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Sputtered Indium Tin Oxide Films for Optoelectronic Applications

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Abstract

High-quality indium tin oxide (ITO) films have been fabricated using a DC sputtering technique in a pure argon atmosphere with a postannealing in an oxygen environment at atmosphere pressure. Structural, morphological, and electro-optical parameters of the ITO films were studied at different annealing temperatures for the films fabricated on two types of glass substrates, soda lime and alkali-free substrates. A comparative analysis shows that low-cost soda lime substrates are suitable for the fabrication of high-quality nanocrystalline ITO films after annealing them at 300°C. This result is of great importance for reducing the cost of thin film solar cells, in which ITO films serve as transparent conducting electrodes. We present a comparison of the properties of sputtered ITO films with those fabricated using a spray pyrolysis deposition technique, which is useful for some optoelectronic applications.

Keywords: thin film, indium tin oxide, DC sputtering, postannealing, soda lime glass, alkali-free glass

1. Introduction

Thin films of transparent conducting oxides (TCOs) have been widely used as a transparent electrode due to their low electrical resistivity and high transparency. As a degenerated semiconductor, ITO can be used as an antireflection layer in n^+-p optical sensors and as a flat panel displays including liquid crystal displays, organic light emitting diodes, and plasma displays [1]. Among the various TCOs, indium oxide films doped with tin (ITO) are widely transparent conducting electrodes in optoelectronic devices [3–6].

ITO thin films can be prepared using a wide variety of techniques such as thermal evaporation, chemical vapor deposition, electron beam evaporation, sol-gel, pulsed laser deposition, magnetron sputtering, and spray pyrolysis [2].

DC and RF magnetron sputtering using ceramic ITO targets is the most widespread method for deposition of ITO films on different substrates. Usually, such sputtering is carried out in a mixed argon-oxygen atmosphere. Normally, sputtering in a pure argon atmosphere is a more simple, cheap and highly controllable technique. However, a practical realization of this process using commercial ITO targets presents some difficulties. Commercial ceramic sputtering targets of ITO usually are fabricated from a mixture of high-purity In_2O_3 and SnO_2 powders (90:10 wt%), according to the solubility limits of Sn in In_2O_3 (12.4–15 mol% SnO_2) [7–9], using hydrothermal processes in the temperature range of 1300–1500°C. A key condition for obtaining ITO films presenting the best parameters is achieved when specific sintering techniques are used, which are also effective to tailor distinct properties of the target materials. X-ray diffractometer (XRD) peaks and the coloration of the ceramic target are references of the complete transformation to indium tin oxide. A thermal treatment in air or oxygen leads to a light-green coloring of the target, whereas a thermal treatment in reducing atmospheres (such as H_2, for instance) results in a black coloring [10]. This specific color of the target can be due to the presence of foreign phases such as metallic tin and/or blue-black tin oxide SnO due to the dissociative decomposition of SnO_2 into SnO + 1/2 O_2. At the same time, the unstable phase SnO can turn into Sn_3O_4 and pure Sn, and Sn_3O_4 will decompose into SnO_2 + Sn.

If only argon is used as the working gas for the sputtering, the deviation in stoichiometry of the target will be duplicated onto the film leading to a poor transparency [11–15]. Therefore, a certain amount of oxygen needs to be added into the working gas, otherwise, the films must be annealed in an air/oxygen atmosphere for obtaining the expected stoichiometry and high transparency; thus, a quasi-reactive deposition in argon mixed with reactive gas O_2 takes the place in contrast to sputtering in pure argon gas. Sputtering target obtained from AJA Corporation and used in this work had a black color that is not usual for light or dark-green color of the ITO targets sintered in an oxygen atmosphere. According to Ref. [15], the use of a reduction atmosphere during the sintering process leads to a black coloring of the target. Thus, the knowledge of the thermal history of the mixed powders, which determines their characteristics, is fundamental in order to determine the target quality, which in turn has a profound impact on the characteristics of the sputtered thin layers [16]. According to Refs. [17, 18], the ITO targets fabricated under laboratory conditions with sintering only in an air atmosphere at 1300°C, during 6 hours, present a light coloring close to that of the initial powders. The stoichiometry of the ITO target confirmed by XRD analysis allows for the fabrication of highly transparent ITO films in a pure argon atmosphere without oxygen adding and a postannealing procedure. Since the chemical content of the as-deposited films, fabricated in a pure argon atmosphere by using the AJA target, was far from stoichiometry and had a very low transparency, for this study, we used a thermal annealing in an oxygen atmosphere at different temperatures. The crystalline structure, the work function, and the optical and electrical properties of the ITO films were investigated.

Another objective of our work was to analyze comparatively the films fabricated with a post-annealing on two types of substrates, namely, low-cost soda lime and expensive alkali-free

glass (AFG) substrates. Currently, the cost of soda-lime glass substrates is 20–30 times lower than the cost of alkali-free borosilicate substrates. The applicability of such inexpensive glass substrates in a large-scale thin film solar cells production is a significant factor for reducing the cost of thin film solar cells, in which the ITO films are an integral part of the solar cell structure as the transparent conducting electrode. Nevertheless, some reported results [19, 20] show that the presence of a large amount of alkali ions (Na^+, K^+) in the soda-lime glass substrates is dangerous for the ITO film parameters if high deposition or annealing temperatures are used in the fabrication process. This is due to the out-diffusion of ions from the substrate into the ITO film, leading to the possible formation of secondary phases such as Na_2SnO_3 or $Na_2Sn_2O_5$ observed in SEM images [21]. These disadvantages from known published results were minimized by intercalating a barrier of TiO_2-SiO_2 composite films [19], or SiO_2 and Al_2O_3 films [20] between the ITO film and the soda-lime glass substrate.

2. Experimental

ITO films with a thickness of 200 ± 10 nm were deposited simultaneously on both, soda lime glass (SLG) (Corning 2947 Plain Microscope Slides) and alkali-free glass (AFG) (Corning 1737 near zero alkali aluminosilicate glass) substrates, using the AJA ATC Orion 5 UHV DC Magnetron Sputtering System with 2 inches heads. SLG and AFG substrates differ on their Na_2O content, which are 14% and less than 1%, respectively. The ITO target (90 wt% In_2O_3, 10 wt% SnO_2) was manufactured by AJA Corporation. Only pure argon was used as the working gas; the detailed deposition conditions can be found in our earlier reports [22, 23]. The substrate surface was not heated and maintained at a temperature below 50°C. The films removed from the sputtering system were thermally annealed during 60 min under a constant flow of a pure oxygen, at temperatures in the range of 200–500°C using a 50°C step, in an improved annealing equipment with a more uniform oxygen flux than the one used in an early published work [22].

The structural characterization of the ITO films was carried out using a Bruker D8 advanced X-ray diffractometer (XRD), with CuK_α radiation (λ = 0.15406 nm). Morphological features of the ITO films were examined using the Ambios USPM atomic force microscopy (AFM). The Keithley's Series 2400 source measurement unit and the Acopia HMS-5000 Hall Effect Measurement System were used for the electrical characterization. Optical measurements were carried out in the 400–1100 nm wavelength range using an F20 Filmetrics spectrometer.

