# Deposition and Ellipsometric Characterization of Transparent Conductive Al-doped ZnO for Solar Cell Application

O. Gençyılmaz, F. Atay, and I. Akyüz

***Abstract*—In this work, aluminum doped ZnO (AZO) thin films doped with different aluminum concentration (0%, 1%, 2%, 3%, 4%, 5%) were grown onto glass substrates by ultrasonic spray pyrolysis technique. Spectroscopic ellipsometeric studies of the AZO thin films were determined by means of spectroscopic ellipsometry and Cauchy-Urbach dispersion model were used to determined extract the thickness and the optical constants (refractive index and extinction coefficient) of AZO thin films. The transmittance, absorbance and reflectance spectra of the films were investigated by using UV-vis Spectrophotometer. The optical band gap of all the films were determined by the measurement of the optical absorbance as a function of wavelength and found to be between 3.22-3.27 eV. Also, surface image and roughness values and electrical resistivity values of AZO thin films were investigated at room temperature by using atomic force microscopy and four-point probe set-up, respectively. According to results, due to good optical, electrical and surface properties of AZO thin films, these films are promising candidates for their use as transparent electrodes in solar cells application.**

***Index Terms*—ZnO thin film, solar cell, spectroscopic ellipsometry, sprays pyrolysis.**

## I. Introduction

ZnO has many inherent advantages such as high conductivity, transparency, wide optical band gap and large exciton binding energy. ZnO has practical benefits that make it on attractive material due to these advantages. It is applied for a variety of important applications e.g. as transparent electrodes and windows materials in solar cells, photodiodes, gas sensors and UV-light emission diode and more [1]-[5]. Undoped ZnO thin films have usually shown n-type conduction and a low resistivity due to oxygen vacancies and zinc intersititials [6]. Also, many researches have been made to reach low resistivity by doping with group-III elements such as aluminum [7], [8]. Transparent conductive oxides (TCO) play an important role in the solar cells is devoted to improving the optical, electrical and surface properties of the TCO materials, such as zinc oxide (ZnO). Specifically, ZnO:Al films have received much attention recently [9]-[11]. AZO thin films have been grown by a variety of techniques such as pulsed laser deposition (PLD), metal–organic chemical vapour deposition (MOCVD), metal–organic vapour phase epitaxy (MOVPE), sputtering, molecular beam epitaxy (MBE), sol-gel and spray pyrolysis [12]-[22]. Among these methods, spray pyrolysis is a simple, scalable and inexpensive method to grow thin films. Also, this method does not require high-quality substrates and vacuum. The growth parameters are relatively easy to control and the stoichiometric synthesis can be obtained [23]-[27]. Although many papers have been reported on ZnO films, the spectroscopic ellipsometric properties of AZO thin films have not been extensively studied for solar cell applications.

In this paper, the AZO thin films were prepared on glass substrates by ultrasonic spray pyrolysis technique at various Al concentrations and investigate the effect of Al concentration on optical, electrical, surface properties and ellipsometric characterization of these films.

## II. Experimental Details

ZnO thin films of various Al doped concentrations (0%, 1%, 2%, 3%, 4%, 5%) were deposited at 350 ± 5°C by ultrasonic spray pyrolysis. The ultrasonic oscillator frequency was 100 kHz, and the droplet size was 20 μm. The substrate temperature was measured using an iron-constantan thermocouple. The nozzle substrate separation used was 30 cm. The spray solution was prepared to 0.1 M zinc acetate [$Zn(CH_3COO)_2.2H_2O$] and 0.1 M aluminum chloride hexahydrate [$AlCl_3.6H_2O$] in a mixture of methyl alcohol and de-ionised water in a volumetric proportion of 1:3 ml. The starting spraying solution was mixed with a magnetic mixer to prevent sedimentation. The solution flow rate was kept at 5 ml $min^{-1}$ and controlled by a followmeter. Totally, 100 ml of solution was used and sprayed for 20 min. Al doped ZnO thin films were named as AZO, AZO1, AZO2, AZO3, AZO4 and AZO5 depending on the increasing Al incorporation, respectively.

