Study of γ-rays Enhanced Changes of the ZnO:Al Thin Film Structure and Optical Properties

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Received: 27 January 2014 / Accepted: 17 February 2014 / Published: 23 March 2014

In this work thin film samples of 300 nm thickness with composition ZnAlO were deposited on glass substrate by DC sputtering technique. The effect of the atmosphere gas contents during sputtering was studied by using Ar for some of the prepared samples and $Ar+O_2$ mixture for the others. Additional investigations were also done for the effect of the heat treatment of the samples for comparison with gamma irradiation. The samples were irradiated by three gradual gamma rays doses using ⁶⁰Co source. The x-ray diffraction patterns showed that the deposited films were of amorphous structure and converted partially to crystalline form by irradiation. Optical properties of the investigated samples were compared with that of unirradiated ones for comparison. In general, it was found that the optical parameters of the samples showed decrease in the transmittance after irradiation and the calculated optical energy gap was decreased. On the other hand, the heat treatment of the samples increased the energy gap. The data were analyzed and discussed in the light of gamma rays interaction with the investigated thin films.

Keywords: AZO thin film, DC sputtering, gamma irradiation, optical properties, TCO materials

1. INTRODUCTION

Transparent conductive oxides (TCO) has extensive potentials in electronic applications due to their suitable optical and electrical properties. The actual and potential applications of TCO thin films include solar collectors, light emitting diodes and flat-panel displays [1-7]. Among those are the oxides

of Indium, Tin, Zinc and Cadmium as well as their doped elements which have been investigated by many groups around the world [8-9]. The ZnAIO was recently used instead of the conventional films like InSnO due to its high transmittance, non-toxicity, suitable resistivity, and low cost [3,4]. Zinc oxide is a II-VI compound semiconductor with optical band gap of 3.37 eV [10]. Its optical properties were efficient for using in several applications such as solar energy, electronic devices [11-17] sensors devices [18]. The properties of the thin film depend on the method of preparation and the treatment conditions like heating or using several surrounding atmospheres during preparation. Sputtering technique is simple and effective method to prepare different TCO materials in homogenous form and with variety of thicknesses (nm- μ m) [19]. This technique allows using Ar gas or other type of gas mixture atmosphere during preparation which can change the crystal structure of the prepared film. It was reported that using O₂ or H₂ fractions during deposition of AlZnO could change the optical transmittance and electrical resistivity [19-22]. Moreover, the wide verity of film thickness and heat treatment could change the microstructure, electrical and optical properties for the ZnO, Al-ZnO [23,24].

In the recent years, modification of materials properties by irradiation gained much interest by several laboratories [25-27]. The effect of γ -irradiation on TeO and TeO₂ thin films was reported [25,26]. In this study physical properties were modified by irradiation with different doses of gamma rays for real-time dosimetry purpose [25]. Furthermore, the effect of electron beam irradiation on structural, optical, and electrical properties for Al doped ZnO with different O₂ fraction have been investigated [28]. Similarly, the effect of gamma radiation on the optical properties of Se₉₀In_{10-x}Sn_x chalcogenide and thin films have been reported [29].

According to the above mentioned studies, it is noticed that little attention has been paid to comparison between irradiation effect and other conditional treatment on the optical conditions of AZO thin film. Therefore, we study in this work the enhanced changes occurred by using several conditions of preparation of ZnAIO thin film and compare these changes with that due to gamma irradiation. This may give added impetus on tuning the structural and optical properties of the AZO films. The results may help in development of the gamma rays dosimeteric tools by evaluating the relation between gamma doses and the tuned parameters of the investigated thin films.