3. Comparison of sputtered ITO films on SLG and AFG substrates

As-deposited, the ITO films grown at room temperature on both glass substrates were amorphous and black colored. The dark color of the films can be explained by nonstoichiometric film contents due to the inclusions of metals and indium, and tin sub-oxides present in the used sputtering target. Crystallization of the films in the (222) and (400) planes (**Figures 1** and **2**) with preferred grains (222) orientation was detected after a thermal annealing in oxygen at a temperature above 200°C.
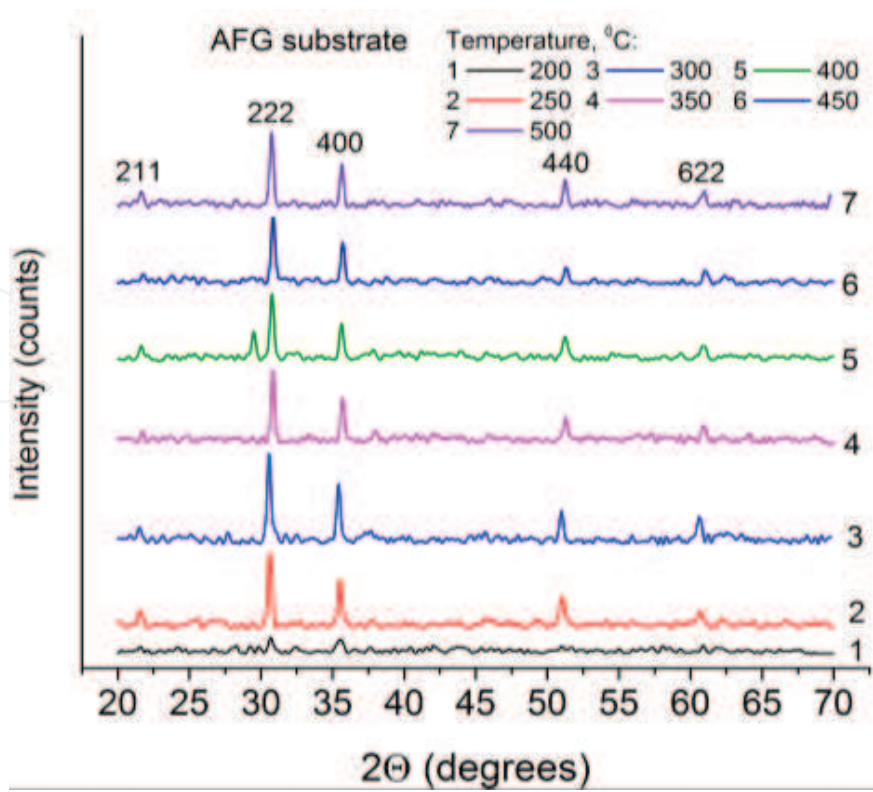
Figure 1. XRD diffraction patterns of the ITO films deposited on AFG substrates and annealed at different temperatures in an oxygen atmosphere.
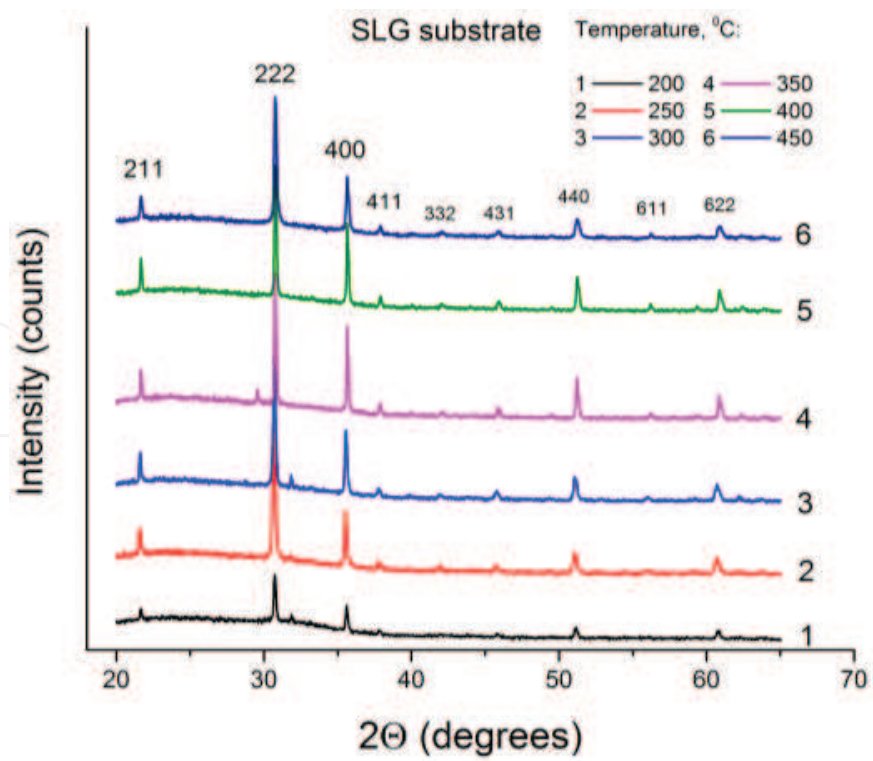

Figure 2. XRD diffraction patterns of the ITO films deposited on SLG substrates and annealed at different temperatures in an oxygen atmosphere.

The films deposited on both substrates and annealed at temperatures 200–450°C in the oxygen atmosphere are polycrystalline. Their XRD spectra (**Figures 1** and **2**) have a little difference in the height of peaks. The preferred grains orientation is (222). This result is consistent with the works presented by Raoufi and Alam [24] and Salehi [25], who obtained the same (222) predominant peak. The XRD peaks for the films deposited on SLG substrates are sharper than those from the films deposited on AFG substrates. The average crystallite size (s) was calculated from the (222) peak, as shown in **Figure 3**, using the well-known Scherrer formula [26].

The surface roughness of the films obtained from AFM analysis is shown in **Figures 4** and **5**. The substrate roughness serves as a reference.

