

Spectroscopic-ellipsometry measurement of the optical properties of zinc oxide thin films prepared by sol-gel method: coating speed effect

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In this work, thin films of zinc oxide were prepared by sol-gel spin coating method with different coating speeds of 3600, 4800 and 6000 rpm. Spectroscopic ellipsometry analysis was used to extract the optical constants such as refractive index n , and extinction coefficient k , in the wavelength range of 300–800 nm. The knowledge of both n and k allows the calculation of the important properties such as real and imaginary parts of the dielectric function, the absorption coefficient and the optical bandgap energy. Ellipsometric measurements were collected at an incidence angle of 70° . Also, a computer programming of Kramers–Kronig (KK) relations was used to calculate the optical properties. The ellipsometric results were compared with the parameters obtained by the KK method. It was found that the coating speed in sol-gel method, noticeably affect optical properties of the films. The films were found to exhibit high transmittance and low absorbance in the visible region. The direct optical bandgap of the films was obtained in the range of 3.4–3.7 eV using the ellipsometric method. A decrease in the void fraction of layers with an increment in the coating speed had been seen.

1. Introduction: Transparent conductive films (TCFs) are usually fabricated from transparent conductive oxides. TCFs properties have a significant influence on the performance of several devices such as light-emitting diodes [1]. ZnO is a unique semiconductor material with a wide bandgap of about 3.3 eV [2]. It has generated enormous research interest because of its stand out optical properties that are useful for piezoelectric for biosensors [3], solar cells [4], gas sensors [2] and optoelectronic [5]. Unlike many of its competitors, ZnO is inexpensive, relatively abundant, chemically stable, easy to prepare, non-toxic and most of the doping materials that are used with it are also available [6]. Several methods have been used to prepare thin films such as molecular beam epitaxy [7], radio frequency magnetron sputtering [8], pulsed laser deposition [9], spray pyrolysis [10–12], evaporation [13], chemical vapour deposition [14] and sol-gel processing [15]. Sol-gel spin coating is one of the simple methods because this technique has distinct advantages such as easy control of chemical composition, low cost and lower crystallisation temperature [16]. The sol-gel method involves many process parameters such as the molar concentration of precursor, chuck rotation speeds, pre and post heat treatment, aging time and drying time, which directly or indirectly influence the quality of thin films [17].

Many methods have been used to study the optical properties of ZnO thin films. Among these, spectroscopic ellipsometry (SE) [18, 19] is a non-destructive method for determining such properties without the limitations of the other methods that have physical contact to the film [18]. Ellipsometry method uses detection of polarisation state to characterise thin films. Due to the dependence of polarisation response on the structure and optical properties of the films, valuable information can be obtained using ellipsometry method [20]. In the SE method, various characterisations including refractive index and dielectric constant are possible. SE has an indirect nature and requires appropriate modelling analysis to extract optical properties. The interpretation of measurement results is rather difficult from the absolute values of (ψ, Δ) . Thus the construction of the optical model is required for data analysis. Ellipsometry data analysis consists of three major parts: dielectric function modelling, construction of an optical model and fitting to measured (ψ, Δ) spectra. The optical model should be selected according to the optical properties of the sample [21]. SE has become a standard technique to obtain optical dielectric functions

and thicknesses of bulk samples and especially layered thin film systems by evaluating the change of the polarisation state of a probe light beam during interaction with the system [19].

In this context, ZnO thin films were prepared on glass substrates by a spin coating method with different coating speeds of 3600, 4800 and 6000 rpm. Optical constants of the films were explored using SE measurement and Kramers–Kronig (KK) method. The effect of coating speed on optical properties of the films was also investigated.

