



# A comparative study on structural, optical, photoconductivity properties of In and Al doped ZnO thin films grown onto glass and FTO substrates grown by spray pyrolysis process

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## ABSTRACT

A comparative study on In and Al doped ZnO thin films grown onto glass and FTO substrates by spray pyrolysis technique has been done. The obtained results show that the structural shape, crystalline, grain size parameter, transmittance, absorption coefficient and optical band gap and photoconductivity of the ZnO films were changed by the nature of the substrate used.

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## 1. Introduction

From all the transparent conducting oxides (TCO's) studied in recent years, zinc oxide has emerged as one of the most promising materials, due to its optical and electrical properties, high chemical and mechanical stability, together with the cost of raw materials, when compared to the currently used TCO's (ITO, SnO<sub>2</sub>). Zinc oxide (ZnO) is a TCO, which has recently been extensively studied. ZnO is a II–VI semiconductor, mostly *n*-type, with a wide band gap of 3.3 eV [1] and recently it becomes *p* type [2] and it is a good photoconductor [3–5]. It has been used in gas sensors [6], surface acoustic wave (SAW) devices [7,8], solar cells [9] and blue light emitting diodes [10]. ZnO films have been grown by several techniques. These methods are spray pyrolysis, either pneumatic [11] or ultrasonic [12,13], chemical vapour deposition [14–16], molecular beam epitaxy [17,18], sol–gel [19], pulsed laser deposition [20], sputtering [21–25], hydrothermal technique [26] and wet chemical process such as chemical vapor deposition (CVD), homogenous precipitation method under solvothermal condition [27–29].

In present study, we report on the structural and optical study, and photoconductivity (PC) effects of zinc oxide thin films grown onto two kinds of glass substrates with different electrical properties: purely insulator and coated with a transparent conductor. We compare the structural parameters, the optical transmittance, the absorption coefficient, the optical band gap and the PC of ZnO grown onto glass and FTO-coated glass, separately.

## 2. Experimental details

Two different substrates were used, corning glass 7059, Near-Zero Alkali baria alumina borosilicate of density 2.76 g/cm<sup>3</sup>, refractive index 1.5440 at 435.8 nm, transmittance (*t* = 0.5 mm) >90% (380–2200 nm) and fluorine-doped tin oxide (FTO) supplied by Pilkington Group Ltd. The starting material used was zinc acetate dihydrated, Zn (CH<sub>3</sub>COO)<sub>2</sub>·2H<sub>2</sub>O [30,31]. Doping sources were aluminium nitrate nonahydrated (Al (NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O) and indium(III) chloride (InCl<sub>3</sub>) [11,12,30]. Both, precursor and doping compound were dissolved in methanol. The zinc acetate concentration was 0.1 M and the doping ratio Al/Zn, In/Zn were 2% in the solution [11,30,32]. Details on the experimental setup are given elsewhere [11,12]. Substrate-to-nozzle distance was fixed at 5 cm. Solution flow rate was kept at 1 ml/min. Deposition time was always 5 min. The substrate temperature was fixed at 300 °C. The thicknesses of the films were determined by Talystep Dekkat 3<sup>st</sup> (profilometer) to be 500 nm onto glass for both, the undoped, AZO and IZO films, 150 nm for undoped ZnO and 1 μm for AZO and IZO onto FTO respectively.

Optical measurements were carried out in the range of 300–1000 nm with a Shimadzu UV-3600 double beam spectrophotometer.

In order to obtain the photocurrent response of the samples, two metals contacts were deposited onto the samples using silver paint. After that, the samples were kept in dark up to 10 h. Immediately after a constant voltage *V* of 10 V is

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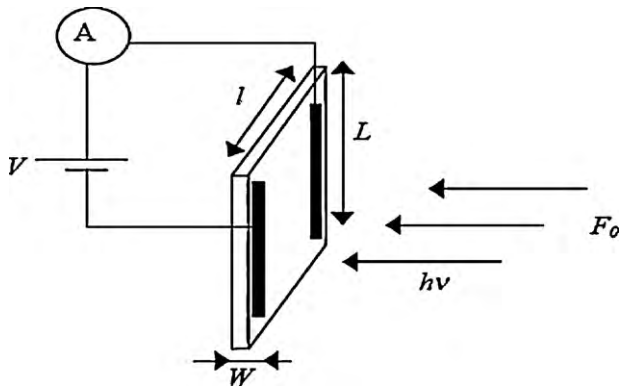