From **Figures 4** and **5**, one can see that the roughness of the AFG substrates is significantly less than that for SLG substrates. However, the roughness of the ITO films deposited on the SLG substrates is less than that of the substrate roughness. We correlate this observed experimental fact with a planarization effect occurring during the film growing on the SLG substrate. A higher roughness of the SLG substrates serves as crystallization centers for the film growth that leads to a bigger crystallite size than that obtained for the ITO films deposited on ALG substrates. The reduction of the grains size of the ITO films deposited on the SLG substrates and annealed at temperatures above 350°C can be correlated with the beginning of the diffusion of Na^+ ions from the SLG substrate with the subsequent formation of secondary phases such as Na_2SnO_3 or $Na_2Sn_2O_5$ that inhibits the normal grain growth [20]. At the same time, the grains size of the ITO films on the AFG substrates is almost independent of the annealing temperature. **Figure 6** shows the dependence of the resistivity of the ITO films deposited on
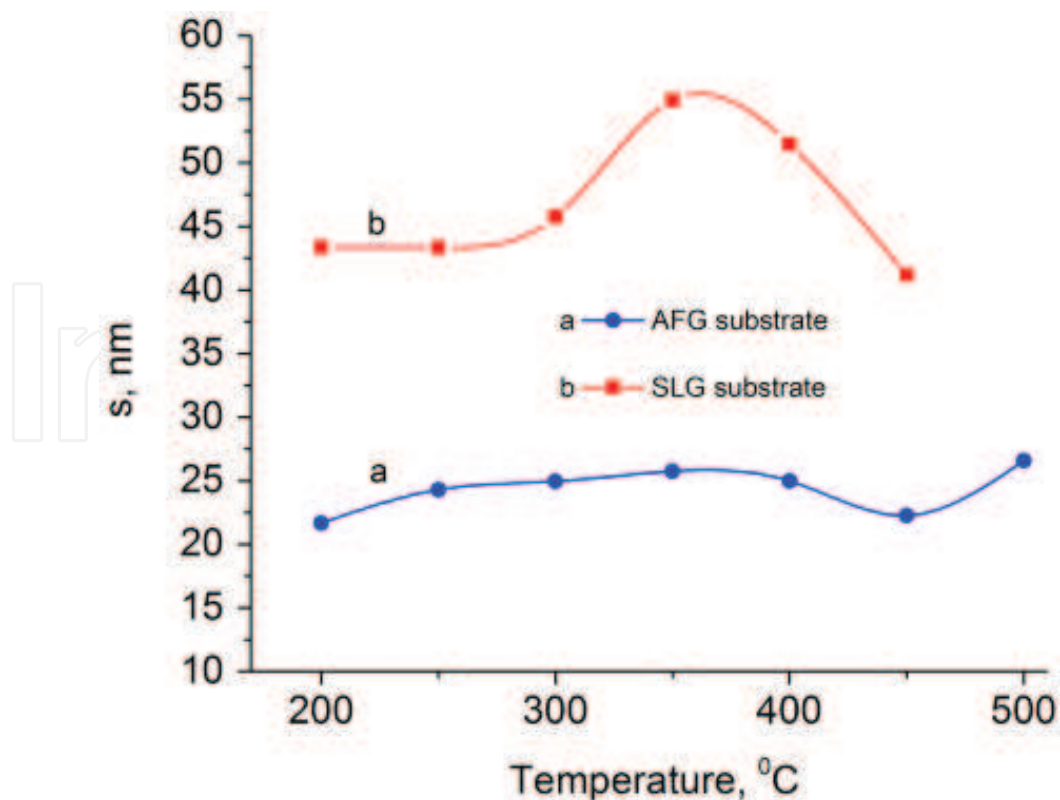

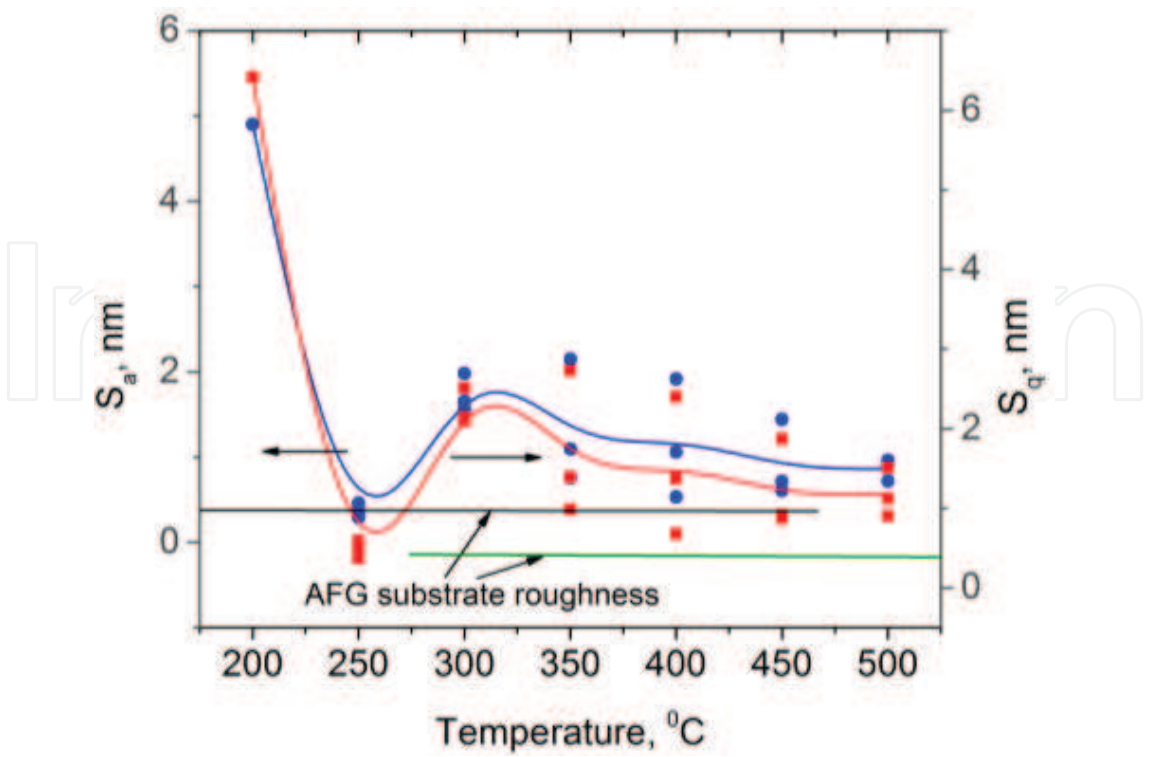**Figure 3.** The average crystallite size calculated from the (222) peak.

Figure 4. Average (S_a) and root mean square (S_q) roughness for the ITO film deposited on the AFG substrate and annealed in oxygen at different temperatures.
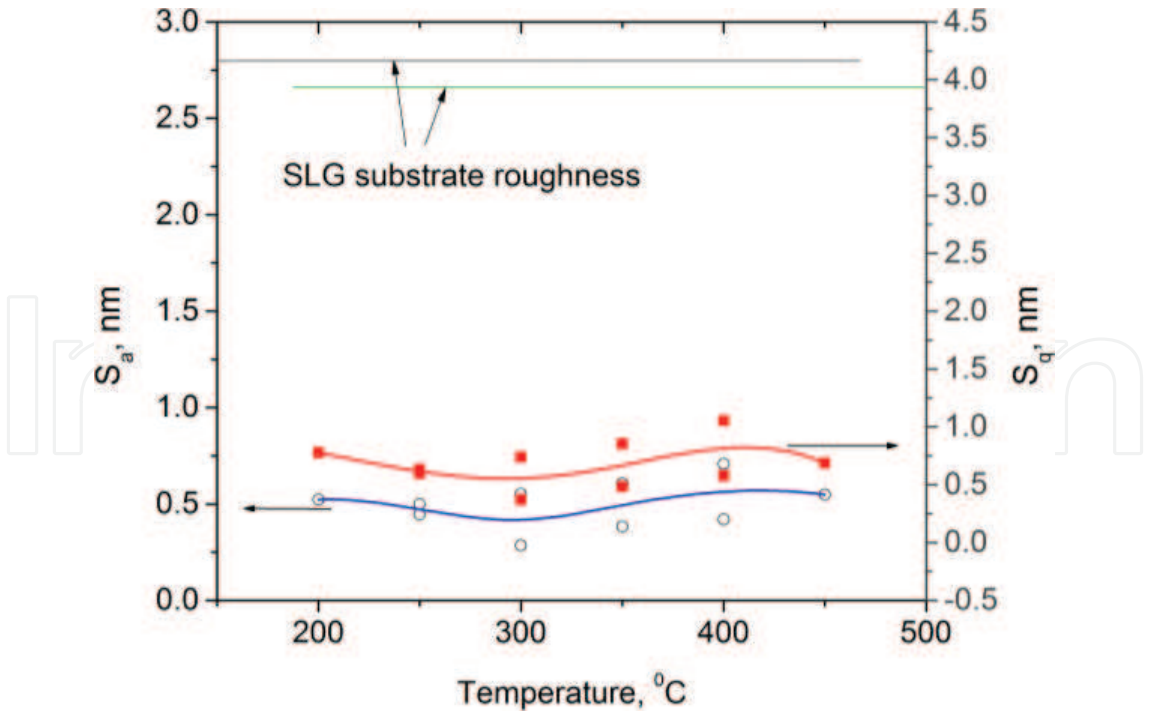