2. Experimental details: ZnO samples were prepared via a sol-gel method by the spin-coating technique using zinc acetate dihydrate as a precursor, 2-methoxyethanol as solvent and monoethanolamine as a stabiliser. The film preparation process in the spin coating method is discussed in our previous works in detail [22, 23]. First, zinc acetate with a concentration of 0.25 M was dissolved in 2-methoxyethanol. The mixture was stirred at 60°C for an hour. MEA was added to the mixture, drop by drop until a clear solution was obtained. The solution was kept for 72 h at room temperature for aging and also for optimum viscosity to be obtained. ZnO thin films were deposited on a glass substrate using spin coater at the coating speeds of 3600, 4800 and 6000 rpm. In order to achieve high quality thin films, it is useful to treat the substrate surface. The glass substrates were treated with chromic acid, cleaned with trichloroethylene, acetone and methanol and dried. After deposition, the films were preheated in the air for 10 min at 200°C to evaporate the solvent and remove organic residuals. Thermal annealing was performed at 500°C in air for 1 h.

In this work, SE 800 DUV (SENTECH) instrument with the measurement wavelength range of 300–800 nm was used for analysing. The measurements were done in Spectra ray software [21]. The steps of spectra ray operation are sample alignment, ellipsometric measurement, modelling, fitting and reporting. In order to calculate the correct incidence angle and (ψ, Δ) data, sample height and tilt aligned accurately. Then the incidence angle, the wavelength limit and the polarisation position were indicated and the measurement was performed. In the modelling step in accordance with the selected material, the proper dispersion formula was used to describe the optical constants dispersion of the thin film. Then the proper model for the sample was made, fit parameters were chosen and the fitting process was done. SE

method was used to extract the optical constants of the layers such as refractive index and extinction coefficient in the wavelength range of 300–800 nm. Ellipsometry parameters (ψ, Δ) were recorded at an incidence angle of 70° . The optical properties of the layers were discussed in two different ways: SE and KK methods.

3. Results and discussion: There are several methods for determining the optical constants of materials. In this work, we have studied the optical constants of ZnO thin films by the SE method using Spectra ray software of SENTECH company and the KK method using a program that we have written in MATLAB computing software.

3.1. KK method: The KK relations are widely accepted tools for investigation of the optical properties of materials. These relations allow performing acquiring knowledge of dispersive phenomena by measuring absorptive phenomena or vice versa. The velocity of propagation of an electromagnetic wave through a solid is given by the complex refractive index, $N(N=n-ik)$. n is called the refractive index and is related to the velocity and k is called the extinction coefficient and is related to the decay of the oscillation amplitude of the incident electric field of an electromagnetic wave. By the interaction between the solid and the electric field, the optical properties of the solid can be deduced. The refractive index (n), and extinction coefficient (k) cannot be measured directly. In the KK method, (n, k) are determined from measurable quantities such as spectroscopic reflectance or transmittance. Refractive index is of critical importance for photonic applications. For more information about KK method, we refer readers to our previous publications [13, 24, 25]. To obtain the refractive index, and the extinction coefficient, using KK relations, we have calculated the phase angle $\theta(E)$ by following relation [24]:

$$\theta(E) = \frac{E}{\pi} \int_0^{E_2} \frac{\ln R(E) - \ln R(E_0)}{E^2 - E_0^2} dE + \frac{1}{2\pi} \ln \left[\frac{R(E)}{R(E_2)} \right] \ln \frac{E_2 + E}{|E_2 - E|} + \frac{1}{\pi} \sum_0^\infty \left[4 \left(\frac{E}{E_2} \right)^{2n+1} \right] (2n + 1) \quad (1)$$

where E denotes the photon energy, E_2 the asymptotic limitation of the free-electron energy, and $R(E)$ the reflectance. Hence, if E_2 is known, the $\theta(E)$ can be calculated. Then the real and imaginary parts of the refractive index were calculated, using the following equations:

$$n = \frac{(1 - R)}{1 + R - 2\sqrt{R} \cos \theta} \quad (2)$$

$$k = \frac{2\sqrt{R} \sin \theta}{1 + R - 2\sqrt{R} \cos \theta} \quad (3)$$

Fig. 1 displays the optical transmittance of ZnO thin films prepared with different coating speeds of 3600, 4800 and 6000 rpm annealed at 500°C in air for 1 h, using a spectrophotometer. The transmission curves of ZnO thin films were taken in the wavelength area of 300–800 nm at normal incidence angle.

