Investigation of dilute aluminum doped zinc oxide thin films: structural and morphological properties for varies applications

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To form a better view of the influences of Al in ZnO, the crystalline structure parameters and morphological (via SEM) of $Zn_{1-x}Al_xO$ (x=0.0, 0.02, 0.04, 0.06, 0.08 and 0.10) thin films organized onto glass substrates using by spin coating technique. The effects of Al doping on the structural ZnO nano crystalline films are investigated using (XRD), (EDAX) and (SEM). Rietveld refinement was used to examine the XRD patterns of Zn₁. _xAl_xO thin films. This was done using Fullprof software. The XRD results showed that Al ions successfully replaced the Zn²⁺ lattice sites without any major change in the structure after Zn²⁺ substitution, and their crystallinity decreased with increasing Al doping content. Also, The XRD analysis confirmed the hexagonal structure. Lattice constant, Cell volume, Atomic Packing Fraction and surface density have been calculated. The microstructural parameters, crystallite size and lattice strain of Zn_{1-x}Al_xO thin films were calculated. The changes in microstructural parameters were discussed as dependent Al concentration. The Zn–O bond lengths and bond angle of Zn_{1-x}Al_xO were determined and have changed.

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1. Introduction

In recent years, many metal oxides such as (ZnO), (ZnS), (SnO₂), and (In₂O₃), whether pure or doped, have been intensively studied. Among all these metal oxides, ZnO has gained prominence, due to its availability, it is non-toxic and low-cost material compared to other materials. ZnO is a binary semiconductor (II-VI). [1-8], and has n-type metal oxide semiconductor [9], and its direct band gap varies between 3.44 eV and 3.37 eV at room temperature. ZnO has a hexagonal crystal structure [10]. The electrical conductivity of Zeno is inherently defective due to its wide band gap, such as interstitial zinc atoms and oxygen vacancies [11]. So far, many different methods have been developed to fabricate ZnO nanopowders and thin film studies [12]. In addition, several methods such as sol-gel [13], co-precipitation method [14], sputtering [15] chamical bath deposition [16], hydrothermal [17], spray pyrolysis [18], thermal evaporation [19] and microwave combination [20] were used. It has various applications such as cathode ray tubes, light emitting diodes, transparent electrodes for solar cells, surface acoustic wave (SAW) devices, hydrogen storage, ceramics, catalysts, antibacterial activity, photoelectric switches, selective filters in gas sensors [21], transparent thin film transistors, UV detectors, and antifungal agents [22]. Addition of impurities to ZnO through proper process changes its electronic and optical properties [23, 24]. The physical properties of ZnO are strongly influenced by the presence of dopants [25]. Some dopants such as Co, Fe, Cr, Al, Cu, Mn, Mg, S, P, etc. can lead to increased area of the of ZnO powders. Al³⁺doped ZnO nanoparticles with lesser crystallinity and high surface area have been reported to have higher catalytic activity and higher sensitivity than pure ZnO or Al2O3 [26].

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Al-doped ZnO (ZnO:Al) films have recently emerged as alternative potential candidates for ITO films, not only because of their optical and electrical properties comparable to ITO films, but also because of their higher thermal and chemical stability when was exposed to hydrogen plasma [27] as ITO.

Moreover, they are abundant, of low cost and non-toxic. In our work, $Zn_{1-x}Al_xO$ (x=0.0, 0.02, 0.04, 0.06, 0.08 and 0.10) thin films on a glass substrate with a fixed thickness (200 nm) by low-cost sol-gel spin coating technique. Morphology of the films has been investigated by Scanning Electron Microscopy (SEM) and various structural parameters e.g. Lattice constant, Cell volume, Atomic Packing Fraction and surface density have been calculated. The microstructural parameters, crystallite size and lattice strain of $Zn_{1-x}Al_xO$ thin films were calculated. The analysis indicates that the $Zn_{1-x}Al_xO$ thin films were uniformly distributed with smooth grain boundaries. Comparison of these parameters of all ZnO films with different dilute Al content leads to in-depth understanding of various physical properties that depends on surface morphology and structural parameters for varies applications.