Spectroscopic ellipsometry (SE) measurements were determined by a SC620 Spectroscopic Ellipsometer over a spectrum range of 250–2300 nm. But, the investigation of AZO films has been performed in the wavelength range of 300-900 nm where Cauchy-Urbach model has been used to obtain the optical constants. Cauchy-Urbach dispersion model can be applied to these films in this wavelength range due to all films are transparent or weakly absorbing in this wavelength region. The thicknesses and optical constants (refractive index ($n$) and extinction coefficient ($k$)) of the films were obtained by analyzing the measured ellipsometric spectra through the Cauchy-Urbach model. Besides, the optical transmittance spectra of the films were taken from a

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Shimadzu-SolidSpec-3700 UV–vis-NIR Spectrophotometer. Also, band gap values of the films have been calculated by optical method. Surface images and roughness values have been taken by Park System XE-70 atomic force microscope (AFM). The AFM measurements have been taken in non-contact mode, ~300 kHz frequency and 0.50 Hz scan rate in air at room temperature. A silicon cantilever which has a spring constant of 40 N/m has been used. Also, root mean square (*rms*, $R_q$), average ($R_a$) and peak valley ($R_{pv}$) roughness values have been obtained using XEI version 1.7.1 software. All the images have been taken from an area of 5×5 μm$^2$. The roughness values belong to whole scanned area. Electrical resistivity values of the films have been investigated using a four-point probe set-up.

## III. Result and Discussion

One of the most important techniques is SE, which is a non-destructive and sensitive technique to measure the optical response of materials, especially semiconductors [28], [29]. SE measuring at several angles of incidence over a wide spectral range produces a wealth of information about the material [30].

SE measurement is to determine the relative phase change of reflected *s*- and *p*-polarized light caused by refractive index change at interfaces. The complex ratio of the total reflection coefficient of *p*- and *s*-components can be written as,

$$\rho = R_p/R_s = tg(\psi)e^{i\Delta} \quad (1)$$

where $R_p$ and $R_s$ are ratios of the reflected wave amplitude to the indecent wave amplitude for the *p*- and *s*-component, respectively. The shape of the ellipse is depicted by $\Delta$, which reflects the change in phase difference between *p*-polarized component $\delta_1$ and the *s*-polarized component $\delta_2$ of the incident and reflected wave. $\psi$ depicts the orientation of the ellipse and $tg(\psi)$ is the absolute value of $R_p/R_s$ [31]. The fundamental equation for the complex reflectance ratio $\rho$ is also described as follows:

$$\rho = f(n_1, n_2, n, \phi, d, \lambda, k) \quad (2)$$

where $n_1$, $n_2$ and $n$ represent the refractive index of air, substrate and film, respectively. $\phi$ and $\lambda$ represent the incident angle and wavelength of incident light, respectively. $d$ and $k$ the thickness and extinction coefficient of thin film [28].

The depolarization effect and incident angle are an important factor for the materials having depolarization effect. So, the measurement were taken in the spectral range of 300-1200 nm at three different angles of incident (50°, 60° and 70°) in steps of 10 nm and the best angle was determined to be 70° using experimental cos ($\Delta$) spectra.

The thickness and optical constants (refractive index and extinction coefficient) of the AZO films was analyzed Cauchy-Urabch model and this model was used to fit the experimental data. In the Cauchy — Urbach model, the refractive index $n(\lambda)$ and the extinction coefficient $k(\lambda)$ as a function of the wavelength are given by,

$$n(\lambda) = A_n + B_n/\lambda^2 + C_n/\lambda^4 \quad (3)$$

$$k(\lambda) = A_k\, e\, (B_k\, (E - E_b)) \quad (4)$$

where $A_n$, $B_n$, $C_n$, $A_k$ and $B_k$ are model parameters [32]. For the samples having depolarization effect, the incident angle is an important factor. Fig. 1 shows the curves of the measured cos ($\Delta$) against wavelength for the AZO films. Thicknesses and model parameters are given in Table I and Table II. The experimental model agrees with the Cauchy-Urbach model, but there are some small deviations on cos$\Delta$ values which becomes probably due to the depolarizing effect of roughness, grain boundaries, morphologies of the films and backside reflection of glass substrates which affect the experimental data.

Fig. 2 and Fig. 3 show refractive indices and extinction coefficients of the AZO films. The refractive indices and extinction coefficients decreased with increasing Al doped concentration. The refractive index of the ZnO film is higher than the other films. We think that this case can probably be due to the thickness effect. We can also see on AFM image, that this film is more compact. The similar results were reported by Qing Hua Li *et al*. [33].

