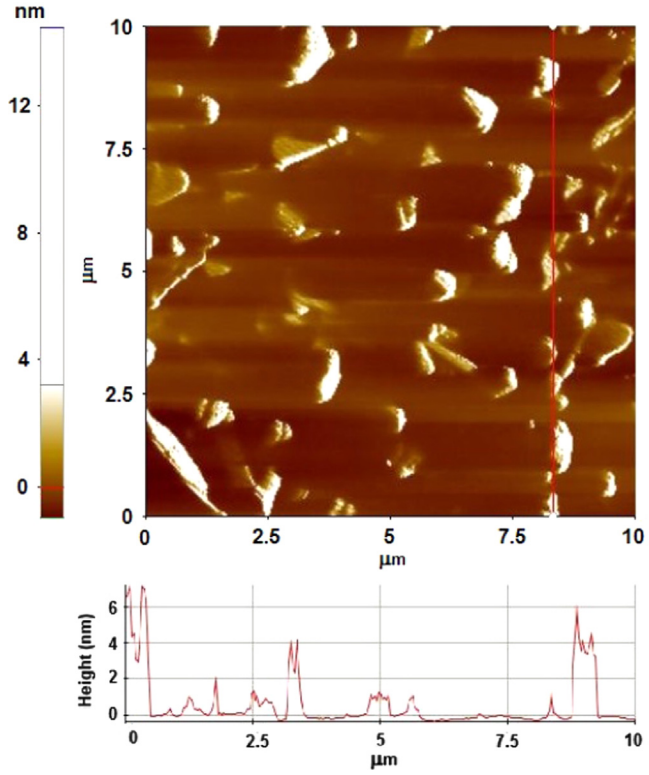

Fig. 7. AFM images of the film annealed at 860 °C for 30 min.

3.6. Calculations of critical current density

The critical current densities, J_C, of the films have been calculated by using the Bean model as given in [30]:

$$J_c = \frac{60a\left|\Delta M\right|}{b(3a-b)}$$

where a and b are the dimensions of the films and $\Delta M = M_+ - M_-$ is a difference of magnetization values measured in electromagnetic units per cubic centimeter.

Fig. 6 shows the calculated critical current densities of the films annealed for 10, 30, and 60 minutes as a function of the applied field, at 10, 15, and 20 K. The maximum J_C values are calculated as 2.99×10^7, 3.34×10^7, and 3.12×10^7 A/cm^2 for the films produced at annealed 860 °C for 10, 30, and 60 min respectively. The calculation of critical current density for Bi-2212 thin film produced via PLD is a rare issue in the literature. However, all maximum J_C values for the film produced are comparable and better than those of some other recent studies [20,31,32]. Calculated J_C value of the thin film annealed for 30 min explicitly demonstrates a better performance with increasing magnetic field up to 1000 Oe, compared to those of other films and then it starts to decrease comparatively. Additionally, it can also be clearly seen that the temperature and field dependence of the film is seen to be higher than those of the others. This may be expected since the large amounts of pores between the grains observed in SEM images for thin film annealed for 30 min behave like a defect and create a field dependent effect in the vortex region.

3.7. AFM analysis

Fig.7 shows the surface morphology and roughness on the film annealed at 860 °C for 30 min observed with AFM. The scanning area was a 10 µm^2. The roughness profile under the selected line demonstrates that the heights of the steps chance in the unit cell level of the 2212 phase. It can be seen that, typical grain structures of Bi-2212 formed in the main matrices.

4. Conclusion

In this study, highly textured and *c*-axis oriented Bi-2212 thin films were deposited on MgO (*100*) single crystal substrate by using the pulsed laser deposition (PLD) technique. The films were firstly deposited on the preheated MgO substrate at 600 °C. Later on, post-annealing heat treatment has performed at 860 °C which was obtained as an optimum temperature in our previous study [20] in argon and oxygen (Ar:93, O_2:7) atmosphere, for 10, 30, and 60 min. The post-annealing heat treatments have been applied on those films for three different annealing times, in order to determine the optimum condition. In this way, the quality and the superconducting properties of the film exhibited a significant improvement. Then the structural and superconducting properties of BSCCO these thin films were investigated.

The optimum annealing time has been obtained as 30 min at 860 °C. The same superconducting transition temperatures, $T_C \approx 78$ K, of the films annealed at 860 °C for 30, and 60 min were obtained.

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