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Structural and optical properties of Al/ZnO thin films deposited by radio frequency sputtering

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Abstract

The effects of annealing temperature and variation of sample thickness on the structural and optical properties of zinc oxide thin films with aluminium contact (Al/ZnO) have been investigated. The study involved the synthesis of a bilayer thin film of Al/ZnO with varied thicknesses on a glass slide substrate by using radio frequency magnetron sputtering deposition technique. 99.99% pure ZnO and aluminium were used as the sputtering target. The films were then annealed in vacuum at annealing temperatures of 200 °C and 400 °C. The structural and optical properties of Al/ZnO thin films grown were characterized by x-ray diffraction (XRD) and optical measurements respectively. The results obtained from the XRD patterns showed that Al/ZnO films (both as-deposited and annealed), exhibits a crystalline structure with (002) preferred orientation. The peak intensity of the preferred plane increases as the annealing temperature increases. The optical studies of the Al/ZnO films showed a maximum value of transmittance ranging from 82% to 91% depending on the condition of the films. A decrease in transmittance as the thickness of the films increases was observed. The transmittance also increased with increasing annealing temperature. The energy gaps (E_g) were determined from the transmittance data and found to be in the range 3.73–3.83 eV. The results obtained from the experiment also show that the optical band gap increases as the thickness and annealing temperature increase.

1. Introduction

ZnO is a very promising material for semiconductor device applications (Look 2001). It has a direct and wide band gap energy ~ 3.3 eV, in the near-UV spectral region (Chen *et al* 1998, Haslinda 2009), and a large free-exciton binding energy (60 meV) (Ogale 2005), so that excitonic emission processes can persist at or even above room temperature. ZnO crystallizes in the wurtzite structure (Rolo *et al* 2007).

Aluminium doped ZnO thin films have high transmittance in the visible region, and the optical band gap can be controlled by using different amount of Al doping. Accordingly they have got potential applications in solar cells, antistatic coatings, solid-state display devices, optical coatings, heaters, defrosters, chemical sensors etc (Jeong *et al* 2007).

Recently there is tremendous interest on the nanoscale semiconductor particles and thin films, due to novel properties generated from quantum confinement effect. The quantum confinement effect changes the physical and chemical properties of the materials (Brus 1984). In the case of II–VI semiconductors, this reduction in size from bulk to the nanoscale results in many size dependent properties such as varying band gap energy, optical and electronic properties (Sravani and Andrew 2010).

Different methods have been employed to deposit ZnO films, these includes, sol-gel, spray pyrolysis (Ivanova *et al* 2010), molecular beam epitaxy (Oh *et al* 2004), metal organic chemical vapour deposition (Wang *et al* 2006), chemical bath deposition (Zhang and Kerr 2007), pulse laser deposition (Zhao *et al* 2006) and rf

Table 1. Sputtering parameters for aluminium deposition.

Thickness (nm)	Sputt. time (s)	Dep. rate (sccm)	Gas flow rate (sccm)	Dep. press (Pa)
10	51	2.04	0.8	3.32×10^{-3}
5	32	1.25	0.8	3.46×10^{-3}
2	5	2.05	0.8	3.06×10^{-3}

magnetron sputtering (Jeong *et al* 2005). Among these methods, rf magnetron sputtering has the advantages of simplicity and working in desire temperature. However, it was reported that the quality of the as-sputtered ZnO films is low and a post annealing process can improve the crystallinity of ZnO thin films (Ozgur *et al* 2005, Shan *et al* 2005, Alivov *et al* 2006). Therefore, beside the sputtering conditions, the annealing factors such as temperature, gas types and time are indeed important parameters for ZnO films deposition.

Hu *et al* (2006) studied the dependence of the Al thickness and annealing temperature on the structural, optical and electrical properties of ZnO/Al multilayers. It was shown that Al thickness and annealing temperature were the key factors to optimize transparency conducting property in ZnO/Al multilayered films. Annealing at elevated temperatures improved the crystallinity and therefore enhanced the x-ray diffraction (XRD) peak intensity. It was also discovered that the transmittance of all multilayers was improved by vacuum annealing process (transmittance above 90% at $T_a = 500^\circ\text{C}$), which is ascribed to the increase of Al atoms diffusing into ZnO layer or the formation of AlO_x . It was noticed that the E_g increases with increasing annealing temperature up to 400°C (Hu *et al* 2006).

