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The effect of post annealing temperature on grain size of indium-tin-oxide for optical and electrical properties improvement

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ABSTRACT

Background (Problem): Indium tin oxide (ITO) is a transparent conductive oxide (TCO) thin film used as a transparent electrode. Given its high demand for the manufacture of transparent electrodes (high visible light transmittance, low resistance) in applications such as liquid crystal displays, touch screens, light emitting devices and solar cells, ITO thin films have attracted immense research interest.
Objectives: This study determines the effect of grain size on the optical and electrical properties of the ITO thin films under different annealing temperatures.
Materials and methods: ITO thin film was deposited at room temperature by a high frequency magnetron sputtering method using a target composed of In_2O_3 and SnO_2. The structural, optical and electrical properties of the thin films annealed at 250 °C, 350 °C, 450 °C and 550 °C for 1 h were then analyzed.
Results: The research shows the grain size of indium-tin oxide thin films is strongly related to annealing conditions. The grain size was found to increase with increasing annealing temperature, although the crystal structure did not change for all the samples. It was observed that the lowest resistivity (500×10^{-4} Ω-cm) and highest optical transmittances (90–98%) of ITO films were obtained at annealing temperature of 450 °C. At low annealing temperatures, the measured resistivity is dependent on the effect of grain size, where it decreases with increasing grain size.
Conclusion: This work showed that the grain size of ITO thin films is strongly influenced by post annealing technique and conditions applied, thus providing a tool for enhancing the optical and electrical properties of the film.

Introduction

With the increasing demand for light emitting diodes (LEDs) and optoelectronic products nowadays, the need for transparent conductive oxides (TCOs) has also risen accordingly. Among the many TCOs, indium tin oxide (ITO) has a unique set of properties, such as high ultraviolet absorption, high reflectance infrared, high microwave attenuation, good processing performance with better acid engravement, lithographic performance, good mechanical strength and abrasion resistance, alkali chemical stability, and a wide band gap (3.5–4.2 eV). ITO also possesses great photo electrolytic properties that include electrical conductivity of 10^3–10^4 O^{-1} cm^{-1} and transparency of 80 to 95% in the visible range [1]. Based on the above-mentioned exceptional intrinsic properties, this material is widely used in the manufacture of flat panel display, solar cells, LEDs, microwave and radio frequency shielding devices, touch switches, architectural glass, and other fields [2]. However, the efficiency of relevant electronic devices has been limited by the large electrical resistivity of the transparent electrodes. Thus, it is imperative to keep the transparency of ITO films without a noticeable drop while decreasing their resistivity [3].

Given the dependence of the physical properties of ITO films (microstructure, electrical and optical properties) on the preparation and

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experimental conditions of ITO films, the crystallinity, surface roughness, impurity levels and band gap of the synthesized thin films may be affected by various growth conditions [4–6]. ITO thin films have been deposited using several methods that include; plasma-enhanced metal organic chemical vapour deposition (PEMOCVD) [7], electron beam evaporation [8], ion beam-assisted deposition (ISD) [9], pulsed laser ablation (PLD) [10,11], dip coating [12], ion implantation [13] and RF magnetron sputtering [14,15].

The fabrication of high-quality ITO thin film depends on two sets of parameters: sputtering process and post deposition treatment parameters. The sputtering process parameters include RF power, gas ambient flow (oxygen, argon, hydrogen, H_2O vapor) flow rate, sputtering working pressure, deposition rate, substrate temperature, substrate type, target to substrate distance and target density [15]. The post deposition treatment parameters include post annealing, ionization irradiation, laser irradiation and interlayers on indium-tin oxide thin films [16]. In addition to these parameters, thickness also influences the properties of the ITO thin film [16,17]. Sitao Yang, et al. [18] studied the effect of oxygen vacancy on the structure and converting the phase from amorphous to crystalline. They found that the absorption intensity of the visible light can be attributed to the formation of uniform distribution of the oxygen vacancy in the crystal bulk of TiO_2x rutile phase by diffusing the oxygen vacancy from the crystal surface to its bulk. This will not be combined with the concentration increase of the oxygen vacancy, because the concentration decrease of oxygen vacancy will caused by the increasing in temperature. The efficiency of visible light adsorption can be enhanced by uniform distribution of the oxygen vacancy. The high temperature annealing is necessary for TiO_2x formation with the uniform distribution of oxygen vacancy, because of the large activation energy of the diffusion of oxygen vacancy (Fig. 1).