2. Experimental

2.1. Thin film prepared by sol-gel electron beam gun method

Zinc acetate dehvdrate $(Zn(CH_3COO)_2.2H_2O),$ isopropanol (C_3H_7OH) and monoethanolamine (C₂H₇NO) were used as precursor, solvent and solution stabilizer, respectively. aluminium acetate (Al(CH₃CO₂)₃) is also used as a doping element. A 0.5 M ZnO solution was prepared by dissolving 1.0975 g of zinc acetate in 10 ml of isopropanol and then adding different amounts of different concentrations of the doping element (iron acetate) to the solution. The doping concentration of Al in the ZnO solution was kept at 0%, 2%, 4%, 6%, 8% and 10%. Stir the solution vigorously for 60 min at 65 °C using a magnetic stirrer on a hot plate. Maintaining a 1:1 molar ratio of zinc acetate to monoethanolamine, the cloudy solution became homogeneous and transparent by adding monoethanolamine dropwise to the solution. The solution was stirred for an additional hour and aged at room temperature for 24 hours. Film deposition was performed using a typical spin coater rotating at 2000 rpm. Preheat the prepared samples by placing them on a hot plate at 150 °C for 10 min. By repeating the spin coating and prebaking process 8 times, the film thickness was increased to 200 nm. Finally, the films were tempered in a conventional muffle furnace at 500 °C for 2 hours.

3. Results and discussion

3.1. Energy-dispersive analysis X-ray (EDAX) spectra

Energy dispersive X-ray studies (EDAX) were performed to probe the elemental composition of the synthesized samples and confirm the successful doping and formation of ZnO NPs. Energy dispersive X-ray analysis of all Aluminium metal-doped metal oxide semiconductor films is shown in the spectra in Fig. 1(a and b). Furthermore, it is interesting to observe the absence of impurities in the spectra. Added confirmation of sample purity. Al peaks along with zinc and oxygen were found in the sample doped with 10 at. % Al. This means that Al has been successfully doped.



Fig. 1. Energy-dispersive analysis X-ray (EDAX) spectrum of (a) ZnO, (b) $Zn_{0.9}Al_{0.1}O$ thin films.

3.2. XRD analysis

Fig.1 shows the X-ray diffraction patterns of pure ZnO powder sample that has polycrystalline phase with a hexagonal wurtzite phase and the diffraction peaks are oriented on the (100), (002), (101), (102), (110), (103), (112) and (201) planes of ZnO. All peaks are consistent with JCPDS data card #01-079-0208. Fig. 3 (a) illustrates the Al-doped ZnO thin films and the XRD patterns of the ZnO samples confirmed the formation of a polycrystalline phase with a hexagonal wurtzite phase. The preferred c-axis orientation of ZnO was found to be along the (002) plane due to the presence of vacuum during preparation of the films, which causes the ease of crystal growth due to minimal internal stress, minimal surface free energy and high atomic density. However, the peak positions for different dopant concentrations of Al:ZnO change slightly, indicating that Al binds to the ZnO material without changing the crystal structure [28, 29]. The full-width at half-maximum (FWHM) corresponding to these planes increases with the increase of Al-doping concentrations as shown in Fig. 3 (b).

The intensities of the (002) peaks gradually decrease with increasing Fe doping concentration, as shown in Fig. 2. The decay of the peak indicated that the substitution of Al ions by Zn ions inhibited the crystal growth of ZnO. This is related to the Al doping concentration that affects the stress of ZnO films [30 replace 28]. The shift of the peak position to lower angles is evident, and the distance between the planes becomes shorter due to the strain within the ZnO lattice. From the above results, it can be concluded that an appropriate Al doping concentration will reduce the crystal quality of ZnO and it is also confirmed that ZnO and Al doped ZnO are the most stable phases [29]. The peak shifts correspond to the elongation of the compounds and the substitution of Al for some of the zinc cations in each compound. Replacing iron in the lattice with zinc changes the elongation, as shown by the peak shift [30]



Fig. 2. XRD patterns for ZnO powder.