2. EXPERIMENTAL

Thin film samples of composition ZnAlO were deposited on a glass substrate using UNIVEX 350 sputtering unit with dc power model TD20 and thickness monitor model INFICON AQM 160. The source target with composition ZnAlO (Al 20wt%, 99.9% purity) from Cathay Advanced Materials Limited, China. The glass substrates were first cleaned with acetone, rinsed in deionized water and then dried in inert atmosphere. Thereafter, the substrates were placed on the rotating holder of the DC sputtering unite. Two sets of the thin film samples were deposited by DC plasma discharge at room temperature using pure Ar (99.999%) and Ar:O₂ (O₂ 2%) mixture gas. The power of the plasma was maintained at 10 W and the gas flow rate of 5 SCCM was introduced through a mass flow controller. The substrate holder was rotated at rate of 2 rpm. The film thickness of 300 nm was

obtained under deposition rate of 0.03 nm/s. After preparation, a group of the prepared samples were annealed at temperature of 500 $^{\circ}$ C for 2 hours in air.

Gamma-rays irradiation was carried out for all the investigated samples in a cavity of ⁶⁰Co irradiator (medical sterilizer type CM-20) in the cyclotron facility, Nuclear Research Center of Egyptian Atomic Energy Authority with average dose rate of 1.4kGy/h. Three groups of the samples were exposed to three different doses: $D_1=201.6$ kGy, $D_2=302.4$ kGy and D3=470 kGy. Finally, the whole groups of the samples were assigned according to the preparation and irradiation conditions as given in table 1.

Sample assignment	Description
R-AR-D0	R: room temperature, AR: Ar gas (99.999%)
R-AR-D1	D0: unirradiated, D1: dose1, D2: dose2, D3: dose3
R-AR-D2	
R-AR-D3	
H-AR-D0	H: heat treatment, AR: Ar gas (99.999%)
H-AR-D1	D0: unirradiated, D1: dose1, D2: dose2, D3: dose3
H-AR-D2	
H-AR-D3	
R-O-D0	R: room temperature, O: (Ar: 98%, O ₂ : 2%)
R-O-D1	D0: unirradiated, D1: dose1, D2: dose2, D3: dose3
R-O-D2	
R-O-D3	
H-O-D0	H: heat treatment, O: (Ar: 98%, O ₂ : 2%)
H-O-D1	D0: unirradiated, D1: dose1, D2: dose2, D3: dose3
H-O-D2	
H-O-D3	

Table 1. Description of the investigated ZnO:Al samples according to treatment conditions.

The phase structural of the unirradiated and irradiated samples was analyzed using XRD diffractometer of type X'Pert Graphics employing Cu K_{α} radiation ($\lambda = 0.15405$ nm). The optical properties were measured using a double beam UV-Vis spectrometers type JASCO-V-670 in the wave length range 200-1500 nm, where a blank glass substrate was used as a reference to eliminate the effect of the glass substrate over which the film was grown. Moreover, a blank glass substrate was also irradiated with the same dose of that corresponding to the investigated sample to be used as base line in the transmittance and absorbance measurement spectra.

3. RESULTS AND DISCUSSION

3.1. XRD spectra

All the XRD patterns showed amorphous structure of the prepared AZO films and no obvious peaks of other phases were detected as shown in Fig. 1. The XRD of R-Ar-D0 sample revealed that

there are two humps around 25° and 30° , while the sample H-Ar-D0 showed broad hump. This broad hump of the unirradiated samples (R-AR-D0 and H-AR-D0) indicates the amorphous structure of the prepared film which reveals that heat treatment didn't improve the crystallinity of the prepared samples (Fig. 1-a,b). After irradiation, the spectra of the irradiated samples R-AR-D1, R-O-D1, H-O-D1, H-AR-D1 indicated that there was a tendency to crystallization around 33° as seen in Fig. 1. The gamma rays irradiation caused partial transformation from amorphous structure to crystalline phase, which is corresponding to the peak (002) of the AlZnO hexagonal Wurtzite structure [24]. On the other hand, there is no change in the spectra before irradiation for the samples that was prepared under mixed Ar+O atmosphere.

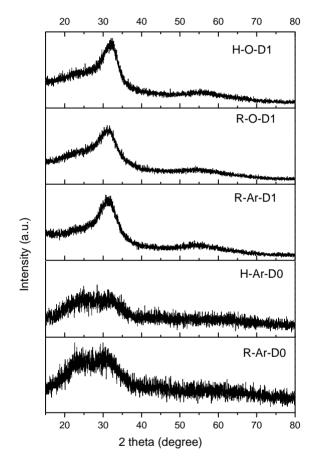


Figure 1. x-ray spectra of the as-prepared and irradiated AZO thin films.