Figure 5. Average (S_a) and root mean square (S_q) roughness for the ITO film deposited on the SLG substrate and annealed in oxygen at different temperatures.
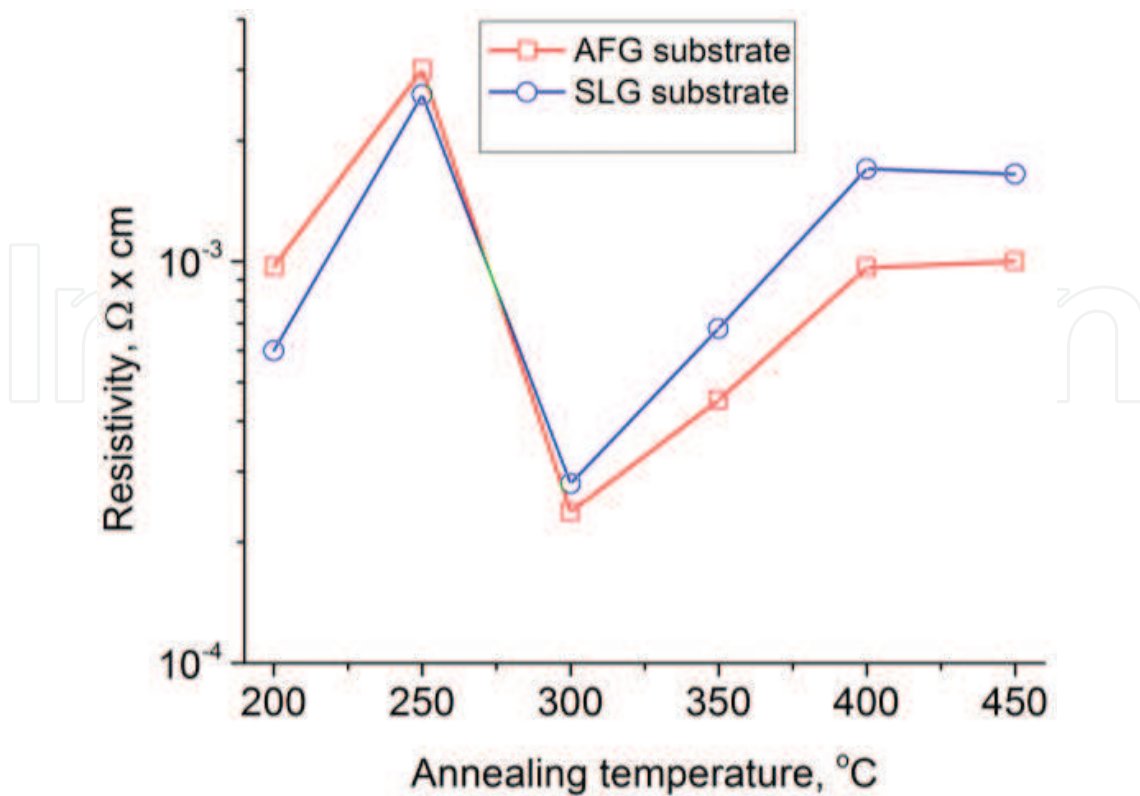
Figure 6. The resistivity dependence of the ITO films deposited on both types of substrates as a function of the annealing temperature in the oxygen atmosphere at different temperatures.

both types of substrates as a function of the annealing temperature in the oxygen atmosphere. The carrier concentration and carrier mobility obtained from Hall measurements are shown in **Figures 7** and **8**, respectively.

