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Effect of Gallium incorporation on the properties of ZnO thin films

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Abstract

Ga doped ZnO thin films were formed by the Ultrasonic Chemical Spray Pyrolysis method onto substrates using zinc acetate and gallium (III) nitrate hydrate as precursors. The structural, optical, surface and electrical properties were studied as a function of increasing Ga doping concentration from 0 to 6 at %. Structural studies were shown polycrystalline with a hexagonal crystal structure. The transparency in the visible range was around ~85% for thin film deposited using 6 at % Ga doping. With the aim of determining surface images and surface roughness of the films atomic force microscope images were taken. Ga doping of ZnO thin films could markedly decrease surface roughness. Electrical resistivity was determined by four point method. The resistivity 2.0% Ga doped ZnO film was the lowest resistivity of 1.7 Ωcm. In the photoluminescence measurements of the films, existence of UV and defect emission band was observed. As a result, Ga doped ZnO films have advanced properties and promising materials for solar cells.

Keywords: Ga-doped ZnO; chemical spray technique; XRD; AFM; Optical properties; Electrical properties.

1. Introduction

Transparent conductive oxide (TCO) thin films have a very important place in optoelectronic applications. The most developed TCO tecnology for practical applications is based on indium tin oxide (ITO) [1]. Recently, zinc oxide (ZnO) has emerged as an important transparent conductive metal oxide capable of replacing commonly used expensive ITOs. ZnO is a transparent semiconductor material composed of lI-VI type with natural n-type conductivity. ZnO is a non-toxic material found abundantly in nature. It has also emerged as one of the most promising materials due to its optical and electrical properties. [2]. Group III

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A elements Al, In, Ga and B have been frequently used as n type dopants for ZnO [3]. Ga-doped ZnO (GZO) has also drawn attraction in the recent times as well as doping-free ZnO [4-7]. Because oxidation of GZO is more stable due to high electro-negativity of gallium [8]. GZO with low electrical resistance and high optical transparency in visible region is one of the most suitable candidates for alternative TCO films. [9]. In order to obtain Ga-doped ZnO films, various thin-film production techniques such as magnetron spraying [10-13], chemical spray pyrolysis[14-21], sol-gel [22-24], chemical steam deposition (MOCVD) [25,26], pulsed laser deposition (PLD) [27-29] and ion plating with DC arc discharge [30,31] are used. The chemical spray technique among these techniques is widely used thanks to its advantages in terms of that the films produced have wide surfaces as well as that it is economic and simple. In this paper, undoped and Ga-doped (2,4 and 6%) ZnO films were produced by using Ultrasonic Chemical Spray technique (USP) and their electrical, optical, structural and surface properties were researched.

2. Experimental Details

USP technique of salts of solid samples and inorganic compounds by mixing the solution at the desired volume prepared in specific concentrations allow the production of thin films and then they are spraying the preheated glass substrate. [32]. The films were produced on glass substrates with USP technique. $Zn(CH_3COO)_2{\cdot}2H_2O$ was used as Zn source and Gallium (III) nitrate hydrate [$Ga(NO_3)_3{\cdot}xH_2O$] was used as Ga source in order to obtain the films. The production parameters and codes of the films are given in Table 1. The solution was sprayed on preheated onto glass substrates at 5 ml/min. Flow rate controlled using flow meter. In order to examine the structural properties of the films, XRD patterns were taken at $30°\leq 2\theta \leq 70°$ by "Rigaku X-Ray Diffractometer" with the powder method by using CuK_α radiation (λ=1.5406 Å). With the "Park Systems XE 100 model", the surface properties of the films were taken. Also, rms (R_q) and average (R_a) roughness values were determined. Thicknesses of the films (t), Δ values, refractive indexes (n) and extinction coefficients (k) were determined by "OPT-S9000 Spectroscopic Elipsometer (SE)" with 250-2300 nm measuring range. Transmittance (T) spectra were taken by "Shimadzu-2550 UV-Vis Spectrophotometer'' with 300-900 nm measuring range. Also, optical band gaps (E_g) of all films were determined by the optical method. "Keithley 2601A LUCAS Labs PRO4" device was used to determine the electrical resistivity of the films. Also, photoluminescence spectra (PL) of the films were taken by "Perkin Elmer LS55 Fluorescence Spectrometer".

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Table 1. Codes and production parameters of GZO.