3.2 Optical properties

3.2.1 Transmission spectra

It was observed that all of the prepared thin films were light-yellow to the eye and highly transparent. The color becomes darker with irradiation. The optical transmittance spectra for the prepared samples with different conditions of heating and irradiation are shown in Fig.2(a-d). All films had high transmittances in the visible region (400–700 nm) and a strong absorption in the UV region.

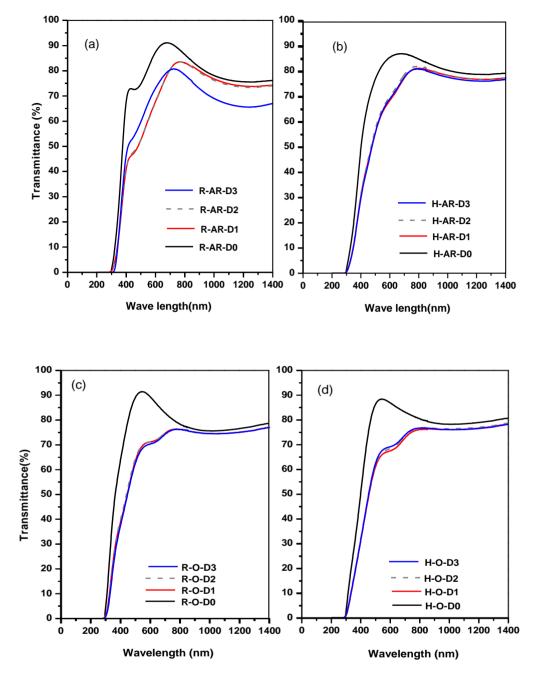


Figure 2. UV spectra of the gamma irradiated AZO samples with different conditions of preparation: (a) as-deposited, (b) annealing, (c) as-deposited using Ar+O mixture during sputtering, (d) annealing after preparation using Ar+O mixture.

The as-deposited ZnAlO film (R-AR-D0) shows one characteristic absorption peak at 600 nm which exhibits shift by irradiation to the longer wavelength by values of 100-160 nm as shown in Fig. 2-a. Comparing with Fig.2-b, smooth hump starts at 500 nm was noticed for the spectra of heat treated samples (H-Ar-D0, D1, D3) may be due to decreasing the surface roughness as was reported in previous studies [30,31]. In general, maximum changes on the transmittance were obtained at dose D1, where the curves showed lower values for all samples (Fig.2c,d). The decrease in transmission with irradiation is due to formation of color centers in the thin film which causes more darkness in

comparison with the unirradiated samples. The absorption edges for the treated samples didn't show any significant change from that of untreated samples whether by heating or irradiation or oxygen atmosphere.

3.2.2 Optical energy gap

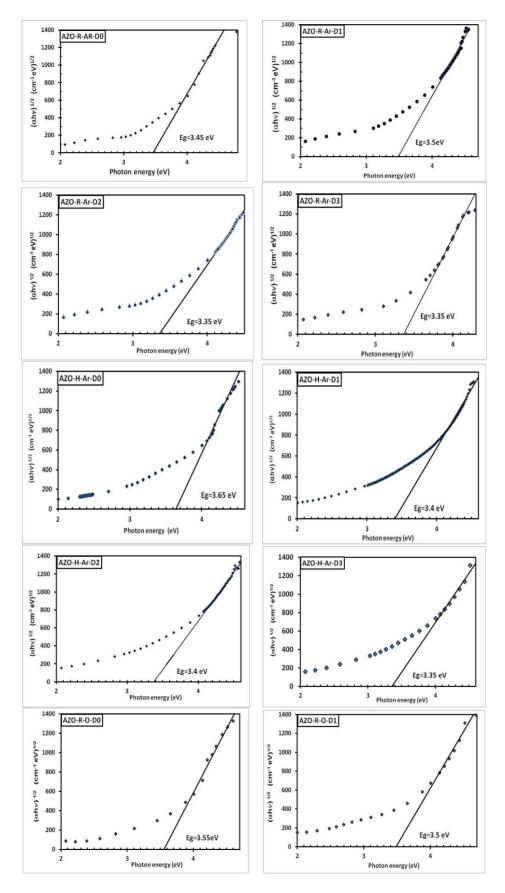
The optical energy gap (E_g) value could be calculated using the formula:

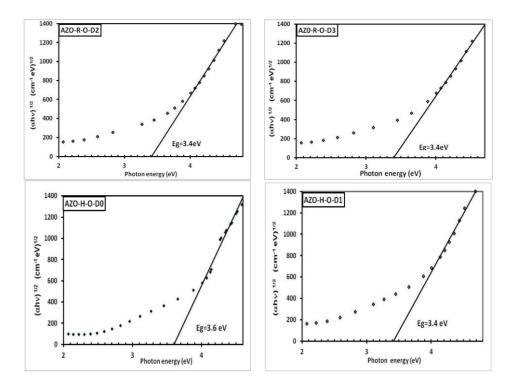
$$\alpha hv = C(hv-E_g)^n$$
, $\alpha = \frac{1}{d} ln(\frac{1}{T})$ (1)

where, the constant C is a parameter that depends on the transition probability and the constant n characterizes the direct (n=1/2) and indirect (n=2) allowed transition, α is the absorbance, T is transmittance and d is the film thickness. By considering the indirect transition (n=2), the $(\alpha hv)^{1/2}$ was plotted as a function of hv as shown in Fig. 3. The optical band gap E_g is determined by extrapolating the linear portion of the obtained curves to zero absorption [32,33]. The determined value of E_g for the as-deposited film in Argon atmosphere at room temperature (R-AR-D0) is 3.45 eV, which is close to that previously reported by Wang et al., (3.345 eV) [34,35]. Table 2 gives all the determined numerical values of the band gaps at different conditions of preparation and irradiation. Fig. 4-a,b represent the values given in Table 2 as a function of the absorbed dose. It is noticed that energy gap of the heated samples (H-Ar-D0, H-O-D0) is higher than that of the as-prepared ones (R-Ar-D0, R-O-D0). This could be explained through Burstein-Moss effect which describe the movement of Fermi level in the conduction band due to heating [24,36,37]. After irradiation with doses D1, D2 and D3 the band gap was decreased for all the samples prepared under the above mentioned conditions (Fig. 4a,b). The decrease percentages in every irradiation dose were amounted to 1.4-6.9 %. This could be attribute to formation of localized states due to structure defects. The increase of carriers localized states lead to decrease in the transition probabilities into the extended state, thus reduce the band gap as described in [38-40]. The following equation calculate the band gap widening (ΔEg) as a function of the carrier concentration (n_e) in a degenerate semiconductor [41,42]:

$$\Delta E_g = \frac{h^2 (3/\pi)^{2/3}}{8m^*} n_e^{2/3}$$
⁽²⁾

where, h is Planck's constant and m* is the electron effective mass in conduction band. Table 2 gives the changes of the numerical value of the carrier concentrations as the band gap increases for as deposited and heat treated samples at every gamma dose. The corresponding reduction in band gap energy by irradiation may be due to excitation of non-bonding electrons into conduction band with their subsequent increases on localized states. In addition, with increasing particle size can lead to quantum confinement [43,44].





- **Figure 3.** Plots of $(\alpha hv)^{1/2}$ as a function of incident photon energy to determine the optical band gap at several conditions of preparations and irradiations.
- **Table 2.** Changes in the numerical values of the band gap and charge density of the investigated samples with irradiation at different doses D1, D2 and D3.