Several studies have reflected that post-annealing treatment can control the structural properties of ITO thin films which have been deposited on different substrates [19,20] by controlling the annealing temperature [21–23]. Other study showed the impact of annealing temperatures (400–600 °C) on specific contact resistance of Ni/Au/ITO thin films [24]. Electrical properties of ITO thin films depend on the physical properties (crystal structure, oxidation level) of the films [25]. However, it is a challenge to optimally develop both optical and electrical characteristics, which are the essential aspects for Transparent conducting oxide (TCO) layers. Optimization of these characteristics can aid the reduction of resistivity.

Therefore, an appropriate optimization demands an understanding of the effect of several parameters on electron transport. For instance, the grain size of thin films is affected by thin film thickness and annealing temperature. Thus, this paper investigates the structural, morphological, optical and electrical properties of ITO thin films based on grain size under different post annealing temperatures. This study also analyzes and explains the dependence of mobility, carrier concentration, sheet resistance, strain, crystalline and the energy gap on the grain size of ITO thin films using a thickness of 200 nm under different annealing temperatures.

Experimental

Grain size and annealing temperature

The resistivity of ITO thin films is subject to diffusion from phonons, point defects, impurities, grain boundaries and film surface [26,27]. To determine the effect of grain size on optical properties and resistivity of ITO film, annealing temperature was varied between (250–550) °C.

ITO film preparation

To obtain a deeper understanding of the impact of grain size on the

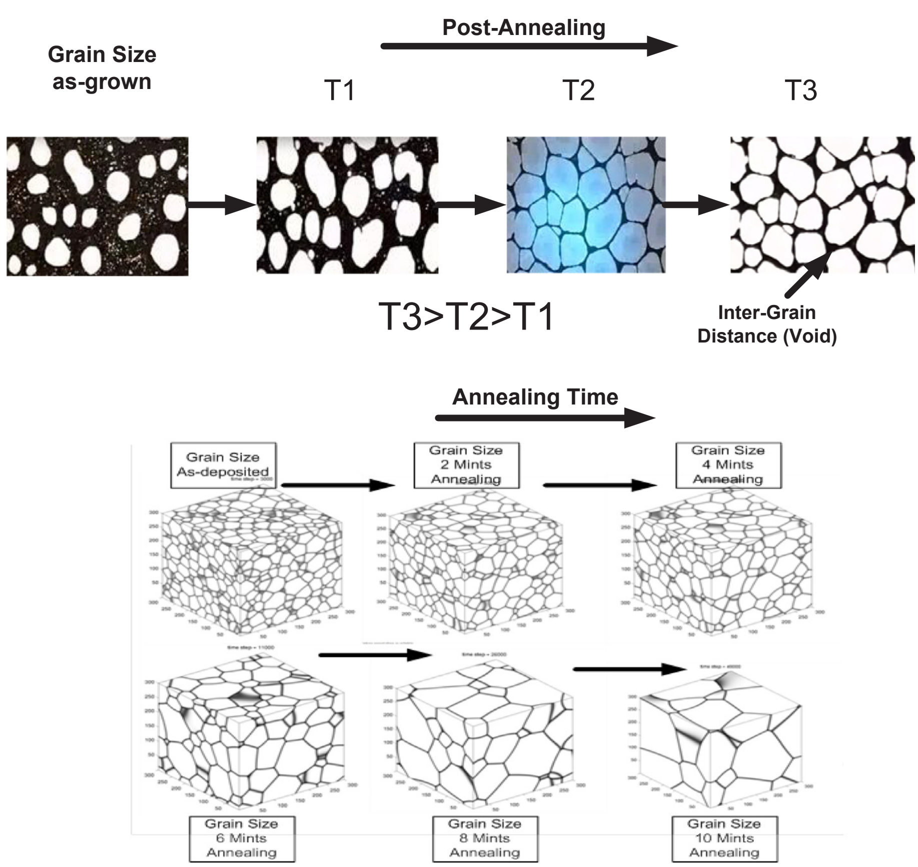

Fig. 1. Thin film Grain size effects of post annealing temperature and annealing time. By: Boettinger.

characteristics of ITO film and to acquire low cost, high quality ITO film with optimal post annealing temperature, the ITO films were deposited at room temperature using RF sputtering method on the glass with 200 nm thicknesses. The effect of annealing temperature on the structural, optical and electrical properties of ITO films was studied using the annealing tube furnace (LENTON VTF/12/60/700). The furnace has a maximum operating temperature of 1200 °C. The glass substrates were cut into $1.2 \times 1.2\,cm^2$ pieces, immersed in aqua regia for two hours, subsequently rinsed with deionized water for 15 min followed by acetone, alcohol and deionized ultrasonic water for 10 min respectively, blow dried using nitrogen gas, and finally oven dried at 120 °C for the purpose of outgassing [24,25,28,29].

