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EFFECTS OF SUBSTRATE TEMPERATURE ON THE STRUCTURAL PROPERTIES OF ZnO:Al THIN FILM DEPOSITED BY ELECTROSTATIC SPRAY DEPOSITION

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ABSTRACT

Zinc Oxide and Al-doped ZnO (AZO) thin films were prepared by Electrostatic spray deposition method on soda lime, glass substrates. The effects of substrate temperatures (300, 350, 400 and 450 ^{O}C) on the Structural properties of the films were investigated. X-ray diffraction analysis indicates that the films were polycrystalline, having hexagonal (Wurtzite) structure irrespective of their substrate temperature and exhibits a preferred orientation along the (002) plane. The micro structural parameters such as particle size, strain and dislocation density were affected and calculated. The particle size of the AZO was found to be within the range of 70.6 nm to 95.34nm. The thickness of the film decreases with increase in substrate temperature and is within the range of 0.23 to 0.15µm.

Key words: Zinc Oxide; Electrostatic spray deposition; Structural properties; TCO; Temperature.

1. INTRODUCTION

Transparent conducting oxides (TCOs) are electrical conductive materials with a comparably low absorption of light. They combine low electrical resistance with high optical transparency in the visible range of the electromagnetic spectrum (Huiyong, Avrutm, Izyumskaya, Ozgar and Morkec, 2010). These properties are sought in a number of applications, notably as electrodes materials in solar cells, light emitting diodes, flat panel displays and other optoelectronic devices where an electric contact needs to be made without obstructing photons from either entering or escaping from the optical active area and in transparent electronics such as transparent field effect transistors (Mugah, Okumu and Njoroge, 2012). Another property of TCO is that although they are transparent in the visible range, they are highly reflective for infrared light. These properties are responsible for today's dominant use of TCOs as an energy conserving material.

In the last four decades the dominant TCOs have been tin oxide (SnO_2), Indium oxide (In_2O_3) and Zinc oxide (ZnO).But ZnO have some advantages over these commonly used materials due to its unique combination of appealing properties such as non-toxicity, good electrical and optical behaviour, abundance in nature which makes it cheap and stability in hydrogen plasma (Tewari and Bhattacharjee, 2010).

ZnO is an n-type semiconductor of hexagonal (wurtzite) structure with a direct energy wide band gap of about 3.37ev at room temperature, whose electrical conductivity is mainly due to intrinsic defect such as interstial zinc atom or oxygen vacancies. But the use of intrinsic ZnO films is limited due to its low reflectance in IR region (Pawar, Jadkar and Takwale, 2005). However its electrical conductivity can be increased by doping thoroughly with appropriate ions using various doping processes, as well as by annealing treatment in controlled atmosphere. The choice of Aluminium is made as a dopant material because of its abundant availability, which makes it cheap, its non-volatility, high potentiality and its gas sensing properties which enhances the gas sensing properties of the ZnO thin films which has immensely important industrial and domestic applications for detecting hazardous gases (Tewari and Bhattacharjee, 2010).

A survey of literatures shows that the properties of doped and undoped ZnO films depend not only on chemical composition but also on the preparative methods. These methods includes Rf magnetron sputtering (Jeong, Lee and Boo, 2003;Abdalla, 2012; Agashe, Kluth, Schope, Siekmann, Hupkes and Rech, 2002); chemical vapour deposition (Buba and Adelabu, 2010; Soderstrom, Domine, Feltrin, Despeisse, Meilland, Bugnon, …Ballif,2010; Kim, Yun and Kim, *2010);* sol gel (Fatima and Devadson, 2011; Alhamed and Abdullah, 2010; Tang and Cameron, 1994; Jun, Park and Koh, 2012); Pulsed laser deposition (Myoung, Yoon, Lee, Yun, Bae and Lee2002), (Diaz, Rodringuez, Duarte, Orrantia-Borunda, Castro-Rodriguez, Perez-Quintana and Iribarren,2011; Kim, Piqu, Horwitz, Murata, Gilmore and Chrisey,2000) and spray pyrolysis (Kaid&Ashour, 2006), (Hung, Henry, Kuznetsov, Basheer, Viadimir, Egdell, … Peter, 2012), (Bakha, Bendimerad&Hamzoui, 2011) among others. Of these methods, highly conductive and transparent doped ZnO films have been prepared by d.c. and r.f magnetron sputtering methods (Martin, Espinos, Justo, Holgado, Yubero and Gonzalez-Elipe,2002). Aluminium (Al)-doped ZnO thin films prepared by these methods, however have important constraints as structural imperfection, non-uniform lateral and volume distribution of the dopant elements and instability of the electrical properties in the films obtained (Martin *et al.*, 2002).