Sample	D_0		D_1		D_2		D ₃	
	Eg(ev)	$n_e(cm^{-3})$	Eg(ev)	$n_e(cm^{-3})$	Eg(ev)	$n_e(cm^{-3})$	Eg(ev)	$n_e(cm^{-3})$
AZO-R-AR	3.45	-	3.5	2.02×10^{50}	3.35	5.72x10 ⁵⁰	3.35	5.72x10 ⁵⁰
AZO-H-AR	3.65	1.61x10 ⁵¹	3.4	2.25x10 ⁵¹	3.4	2.25x10 ⁵¹	3.35	2.96x10 ⁵¹
AZO-R-O	3.55	5.72×10^{50}	3.5	2.02×10^{50}	3.4	1.05×10^{51}	3.4	1.05x10 ⁵¹
AZO-H-O	3.6	$1.05 \mathrm{x} 10^{51}$	3.4	1.61x10 ⁵¹	3.4	1.61x10 ⁵¹	3.35	2.25x10 ⁵¹

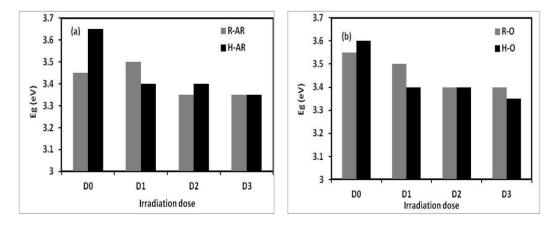


Figure 4. Variation of the optical band gap of the AZO samples due to irradiations effects and other treatment conditions: (a) Using Ar atmosphere (b) Using Ar+O₂ mixture.

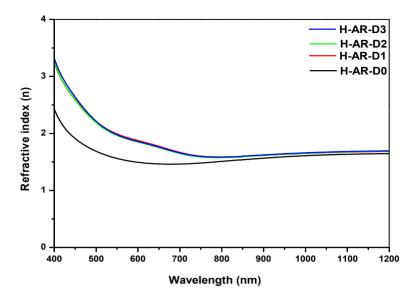


Figure 5. Curves of the refractive index as a function of wavelength for the AZO samples.

3.2.3 Refractive index

The refractive index can be obtained using the relation [45,46]: $n = \frac{1 + \sqrt{R}}{1 - \sqrt{R}}$ (3)

where, R is the reflectance. Fig. 5 represents the obtained refractive index as a function of the wavelength (nm) which can be considered normal dispersion behavior. It is seen that after irradiation, the values of refractive index showed increments with a value 40 % at 400 nm and 6% at 1200 nm. This increase was observed for the heat treated samples that were irradiated by three doses D1, D2 and D3. This may be attributed to the crystal defects caused by irradiation which changed the color behavior of the films. Similar behavior was reported in a study on gamma irradiation of Fe-doped barium titanate thin films [47]. As an extended calculations for the optical parameters, we used Wample and Didomenico model [48,49] which is based on single oscillator formula to calculate the oscillator energy (E_0) and dispersion energy (E_d). The formula could be written in the form:

$$(n^{2}-1)^{-1} = \frac{E_{o}}{E_{d}} - \frac{1}{E_{o}E_{d}}(h\nu)^{2}$$
(4)

where, n is the refractive index and hv is the photon energy in eV. Plots of $(n^2-1)^{-1}$ versus $(hv)^2$ for the heat treated sample, as an example, at irradiation doses D1, D2, and D3 are shown in Fig. 6 together with that of unirradiated one H-Ar-D0. From the slope $(1/E_dE_o)$ and the y-axis intercept (E_o/E_d) the values of E_d and E_o were determined for all the linear curves. The obtained values are given in Table3 and represented in Figs. 7a and b for comparison. It is noticed that by irradiation with the dose D1 the oscillator energy (E_0) was decreased by a factor ~11% for all the samples (R-AR, H-AR, R-O, H-O) and no further changes was observed after irradiation with doses D2 and D3 except that of the sample (R-AR) which was increased at D3. This effects indicate that the bounded charges becomes more

localized after irradiation due to the crystal defects occurred by irradiation. From Fig. 7b it is noticed that the dispersion energy (E_d) was increased by irradiation with dose D1 for the samples H-AR, H-O and R-O, and no further changes was observed for the doses D2 and D3 where E_d reached maximum value. For the sample (R-AR) E_d showed significant increase at D3 in comparison with the other samples. This changes reflects the degree of dispersion due to irradiation effects.