The as-deposited films are amorphous with a high concentration of nonstoichiometric contents such as metals and sub-oxides inclusions. This leads to a significant reduction of the resistivity in spite of the fact that the films were opaque and presented a black color. The film crystallization takes place at temperatures above 200°C. Higher annealing temperatures promote tin doping of the ITO films due to an effective replacement of the three-valence indium atoms by four-valence tin atoms in the crystal lattice of In_2O_3. A minimum value of the resistivity, $(2–3) \times 10^{-4}$ $\Omega \times cm$ (**Figure 6**), is obtained at 300°C for both types of substrates. At higher annealing temperatures, an increasing resistivity is observed due to a reduction of oxygen vacancies that are doubly charged donor impurity in the ITO films; it is higher for the ITO films deposited on the SLG substrates, which can be due to the effect of alkali ions out-diffusion from the SLG substrate [19, 20]. The same explanation can be applied to the carrier concentration reduction (**Figure 7**) at annealing temperature above 300°C. The increment of the carrier mobility (**Figure 8**) with annealing temperatures for the ITO films deposited on both types substrates comes from an enhancement of the films structural morphology as well as a reduction of defects at the grain boundaries.
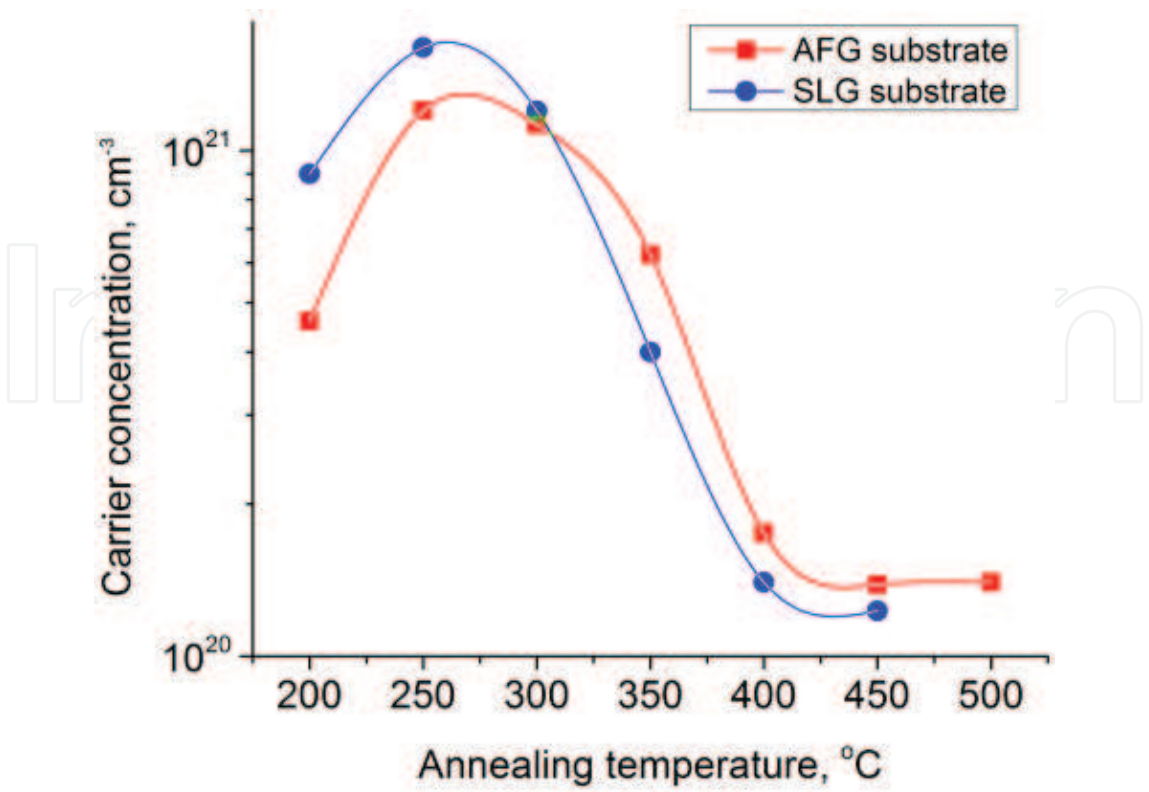
Figure 7. Dependence of carrier concentration on the annealing temperature for the ITO films deposited on AFG and SLG substrates.
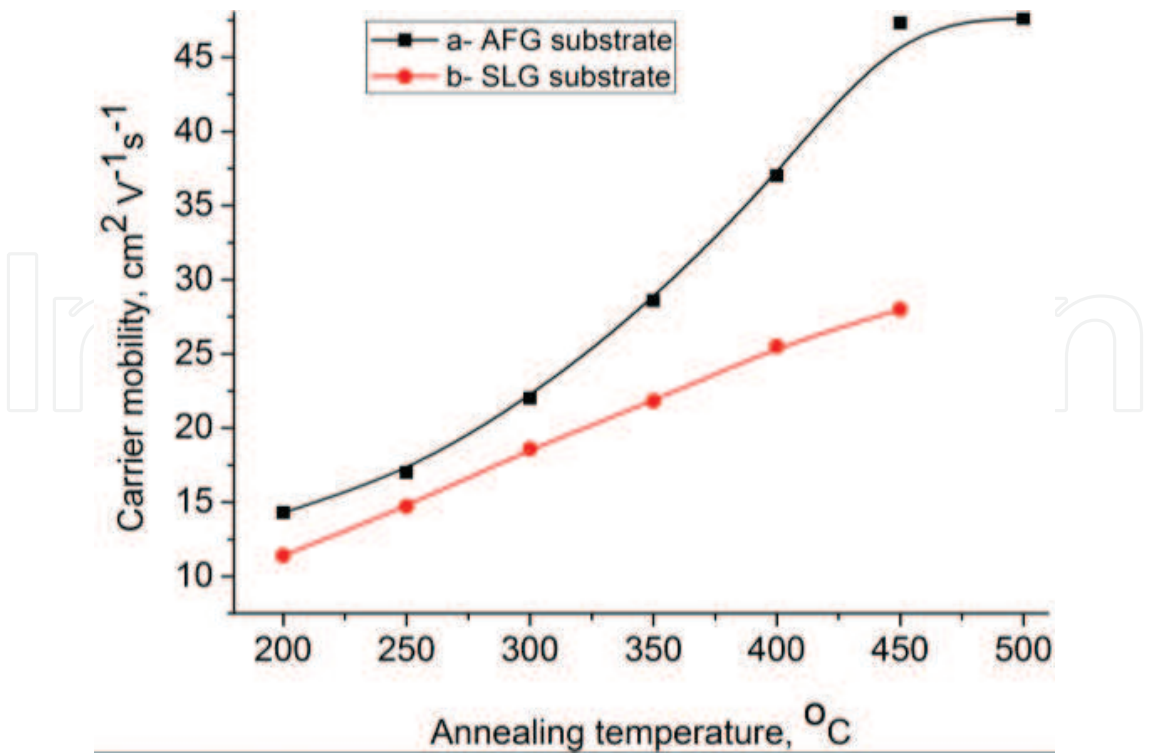

Figure 8. Dependence of carrier mobility on annealing temperature for the ITO films deposited on AFG and SLG substrates.

From the above-mentioned experimental results, we conclude that the highest structural and electrical parameters of the ITO films are obtained after a postannealing in the oxygen atmosphere at 300–350°C temperature range.

For practical applications of the ITO films as transparent conducting electrodes, the fabrication process needs to be optimized for obtaining a better balance between the transmittance (T) and the sheet resistance $R_s = \rho/d$, where ρ and d are the resistivity and the thickness of the film, respectively. However, a simultaneous obtaining of maximum transmission and conductivity is not possible to achieve in most cases. Haacke [27] proposed the figure-of-merit (FOM) $\phi_{TC} = T^{10}/R_s$ for comparison of films with different thickness and fabrication history. A higher FOM indicates a higher performance, this characterizes a film showing simultaneously a low sheet resistance (R_s) and a high transparency (T). However, the value of one of these parameters can compromise the value of the other. To avoid the influence of an interference effect in the thin films, the average integrated value of the films transmittance (T) at wavelengths in the range of 400–700 nm was used for the FOM value calculation. The dependence of integrated optical transmittance in visible spectral range (400–700 nm) of the ITO films deposited on AFG and SLG substrates is shown in **Figure 9** for different annealing temperatures.