Electrostatic spray deposition (ESD) is a useful alternative to this commonly used methods for obtaining ZnO:Al thin films and has been rarely reported for depositing doped and undoped ZnO. ESD is of particular interest because of its simplicity, low cost and minimal waste production. ESD involves the generation of an aerosol from a precursor solution when a strong electric field is applied between a metal nozzle and a substrate. Hence we report for the first time the effects of substrate temperature on the Structural properties of ZnO:Al thin film using Electrostatic spray deposition.

2. EXPERIMENTAL DETAILS

Deposition Parameters

S/no	Parameter	Deposition details
1	Substrate	Soda lime glass (5 Samples)
2	Substrate to nozzle Distance	10cm
3	Substrates temperature	300, 350,400 and 450^{0}C for AZO and 400^{0}C for ZnO.
4	Deposition time for each sample	15 minutes
5	Volume of spray	0.8 ml
6	Flow rate	0.05 ml/min
7	Atomizing Voltage	12.2 kv
8	Concentration of solution	0.4M

PROCEDURE

The soda lime glass substrate is first cleaned with distilled water and air dried for about 10 minutes, after which it was placed on the substrate heater via the substrate holder. 0.4M Zinc Acetate (99.99%, BDH) solution prepared by dissolving a solute quantity of 1.756 of Zinc Acetate in 40ml of solvent (Ethanol: water (80:20v/v)) was used in depositing ZnO thin film while Aluminium doping was achieved by adding aluminium chloride (99.50%, BDH) at 3at % to the starting solution. This is enhanced by using a magnetic stirrer for about 15minutes with addition of a drop of acetic acid to facilitate the complete dissolution of the solute in the solvent and to obtain a homogeneous transparent solution. A syringe pump (5ml- Hypodermic syringe, manufactured by Shandong Zibo Shanchuan medical instrument Co. Ltd, Shandong China- LOT NO: 201312) was used to feed the precursor solution through silicon tubing connected to a small stainless steel needle (0.184mm and 0.3366mm, inner and outer diameter respectively) for atomization. D.C (5-20Kv) voltage is applied between the needle tip and the hot plate to get a stable cone-jet mode. The distance between the nozzle and the substrate was kept constant at 10cm for all depositions.

The Electrostatic spray was conducted at varying substrate temperatures of 300-450 ^{o}C (i.e 300, 350, 400 and 450 ^{o}C) for five samples during the deposition. The substrate temperatures were maintained at the desired temperature using an Aluminium heater and type K thermocouple attached to a digital temperature indicator. The spray rate of precursor solution was maintained at 0.05mL / min throughout the experiment. After the deposition the deposited films were allowed to cool down to room temperature before being characterised.

The thickness of the deposited films was determine by surface profilometry using VEECO DEKTAK 150 Profilometer while the crystal phase analysis of the ZnO and AZO was carried out at room temperature with an Xray diffractometer PANalytical XPERT-PROMPD, at 40mA and 45Kv with CuKα radiation of wavelength λ=1.54060A^0. A scanning range 3.0020-79.9980(2Θ) with a step size of 0.0020^0 was used. In order to obtain good resolutions, 5.08sec was used as count time per step as well as small specimen length of 10mm. Results were analysed with the scientific graphing analysis software and phase identification was done with the inorganic crystal structure data (ICSD) pattern. (Albertson, 1989). The precise lattice parameters were calculated with ICSD standard, after which the grain size, d-spacing, lattice parameter, dislocation density and micro strain were calculated as follows.

Lattice Parameter

The lattice parameters, a and c value for Hexagonal crystallographic system can be calculated from the following relations (Saleem, Fang, Wakeel, Rashad and Kong, 2012) :

$$a = \sqrt{\frac{1}{3}} \times \frac{\lambda}{sin\theta} \quad \text{and} \quad c = \frac{\lambda}{sin\theta} \tag{2.0}$$

d-Spacing

The atomic spacing parameter d is estimated from the Bragg's equation:

$$2d \sin\theta = n\lambda \tag{2.1}$$

$$d = \frac{\lambda}{2sin\theta} \tag{2.2}$$

Where n=1, λ=1.5406A^0

Crystalline Grain Size

The average grain size of the film is calculated using Debye Scherer's formula (Barret 1956)

Grain size = $\frac{K\lambda}{\beta Cos\theta}$, which can also be written as D = $\frac{0.9\lambda}{\beta cos\theta}$ (2.3)

Where β = full width at half maximum (FWHM), θ = diffraction angle, k = Shape factor and λ = wavelength of the X-rays (1.5406 Å) and D= grain size respectively.