The synthesis process of ITO thin films can be summarized as follows; firstly, the use of RF sputtering to deposit ITO onto a glass substrate from a ceramic spattering target containing 90 wt% In_2O_3 and 10 wt% SnO_2. The prior pressure was maintained at approximately 10^{-7} mbar. The ITO thin films were synthesized at an RF power of 100 W to achieve a thickness of 200 nm. The annealing process was performed under ambient air at various temperatures (250 °C – 550 °C) for 1 h.

Characterizations

The synthesized ITO thin films were subsequently characterized using various tools. The structural characteristics of the films were studied using high-resolution X-ray diffraction technique and field emission scanning electron microscopy (FESEM). The X-ray diffractometer (HR-XRD) model PANalytical X'pert Pro MRD used the 2θ mode and Cu Kα radiation source at a wavelength of 1.54 A°. The resistivity values were measured using KETHLEY, 2400 Source Meter. Finally, the optical transmission spectra curves were gained using a Varian-Cary system 5000 UV- Vis-NIR spectrophotometer (Agilent Technologies, Product No. G9825AA).

Results and discussions

X-Ray results

XRD analysis was executed to obtain comprehensive data regarding the crystalline properties of the ITO thin films (Fig. 2). For the as-grown ITO thin film, no diffraction peaks were observed for the amorphous phase of the film. For the ITO thin films annealed at >400 °C, the observed diffraction peaks of (2 1 1), (2 2 2), (4 1 1), and (4 4 0) planes confirm the bixbyite structure of the ITO thin films. In addition, all the

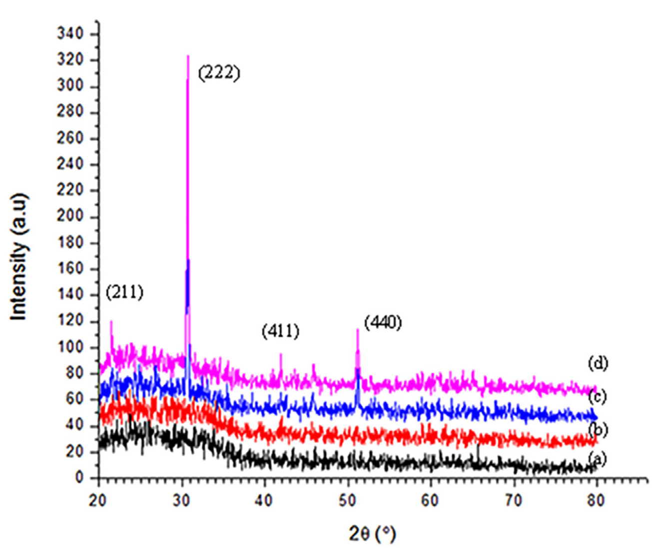

Fig. 2. X-ray diffraction image of ITO thin films: (a) as-deposited, (b) annealed at 250 °C, (c) annealed at 350 °C, and (d) annealed at 450 °C.

spectra showed no diffraction peaks for impurity phases. The presence of the characteristic peak for SnO_2 indicates the Sn^{4+} atoms were effectively integrated into the In^{3+} lattice sites.

The crystallite size was determined from calculations of full width at half maxima (FWHM) values obtained from the XRD spectra using Scherrer's formula. Increasing the annealing temperature concomitantly increases the intensity and straightness of the spectral peaks of the ITO structure, which is consistent with the enlarged grain size of ITO nanocrystals. This confirms that the crystallinity of the ITO thin films is enhanced with increasing annealing temperature. Table 1 shows the average crystallite sizes (D) of the ITO thin films which were obtained from the width of spectral peaks using Scherrer's formula [30], which is expressed below (Eq. (1)):

$$D = K\frac{\lambda}{\beta cos(\theta)} \tag{1}$$

Where λ denotes the X-ray wavelength (λ = 1.5406 Å), β (in radian) refers to Full Width at Half Maxima (FWHM) of X-ray peaks (obtained from the peak (2 2 2)), K symbolizes the shape constant, which is = 0.94, and θ (in degree) is the diffraction angle at which the peak of an orientation (2 2 2) arises (obtained from Fig. 2 by dividing the value of 2θ over 2). Applying all these parameters to Eq. (1), Table 1 can be achieved.