In this research, the effects of annealing temperature and thickness on the structural and optical properties of Al/ZnO thin films prepared by rf sputtering method were investigated.

2. Experimental details

The Al/ZnO thin films used in this study were prepared on glass slide substrates using rf magnetron sputtering system. Before use, the glass slides were cleaned in deionized water and then in an ultrasonic bath with methanol, acetone and isopropyl alcohol for 10 min. After cleaning, the glass slides were set in a metal mask. The mask and the glass slides was loaded into the chamber of an Edward FL400 model rf sputtering and rate thickness monitor model Inficon AQM160 machine. ZnO films of thickness 100 nm were the first to be deposited on the glass slides. The target used was ZnO with purity of 99.99%. 99.99% pure argon and oxygen gas mixture are used as the sputtering gas. The flow rate of Ar/O_2 was 0.8 sccm. The base pressure and the target to substrate distance used were 3.19×10^{-3} Pa and 12 cm respectively. RF power was 100 W and the sputtering pressure was fixed on 4.65×10^{-1} Pa. The deposition rate was about 1.2 sccm.

Following the same procedure, aluminium layer of varied thicknesses of 2, 5 and 10 nm were deposited on the ZnO film. The target used was an aluminium with purity 99.99% with rf power at 500 W.

The detailed sputtering conditions are reported in table 1.

After deposition, the samples were cut into several pieces, each of 2.5 cm long for vacuum annealing and characterization. Two samples each were annealed in vacuum using a Carbolite furnace, MTF/12/38/400 model at 200°C and 400°C for a fixed time of 90 min. The crystal structure was characterized by XRD pattern using PANalytical multipurpose diffractometer model X, Pert-Pro which utilizes monochromatic $\text{CuK}\alpha = 1.5406 \text{ \AA}$ radiation operated at 40 KV and 30 mA. The measurement was carried out in the 2θ range from 30.01° to 59.97° . The optical transmittance of the Al/ZnO thin films was measured by Avantes-SAI-07086751 model UV spectrophotometer in conjunction with Avasoft 7.1 software.

3. Results and discussion

3.1. Structural characterization

Figure 1 show the XRD patterns for the as-deposited and annealed Al/ZnO at 200°C and 400°C . The dominant (002) diffraction peaks corresponding to the as-deposited and the annealed Al/ZnO thin films are clearly observed.

The XRD patterns indicate that the as-deposited and annealed Al/ZnO films are crystalline, indicative of a (002) *c*-axis preferred orientations at $2\theta = 34.07^\circ$, 34.14° and 34.43° , for as-prepared, 200°C annealed and 400°C annealed films respectively. Similar result has been observed by different authors according to literature (Hu *et al* 2006, Chun *et al* 2010, Guillen and Herrero 2010). These values fit well with that of the hexagonal ZnO

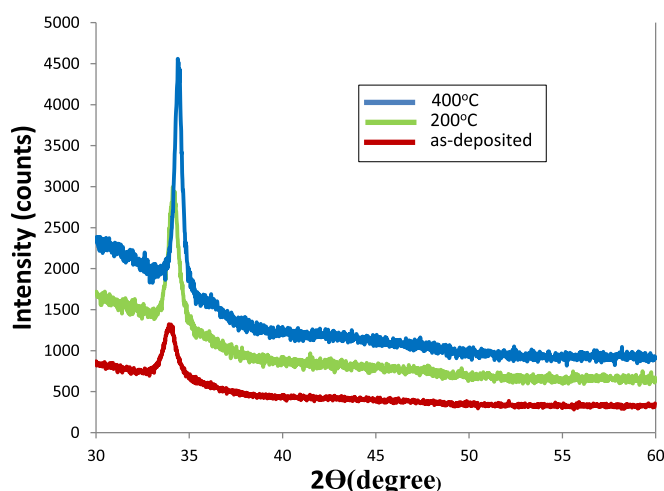


Figure 1. X-ray diffraction patterns for 110 nm thick as deposited and annealed Al/ZnO thin films at different annealing temperatures.