Fig. 3. (a) XRD patterns of $Zn_{1-x}Al_xO$ films with different Al composition (x=0, 0.02, 0.04, 0.06, 0.08 and 0.1mol.), (b) peak shift of (002) plane.

It should be revealed that the lattice parameters are improved due to Al doping. In addition, a Rietveld modification was performed to analyze the XRD patterns of the thin films. This is done using software of Fullprof software. The hexagonal-type structure was found to be most suitable for both undoped and doped samples. The intensities within different diffraction peaks are changed, indicating that the growth of ZnO on different planes is different and the growth is anisotropic. The Rietveld XRD patterns of (a) ZnO and (b) $Zn_{0.96}Al_{0.04}O$ thin films observed in Figure 4 show different degrees of crystallinity without any impurity phase in the films.



Fig. 3. Reitveld refined XRD pattern for (a) ZnO and (b) Zn_{0.96}Al_{0.04}O thin films.

3.3. The lattice parameters

The lattice parameters of hexagonal 'a' and 'c' of ZnO and Al doped ZnO films can be calculated using the next equation [31].

$$\frac{1}{d_{hk\ell}^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{\ell^2}{c^2}$$
(1)

where d is the interplanar space and (hkl) are the Miller index. Using the Bragg equation $n\lambda = 2d_{nk\ell} \sin\theta$ The values of 'a' and 'c' is obtained from the relations

$$a = \frac{\lambda}{(\sqrt{3}\sin\theta_{100})}$$
 & $c = \frac{\lambda}{\sin\theta_{002}}$ (2)

The values of lattice parameters "a" and "c" are in good agreement with the reference values for ZnO crystals (a = 3.250 Å & c = 5.207 Å), indicating the homogenous of the ZnO thin films. The values of lattice parameters "a" and "c" are lower than those of bulk that are strong indicated of the presence of stresses in the films [34]. In Fig. 5, it can be observed that the lattice parameters "a" and "c" increase with increasing Al content in $Zn_{1-x}Al_xO$ thin films. The lattice parameter c/a ratio is almost equal to 1.60, which indicates that the shape of ZnO and Al doped ZnO wurtzite structures is reasonably perfect. The values of lattice parameters "a", "c" and c/a are listed in Table 1.



Fig. 4. Lattice parameters "a" and "c" against Al concentration for Zn_{1-x}Al_xO thin films.

3.4. Atomic Packing Fraction (APF)

It defines as the fraction of volume in a crystal structure that is occupied by constituent particles. It is a dimensionless quantity and always less than unity. Atomic packing fraction (APF) for hexagonal was calculated by using the formula [32]

$$P = \frac{2\pi a}{3c\sqrt{3}} \tag{5}$$

where a and c are lattice parameters [33] .The results show that the APF increases with the iron content, which may be due to the increase of voids in the samples. The APF of the bulk hexagonal ZnO material is about 75%, but in this study, the APF of the $Zn_{1-x}Fe_xO$ film is greater than 75%, indicating that the APF in the nanocrystals is slightly larger than that in the bulk material. This is attributed to size effects in nanocrystalline thin films. The APF increases with the Fe content in the $Zn_{1-x}Al_xO$ films, indicating uniform substitution of Fe ions in the zinc sites of the ZnO structure [34]. The atomic packing fractions (APFs) of the $Zn_{1-x}Al_xO$ films are listed in Table 2.

3.5. Volume of unit cell and X-ray density

The volume of unit cell was calculated using the equation [35]

$$V = \frac{\sqrt{3}}{2}a^2c \tag{6}$$

where a & c are lattice parameters. It is found that the unit cell volume increases with increasing Al content, which can be attributed to the increase of lattice parameters a and c, and the unit cell volume values are shown in Table 1. This suggests that Al ions have moved to available zinc sites in the structure.