Fig. 1. Schematic set up of photoconductivity.

applied between two bars of aluminium evaporated on the sample as illustrated in Fig. 1. In the dark, the current  $I$  pass between two contacts. When  $F_0$  the photon flow (photon/s) uniformly illuminates the thin layer of the dimension  $l \times L \times W$ , an increase of  $\Delta I_L$  dark current is observed directly linked to increased conductivity of the material. The contacts are not illuminated during the experiment to ensure that the measures describe the properties of the film rather than photovoltaic effect level contacts. The parallels aluminium bars contacts have dimensions of 4 mm of separation and 15 mm length are investigated during 20 s under dark conditions, 20 s under  $100 \text{ mW/cm}^2$  illumination by xenon power and 20 s under dark again [12,30]. The current across the samples was recorded automatically. The process of PC measurements for the samples grown onto FTO is 60 s under dark, 120 s under light and 60 under dark.

### 3. Results and discussion

#### 3.1. Structural characterizations

X-ray diffraction patterns show that the zinc oxide, undoped and doped, grown onto FTO present a high crystallinity, as seen in Fig. 2 compared to those grown onto glass and the crystallinity improves for AZO and IZO, respectively, as evidenced in Fig. 2(b) and (c) [13]. We conclude that IZO presented a good crystallinity when it was deposited on glass substrate whereas AZO enhanced its crystallinity on FTO substrate. The diffraction peaks related to FTO-coated glass are signed by \* (star) as shown in Fig. 2. Using Scherer's formulae (1), the grain size  $G$  calculated was listed in Table 1 and shown in Fig. 3 when both the substrate glass and FTO were used.

$$G = \frac{0.94\lambda}{\beta \cos \theta} \quad (1)$$

We remarked that ZnO/FTO has a high value of grain size than ZnO/glass in the direction (100) and (101), but in the direction (002) IZO/glass exhibits a maximum of grain size, as listed in Table 1. Thus, this result concorded with the best photoconductivity  $400 (\Omega \text{ cm})^{-1}$  [31] and high transmittance equals to 100% in visible range for IZO/glass [33].

The preferred orientation of ZnO films was quantitatively evaluated by texture coefficient (TC), which was calculated, for the mains peaks from X-ray diffractograms. It is expressed as follows for a plane ( $hkl$ ),

$$TC = \frac{I_{hkl}}{\sum_N I_{hkl}} \quad (2)$$

When  $I_{hkl}$  is the measured intensity of the plane ( $hkl$ ) which is normalized, and  $N$  is a reflection number. As listed in Table 1, the AZO, IZO grown onto glass exhibit TC equal to 0.85 respectively for [002] direction and undoped onto glass shows TC equal to 0.58 for [100] direction.