Table 2. Variation of the (002) plane crystallite size and full width at half maximum (FWHM) of Al/ZnO thin films with annealing temperature.

Sample	Annealing temp. (°C)	2θ (degree)	Crystallite size (nm)	FWHM (degree)
Al/ZnO	As deposited	34.07	27.34	0.5304
Al/ZnO	200	34.14	29.63	0.4896
Al/ZnO	400	34.43	43.38	0.3346

thin films with the wurtzite structure (34.45), (according to the Joint Committee on Powder Diffraction Standard). The (002) *c*-axis orientation occurs due to a minimization of the internal stress and surface energy (Bao *et al* 1998). It could also result from an easy growth because of the high atomic density along (002) plane (Amirhaghi *et al* 1994).

The figure shows that the diffraction angles of all the Al/ZnO thin films depart from 34.45° toward the lower angles. This may be due to the doping of Al to Zn leading to an aberration in ZnO crystal lattice (Hong-ming *et al* 2007).

It was also revealed from the figure that the intensity of the peak representing the preferred plane was found to increase with increase in the annealing temperature. This can be interpreted as due to (i) the relaxation of the residual tensile strain and stresses that exist in the Al/ZnO thin films and (ii) an improvement of the crystallinity of the films (Oh *et al* 2005, Hong-ming *et al* 2007).

The average grain size is calculated using Debye Scherer's formula (Cullity 1978)

$$D = \frac{K\lambda}{\beta \cos \theta}, \quad (1)$$

where $K = 0.9$, is the shape factor. $\lambda = 1.54060 \text{ \AA}$, is the x-ray wavelength, θ is the maximum of the Bragg diffraction peak, and β is the full width at half maximum (FWHM) of the XRD peak. We observed that an increase in the annealing temperature of the films introduces variations in the crystallite sizes and FWHM. These variations are shown in table 2 for the (002) plane of the films with thickness 110 nm. Table 2 shows that increasing the annealing temperature results in Al/ZnO films with higher crystallite size and lower width of the diffraction peak (FWHM). Decrease in the width of the diffraction peaks suggests that annealing at higher temperatures produces better quality crystal (Ezema and Nwankwo 2010, Oh *et al* 2011). Similar result has been achieved by Ga-doped ZnO films prepared by radio frequency magnetron sputtering (Xuhu *et al* 2005).

3.2. Optical characterization

3.2.1. Effects of thickness of Al/ZnO thin films on optical properties

Figure 2 reveal the variation of transmittance with the incident wavelength in the visible region (300–800 nm) for the as-deposited Al/ZnO thin films of various aluminium layer thicknesses. All films exhibits high transmittance (above 82%) in the wavelength range 400–550 nm. The high transmittance depicts an excellent