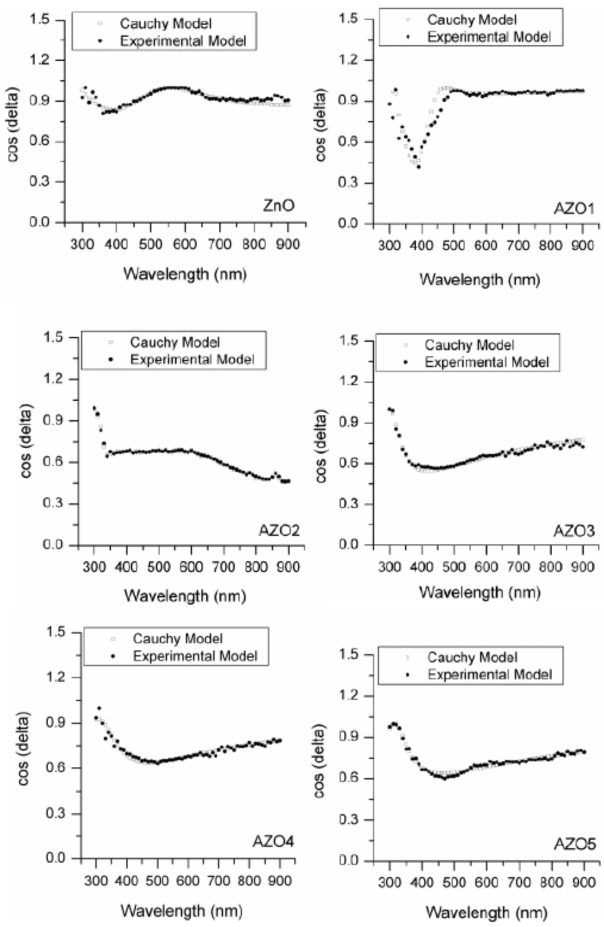


Fig. 1. cos$\Delta$ spectra of the AZO thin films.

TABLE I: The Thickness and the Cauchy Model Parameters of AZO Thin Films

| Film | $d$ (nm) | $A_n$ | $B_n$ (nm$^2$) | $C_n$ (nm$^4$) |
|---|---|---|---|---|
| ZnO | 56.77 | 2.21 | 0.041 | 0.019 |
| AZO1 | 53.83 | 2.01 | 0.079 | 0.073 |
| AZO2 | 49.91 | 2.06 | 0.013 | 0.010 |
| AZO3 | 45.71 | 2.03 | 0.051 | 0.015 |
| AZO4 | 44.89 | 2.12 | 0.026 | 0.021 |
| AZO5 | 43.70 | 2.07 | 0.099 | 0.042 |

TABLE II: THE URBACH MODEL PARAMETERS AND BAND GAP VALUES OF AZO THIN FILMS

| Film | $A_k$ | $B_k$ (nm) | MSE | $E_g$ (eV) |
|---|---|---|---|---|
| ZnO | 0.014 | 0.019 | 0.06 | 3.22 |
| AZO1 | 0.029 | 0.068 | 0.12 | 3.24 |
| AZO2 | 0.016 | 0.078 | 0.04 | 3.24 |
| AZO3 | 0.024 | 0.015 | 0.05 | 3.25 |
| AZO4 | 0.053 | 0.092 | 0.08 | 3.26 |
| AZO5 | 0.012 | 0.042 | 0.02 | 3.27 |

The transmission and reflectance of AZO thin films on glass substrate were studied in wavelength 300-900 nm and shown Fig. 3, respectively. All AZO thin films present high transparency ~70 % in the visible region from 550 to 850 nm, making the possible to be used as window layers in photovoltaic solar cells. The transmittance values can be improved with different process as thermal annealing, different Al concentration and variable experimental parameters. Reflectance spectra were determined that the average reflection value of AZO thin films is about 4%.

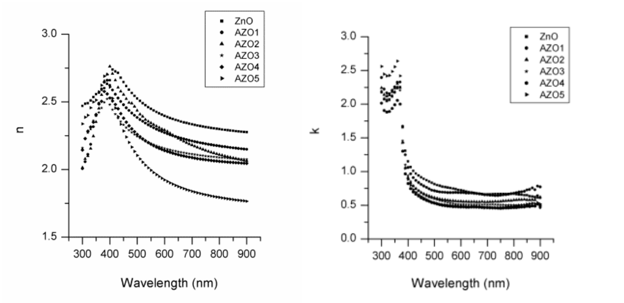


Fig. 2. Refractive index and extinction coefficient of the AZO thin films.