Film	Kod	Molarite	Püskürtme Süresi	Taban Sıcaklığı
ZnO	Z0	0.1 M	20dk	350±5 °C
ZnO:Ga %2	GZO-2	0.1 M	20dk	350±5 °C
ZnO:Ga %4	GZO-4	0.1 M	20dk	350±5 °C
ZnO:Ga %6	GZO-6	0.1 M	20dk	350±5 °C

3. Results and Discussions

3.1. Structural properties

Figure 1 shows the XRD patterns of the films the existence of a strong peak corresponding to (0 0 2) and weak peaks corresponding to (1 0 1), (1 0 2) and (1 0 3) of the wurtzite phase of ZnO. Existence of peaks having different intensity and widths is an indicator of that the films form at a polycrystalline structure [33]. As the amount of Ga in the film increases, the density of the peaks decreases. This behaviour indicates that deteriorates the crystallinity of the films. This is due to the stresses caused by the difference in the ionic radii of zinc and gallium. In Table 2, it draws attraction that 2θ and d values belonging to the films show difference in accordance with the values given in JCPDS (00-036-1451) [34]. In Table 2, it is seen that (0 0 2) refraction peak in the films peak shifted towards higher angles compared to Z0 peak. This case indicates that Ga^{+3} (0.62Å) having a small ionic radius took the place of Zn^{+2} (0.74 Å) ion having a bigger ionic radius [35]. Grain size (D) values can be calculated using the Debye-Scherrer formula given below;

$$D = \frac{0.94\lambda}{B\,Cos\theta} \qquad (1)$$

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where λ is the x-ray wavelength used, β is the full-width at half maximum (FWHM) of the diffraction peak and θ is the Bragg's angle [36, 37]. Table 2 shows that disrupt the crystallinity of the contribution from Ga.

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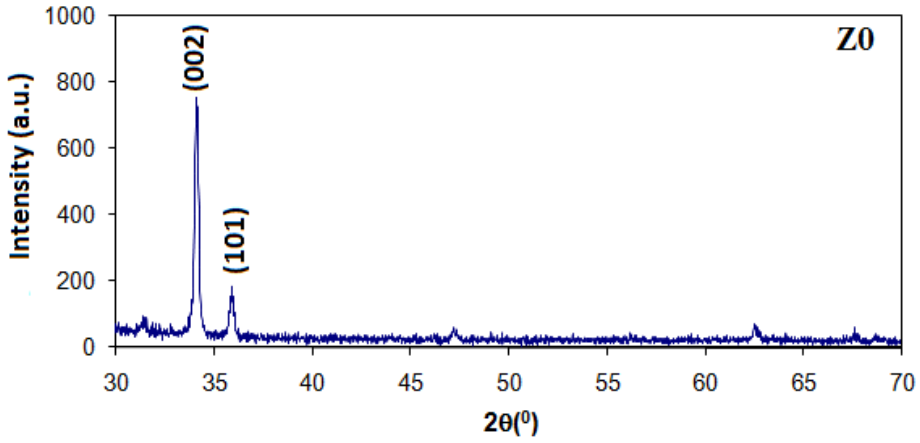