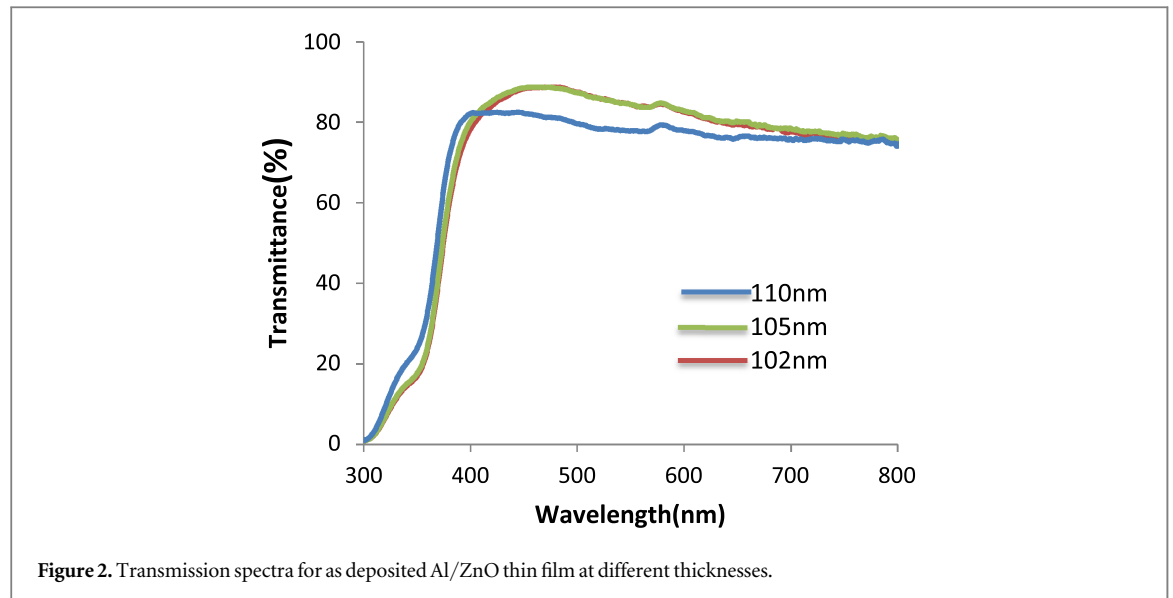


Figure 2. Transmission spectra for as deposited Al/ZnO thin film at different thicknesses.

optical transparency of the films which plays an important role for potential applications in transparent optoelectronic devices. The high transmittance is due to fairly smooth surface and relatively homogeneity of the films (Yanfeng *et al* 2003).

The figure reveals that the transmittance of the as deposited films decreases with increasing thickness (aluminium layer thickness of 2, 5 and 10 nm) within the visible range of wavelength. The transmittance decreases from 89% to 83% when the thickness increases from 102 to 110 nm (i.e. aluminium thickness increase from 2 to 10 nm). Similar transmittance changes in the visible range with increasing film thickness have been observed and attributed to an increase in both reflection and absorption in the films. In addition, films structure may lose its homogeneity with increasing film thickness due to accumulation of different types of structural faults hence increasing the films absorption (Al-Ofi *et al* 2012). The decrease in transmittance as a result of an increase in aluminium layer thickness from 2 to 10 nm may also be attributed to the formation of aluminium cluster or discontinuous aluminium layer at the Al/ZnO interface, which in turn enhances reflection or absorption of the visible light (Hu *et al* 2006). It can also be attributed to optical scattering arising from longer optical paths and also to changes in the carrier concentration (Agashe *et al* 2003, Banerjee *et al* 2006).

The direct E_g was determined from the equation (Blatt 1968)

$$\alpha = (h\nu - E_g)^{1/2}, \quad (2)$$

where α is the absorption coefficient, $h\nu$ is the photon energy and E_g is the energy gap.

By squaring both sides of the equation we have

$$\alpha^2 = h\nu - E_g.$$

The values of the direct optical energy gap, E_g , was obtained by extrapolating the linear portion of the plot of $(\alpha)^2$ versus $(h\nu)$ to $\alpha^2 = 0$.

Figure 3 shows the relation $(\alpha)^2$ versus $(h\nu)$ for as deposited films with different thicknesses.

The values of the optical E_g from the plot are shown in table 3. The estimated values of the optical E_g were found to increase with increasing thickness of the films. This can be attributed to an improvement in the crystal, in morphological changes of the films, in changes of atomic distances, grain size and structural defects in the films (Al-Ofi *et al* 2012).

3.2.2. Effects of annealing temperature on the optical properties of Al/ZnO thin films

Figures 4–6 shows the spectral behaviour of the transmittance, T in wavelength range (300–800 nm) for Al/ZnO thin films of thicknesses 110 nm, 105 nm and 102 nm, respectively at different annealing temperatures. It is clear from the figures that the optical transmittance increases with increasing annealing temperature.

The increase in transmittance may be as a result of increase in the particle size and the number of voids present in the films due to increasing annealing temperature which may lead to a decrease in optical scattering (Hong-ming *et al* 2007).