Table 1 shows the structural, electrical parameters, the FOM for ITO films deposited on both glass substrates, AFG and SLG, after postannealing in the oxygen atmosphere at two different temperatures that are close to the optimal value.
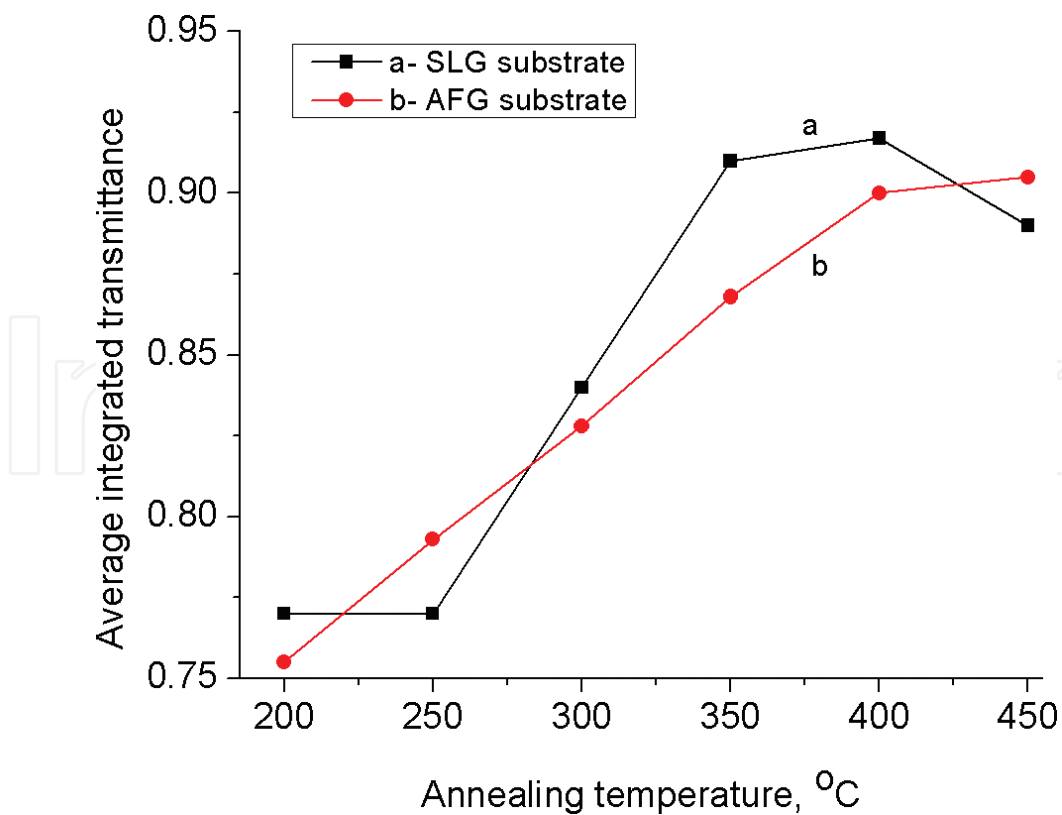

Figure 9. Dependence of the transmittance on annealing temperature for the ITO films deposited on SLG and AFG substrates.

Parameters/substrate	AFG	SLG	AFG	SLG
Annealing temperature (°C)	300	300	350	350
Substrate roughness (nm)	0.37	2.8	0.37	2.8
Ratio XRD peaks (222/400)	1.16	1.23	1.11	1.16
Grain size (nm)	25.0	45.0	25.0	55.0
ITO roughness (nm)	1.74	0.43	1.55	0.52
Specific resistance (10^{-4} Ω-cm)	2.2	2.8	4.2	6.8
Carrier concentration (10^{20} cm^{-3})	11.0	11.8	6.2	4.0
Mobility (cm^2 V^{-1} s^{-1})	25.0	17.5	28.0	23.0
Integral transparency	0.83	0.84	0.87	0.92
FOM (10^{-3} Ω^{-1})	14.1	12.5	11.8	12.8

Table 1. Comparison of properties of the ITO films deposited on AFG and SLG glass substrates after annealing in oxygen at 300 and 350°C.

From **Table 1**, the maximum $_{\phi TC}$ is observed for the films after postannealing in oxygen at 300– 350°C. At 300°C, where a minimum resistivity is observed, the films deposited on both substrates do not show a large difference in their FOM. Usually, ITO films present an FOM of the order of 10^{-3} Ω^{-1}. The highest known value of the FOM for commercially available sputtered ITO films is $(14–17) \times 10^{-3}$ Ω^{-1} [28, 29]. Also, we can compare the maximum value of the FOM obtained in this work (12×10^{-3} Ω^{-1}), with those reported for ITO films fabricated using other alternative methods: 6.6×10^{-3} Ω^{-1} by electron beam evaporation [30], 13×10^{-3} Ω^{-1} by sputtering [31], 13.6×10^{-3} Ω^{-1} by pulse laser deposition [32], 3.9×10^{-3} Ω^{-1} by spray pyrolysis [33], and 0.3×10^{-3} Ω^{-1} by sol-gel deposition [34].

We conclude that the ITO films sputtered in a pure argon atmosphere on low-cost SLG substrates, after annealing in oxygen at 300°C, are suitable for obtaining ITO films with high electro-optical parameters without the creation of a diffusion barrier between the substrate and the ITO film. The use of low-cost SLG substrates is an important factor for a significant reduction in the cost of thin film solar cells and modules for large-scale production. Moreover, a low annealing temperature allows for the fabrication of the ITO film on flexible plastic substrates. The ITO films, whose XRD spectra are shown in **Figure 10**, have been successfully fabricated on the Kapton substrate with annealing in oxygen at 300°C during 1 hour [23].

The incomprehensible preferred grains orientation (231) characteristics of a cubic film structure, or the (110) for hexagonal structure, need a further investigation. The electrical parameters of the 215-nm thick ITO films, deposited on Kapton plastic substrates at room temperature after annealing in an oxygen atmosphere during 1 hour at 300°C, are resistivity $\rho = 3.2 \times 10^{-4}$ Ω-cm, carrier concentration $n = 1.4 \times 10^{21}$ cm^{-3}, and mobility $\mu = 13.7$ cm^2/V-s.
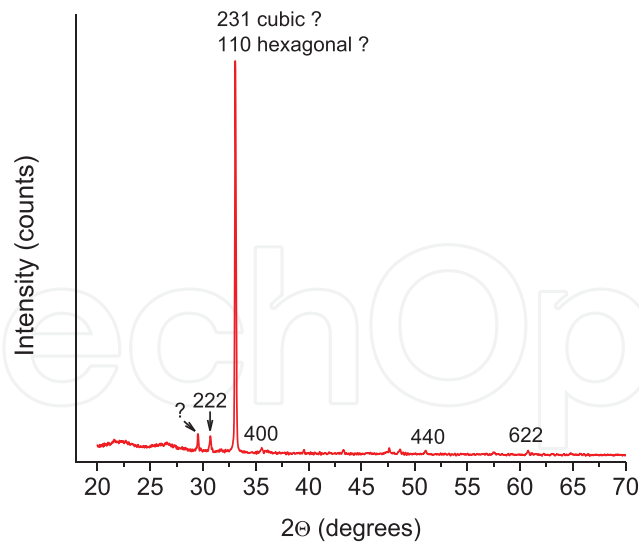
Figure 10. XRD spectra of the ITO film deposited on a Kapton substrate and annealed for 1 hour in oxygen at 300°C.

4. Comparison of sputtered and spray deposited ITO films

In this section, we present a comparison of the properties of the ITO films, fabricated using a two-step process for fabrication of films (sputtering an annealing), with those properties obtained using a one-step spray pyrolysis process [6, 23]; the second option is no expensive because it does not demand the use of sophisticated technological equipment. The spray pyrolysis is a stable and repetitive process very convenient for laboratory purposes and also for sufficiently large-scale production in the frame of small enterprises.