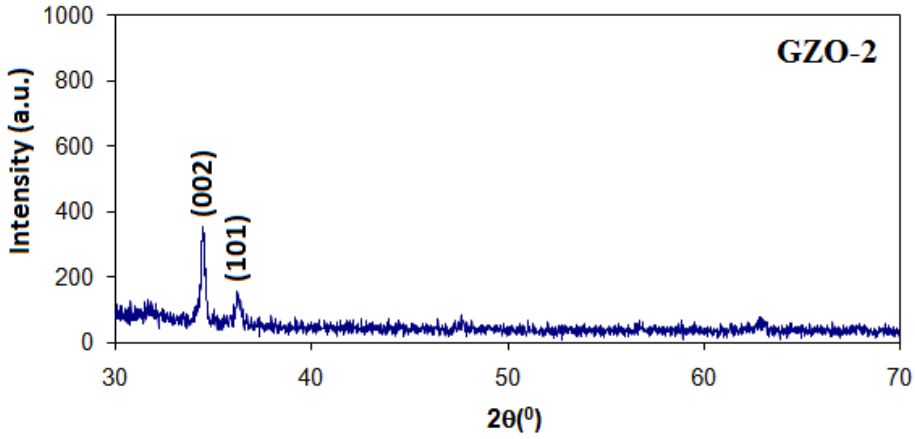

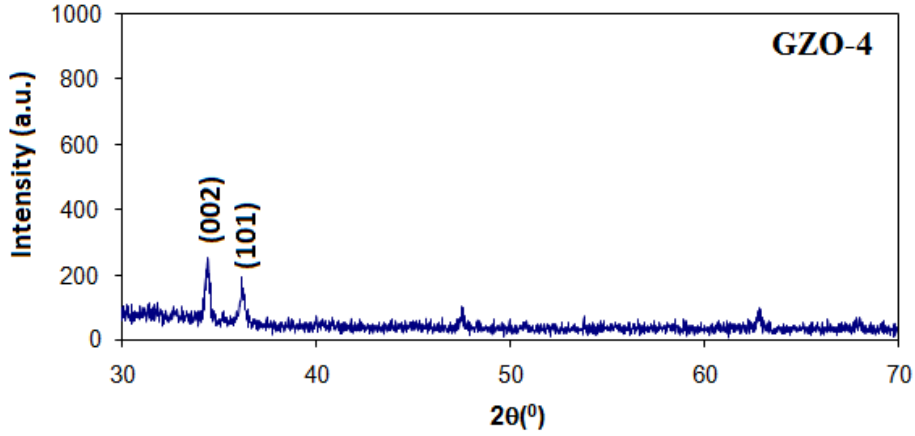

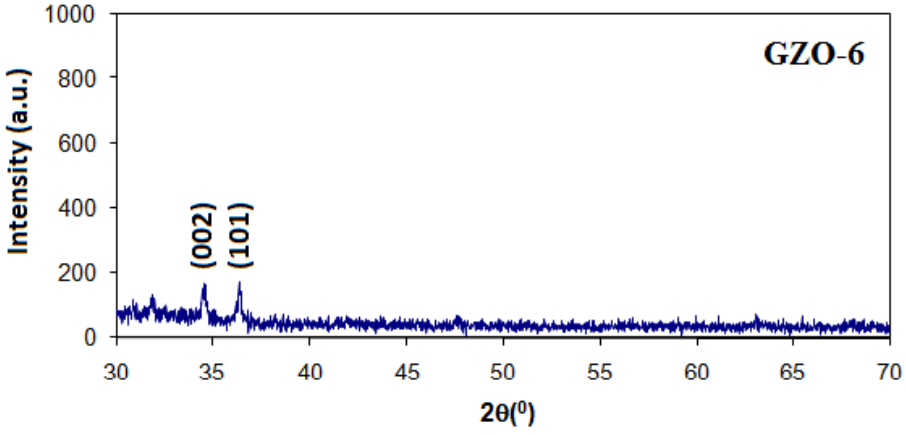

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Fig 1.

Table 2. The data and structural parameters belonging to XRD patterns of GZO films.

Film	2θ (°)	d (Å)	$2\theta_0$ (°) (ASTM)	d_0 (Å) (ASTM)	(hkl)	D (nm)
Z0	34.22	2.618	34.42	2.603	(002)	35
	36.06	2.489	36.25	2.476	(101)	30
GZO-2	34.48	2.599	34.42	2.603	(002)	29
	36.42	2.477	36.25	2.476	(101)	24
GZO-4	34.40	2.605	34.42	2.603	(002)	18
	36.20	2.479	36.25	2.476	(101)	18
GZO-6	34.54	2.595	34.42	2.603	(002)	26
	36.36	2.469	36.25	2.476	(101)	26

3.2. Optical properties

The thicknesses and optical constants of the films are determined by SE. The polarization state of the electromagnetic wave is given by Ψ and Δ , which are known as ellipsometric parameters. Thus, information about the sample that changes the polarization state is obtained. ψ and Δ parameters are measured as wavelength function in SE measurements [38]. Elipsometric data analysis requires an optical model that defines the layer thickness and optical constants of the sample. The Cauchy model is suitable for areas where the extinction coefficient is zero. The Urbach equation represents the extinction coefficient of the region where the material has little absorption [39]. Cauchy-Urbach model is used for fitting the experimental data. SE measurements have been taken at a wavelength range of 1200-1600 nm. In order to determine the thickness of all films with high precision, polarized light are sent at different angles (60°, 65°, 70°) on the material, and Δ spectra are taken. The best angle for Δ spectra was determined as 70°. Figure 2 are showed Δ spectra of the films.