The ZnO thin films at the spinning speed of 6000 rpm had the highest transmittance of 0.55 in the visible region. As shown in Fig. 1, by increasing coating speed, the absorption edge shifted towards the lower wavelength region (the blue shift). The blue shift of the absorption edge, with the rise in coating speed, shows the growth in the optical bandgap. In Fig. 2, the refractive index (n) and the extinction coefficient (k) of the layers calculated by

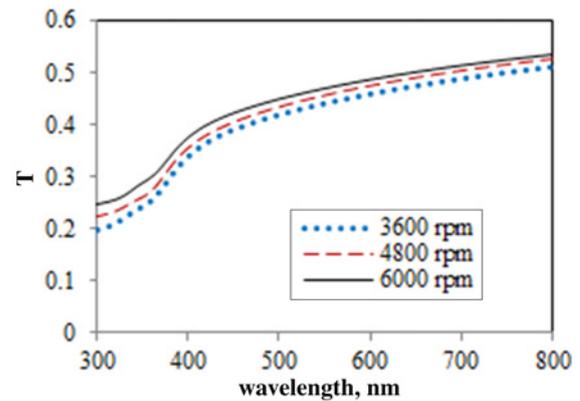


Fig. 1 Transmittance of ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm

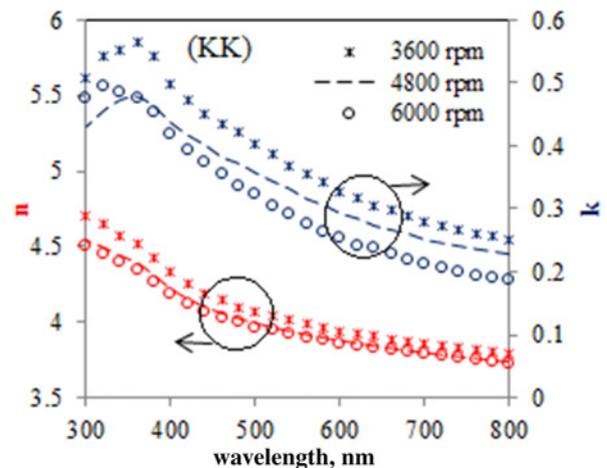


Fig. 2 Refractive index (n), and the extinction coefficient (k), of ZnO thin-films prepared with coating speeds of 3600, 4800 and 6000 rpm calculated by KK method

the KK method, can be seen. With increasing coating speed, both the refractive index and the extinction coefficient decrease.

According to Fig. 2 and the lower values of k in the visible wavelength region, the higher transparency of the films can be deduced. Knowing (n, k), the real and imaginary parts of dielectric function (ϵ_1, ϵ_2) are calculated using the following equations:

$$\epsilon_1 = n^2 - k^2 \quad (4)$$

$$\epsilon_2 = 2nk \quad (5)$$

where n and k are the refractive index and the extinction coefficient, respectively. The results for (ϵ_1, ϵ_2) in the wavelength range of 300–800 nm are displayed in Fig. 3.

It can be seen that for the results obtained by KK relations, by increasing the coating speed, there is a decreasing trend for both (ϵ_1, ϵ_2). The higher value of the real part of dielectric constant in comparison with the imaginary part shows low energy loss of light through ZnO thin films in the visible spectral range. The absorption coefficient (α), of ZnO films, is obtained using (6) and the data is displayed in Fig. 4.

$$\alpha = \frac{4\pi k}{\lambda} \quad (6)$$

where k is the extinction coefficient and λ is the wavelength.

As it can be understood from Fig. 4, the absorption coefficient was decreased with increasing wavelength. Near the band edge, the absorption coefficient shows an exponential dependence on photon energy. The optical bandgap energy was determined by

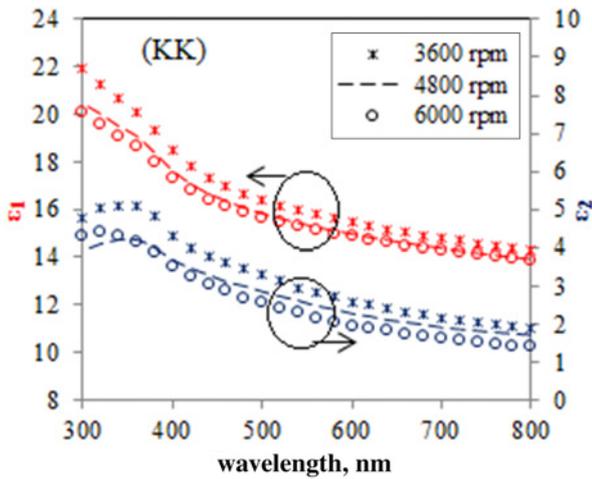


Fig. 3 Real and imaginary parts of the dielectric constant (ϵ_1 , ϵ_2) of ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm calculated by KK method