The spray pyrolysis technique was used for the deposition of 175–220 nm ITO films on an SLG substrate (Corning 1747). A glass atomizer was designed in order to produce small-size droplets. The SLG substrates were placed on a heater covered with a carbon disk in order to assure a uniform temperature, and the spraying was conducted using compressed air. The deposition rate was high at around 200 nm/min. For the ITO films deposition, 13.5 mg of $InCl_3 \times 4H_2O$ were dissolved in 170 ml of 1:1 water and ethylic alcohol mixture, with an addition of 5 ml of HCl to prevent hydrolysis. The different ratios of Sn/In achieved in the ITO films were controlled by adding in the solution a calculated amount of tin chloride ($SnCl_4 \times 5H_2O$). The substrate temperature was controlled using a thermocouple at $480 \pm 5°C$. The optimum distance from the atomizer to the substrate and the compressed air pressure were 25 cm and 1.4 kg/cm^2, respectively. We achieved a high deposition rate at around 200 nm/min. In order to prevent the deposition of the films under nonequilibrium thermal conditions, which can be due to a possible fast cooling of the substrate surface with the stream of the precursor and the compressed gas, periodical cycles of the deposition with durations of 1 s and intervals of 10 s were employed.

The X-ray diffraction patterns of the ITO films, prepared by spray pyrolysis at a substrate temperature of 480°C (**Figure 11**), show that these films are polycrystalline in nature for all the Sn/In ratios in the precursor and have a strong preferred columnar (400) grains orientation. Such

preferred grains orientation significantly differs from grains orientation for sputtered and annealed ITO films (**Figure 1**). The average grain size estimated from full width at half maximum (FWHM) of the (400) peak using the Scherrer formula [26] is 160 nm, this is bigger than the one obtained in sputtered and annealed films as well as the average roughness, $S_a = 30$ nm, determined from the analysis of AFM measurements.

Figure 12 shows the thickness and the sheet resistance (R_s) of the spray deposited ITO films prepared from the precursors with different values of the Sn/In ratio. The minimum $R_s = 12$ Ω/square or resistivity $\rho = 2 \times 10^{-4}$ Ω-cm was obtained for the films deposited using the solution with Sn/In = 5%. The dependencies of the carrier concentration and mobility on the Sn/In ratio are shown in **Figure 13**. Both parameters present their maximum for Sn/In = 5%. The posterior reduction of these parameters for a Sn/In ratio above 5% can be explained as due to the limit of solubility of tin in indium oxide and a possible formation of neutral complexes of tin inside the indium oxide lattice.
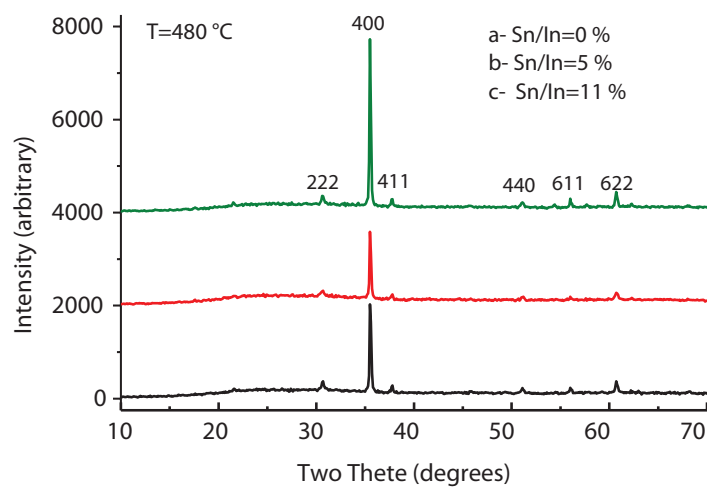

Figure 11. XRD diffraction patterns of the ITO films fabricated using solutions with different Sn/In ratios.
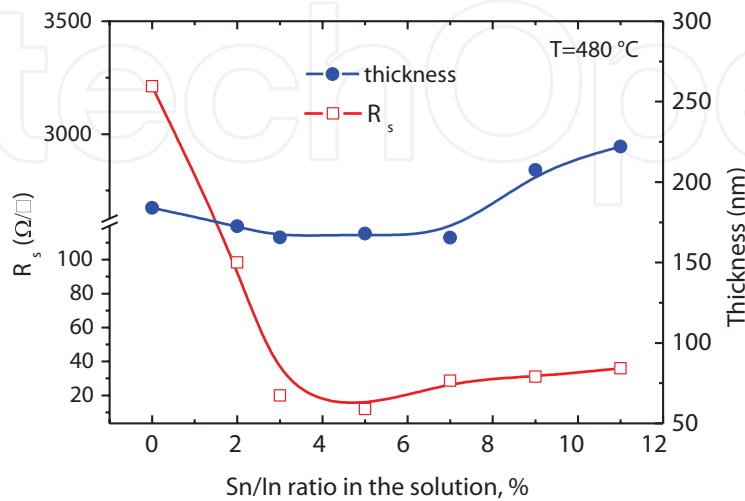

Figure 12. Sheet resistance and thickness of the sprayed ITO films fabricated using solutions with different Sn/In ratios.

The spray films deposited at 480°C, using the solution with a Sn/In = 5% ratio, showed the best electrical parameters, namely, a resistivity of 2×10^{-4} Ω-cm, a carrier concentration of 1.15×10^{21} cm^{-3}, and a mobility above 26 cm^2/V-s.

The optical transmittance (**Figure 14**) of the spray-deposited ITO films exceeded that of the sputtered/annealed films leading to a much higher FOM (**Figure 15**).

Comparison of properties of the ITO films deposited by sputtering and annealed in oxygen at 300°C with the properties of spray deposited films is shown in **Table 2**.

The practical optoelectronic applications (solar cell, hetero-photodiodes, and light emitting diode on silicon) of thin ITO films fabricated by spray pyrolysis are presented in our recently published articles [3–6].
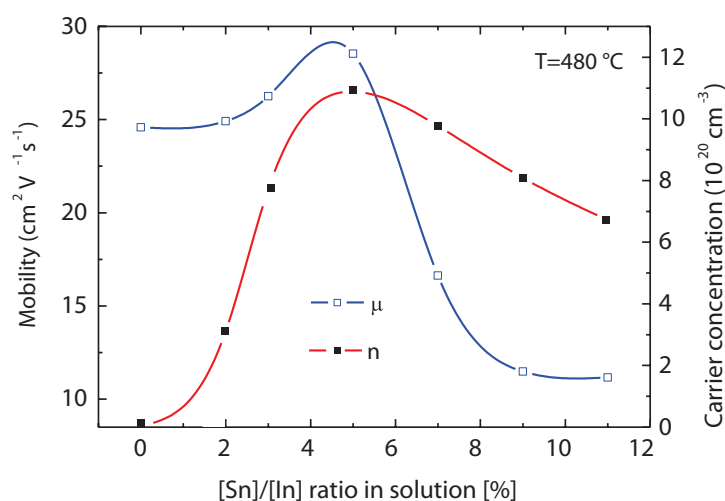