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The coherence between theoretical model and experimental data is good. But in the films, it draws attraction that there are deviations at experimental and theoretical Δ values. Such a deviation may have been occurred due to the data taken in spectroscopic elipsometer technique which is sensitive to surfaces of the films. Another reason of the deviation may have been the grain boundaries. It is previously seen that there is a decrease at crystallization level with addition of Ga element. Thicknesses (t) and modeling parameters of all films are given in Table 3. It is noteworthy that the thickness values decrease with Ga incorporation.

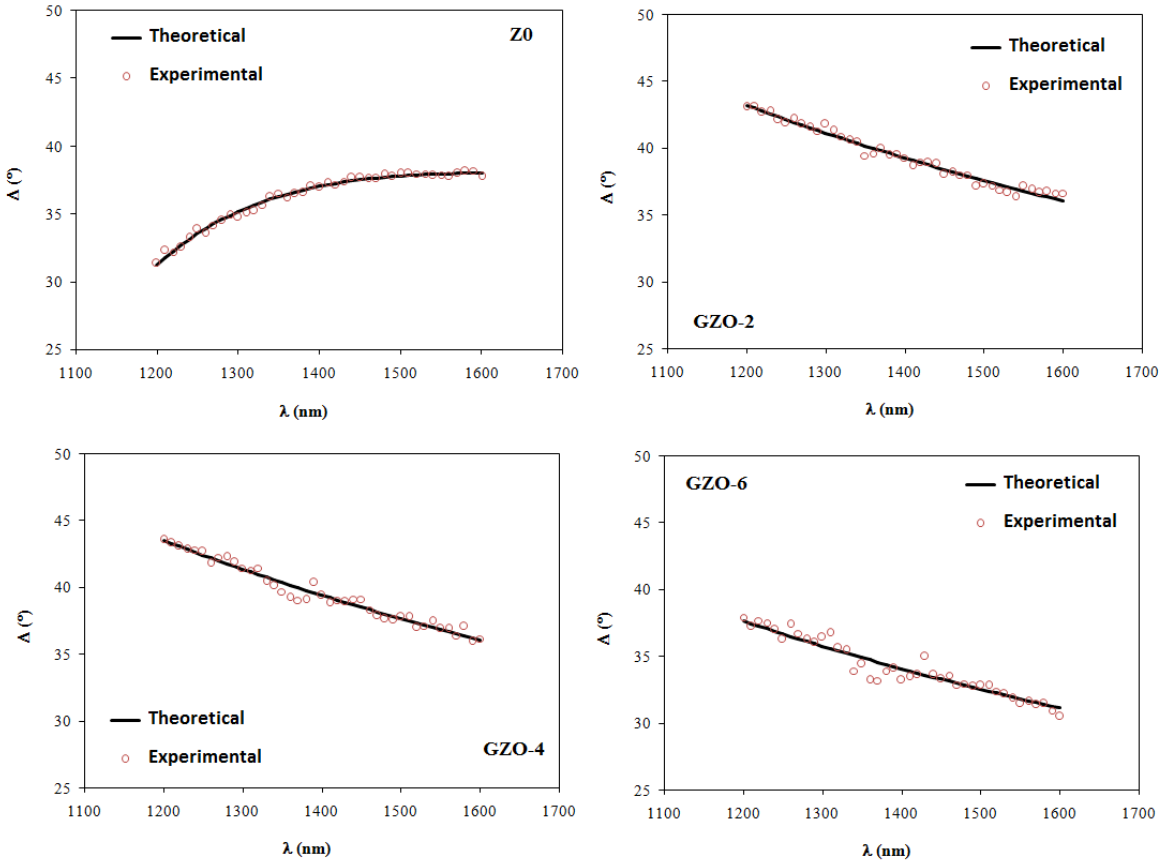

Fig 2.

Table 3. Cauchy-Urbach model parameters, thickness values and optical constants of GZO films.