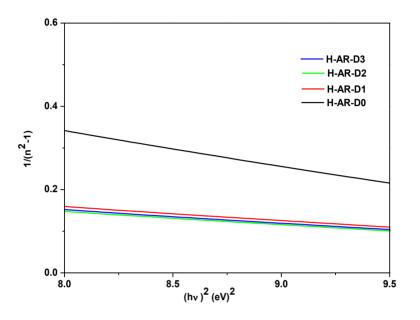


Figure 6. Plots of $(n^2 - 1)^{-1}$ versus $(hv)^2$ for the heat treated sample.

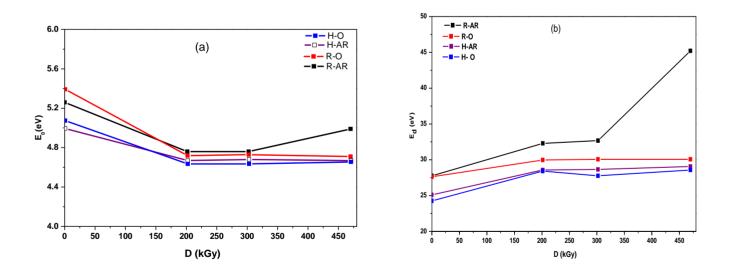


Figure 7. Variation of oscillation energy E_o (a) and desperation energy E_d (b) with increasing the gamma irradiation dose.

Sample	D0		D1		D2		D3	
	E _d (ev)	E _o (ev)						
R-AR	27.75	5.22	32.29	4.62	32.68	4.61	45.21	4.85
R-O	27.65	5.38	30.01	4.53	30.1	4.54	30.1	4.53
H-O	24.26	4.99	28.42	4.47	27.75	4.47	28.57	4.49
H-AR	25.1	4.91	28.57	4.52	28.65	4.54	29.1	4.53

Table 3. Changes in the values of the dispersion energy E_d and oscillator energy E_0 .

3.2.4. Optical conductivity

The optical conductivity σ_{op} was calculated from the relation [45,50]:

$$\sigma_{op} = \frac{\alpha n c}{4\pi} \tag{6}$$

where, c is the velocity of light, n is the refractive index, α is the absorption coefficient. The value of optical conductivity (σ_{op}) was plotted as a function of the photon energy as shown in Fig. 8. It is noticed that the optical conductivity increases with increasing photon energy before and after irradiation for all the samples. The curves for the irradiated samples showed higher values of optical conductivity for the doses D1, D2 and D3 without any differences between the three doses effects. This confirms that the increasing of carries charges that was calculated in table 2 increases the optical conductivity of the samples by irradiation.

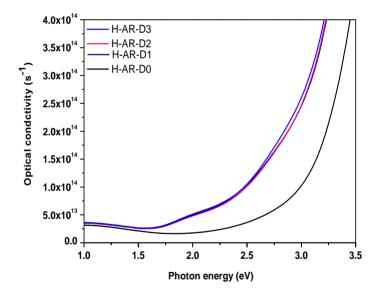


Figure 8. Optical conductivity as a function of the incident photon energy.

4. CONCLUSIONS

The following points were concluded:

- Thin film of ZnO:Al (Al: wt 20%) was prepared by DC sputtering technique and the XRD confirmed the amorphous state of the deposited film.
- The crystallinity of the prepared films was changed by irradiation and heating.
- The transmission properties were changed due to surface modification by irradiation and heating.
- The optical band gap of the prepared films showed linear decrease with the irradiation doses which is useful in using the prepared films in dosimeter purposes.
- Gamma irradiation causes changing in the optical parameters such as refractive index, oscillator energy and dispersion energy which may be useful in tuning these parameters by applying suitable absorbed dose.
- Finally it can be noticed that the AZO thin films are highly sensitive to gamma irradiation which should be taken into consideration in the applied electronic industry.

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