The allowed direct optical energy gap, E_g obtained by extrapolating the linear portion of the plots of $(\alpha)^2$ versus $(h\nu)$ to $\alpha = 0$ for annealed 102 nm, 105 nm and 110 nm thick Al/ZnO thin films is shown in figures 7, 8 and 9, respectively. The obtained values of E_g for all the films at different annealing temperatures are listed in

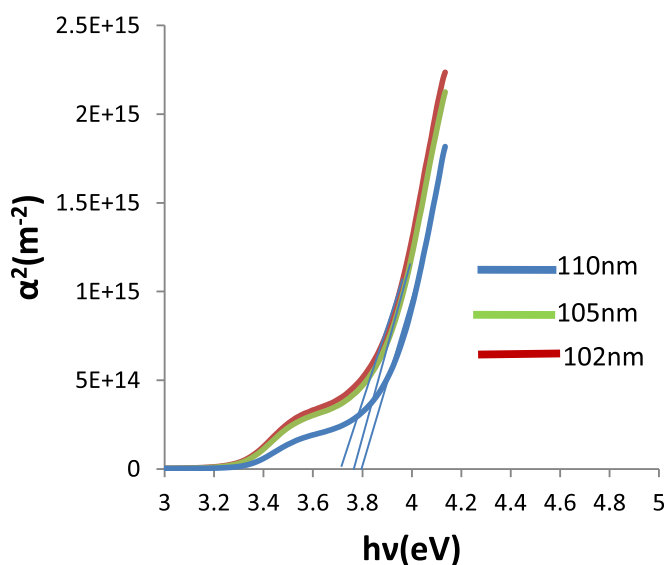


Figure 3. Plot of $(\alpha)^2$ versus $(h\nu)$ for as deposited films with different thicknesses.

Table 3. Values of E_g for as deposited Al/ZnO thin films at different thicknesses.

Thickness (nm)	Energy (eV)
102	3.73
105	3.76
110	3.80

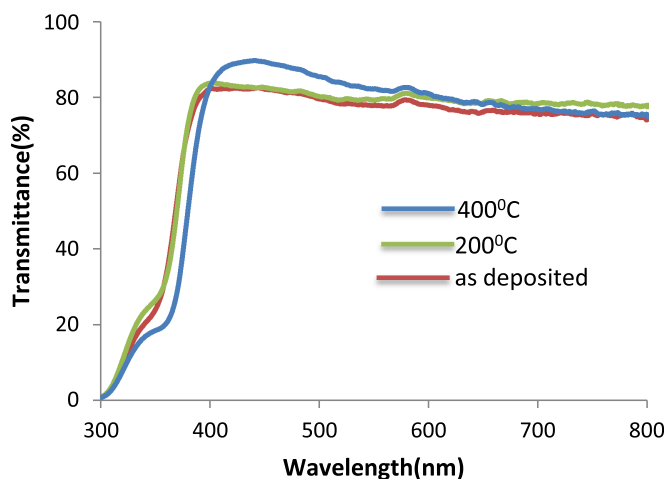


Figure 4. Transmission spectra for 110 nm thick Al/ZnO thin film at different annealing temperature.

table 4 where E_g increases with increasing annealing temperature. This observation is similar to the result of Al-Ofi *et al* (2012) who suggested that the effect can be interpreted by assuming that during the annealing process the films will have time for some atomic rearrangement to take place. So, some defects will be removed which reduces the density of dangling bonds, redistributing atomic distances and bond angles and the optical gap will then increase. It is generally believed that the Burstein–Moss shift plays a key role in this phenomenon (Shan and Yu 2004), it states that the Fermi level inside the conduction band moves upward, and hence the optical band gap is widened, with increasing carrier concentration due to filling of the conduction band by increase of electron carriers.