Film	t (nm)	A_n	B_n (nm)^2	C_n (nm)^4	A_k	B_k (eV)$^{-1}$	n	k
Z0	135	1.914	0.0100	0.011	0.29	1.09	1.922	0.0233

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GZO-2	39	2.201	0.0106	0.0014	0.35	1.386	2.206	0.0135
GZO-4	59	2.079	0.0102	0.0015	0.36	1.386	2.085	0.0136
GZO-6	51	2.031	0.0100	0.002	0.36	1.385	2.037	0.0139

Figure 3 are showed transmittance spectra of the films. When the films are looked to naked eye, they are transparent and homogenous [40]. This has been also validated with transmittance spectrums. The transmittance in the visible area is approximately %80. An increase is also discusses at average transmittance values in visible area of the films with Ga doping. It is good for device application that the transmittance of the doped films are increased in the visible area.

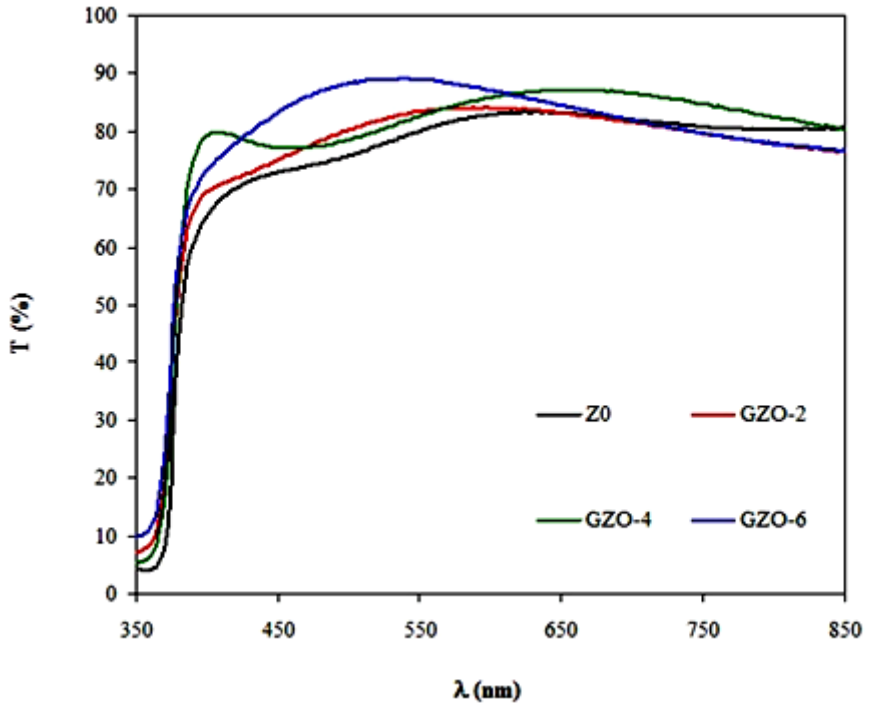

Fig 3.

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$(\alpha h\nu)^2 \sim h\nu$ graphs of the films are in Figure 4. Optical band gap values of the films have been determined by optical method [41]. Optical band gap values of the films Z0, GZO-2, GZO-4 and GZO-6 have been calculated to be 3.27, 3.28, 3.28 and 3.28 eV, respectively. All films have direct band gap transitions.