The number of unit cells in the particle is calculated from the equation [36]

$$N = \frac{4}{3\pi \frac{D}{2V}}$$
(7)

The X-ray density of ZnO sample was calculated by using the formula [37]

$$\rho = \frac{nM}{N_A V} \tag{8}$$

where, ρ is X-ray density, *n* is the number of atoms per unit cell, *M* is the molecular weight of the sample N_A is Avogadro's number and *V* is the volume of unit cell. The X-ray density reduces with increasing Al content of Zn_{1-x}Al_xO nanoparticles. This may be due to a increase in unit cell volume and a decrease in sample molecular weight. The molecular weight of Al ions (26.98 amu) is smaller than that of Zn ions (65.38 amu). As the Al content increases. The volume of the unit cell increases and the X-ray density decreases, this means that Al ions enter the Zn sites in the ZnO structure [38].

Conc. x %	a (Å)	c (Å)	c/a	$V(Å)^3$	E _a	ε _c
0	3.0181	5.0846	1.6847	40.109	5.6591E-4	0.0333
2	3.0189	5.0963	1.6881	40.224	8.4911E-4	0.0357
4	3.0215	5.1058	1.6898	40.3672	0.0017	0.0376
6	3.024	5.1117	1.6903	40.4826	0.0026	0.0388
8	3.0266	5.1175	1.6908	40.5983	0.0034	0.04
10	3.0292	5.1224	1.691	40.7063	0.0043	0.041

Table 1. Morphological parameters of $Zn_{l-x}Al_xO$ *thin films.*

3.6. Specific surface area

It is a property of solids defined as the total surface area of a material per unit of mass and can be used to determine the type and properties of a material. The values obtained for specific area depend on the method of measurement. The specific surface area can be calculated from particle size given by [39]

$$S = \frac{6x\,10^8}{Dx\,\rho} \tag{9}$$

The specific surface area decreases with iron incorporated as shown in Table 2.

3.7. Crystal size and lattice strain

The average crystallite size D of the films has been identified by Debye–Scherer's Eq. [40]

$$D = \frac{k\lambda}{\beta_{Crystallite} \cos\theta}$$
(10)

The average crystallite size of Al-doped ZnO sample was reduced with rising in the concentration of Al dopant owing to the deformation of ZnO matrix affected by the accumulation of Al ions which can lead to decrease the nucleation and growth rates of Al doped ZnO nanoparticles. Drop of crystallize size signifies combining from good crystallinity. The calculated crystallite size values are organized in Table 2.

From this study, dislocation density (δ) of the films are determined by using following equation:

$$\delta = \frac{1}{D^2} \tag{11}$$

The dislocation density of the films determines the quality of the film and its defect structure. In this study, Al doping was incorporated into the ZnO matrix, and the dislocation density gradually increased, indicating its defect structure. Strain towards c-axis and towards a-

axis can be calculated from the formula as below as $\varepsilon_c = ((c-c_\circ) / c_\circ) \times 100\%$ and $\varepsilon_a = ((a-a_\circ) / a_\circ) \times 100\%$. The δ and " ε_c , ε_a " data are tabulated in Table 2 and 1 respectively.

The lattice strain (ϵ) can be calculated from Stoke and Wilson Eq. [41]

$$\varepsilon = \frac{\beta_{strain}}{4tan\theta} \tag{12}$$

The β can be corrected form the following relationship

$$\beta = \sqrt{\beta_{obs}^2 - \beta_{std}^2} \tag{13}$$

where β_{obs} is the integral peak width of film, and β_{stat} is the peak width of standard (silicon). The average crystallite size and lattice strain of the pure ZnO and Al/ZnO films are shown in Fig. 5. The crystallite size reductions with growing Al incorporation but the lattice strain increases. Both trend of *D* and ε may attribute to the change in the lattice size with Al substitution by Zn because the ionic radius of Al (Al³⁺: 0.53 Å) is slightly smaller the ionic radius of Zn (Zn²⁺: 0.60 Å). The observed decrease in *D* and increase in ε may attribute to (i) the combined effect of lattice distortion of the host ZnO crystal due to the substitution of smaller ion-sized iron atoms [42] and (ii) It was reported that a thin Al-O-Zn layer was formed on the surface of the doped samples due to the presence of excess iron ions in the precipitation solution, which hindered the crystal growth [43].