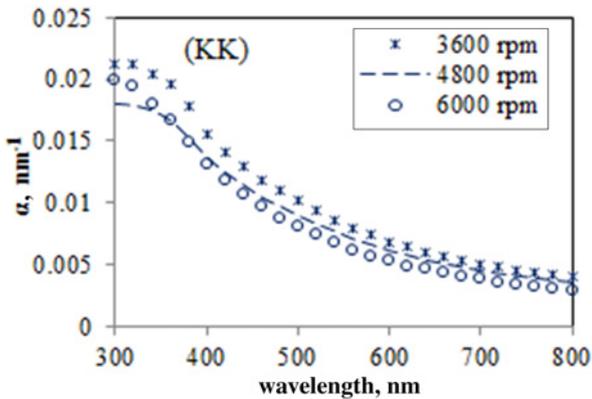


Fig. 4 Absorption coefficient of ZnO films prepared with coating speeds of 3600, 4800 and 6000 rpm calculated by KK method

applying the Tauc relation as given below [26, 27]:

$$\alpha E = A(E - E_g)^n \quad (7)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, A is a constant and n is 1/2, 2, 3/2 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect bandgap semiconductors, respectively. Due to the direct E_g for ZnO thin films, $n = 1/2$ is more suitable. An extrapolation of the linear region of a plot of $(\alpha E)^2$ on the Y-axis versus photon energy (E) on the X-axis gives the value of the optical bandgap (E_g) [26, 27] (as shown in Fig. 5). The optical bandgap energy data obtained by the KK method are shown in Table 1.

3.2. SE method: SE is an indirect optical technique in which information about the physical properties of a sample is obtained through modelling analysis. The mechanism of SE is that it measures the change in polarised light when it is reflected in a film or transmitted through the sample. Ellipsometry measures the two values (ψ , Δ) using the following equation:

$$\rho \equiv \tan \psi \exp(i\Delta) \equiv \frac{r_p}{r_s} \quad (8)$$

(ψ , Δ) values represent the amplitude ratio and phase difference between p and s polarised light waves respectively. r_p and r_s are the amplitude reflection coefficients for p and s polarisations

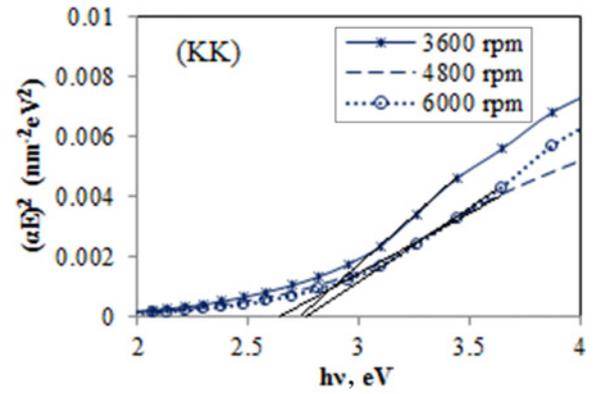


Fig. 5 Optical bandgap energy of ZnO films prepared with coating speeds of 3600, 4800 and 6000 rpm calculated by KK method

Table 1 Optical bandgap energy data obtained by both SE and KK methods for the ZnO thin films at different coating speeds of 3600, 4800 and 6000 rpm