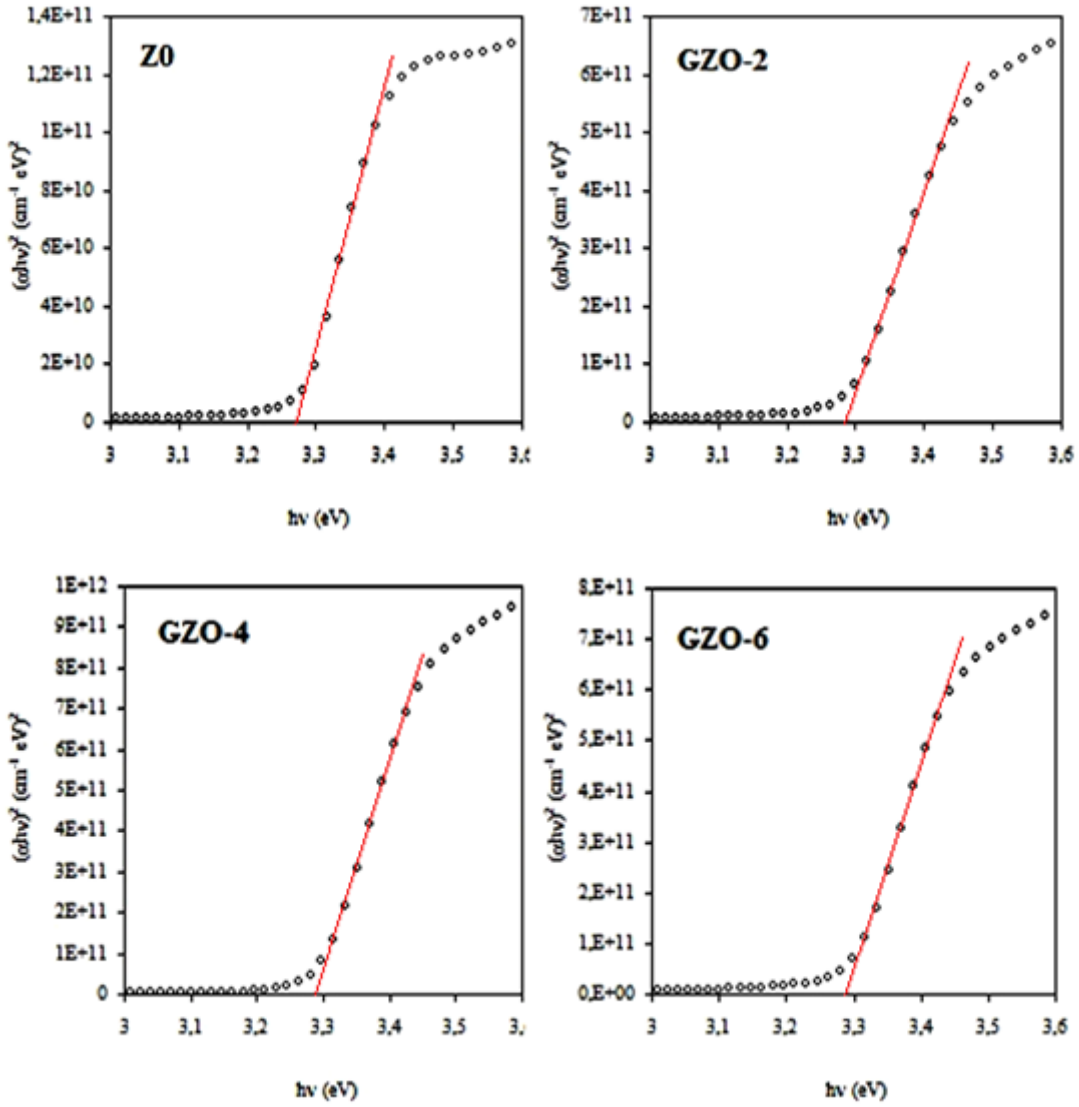

Fig 4.

PL spectroscopy is an important tool to characterize defects in semiconductors [42]. PL spectrums of the films taken with 3.82 eV excitation energy at room temperature are given in Figure 5. When PL spectrums are examined, it is determined that UV emission are at ~390 nm (3.18 eV) (1^{th} peak); blue emission is at ~440 nm (2.76 eV) (2^{nd} peak); green emission is at ~490 nm (2.5 eV) (3^{rd} peak); yellow-orange emission is at ~550 nm (2.25 eV) (4^{th} peak) and orange emission is at ~590 nm (2.11 eV) (5^{th} peak) [43,44]. The intensity of 1^{th}

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peak defined as UV emission peak decreased with the increase of Ga amount in GZO-4 and GZO-6 films compared to Z0 film. The intensity of 2nd peak increased in GZO-2 film compared to others. We may say that this case was resulted from the interstitial zinc (Zn_i) [19, 20]. We think that weakening of this peak in GZO-4 and GZO-6 films are resulted from the changes on distributions of Zn_i atoms in the optical band gap along with Ga doping. 3rd peak corresponding to the green emission in PL spectra is an indicator for existence of oxygen vacancies (V_o) that act as a deep visible trap in the structure [40]. The increase in the intensity of this peak draws attraction especially in GZO-2 films. 4th and 5th peaks that show yellow-orange and orange emissions are indicators of intersitial oxygen (O_i). Increase of peak intensity representing the orange emission in the films brings the idea of increase of density of the oxygen vacancies to the mind. This case is clearly seen in GZO-2 film. In GZO-4 and GZO-6 films, it may be said that interstitial oxygen had decreased.