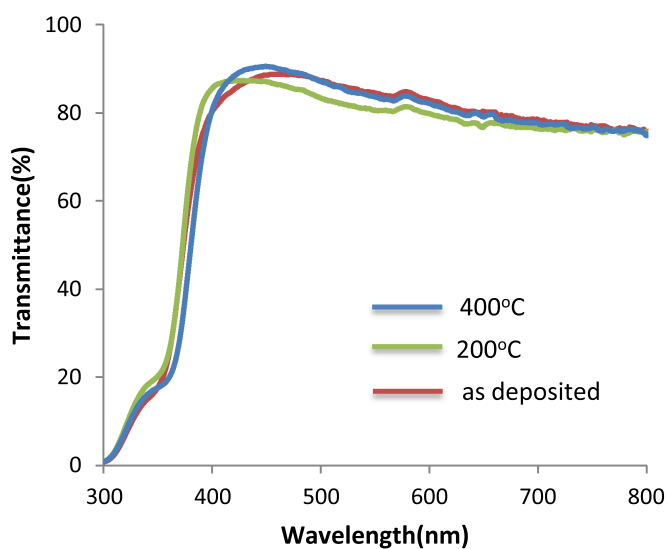


Figure 5. Transmission spectra for 105 nm thick Al/ZnO thin film at different annealing temperature.

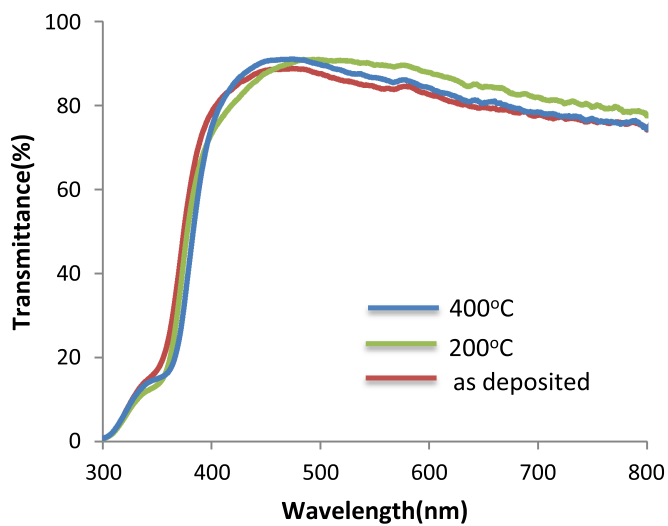


Figure 6. Transmission spectra for 102 nm thick Al/ZnO thin film at different annealing temperature.

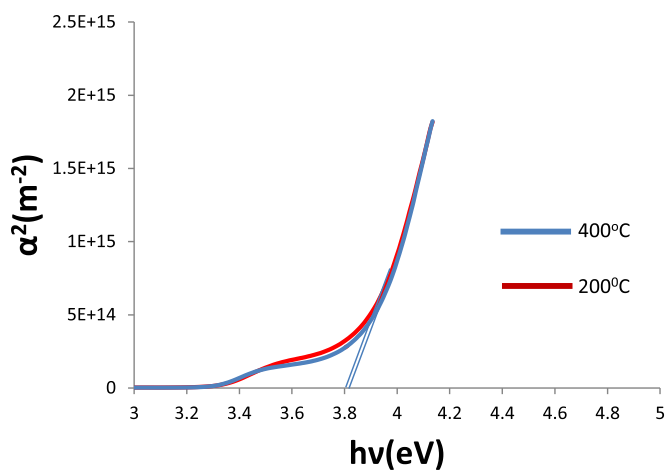


Figure 7. Plot of α^2 against $h\nu$ for 110 nm thick Al/ZnO thin film at different annealing temperature.

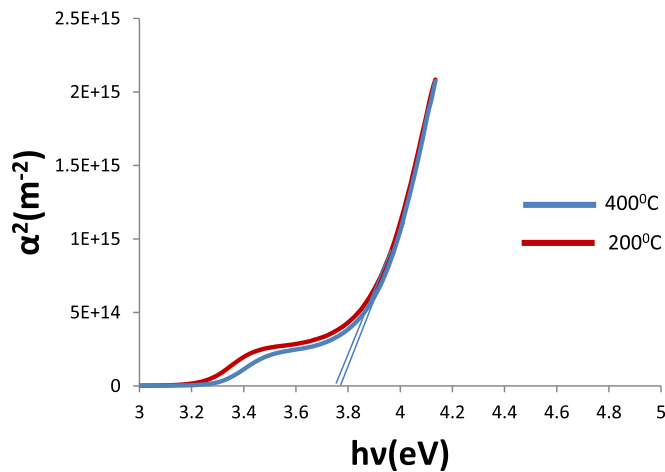


Figure 8. Plot of α^2 against $h\nu$ for 105 nm thick Al/ZnO thin film at different annealing temperature.