ZnO thin films	Spin coating speed	Energy bandgap, eV
i	3600 rpm (SE)	3.38
i	3600 rpm (KK)	2.77
II	4800 rpm (SE)	3.35
II	4800 rpm (KK)	2.66
III	6000 rpm (SE)	3.40
III	6000 rpm (KK)	2.80

respectively. In the SE method, linear regression analysis is used for determining the optical properties by minimising fitting errors. In this method, the data analysis procedure is the construction of an optical model, selection or modelling of dielectric functions of the layers, fitting calculated (ψ , Δ) to experimental spectra, evaluating and minimising the fitting error and determination of optical properties, respectively. One of the remarkable features of the SE is the high precision of the measurement, and very high thickness sensitivity ($\sim 0.1 \text{ \AA}$). The mean square error (MSE) is used to qualify the difference between the experimental and predicted data.

$$(\text{MSE})\chi^2 = \frac{1}{N} \sum_{i=1}^N \frac{(\text{Mes}_i - \text{Th}_i)^2}{\sigma_i^2} \quad (9)$$

where σ_i is the standard deviation of the i th data point, N is the number of data points, Mes_i is the i th experimental data point and Th_i is the i th calculated data point from the assumed theoretical model. When the model matches the experimental data as closely as possible the MSE exhibits a minimum value. SE method is sensitive to surface structures so it is necessary to incorporate these structures into the optical model in data analysis.

Two different optical models are assumed in this section: The first optical model (model 1) included a glass substrate and a single film ZnO on the surface (Fig. 6a).

In the second optical model (model 2), the effective optical constants have been calculated using effective medium approximation (EMA) (Fig. 6b)

$$\sum_i f_i \left[\frac{\epsilon_i - \epsilon}{\epsilon_i + 2\epsilon} \right] = 0 \quad (10)$$

where ϵ_i is the dielectric function of the i th component and f_i is the volume fraction for the i th component in the composite.

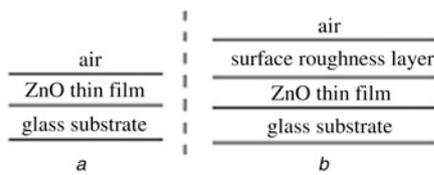


Fig. 6 optical model included
a Glass substrate and a single film ZnO on surface
b Glass substrate, a single film ZnO and a ZnO layer+void (surface roughness layer) on the surface

When a ZnO thin film is formed on the glass substrate, the effect of backside reflection leads to complications in the SE analysis. To eliminate the backside reflection, in measuring the properties of Fe-doped ZnO thin films, the rear surface of the substrates were roughened and the Scotch tape was used during the measurement. In this case, when light enters the Scotch tape, scattering by the cloudy translucent material and backside reflections are suppressed. Several dispersion models can be used to derive the real and imaginary parts of the dielectric function (ϵ_1 , ϵ_2), such as Lorentz, Tauc-Lorentz, Couchy, Sellmeier and Leng-Lorentz oscillator model. In this work, Leng-Lorentz oscillator model [28] was used. SENTECH has expanded the formula published by Leng in the following form:

$$\epsilon(E) = \epsilon_\infty + \sum_{i=1}^N \left(\frac{C_{0i}}{E^2} \left[e^{i\beta_i} (E_{gi} - E - i\Gamma_i)^{\mu_i} + e^{-i\beta_i} (E_{gi} + E + i\Gamma_i)^{\mu_i} - 2\text{Re} \left[e^{-i\beta_i} (E_{gi} + i\Gamma_i)^{\mu_i} \right] - 2i\mu_i E \text{Im} \left[e^{-i\beta_i} (E_{gi} + i\Gamma_i)^{\mu_i - 1} \right] \right] \right) \quad (11)$$

For a single critical point, C_0 is the amplitude, β is the phase, μ is the order of the pole, E_g is the critical point energy and Γ is the broadening parameter of the oscillator. Fig. 7 shows the experimental and fitted ellipsometry parameters ψ , as a function of wavelength for