Fig. 5. Crystallite size and lattice strain $Zn_{l-x}Al_xO$ films as a function of Al content.

Table 2. Corrected (FWHM), crystallize size, lattice strain, dislocation density, X-ray density,
packing factor and surface energy, respectively for $Zn_{1-x} \mathcal{A}l_x O$ thin films.

Conc. x %	Corrected β	D (nm)	æ	δ x 10 ⁻⁴ (nm) ⁻²	ρ (g/cm³)	Р	S (cm²/g)
0	0.378411	22.94538	0.005354	18.99371	6.738441	71.77394	38.8058
2	0.442274	19.63161	0.006259	25.94705	6.719176	71.62952	45.48616
4	0.50423	17.21802	0.007143	33.73133	6.695338	71.55753	52.04698
6	0.549982	15.7844	0.007798	40.13689	6.676267	71.53596	56.93633
8	0.588109	14.75991	0.008346	45.90208	6.657235	71.51441	61.06236
10	0.602407	14.40844	0.008557	48.16883	6.639575	71.50723	62.71826

3.8. Bond length and bond angle

Different atoms all combine with each other and become stable. This bonding is achieved by forming bonds. There are different types of bonds, namely ionic or electrovalent bonds, covalent bonds and coordinate bonds. This in turn indicates that each bond is associated with a specific property. Bond length describes the distance between the nucleus centers of two bonded atoms in equilibrium. The stronger the attraction between the bonded atoms, the smaller the bond length. However, the larger the atomic size, the longer the bond length. Bond angle refers to the angle between two bonds, that is, the angle between two orbitals that contain a pair of bonding electrons around the central atom of a complex molecule or ion.



Fig. 6. Unit cell of ZnO.

The Zn–O bond length L is given by [44]

$$\mathbf{L} = \sqrt{\left(\frac{\mathbf{a}^2}{3} + \mathbf{c}^2 \left(\frac{1}{2} - \mathbf{u}\right)^2\right)} \tag{14}$$

where a and c are lattice parametes, u is the positional parameter in the wurtzite structure and measures the amount which each atom gets displaced with respect to the next [45]. and u is given by

$$\mathbf{u} = \frac{\mathbf{a}^2}{3\mathbf{c}^2} + \frac{1}{4} \tag{15}$$

The Zn–O bond length calculated according to this study is from 1.9758 Å to1.9763Å. whereas the reported Zn–O bond length in the unit cell of ZnO and neighboring atoms is 1.9767 Å [46]. There is a good agreement of the calculated & actual bond length which supports the results of present study. The value of bond length is given in Table 3.

In additional, there are three types of second-nearest neighbors, which designated as b_1 (one along the direction), b_2 (six of them) and b_3 (three of them) with the bond lengths, shown in Fig. 6 are calculated as [47]

$$\mathbf{b}_1' = \mathbf{c}(1 - \mathbf{u}) \tag{16}$$

$$\mathbf{b}_{2}' = \sqrt{\mathbf{a}^{2} + (\mathbf{uc})^{2}}$$
 (17)

$$b_3' = \sqrt{\frac{4a^2}{3} + (\frac{1}{2} - u)^2 c^2}$$
(18)

As the Al doping concentration increases, the distance of the second-nearest neighbors also decreases. This is due to the increased occupancy of the dopant. The variation of the second-nearest neighbor as a function of Al doping concentration is Table 3. It is interesting to note that both distances b_2^{*} and b_3^{*} are equal for Wurtzite geometry. The bond angles are given by,

$$\alpha = \frac{\pi}{2} + \cos^{-1} \left[1 + 3\left(\frac{c}{a}\right) \left(-u + \frac{1}{2} \right)^2 \right]^{-1} \tag{19}$$

$$\beta = 2\sin^{-1} \left[\frac{4}{3} + 4\left(\frac{c}{a}\right)^2 \left(-u + \frac{1}{2} \right)^2 \right]^{-1}$$
(20)