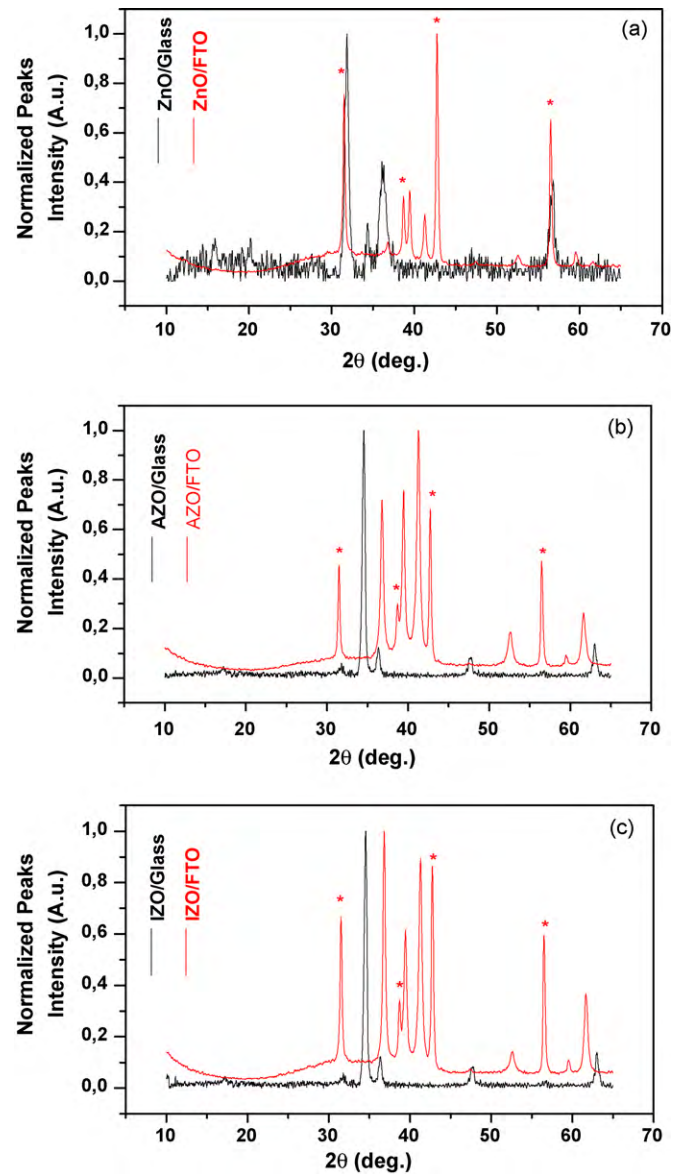


Fig. 2. Structural properties of ZnO/glass and ZnO/FTO (a), AZO/glass and AZO/FTO (b) and IZO/glass and IZO/FTO (c).

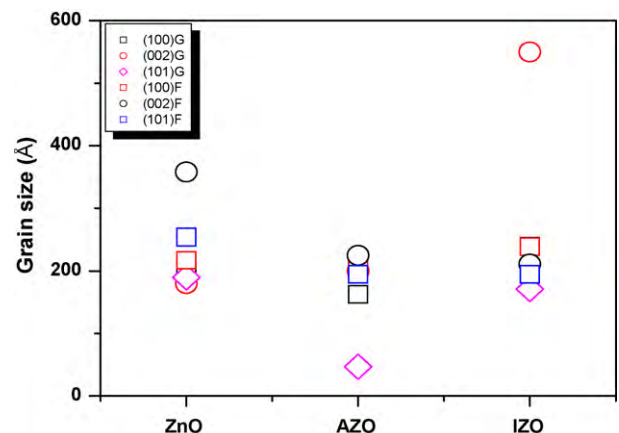


Fig. 3. Grain size variation of the peaks (100), (002) and (101) of doped and undoped zinc oxide grown on the glass (G) and FTO (F) substrates.

**Table 1**  
Structural, optical and photoconductivity parameters of undoped, Al and In doped ZnO films grown on glass and FTO substrates [11–13,30].

Substrate material	$(hkl)$	$G$ (Å)			TC			$T_{\max}$ (%)			$E_g$ (eV)			$\eta$		
		ZnO	AZO	IZO	ZnO	AZO	IZO	ZnO	AZO	IZO	ZnO	AZO	IZO	ZnO	AZO	IZO
Glass	(1 0 0)	460	163	240	0.58	0.04	0.05	66	88	100	3.25	3.29	3.28	15.66	1.19	2.38
	(0 0 2)	260	200	550	0.13	0.85	0.84									
	(1 0 1)	150	47	171	0.27	0.10	0.10									
FTO	(1 0 0)	217	206	239	0.21	0.29	0.40	82	65	66	3.26	3.23	3.29	1.31	1.14	1.11
	(0 0 2)	358	225	211	0.45	0.30	0.24									
	(1 0 1)	254	195	195	0.34	0.40	0.36									