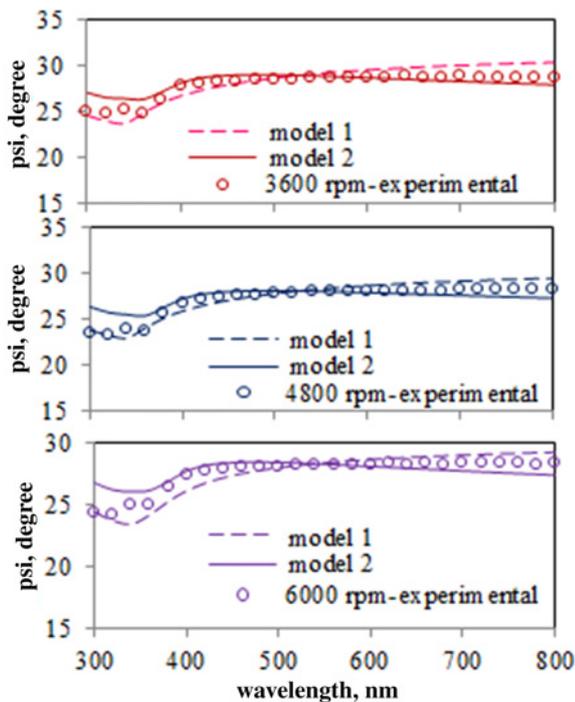


Fig. 7 Experimental and fitted ellipsometry parameter (ψ) as a function of wavelength for ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm for models 1 and 2

ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm for models 1 and 2. In Fig. 8, the experimental and fitted ellipsometry parameters Δ , as a function of wavelength for ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm for models 1 and 2 is shown. We have mentioned that the SE method is sensitive to the surface roughness of the film, so it is necessary to incorporate these structures into the optical model in data analysis. As it can be seen by adding EMA, the experimental and fitted data have a better agreement, so to calculate the optical properties we have used the model with the EMA (model 2) to calculate the optical properties. Table 2 shows the MSE obtained by the SE method for the ZnO thin films at different coating speeds of 3600, 4800 and 6000 rpm for both models.

As it can be seen, for all layers with different coating speeds with considering EMA (model 2), the MSE is smaller. It means that model 2 can be a better choice for this work. Fig. 9 shows the real and imaginary parts of the dielectric constant (ϵ_1 , ϵ_2) calculated by the SE method using model 2. For ϵ_1 data obtained by the SE method there is a peak at about 380 nm for all layers which was not seen in the data obtained by KK relations. As it can be seen, by increasing spin coating speed ϵ_1 was decreased but there was no specific trend for the ϵ_2 data obtained by the SE method.

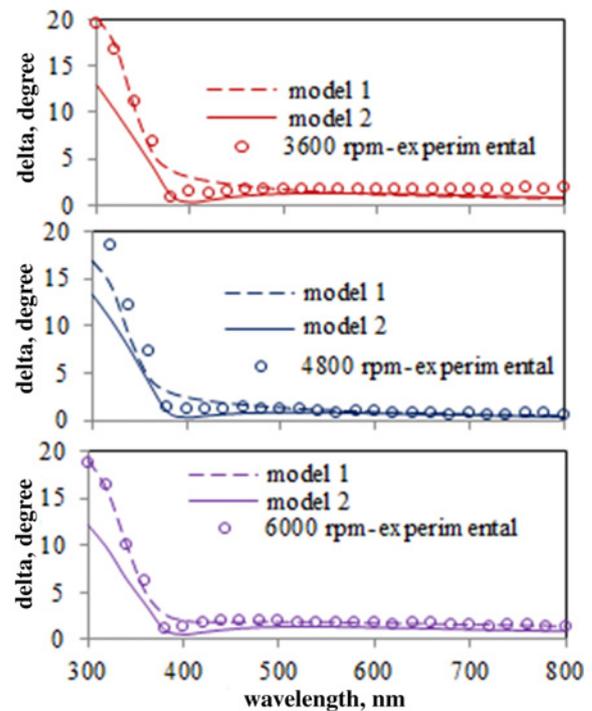


Fig. 8 Experimental and fitted ellipsometry parameters (Δ), as a function of wavelength for ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm for models 1 and 2

Table 2 MSE obtained by SE method for the ZnO thin films at different coating speeds of 3600, 4800 and 6000 rpm in two models without EMA (model 1) and with considering EMA (model 2)

Spin coating speed, rpm	Model	MSE
3600	1	1.2968
	2	0.6568
4800	1	1.0245
	2	0.8839
6000	1	0.8631
	2	0.7717