The improvement in the crystalline structure of thin film causes decreasing on its band gap. Such narrow in the energy band gap with post-annealing treatment could be attributed to the possibilities of oxidation, more realignment and strong interaction between film and substrate [31].

The observed structural results matched with that of the cubic lattice of JCPDS standard card no. 89–4598. No additional peaks were observed correspond to additional phases; which confirms no impurities existed. This result denotes that the ITO phase formation was achieved as a single-phase material. This structure was synthesized through the solid-state reaction path only after lengthened calcinations of the reaction mixture at comparatively high temperatures [31–34]. The crystallite size (D) increment lightly within a very narrow limit with rising annealing temperature, as follows; 35.05, 40.98, 41.17, 41.54 and 51.62 nm for as grown, 250, 350, 450 and 550 °C, respectively. These results indicate that the annealing temperature of 550 °C increases the grain size to 51.62 nm, although with voids emerging between the grains (see FESEM image). Fig. 2c shows the peak intensity of the (2 2 2) and (4 4 0) starts to appear with increasing annealing temperature to 350 °C, which keep rising with higher annealing temperature. ITO thin films with higher intensity peaks exhibit lower resistivity. The ITO thin film with growth direction along the (2 2 2) plane contains larger amount of interstitial oxygen atoms into the thin film particles, this can be attributed to the non-uniform distribution of oxygen vacancies [32].

Field emission scanning electron microscopy (FESEM)

The as-deposited films were annealed using different temperatures. The structure of the as-deposited ITO thin films is transformed from amorphous to crystalline as annealing temperature increased. However, as the annealing temperature extends beyond 550 °C, the excessive heat input adversely affects the film structure by creating voids between the grains. The texture and surface morphology of the thin films were characterized using field emission scanning election microscopy (FESEM). Fig. 3(i-a) shows the grains are tight (closely connected) and regularly shaped, while Fig. 3(i-b) depicts the onset of development and formation of granular structures. As annealing temperature is increased from 250 to 550 °C, the grain sizes of the ITO thin films concomitantly increase (Table 1). The ITO thin films annealed at 250 °C display amorphous structures, while the crystallization was revealed at 450 °C. Up to 550 °C, crystal growth via slight increase in grain size can be observed, but at >550 °C the grain size increased rapidly, and voids

Table 1
The average crystallite size of the ITO thin films annealed at As grown, 250, 350, 450 and 550 °C.

Time (min)	Orientation	FWHM (Degree)	FWHM (rad)	Degree (degree)	Rad (Degree)	Cos (θ)	D (nm)
As-grown	(222)	0.235	0.004	30.63	0.267	0.964	35.05
250	(222)	0.201	0.004	30.58	0.267	0.965	40.98
350	(222)	0.200	0.003	30.63	0.267	0.964	41.17
450	(222)	0.198	0.003	30.63	0.267	0.964	41.54
550	(222)	0.16	0.003	30.63	0.267	0.964	51.62

were created between the grains. The composition of ITO thin films has been displayed by EDX result shown in Fig. 3(ii).

Effect of grain size on visible light transmission (300 nm ~ 800 nm)

Fig. 4 shows the transmittance spectra of the ITO films in the wavelength range from 300 to 800 nm. The optical transmittance spectra of the treated films found to be varied with post annealing which might be due to improvement in crystalline behavior (from amorphous to a cubic structure) [31]. Fig. 4 shows high transmittance in the visible wavelength which enhanced significantly with the temperature, but it was at its maximum for films annealed at 450 °C, which might be ascribed to the ordered structure and variation in the corresponding average grain size [35]. As-deposited films showed the minimum transmittance, also at annealing temperature $^{>}$450 °C, the surface visible light transmittance slightly decreased. The crystallization of the film improved with increasing temperature, which subsequently increased both the mobility of the film and density of Sn^{4+} carriers, thereby reducing the surface resistance. The increased density of Sn^{4+} carriers reduce the generation of black InO, thus improving the visible light transmittance.

Analysis of electrical resistivity

Carrier concentration and mobility can influence the resistivity of ITO films. Oxygen vacancies and Sn synthesis in ITO thin film basically affect the carrier concentration, while strong diffusion centers with free carriers, surface, grain boundaries, acoustical phonon, etc. can influence the mobility of the film [27].