### 3.2. Optical properties

The transmittance comparison of zinc oxide undoped and doped onto glass and FTO substrates was considered in the wavelength range 300–1000 nm as depicted in Fig. 4. Mutually, for ZnO/glass and ZnO/FTO films, transmittance increases rapidly in a narrow range of wavelength from 300 to 400 nm [34], and then, the transmittance varies slowly and the difference between two curves was distinctly observed as described in Fig. 4(a), and was confirmed by the average line in visible and near infrared range. The curves joined at 68% for 922 nm which corresponds to infrared. This is due to difference in film thickness, roughness and material substrate. Excluding ZnO, transmittance of the AZO and IZO films indicates oscillation only with glass substrate, caused by film thickness which was varied from 0.3 to 0.8  $\mu\text{m}$  [11,12,30]. Transmittance obtained around 80% was for ZnO/FTO [13], and Subramanyam has found a transmittance around 80–90% for undoped zinc oxide [35] which was in agreement with our result. For the AZO, the transmittance parameter reached 88 and 65%, and for IZO 100 and 65% respectively for glass [12] and FTO [13]. The transmittance reached 85% as cited in Jayaraj et al. paper [36]. Tiburcio obtained a transmittance equals to 95 for IZO [37].

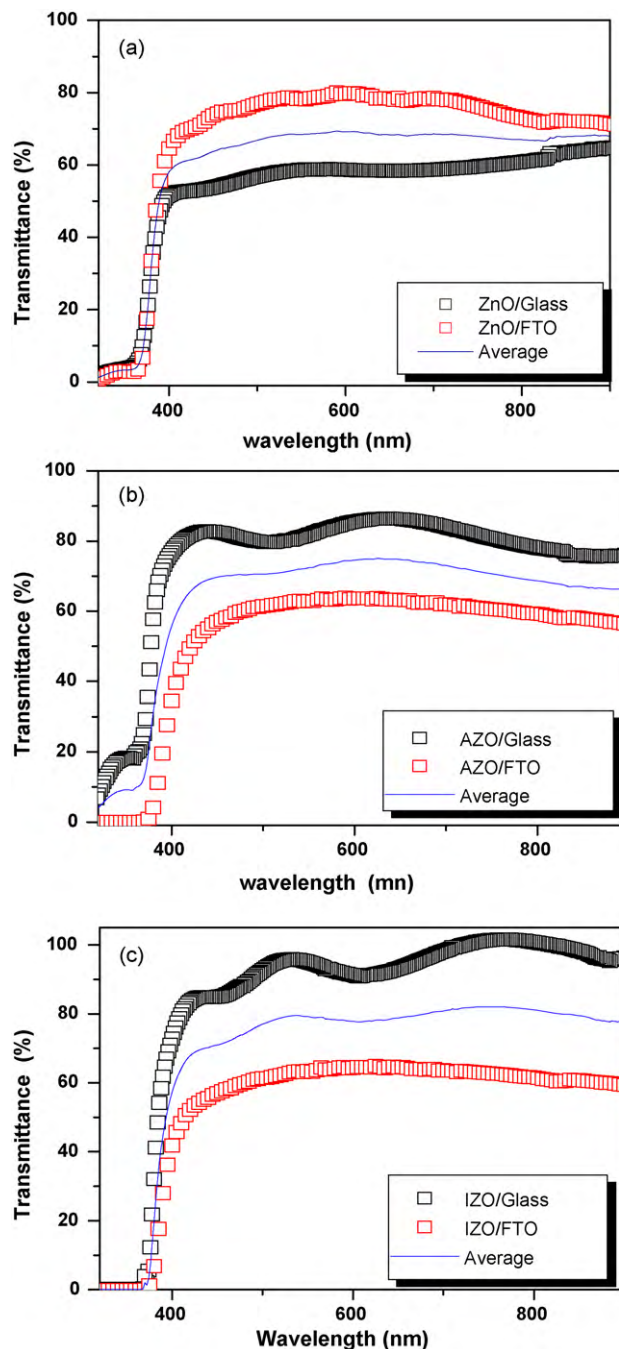
We can confirm that both AZO and IZO grown on the glass substrate exhibit the high transmittance, while for the undoped zinc oxide, the reverse was remarked [31], this fact was due to doping. Thus the choice of material candidate as TCO may be the undoped ZnO onto FTO and doped ZnO with Al [31] and In [11,12] onto glass in the range of UV, VIS and IR. These differences were also caused by the film thickness [38,39]. We concluded that ZnO had good optical transparency in the visible region [40,19]. The absorption and optical band gap are depicted in Fig. 5. We plotted  $(\alpha h\nu)^2$  versus photon energy (eV), the extrapolation intercepting the photon energy axis given the optical band gap  $E_g$ . It varied between 3.23 to 3.29 and 3.25 to 3.29 eV respectively for zinc oxide on FTO and zinc oxide on glass substrates [31]. These ranges of optical band gap were also found by Subramanian for ZnO in the range 3.20–3.32 eV, for ZnO deposited by DC reactive magnetron sputtering onto glass substrates held at a temperature of 663 K and oxygen partial pressure of  $1 \times 10^{-3}$  mbar [35], by Jayaraj for AZO (Al/Zn was 2%) prepared by RF sputtering,  $E_g$  is found to be 3.30 eV [36] and by Tiburcio for IZO, with In/Zn was 1, 2 and 6% in solution, grown on soda-lime glass substrates via spray pyrolysis,  $E_g$  is around 3.27 eV [37].