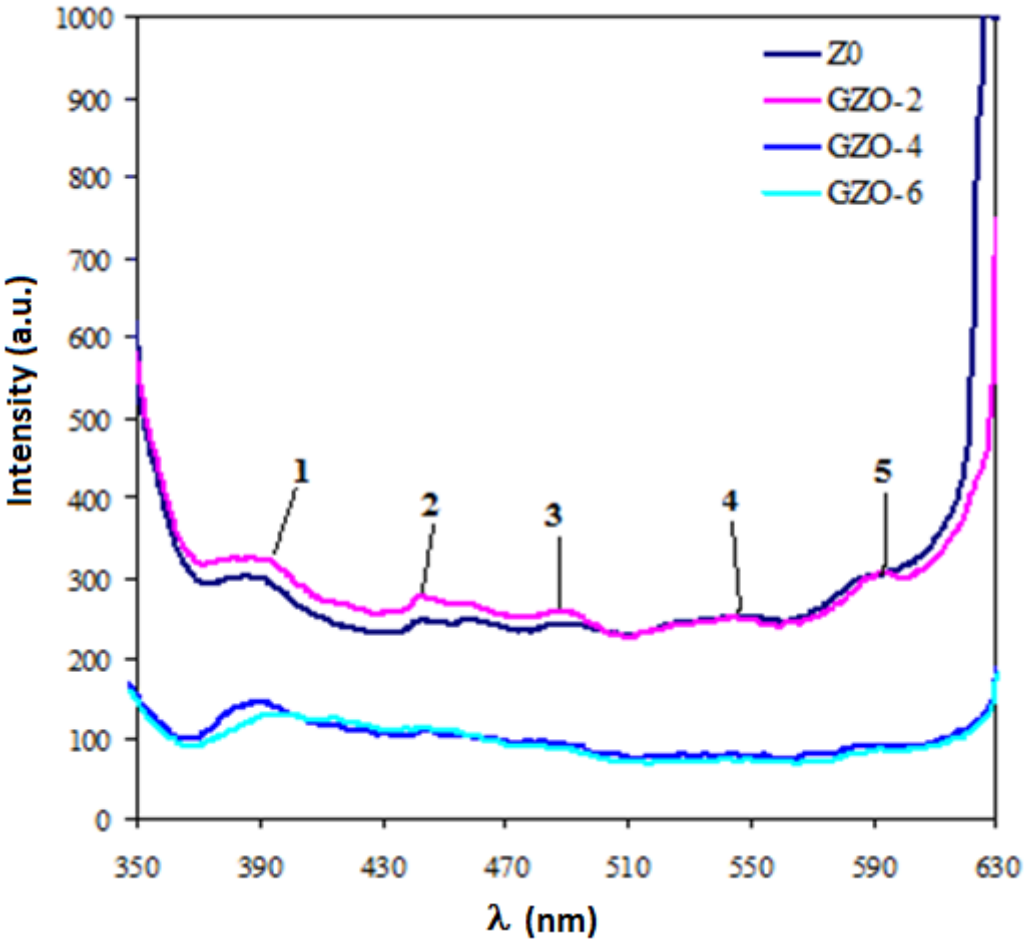

Fig 5.

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3.3. Surface properties

Figure 6 are showed AFM images of the films. When images are examined for all films, the formations randomly distributed on the surface that have different heights draw attention [41]. Ga doping concentrations affected the surface roughness levels of the films When R_q (rms roughness) and R_a (roughness) roughness values given in Table 4. It is seen that roughness decreased in Ga-doped films.