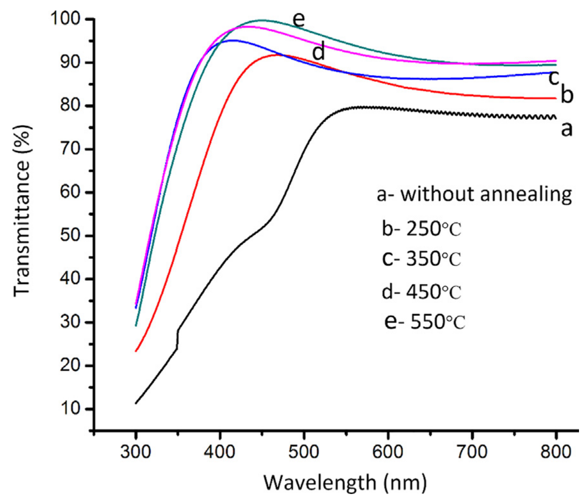

Fig. 4. Optical transmittance spectra of ITO thin films.

Generally, the high resistivity of the as grown film decreases with annealing temperature. The peak resistivity of sample (a) is 9500×10^{-4} ohm.cm, which is the highest because of the many dislocations and defects in the film. The resistivity value of ITO thin film annealed at 250 °C (sample b) is 4500×10^{-4} ohm.cm, which is lower compared to as grown sample because of the larger grain size. The resistivity values of 1000×10^{-4} and 500×10^{-4} (ohm.cm) for samples (c) and (d), respectively, are the lowest. The resistance and electrical current are related to the ITO grain size, since the sizes of crystalline grains

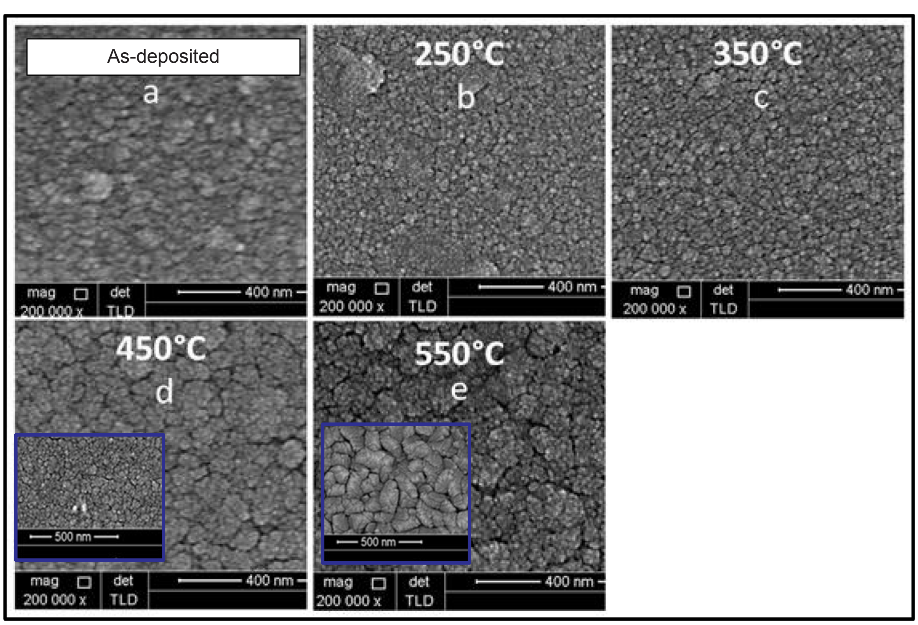

Fig. 3. (i) FESEM micrographs of ITO film with various annealing temperatures, the last image showing grains with voids between neighbouring grains (ii) EDX of all ITO thin films.