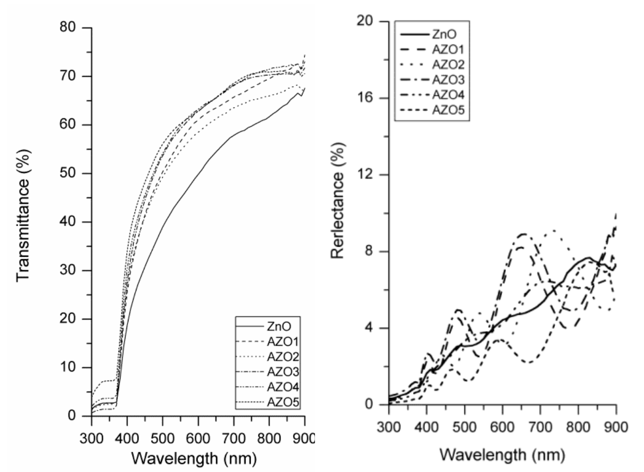


Fig. 3. Transmittance and reflectance spectra of AZO thin films.

The optical band gaps AZO thin films were calculated from the absorption spectrum. The relation between the absorption coefficient α and the incident photon energy $h\nu$ is given by eq. (1);

$$(\alpha h\nu)^2 = A\,(h\nu - E_g) \quad (5)$$

where $A$ is a constant and $E_g$ is the band gap energy. The optical energy gaps $E_g$ could be obtained from the intercept of $(\alpha h\nu)^2$ vs. $h\nu$ direct allowed transitions. The plots of $(\alpha h\nu)^2$ vs. $h\nu$ for different Al doped concentration are shown in the Fig. 4. The straight-line portion was extrapolated to the energy axis at $\alpha$=0, to obtain the band gap of AZO thin films. The optical band gap of AZO thin films was shifted from 3.22 eV to 3.27 eV. All films were found to be direct band gap materials, which is a desired property for photovoltaic solar cell applications. The variation of band gap energy with Al doped concentration is depicted in Fig. 4, and it was seen that doping caused the optical band gap values of the films to increase.

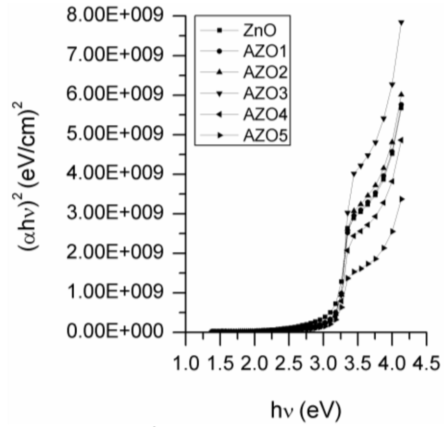


Fig. 4. $(\alpha h\nu)^2$~$h\nu$ variations of AZO films.

The surface morphology of AZO thin films significantly influence their various properties due to which they play a key role in many applications linked to optoelectronics [34]. In Fig. 5 shows that there are randomly distributed mount type formations and regions with different height and width. Al doping caused the films have an island like surface with decreasing number of cracks. Al doped ZnO thin films look dense with a smoother surface when compared to ZnO thin films. This granular structure may probably have positive effect on the optical properties of this sample, as we have mentioned before. Roughness values of the films are given in Table III. It is clear that Al doping process affected the roughness values of ZnO films. The roughness value of the AZO thin film slightly decreases with the Al doping. We think that, this decrease is manly related to the surface combination and surface form.

Fig. 6 shows the electrical resistivity and conductivity of the AZO thin films versus dopant concentration. The electrical resistivity of AZO thin films decreases with Al doping. In the Fig. 6, in our experiments ZnO films showed electrical resistivity of about $1.08\times10^4$ Ωcm while Al (at. 5%)-doped ZnO showed $1.00\times10^3$ Ωcm. The decrease in resistivity with Al doping is due to the fact that $Al^{+3}$ going into $Zn^{+2}$ sites will have one extra electron and these electrons were used as conduction electrons [35], [36]. In many applications requiring transparent conducting films, the optical transmission and electrical conductivity should be as high as possible. This is particularly important for solar cell applications because high optical transmission in the visible region enhances the photogenerated current and low sheet resistance reduces the series resistance of the cell, but in various applications of transparent conductive films either the electrical or optical properties are more critical [37]. The interrelationship between average transmission and conductivity is expressed by Haacke's figure of merit:

$$\phi = (T_{ave}^{10})/R_s \quad (6)$$

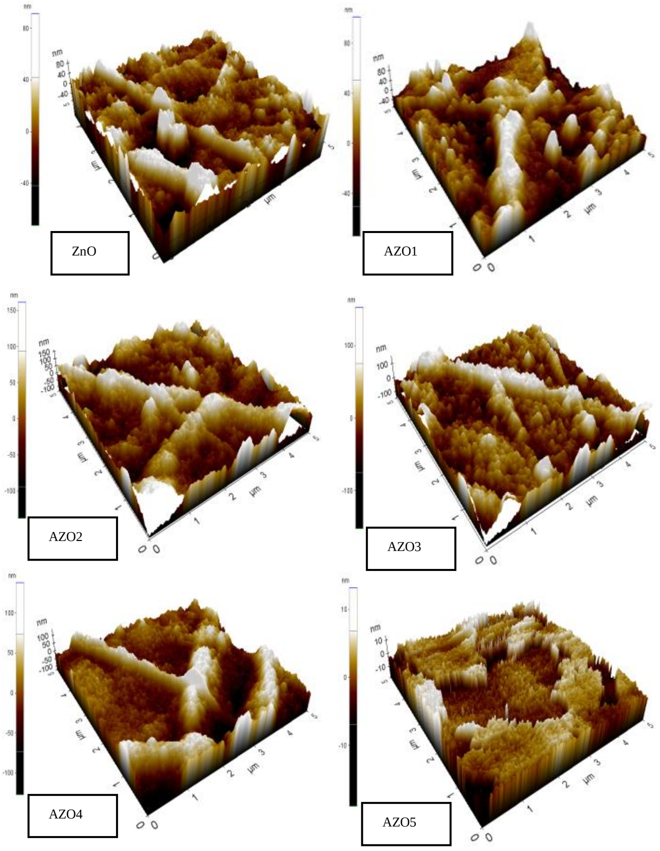


Fig. 5. AFM images of AZO thin films.

TABLE III: ROUGHNESS VALUES OF AZO THIN FILMS

| Film | $R_a$ (nm) | $R_q$ (nm) | $R_{pv}$ (nm) |
|---|---|---|---|
| ZnO | 39 | 47 | 304 |
| AZO1 | 29 | 36 | 299 |
| AZO2 | 28 | 35 | 175 |
| AZO3 | 27 | 25 | 264 |
| AZO4 | 20 | 21 | 164 |
| AZO5 | 16 | 12 | 132 |

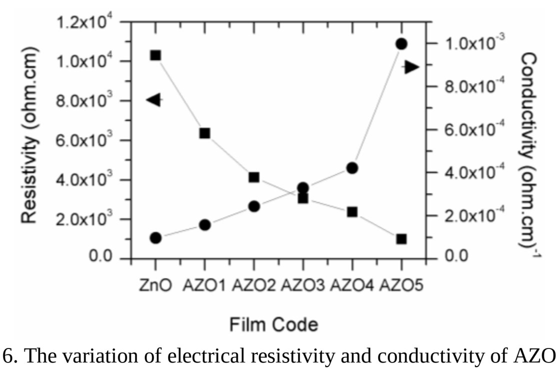


Fig. 6. The variation of electrical resistivity and conductivity of AZO thin films.

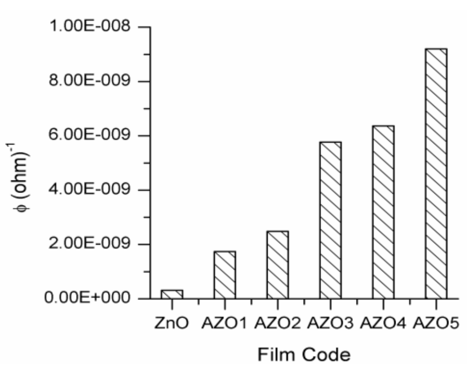


Fig. 7. Haacke's figure of merit of AZO thin films.

Fig. 7 is seen that the AZ05 thin films have a high figure of merit value.

## IV. CONCLUSION

AZO thin films were produced by the ultrasonic spray pyrolysis method by which is an inexpensive and rapid technique for different aluminum doping. The effects of aluminum doped concentration were investigated the optical, electrical and surface properties of the films. AZO5 thin film caused the transmittance values to increase which is a desired development for transparent conducting oxide industry. The spectroscopic ellipsometry and UV-vis results showed the Al doping effects on optical properties of deposited thin films. The refractive index ($n$), extinction coefficient ($k$), and optical band gap energy ($E_g$), absorbance, transmittance of the AZO thin films varied with different Al doping concentration. The Al doping also showed some interesting effect on surface properties of the deposited films. The roughness values of AZO thin films decreased at high Al doping concentration. The AZO5 thin films showed high conductivity and low resistivity. The figure of merit values of the samples for different growth temperature was calculated and high figure of merit value was obtained for the AZO5 thin films. These films (especially AZO5) grown via ultrasonic spray pyrolysis deposition could be a good candidate for solar cell applications.

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