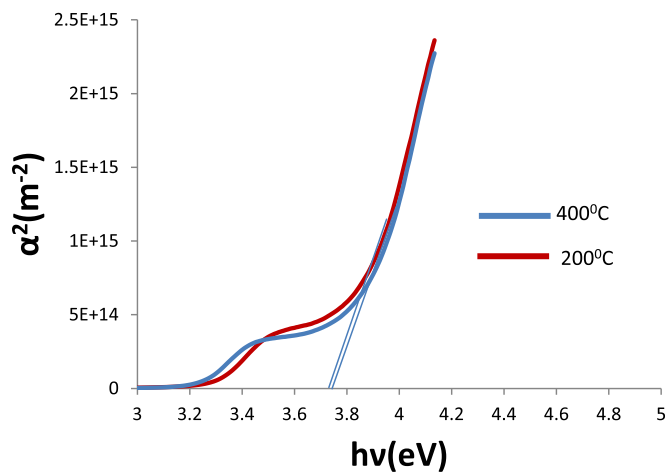


Figure 9. Plot of α^2 against $h\nu$ for 102 nm thick Al/ZnO thin film at different annealing temperature.

Table 4. Values of E_g for Al/ZnO thin films with different thicknesses at different annealing temperatures.

Annealing temp. (°C)	Thicknesses		
	102 nm	105 nm	110 nm
200	3.74	3.77	3.81
400	3.76	3.79	3.83

The energy band gap broadening (ΔE_g) can be expressed in the following (Wang *et al* 2011):

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

where h is Planck's constant, m^* is the electron effective mass in the conduction band and n_e is carrier concentration. The higher the carrier concentration the higher the band gap (Hao Chen *et al* 2009, Kumar Rajesh and Rao Subba 2011).

4. Conclusion

Al/ZnO thin films were prepared on glass substrates by rf sputtering method, and the effects of thickness and annealing on the films were studied by measuring structural and optical properties. The films showed a crystalline structure with (002) preferred plane which have its peak intensity increased as the annealing

temperature is increased. The increase in peak intensity is due to the relaxation of the residual tensile strain and stresses that exist in the Al/ZnO thin films and an improvement in the crystallinity of the films. Increasing the annealing temperature also results in Al/ZnO films with higher crystallite size and lower width of the diffraction peak (FWHM). The optical transmittance of the thin films, both as deposited and annealed, was in the range of 82%–91%. The high transmittance depicts an excellent optical transparency of the films which plays an important role for potential applications in transparent optoelectronic devices. It was also observed that the thin films show a decrease in transmittance as the aluminium layer thicknesses of the films increases, but an increase as the annealing temperature is increased. The decrease in transmittance with increasing aluminium thickness may be attributed to the formation of aluminium cluster or discontinuous aluminium layer at the Al/ZnO interface, which in turn enhances reflection or absorption of the visible light. The increase in transmittance with increasing annealing temperature may be as a result of increase in the particle size and the number of voids present in the films due to increasing annealing temperature which may lead to a decrease in optical scattering. The optical E_g increases as the thickness and annealing temperature increases. The range of the E_g is 3.73–3.80 eV when the thickness increases from 102 to 110 nm and 3.74–3.83 eV when the annealing temperature increases from 200 °C to 400 °C depending on the thickness. The increase in E_g due to increasing thickness can be attributed to an improvement in the crystal and changes in the morphology of the films. All the samples investigated are characterized by wide band gaps which make them attractive for the fabrication of higher efficient optoelectronic devices.

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