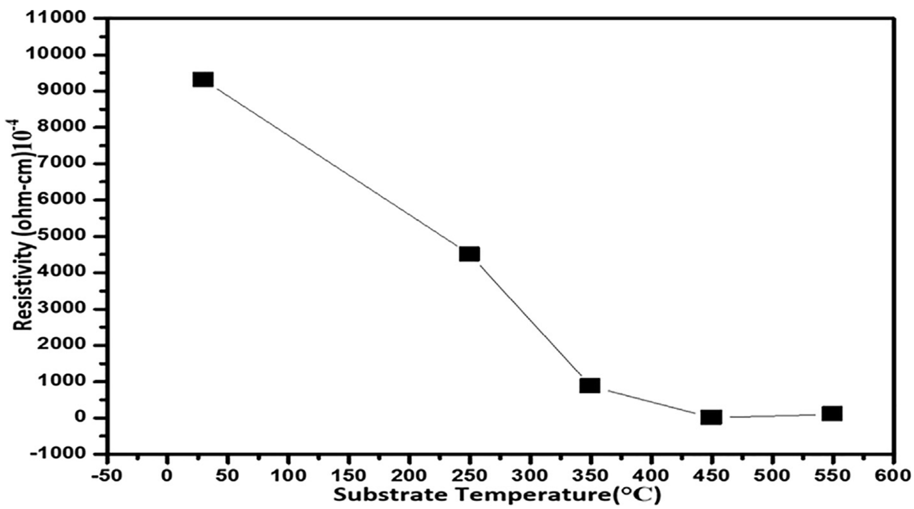

Fig. 5. Resistivity of ITO thin films with different annealing temperatures.

contribute to defining the electrical resistance of a material. These values were determined and plotted against temperature, as shown in Fig. 5

In general, the electrical conductivity of semiconducting thin films is increased with annealing. The electrical conductivity of thin films is robustly affected by the tunneling of the charge carriers through the barriers of the grain boundary and recrystallization of grains during the annealing treatment. The grain boundary model can be considered to explain the electrical conduction and charge transport in polycrystalline thin films, since this model is based on the flow of the majority and minority charge carriers, which might be either normal or parallel to the grain boundaries [35]. In the first case, for flowing the current across a grain boundary, the transport of majority carriers has been slowed down by the potential barriers, the majority carrier mobility has been limited, whilst the minority carriers have been driven by the potential wells towards recombination centers at the grain boundary, diminution the minority carrier diffusion lifetime and length. The size of these effects depends on many parameters; the density of interface states, the photogenerated carrier density, and the doping. While for the parallel case, the minority carriers will be principally affected by grain boundaries that lie parallel to the direction of current flow. In other wise, the majority carriers transporting parallel to the grain boundary are not affected (no barrier), but the minority carriers are continue trapped in the potential well and recombine [36].

The medium effective conductivity can be described as the current crossing the grain boundary per unit applied field. Furthermore, the effective mobility can be described as the conductivity per unit charge. Hence, they both depend on the mean current density crossing the grain boundary [36].

The grain size is considered a significant factor that controls resistance. The size of crystallites is determined by directly manipulating the active surfaces (spread out) of thin films [37]. It appears that as annealing temperature increases, the average diameter of the crystallites will increase, which decreases the spread-out surface of the layer formed by these nanocrystallites. Furthermore, when the created crystallites attain a specific cut limit (after 1-hour annealing), the grain size becomes 40.98 nm and 41.54 nm for annealing temperatures of 250 °C and 450 °C, respectively. Therefore, the spread out (active) surface increases with annealing, which in turn decreases the value of resistance.

Based on the characterizations accolmplished in this study, the best ITO thin film which is suitable for the technology concerned was found to be the film annealed at 450 °C. As it acquired the best structure, large grain size, highest transmittance and highest conductivity.

Conclusion

This study shows annealing temperature effects on the grain size of ITO thin films, thereby influencing their structural, optical and electrical properties. The ITO films were deposited at room temperature using RF sputtering method on the glass with 200 nm thicknesses. Then post annealing treatment was performed for these thin films at 250, 350, 450 and 550 °C. It can be concluded that the size of crystallites is related directly to annealing temperature, i.e., the size of the grains increases with temperature. As the annealing temperature was increased, the microstructure of the ITO film improved (larger grain size and uniform surface morphology). The output of this study displayed that the annealing temperature 450 °C is the optimum condition for getting thin film with optimum characteristics, which can be suitable for the technology concerned. The ITO film annealed at 450 °C showed the best structure, large grain size, highest transmittance, highest conductivity. However, at annealing temperature $^{>}$550 °C, the particle sizes and shapes, agglomeration and surface morphology of the ITO film were destroyed. The dependence of electrical resistivity on temperature accounts for the significant influence of grain size on the electrical conductivity characteristics of the ITO film. It is observed that the smaller grain size results in a low electrical conductivity. Therefore, the grain size of the ITO film is strongly influenced by post annealing temperature, thus providing an approach to enhancing the optical and electrical properties of the film.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.rinp.2019.102159.

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