Figure 13. Hall mobility and carrier concentration of the sprayed ITO films fabricated using solutions with different Sn/In ratios.
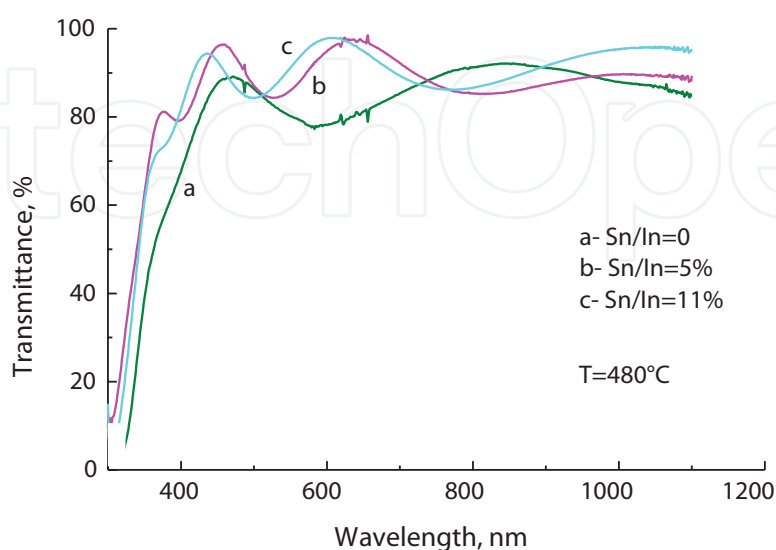

Figure 14. Optical transmittance spectra of the ITO films fabricated using solutions with different Sn/In ratios.
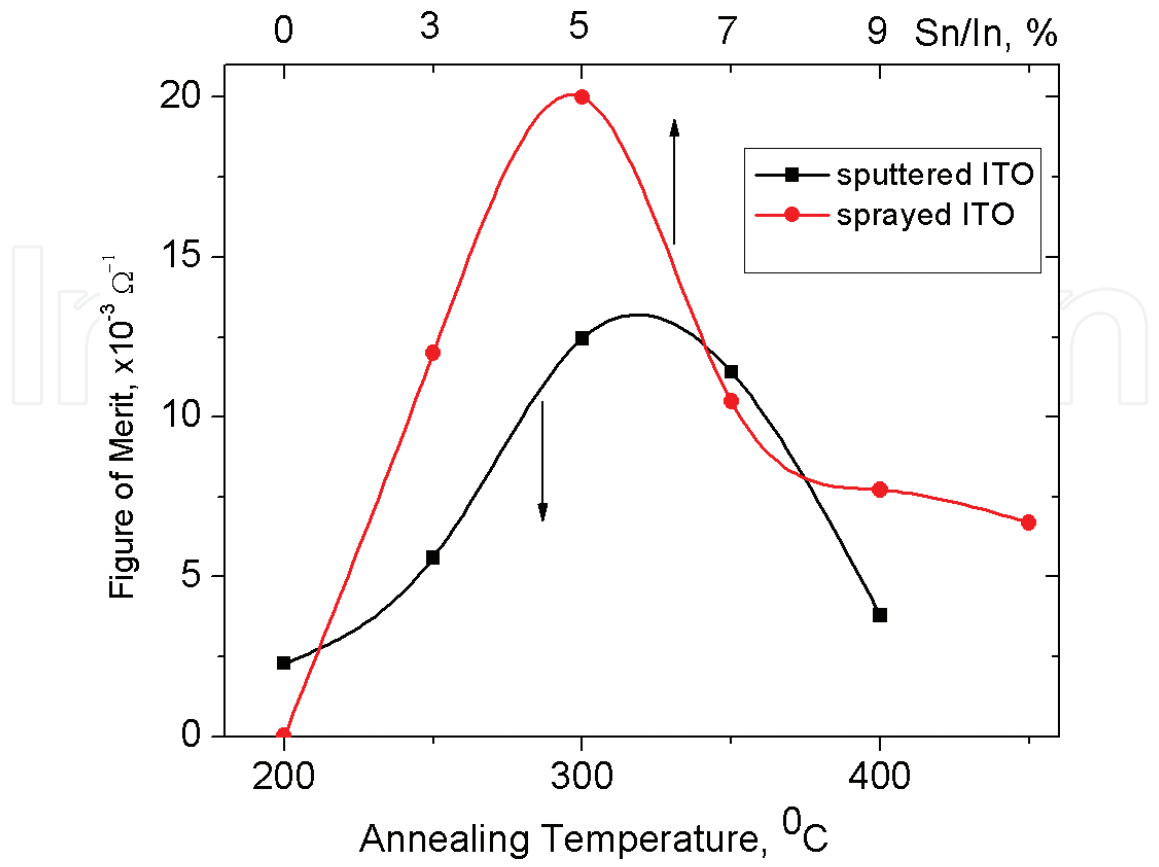
Figure 15. Comparison of the figure-of-merit (FOM) of the ITO films fabricated by sputtering with a subsequent annealing in oxygen at different temperatures, with the FOM of the films fabricated by spray pyrolysis using solutions with different Sn/In ratios.

Parameters	Sputtered and annealed ITO film on SLG	Spray deposited ITO film
Annealing temperature (°C)	300	-
Ratio Sn/In in solution (%)	-	5
Substrate roughness (nm)	2.7	2.7
Ratio XRD peaks (222/400)	1.23	0.032
Grain size (nm)	45.0	160
ITO roughness (nm)	0.43	30
Specific resistance (10^{-4} Ω-cm)	2.8	2
Carrier concentration (10^{20} cm^{-3})	11.8	11.5
Mobility (cm^2 V^{-1} s^{-1})	22.0	28
Integral transparency	0.83	0.85
FOM (10^{-3} Ω^{-1})	12.5	20

Table 2. Comparison of properties of the ITO films deposited by sputtering and annealed in oxygen at 300°C with the properties of spray deposited films.

5. Conclusions

The structural, electrical, and optical properties of sputtered and oxygen-annealed ITO films, deposited on alkali-free glass (AFG) and soda-lime glass (SLG) substrates, were studied. An important result of this work is the evidence that the sputtered ITO films in a pure argon atmosphere on low-cost SLG substrates, after annealing in oxygen at 300–350°C, show high optoelectronics parameters without the need of a diffusion barrier between the substrate and the ITO film. The electrical parameters of ITO films deposited on SLG substrates show a slight difference when compared to those obtained for the films deposited on AFG substrates at annealing temperatures no exceeding 350°C; the films deposited on SLG substrates exhibit better optical parameters (transmittance and value of FOM). Our conclusions are contrary to those reported in Ref. [20], where the ITO films on SLG substrates were annealed in a nitrogen atmosphere at 400°C. Our films were annealed in an oxygen atmosphere that leads to a binding of the diffused alkali ions with oxygen atoms diffused through the ITO films forming a stable oxide or peroxide at the film-substrate interface.

According to our results, we can conclude that SLG substrates are suitable for obtaining ITO films with high electro-optical parameters and small roughness due to a planarization effect. The use of such low-cost substrates is an important factor for a significant reduction in the cost of thin film solar cells and modules for large-scale production.

A comparative study shows that films deposited by spray pyrolysis present better electrical and optical properties than those obtained by sputtering. Hence, such a simple and low-cost deposition method is very promising for the fabrication of high-quality ITO films working as the ohmic contact in thin film optoelectronic devices as well as the active transparent conducting electrode inefficient solar cells and photodetectors with potential surface-barriers.

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