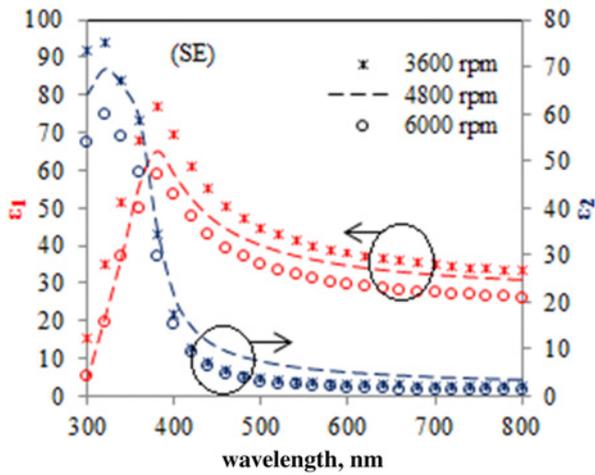


Fig. 9 Real and imaginary parts of the dielectric constant of ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm calculated by SE method

By knowing (ϵ_1, ϵ_2) we can find refractive index and extinction coefficient (n, k) using following equations:

$$n = \{ [\epsilon_1 + (\epsilon_1^2 + \epsilon_2^2)^{1/2}] / 2 \}^{1/2} \quad (12)$$

$$k = \{ [-\epsilon_1 + (\epsilon_1^2 + \epsilon_2^2)^{1/2}] / 2 \}^{1/2} \quad (13)$$

Fig. 10 shows the dependence of the refractive index and extinction coefficient of ZnO thin films on wavelength, calculated by the SE method. It can be seen that increasing coating speed, leads to a reduction in (n, k) . This is in agreement with data obtained by KK relations (Fig. 2).

There is an anomalous behaviour in n curves for all films in the wavelength around 370 nm which is associated with excitonic-like transitions. For all samples, the extinction coefficient (k), is high at the high ultraviolet region, but the value leads to falling at the low visible region. The lower data of k in the visible region show the high transparency of the ZnO thin films.

Fig. 11 shows the absorption spectra for the ZnO thin films at different coating speeds of 3600, 4800 and 6000 rpm using the SE method. The absorption coefficient decreases with increasing wavelength. The layers sharp absorption edge is located at about 380 nm which is due to the fact that ZnO is a direct bandgap semiconductor.

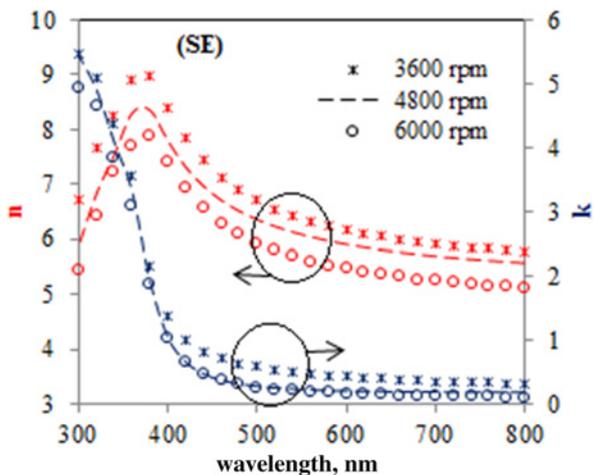


Fig. 10 Refractive index and the extinction coefficient of ZnO thin films prepared with coating speeds of 3600, 4800 and 6000 rpm calculated by SE method

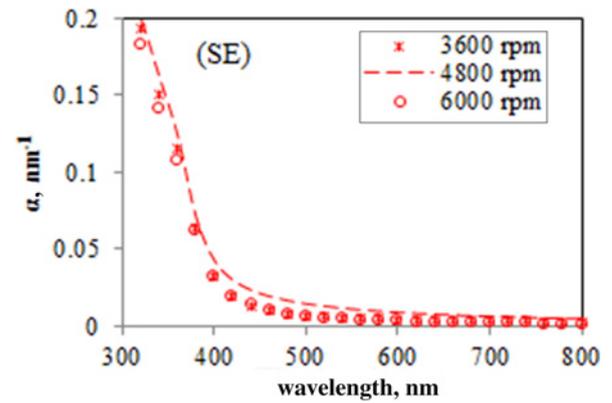


Fig. 11 Absorption coefficient of ZnO films with coating speeds of 3600, 4800 and 6000 rpm calculated by SE method