The calculated bond angles are coinciding with the standard value ($\alpha = \beta = 109.47$). The ratio between α and β is one for the ideal case. But the ratio is 0.98 for the Al doped ZnO nanoparticles irrespective of the Al doping concentration. All the evaluated values are presented in Table 3.

Table 3. Potential parameter, bond length, sec nearest neighbor bond length, one along c-direction, ecnearest neighbor bond length six along c-direction, sec nearest neighbor bond length three along c-direction and both bond angle for $Zn_{1-x}Al_xO$ thin films.

Conc. x %	u	b (Å)	$b_{1}'(Å)$	$b_{2}'(Å)$	$b_{3}'(Å)$	α(°)	β(°)
0	0.3674401	1.868291	3.216323	3.549528	3.549528	111.1472	107.7444
2	0.366968	1.870181	3.226126	3.55125	3.55125	111.2548	107.6302
4	0.366733	1.872454	3.233316	3.554628	3.554628	111.3085	107.5731
6	0.3666626	1.874252	3.237401	3.55776	3.55776	111.3246	107.5559
8	0.3665923	1.876054	3.241495	3.560898	3.560898	111.3407	107.5388
10	0.3665689	1.877722	3.244703	3.563969	3.563969	111.3461	107.5331

3.9. Scanning Electron Microscopy (SEM)

The morphology and particle size of Al/ZnO films were examined by using SEM are shown in Fig. 7. The entire SEM images shows the films comprises of mostly spherical formed and the average size of the films is of the order of nano-meter size. The SEM images exhibit particle size of $Zn_{1-x}Al_xO$ films which are in the range 24, 17 and 12 nm (x=0, 0.06 and 0.10). Another logical point of view from the SEM images is that increasing Al doping results in a regular decrease in average particle size. In our current study, increasing the doping percentage resulted in a decrease in interstitial zinc due to the dopant concentration. Therefore, the grain growth of ZnO is suppressed due to the decrease in diffusivity.

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Fig. 7. SEM micrographs of $Zn_{1-x}Al_xO$ thin films with (x=0, 0.06 and 0.10).

4. Conclusions

In this paper, different composition of dilute semiconductor 0f $Zn_{1-x}Al_xO$ thin films undoped and Al doped ZnO thin films were prepared onto glass substrates by sol-gel coating technique in order to investigate the ZnO based thin films' behavior as a function of Al concentration. The XRD result indicated that the Al ions were successfully diffuse substantially into the ZnO lattice without any significant change in the structure and their crystallinities were deteriorated with increasing Al doping content. The calculated values of lattice parameters "a" and "c" are in good agreement with the standard values for ZnO single crystals (a = 3.250 Å and c = 5.207 Å), indicating the quality of the ZnO thin films.

The lattice parametes "a" and "c" increase with increasing Al content in $Zn_{1-x}Al_xO$. The lattice parameter c/a ratio is almost equal to 1.6 which indicates that the shape of ZnO and Al doped ZnO wurtzite structures is reasonably perfect. The crystallite size decreases with increasing Al incorporation, but the lattice strain increases. Both trend of *D* and ε may attribute to the change in the lattice size with Al diffuse substantially in Zn lattice because the ionic radius of Al (A3⁺: 0.53 Å) is slightly smaller the ionic radius of Zn (Zn²⁺: 0.60 Å). The observed decrease of *D* and increases in lattice strain can be attributed hindering of the crystal growth due to the formation of a thin layer of Al–O–Zn on the surface of the doped samples due to the presence of excessive iron ions in the precipitation solution. The second-nearest neighbor distances were calculated for Al doping ZnO nanoparticles for the first time.

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