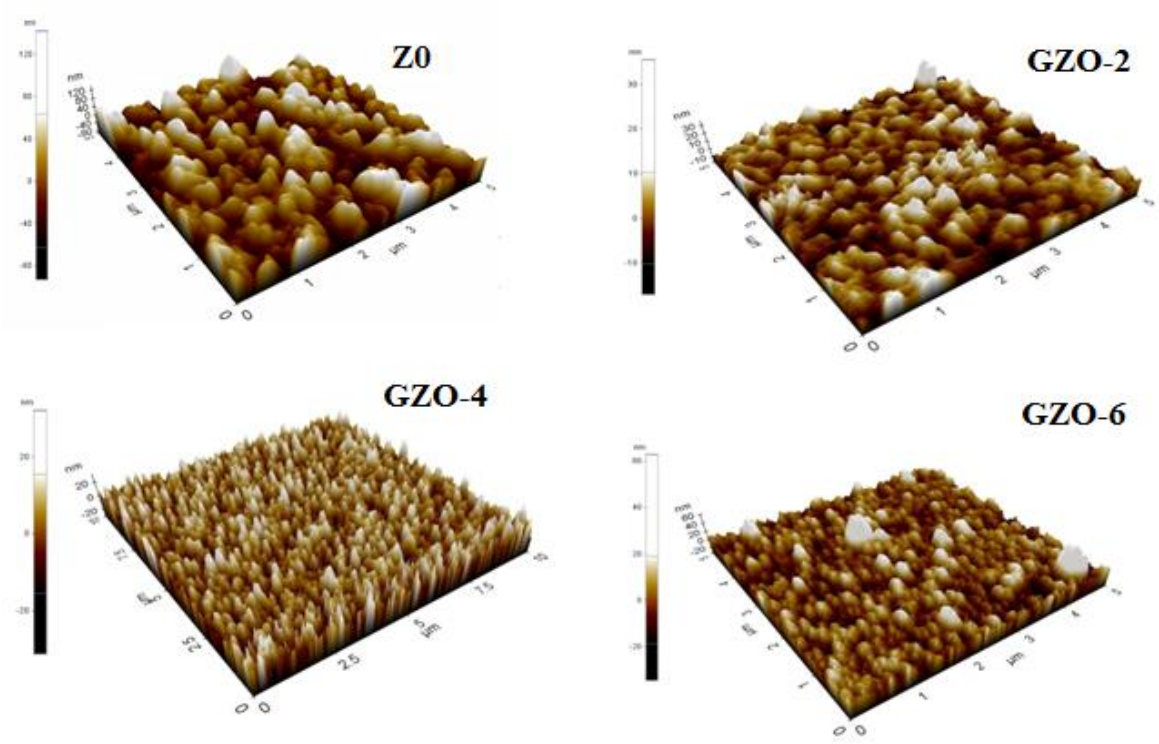

Fig 6.

Table 4. Roughness values of GZO films.

Film	Z0	GZO-2	GZO-4	GZO-6
R_a(nm)	26	4	6	7
R_q(nm)	32	5	8	10

3.4. Electrical properties.

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Resistivity values of the films ZO, GZO-2, GZO-4 and GZO-6 have been determined to be 29.4, 1.7, 1.9 and 3.0 Ωcm, respectively. The resistivity of the Z0 is higher than that of the Ga doped films. We think that this case is related to additional electron as a result of replacement of Ga^{+3} ion entering into the structure with Zn^{+2} ion. Also, the defects such as interstitial Ga^{+3} or interstitial zinc (Zn_i) or oxygen vacancies(V_0) as well as Ga doping may take a role in the increase of carrier densities by playing a role in this case [46].

4. Conclusions

Ga doped ZnO films have been deposited onto glass substrates by USP technique. The Ga element doping procedure has been selected at three different rates of 2%, 4% and 6%. Structural, optical, surface and electrical properties of the films have been researched. XRD patterns of the films have a polycrystalline structure. x-ray diffraction studies have shown that the level of crystallinity is impaired by the effect of the element Ga. That is, successfully substitution of Ga^{+3} ions with Zn^{+2} ions has been applied. Spectroscopic ellipsometry technique is used to determine the thicknesses and optical constants of the films. It is seen that the optical permeability of the films is the contribution of gallium. It appears that Ga does not have an effect on the optical band gap values. The minumum resistivity achieved for Ga doped ZnO thin films. As a result of reviewing optical, structural, surface and electrical properties of all films obtained, it was determined that amount of the doping element significantly affected physical properties of ZnO films. It was also concluded that the films may be used in solar cells as transparent front contact due to their opto-electronic properties.

Acknowledgments

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FIGURE CAPTIONS

Fig. 1. XRD patterns of GZO films.

Fig. 2. Δ spectrums of GZO films.

Fig. 3. Transmittance spectra of GZO films.

Fig. 4. $(\alpha h\nu)^2 \sim h\nu$ alteration graphics of GZO films.

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Fig. 5. PL spectra of GZO films

Fig. 6. AFM images of GZO films.

TABLE CAPTIONS

Table 1. Codes and production parameters of GZO films.

Table 2. The data and structural parameters belonging to XRD patterns of GZO films.

Table 3. Cauchy-Urbach model parameters, thickness values and optical constants of GZO films.

Table 4. Roughness values of GZO films.