### 3.3. Electrical photoconductivity

Fig. 6 shows the photoconductivity results of ZnO, AZO and IZO deposited onto glass and FTO substrates respectively. The PC variation follows the same shape for the undoped zinc onto glass [12] as well as for ZnO onto FTO. The ratio of photo to dark conductivity is denoted  $\eta$  [48,49], which is listed in Table 1.

#### 3.3.1. The samples deposited on glass

The doped zinc oxide deposited onto glass shows an increasing variation till a saturation around 120 and 400  $(\Omega \text{ cm})^{-1}$  respectively



**Fig. 4.** Transmittance spectra, ZnO/glass and ZnO/FTO (a), AZO/glass and AZO/FTO (b), IZO/glass and IZO/FTO (c) [13].

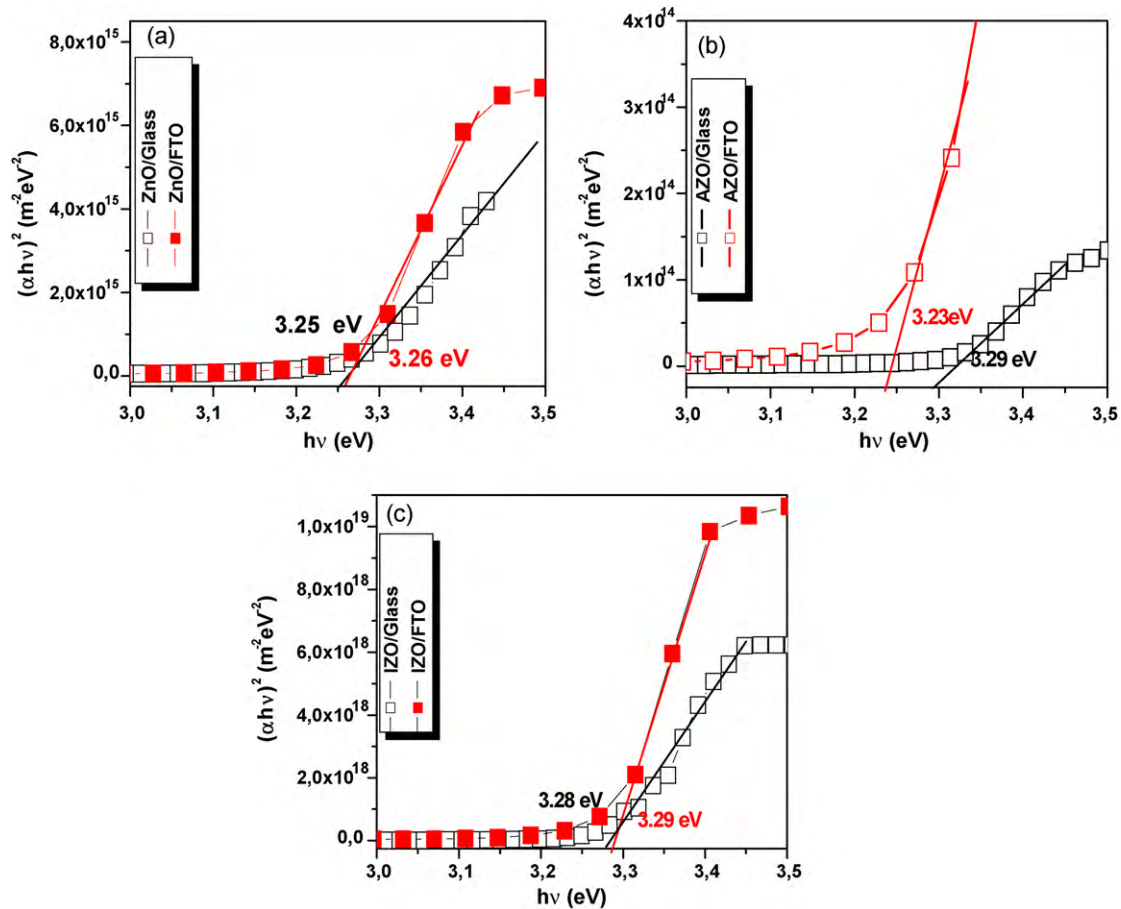


Fig. 5. Absorption parameter, band gap  $E_g$ , ZnO/glass and ZnO/FTO (a), AZO/glass and AZO/FTO (b), IZO/glass and IZO/FTO (c).

for AZO and IZO [31]. An increase in photoconductivity of ZnO was observed and then reaches a maximum of  $64 (\Omega \text{cm})^{-1}$  and it is decreased in dark. AZO film gives a PC two times more than those of ZnO, as shown in Fig. 6(a) and (b). We remark that the photoconductivity increases with the doping. The effect of illumination is to neutralize negative charge in the surface states, Electron-hole pairs created in the bulk of crystallites mainly recombine via radiative of Shockly-Read Hall mechanisms. Holes can be captured by deep traps at the grain boundary resulting an increase in number of free electrons which are unable to recombine. This detail can be explained by the removed oxygen from the grain boundary and this caused a decrease in the density of acceptors states at the grain boundary, resulting in the capture of a small numbers of bulk electrons by these state acceptors. For undoped films, the decay observed in the photoconductance is due to adsorption of oxygen. Absorbed oxygen captures an electron from the conduction band and became chemically absorbed, accompanied by a decrease in photoconductivity. We observed a very slow decay photoconductivity in