The optical bandgap energy of the films with the data of SE method is obtained by Tauc relation (Fig. 12). Tauc relation is discussed in Section 3.1. The slight deviation in the energy bandgap from this work compared with the previous works [16, 19–21, 28] may be due to the fact that the values of bandgap depend on many factors like coating speed (rpm), the granular structure, the nature and concentration of precursors, the structural defects and the crystal structure of the films.

As it can be understood from Table 1, the data obtained by the SE method have more agreement with the data of previous works [16, 29–32]. It may be deduced that SE method is more precise than KK relations. The bandgap difference between the film and bulk ZnO is due to the grain boundary, the stress and the interaction potentials between defects and the host materials in the films [29]. The ZnO

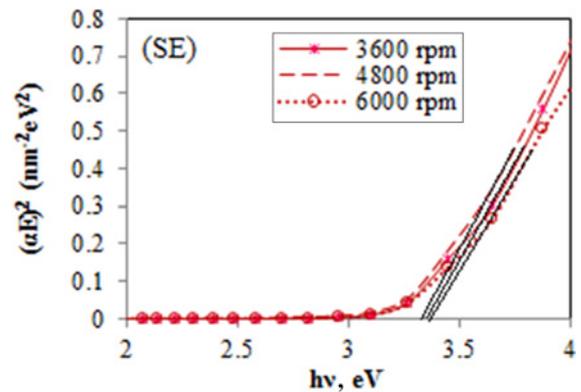


Fig. 12 Optical bandgap energy of ZnO films with coating speeds of 3600, 4800 and 6000 rpm calculated by SE method

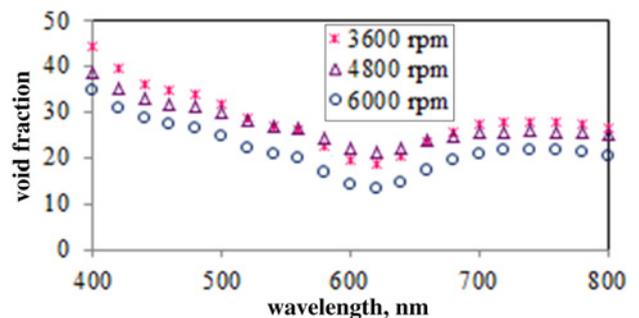


Fig. 13 fraction of voids in SE method by Aspnes theory for the ZnO thin films at different coating speeds of 3600, 4800 and 6000 rpm

thin films with coating speed of 6000 rpm have the highest optical bandgap in both SE and KK methods.

The effect of voids on the optical properties of thin films maybe investigated by the Bruggeman effective-media approximation [33] or its developed version by Aspnes *et al.* [34]. The void fraction of ZnO thin films is measured by Aspnes theory. The void fractions of ZnO thin films calculated for the layers prepared spin coating speeds of 3600, 4800 and 6000 rpm are shown in Fig. 13 as a function of wavelength. In the general migration of grains should lead to a decrease in the fraction of voids. With changing coating speed, the fraction of voids changes too. As it can be seen in Fig. 13, the ZnO thin film with coating speed of 6000 rpm has the least void fraction.

4. Conclusion: Optical properties of ZnO thin films with varying coating speed have been investigated. The optical constants of the layers were calculated by two different methods, SE and KK. In the SE method, two models were investigated to determine the optical constants of ZnO thin films. First, a model without considering the surface roughness of the films and second a model with the surface roughness. According to MSE results, the data obtained for the model with considering the surface roughness had smaller MSE and consequently had better fit with experimental data. Also, comparing with previous works, the optical constants obtained by SE was more accurate than KK data. The ZnO films with coating speed of 4800 rpm had the least and the ones with coating speed of 6000 rpm had the most bandgap energy using both SE and KK methods. Also, the films with 6000 rpm spin coating speed, showed the least fraction of voids.

5 References

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