doped films [11,12,30]. This is attributed to a slow electron-hole recombination. The same conclusion was reported by Studenikin and Zhang [50,51]. The conductivity was not assigned by the exposition of the thin films to illumination in the AZO and IZO cases as shown in Changhyun paper [52]. As a consequence, under illumination step the current generated was so important in IZO and AZO, became weak in ZnO. Photoconductivity of ZnO increases and a fast decay was remarked [43–46]. As listed in Table 1, the calculated values of  $\eta$  are higher for ZnO and IZO.

### 3.3.2. The samples deposited on FTO

When the absorption of radiation results in a decrease in the dark conductivity of a material, the phenomenon is called negative photoconductivity. The negative PC was only depicted in IZO/FTO sample in the time range 60–90 s under illumination as shown in Fig. 6(f), negative PC has been reported by others authors such as Hye Ryong Kim [41]. This negative PC in ZnO may due to the exciton [44]. It was anomalous, negative PC in IZO/FTO was caused by existence of traps level energy  $E_t$ , which was higher than the conduction band level  $E_c$ , and some of the electrons in the conduction band have then enough energy to hop into the trapping levels under illumination exposure, so the charge carrier concentration fall in the conduction band [40,46]. Borshchevskii suggests that this effect is due to defects and impurities in the crystals, these imperfections increasing the volume polarization under illumination and hence decreasing the apparent conductivity [53], the negative PC for ZnO has been also reported by Borisov [46,54]. He contended that the action of illumination was to create excitons which could collide with thermally ionized excess Zn atoms, raising electrons from the valence band to the levels corresponding to the Zn atoms, and thus freeing holes which became available for recombination with free electrons to reduce the dark conductivity. The occurrence of negative photoconductivity requires that:

- (1) The thermal ejection of electrons from levels II ( $E > E_F$  where  $E_F$  is Fermi energy level) must be slower than the recombination of electron and hole at levels I ( $E < E_F$ ) as sketched in Fig. 7,

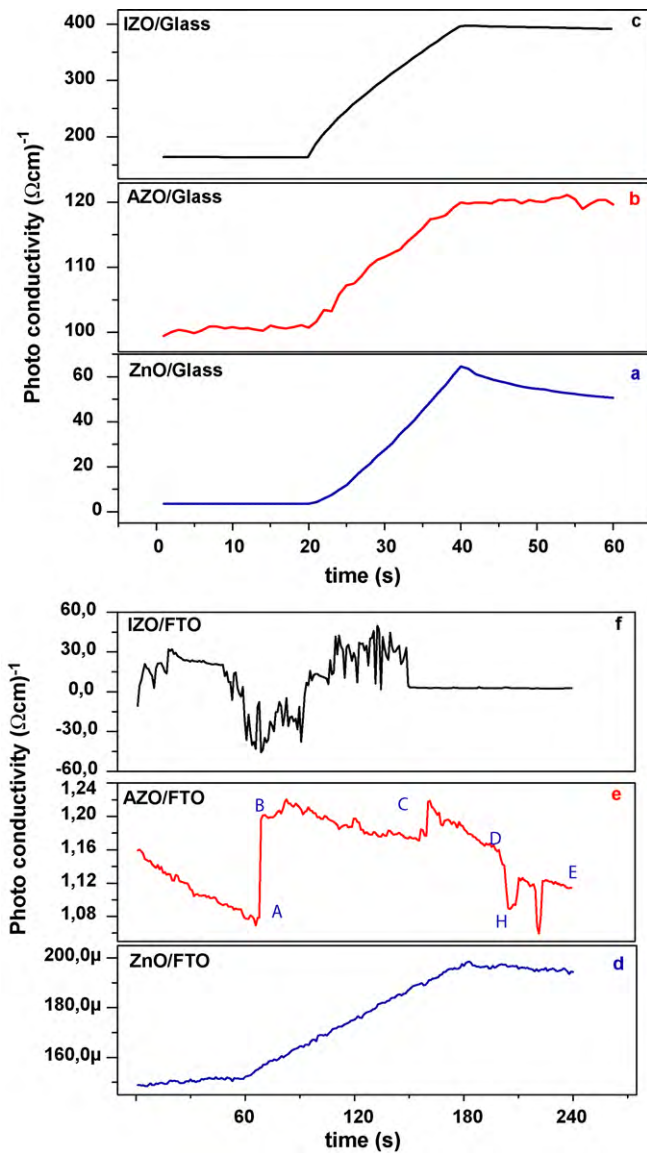


Fig. 6. Photoconductivity spectra versus time of ZnO/glass (a), AZO/glass (b), IZO/glass (c) and ZnO/FTO (d), AZO/FTO (e), IZO/FTO (f).

- (2) holes must not recombine directly with electrons in levels II,
- (3) levels II must lie above the Fermi level,
- (4) the cross section of centers II for majority carriers must be much less than the cross section of centers I, and
- (5) the density of centers I and their cross section for minority carriers must not be too small.

The necessity for the fulfilment of most of these conditions explains why negative photoconductivity is observed so rarely.

In practice, the conditions for negative photoconductivity are most nearly satisfied by centers which can exist in the crystal either singly or doubly charged. In an *n*-type material, such as is indicated in Fig. 7, for example, if the centers II correspond to doubly negative centers when occupied, requirements (1) and (4) are automatically fulfilled. Negative photoconductivity is associated with the presence of multiply charged centers above the Fermi level, whereas positive high-sensitivity photoconductivity is associated with the presence of these same types of centers below the Fermi level. FTO material may create these branches in the PC for AZO and IZO. The defects caused by oxygen desorbed might produce these branches [44]. AZO/FTO exhibited a huge increase by a factor up to 6000 in comparison with ZnO/FTO. Under illumination the undoped ZnO presented a quasi linear increase except AZO and IZO. This persistent PC was a phenomenon observed in ZnO/glass case also cited in literature [39,42,43,48], and also for undoped zinc oxide on FTO.

The grain size, the preferred orientation coefficient, the transmittance, the optical band gap, as well as the PC effects could be correlated as summarized in Table 1. IZO Films with thickness 500 nm shows the better photoconducting properties due to presence of optimum grain size (550 nm), in agreement with result found by Mridha [47], the preferred orientation coefficient assessed at 0.84 the transmittance reached 100%,  $E_g$  is equal to 3.28 eV and PC ratio  $\eta$  found for IZO deposited on glass is 2.38.

For AZO/FTO, the shallow traps have a decay time of about 40 s as can be seen in Fig. 6(e). These are hole traps with levels lying above the top of the valence band. Typical rise and decay curves, illustrating the behaviour of these traps are shown in Fig. 6(e). The effects of the shallow traps are shown in Fig. 6(e). The light is turned on at point A. Between A and B, the electrons and holes produced by light reach equilibrium in a hole lifetime between B and C, hole trapping occurs. When the light is turned off at point C, the current drops to D because of the recombination of excess electrons and holes. The shallow trap decay manifests itself in the continuing current in the region D to E. The magnitude of the difference in current between points C and B is about the same as the difference in current at D above the dark current H.

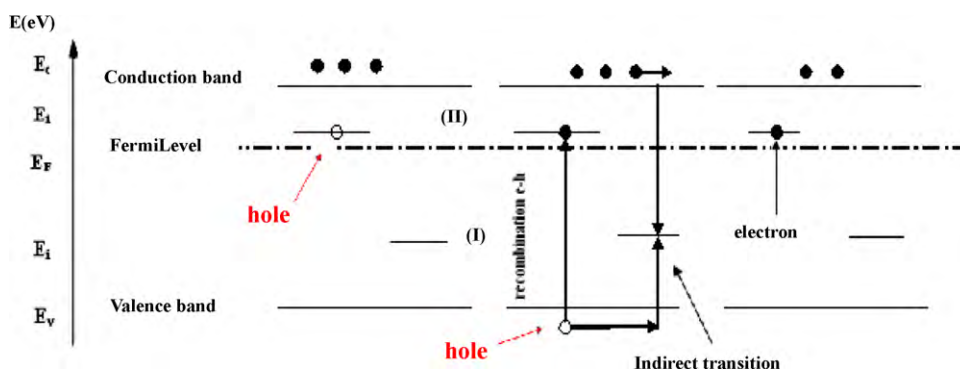


Fig. 7. Energy-level diagram of the steps involved in negative photoconductivity according to Stöckmann [46] (conduction band ( $E_c$ ), crystal imperfection level ( $E_i$ ), Fermi level ( $E_F$ ), valence band ( $E_v$ ), energy level is given in eV).

#### 4. Conclusions

This study was beneficent because it demonstrated difference in properties of thin film of zinc oxide undoped and doped grown separately on glass and FTO. These differences showed firstly the structural shape and the crystallinity, the grain size parameter. Secondly the optical parameters were affected such as transmittance, absorption coefficient and optical band gap. Finally the photoconductivity was varied with substrate material. Indeed, these properties differences were caused by the influence of material and level doping, film thickness which were investigated by many researchers recently and also by material substrate. IZO/glass exhibited grain size around 550 Å according to (002), a transmittance of 100%,  $E_g = 3.28$  eV, PC equals to  $400 (\Omega \text{ cm})^{-1}$ . However, IZO/FTO demonstrated a negative PC response under illumination— $45 (\Omega \text{ cm})^{-1}$  at 70 s, according to (002) grain size was 211 Å, transmittance reached 66% for red light (~663 nm) and optical gap was 3.29 eV. We conclude that the high grain size, transmittance and photoconductivity values were obtained with In: ZnO/glass. Finally both substrate material and doping affect the structural, optical and photoconductivity parameters.

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