Dislocation Density δ

Dislocation density δ is the primary crystal defect in multicrystalline silicon, and it plays an important role in influencing the photovoltaic properties of multicrystalline (Chen *et al*., 2010). They are line defect which arise during crystal growth or as a result of mechanical deformation of a crystal and is determined from (Ilican *et al*., 2008)

$$\delta = \frac{1}{D^2} \tag{2.4}$$

Where D is the grain size of the film

Micro Strain ε

The micro-strain ε is the deformation of an object divided by it`s ideal length. It is estimated using the equation,

$$\varepsilon = \frac{\beta}{4tan\theta} \quad (2.4)$$

Where β = FWHM

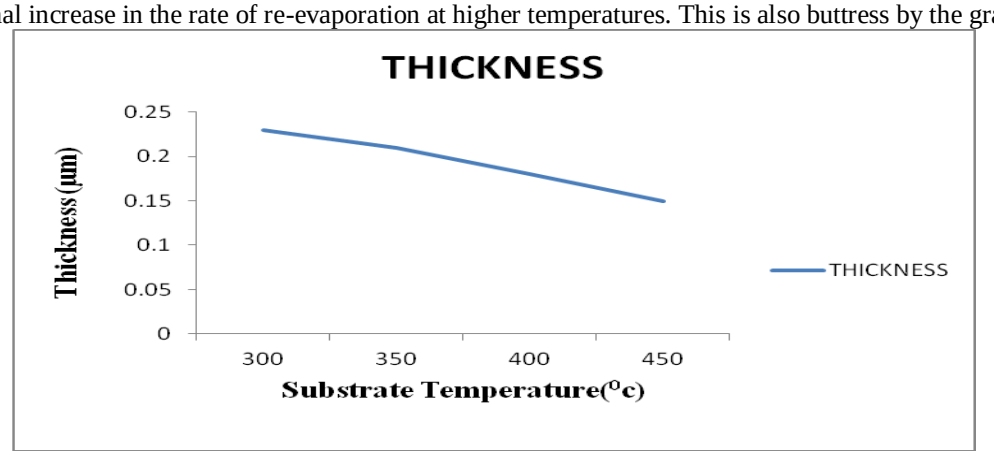
3. RESULTS AND DISCUSSION

3.1. SURFACE PROFILOMETRY

Table1. EFFECTS OF SUBSTRATE TEMPERATURE ON THE THICKNESS OF DOPED AND UNDOPED ZnO THIN FILM

SAMPLE	TEMPERATURE	THICKNESS (µm)
Zno	400	0.20
ZnO:Al	300	0.23
ZnO:Al	350	0.21
ZnO:Al	400	0.18
ZnO:Al	450	0.15

From table 1, the thickness of the deposited AZO films is observed to decrease as the substrate temperature increases, even though the kinetics of the film forming process/reaction suggest an increase in film thickness with increase in substrate temperature This decrease in the film thickness may be inferred to be due to the diminished mass transport of atomic particles to the substrate at higher temperature. Since temperature is a measure of the average kinetic energy of a system of particles, as temperature increases the film particles again more kinetic energy to move thereby reducing mass transport to the substrate. The decrease in film thickness with substrate temperature can also be attributed to a proportional increase in the rate of re-evaporation at higher temperatures. This is also buttress by the graph below



Similar results have been reported by Rajashree, Balu and Nagarethinan (2014) ;Afifi, Aly, Abdal-Rahman and Adbal-thady (2000). An implication of the low values of thickness especially at 400 and 450, is the reduction of grain boundary scattering which will results in the improvement of crystallinity. This is buttress by the xray diffractometry analysis.

3.2. X-ray diffraction (XRD)

Table 2. Comparison of observed and standard d values for ZnO and AZO at different substrate temperatures.

STANDARD (ICDS)	CALCULATED	hkl
	For AZO @ 300 ^{0}c	
2.82740	2.82741	100
	For AZO @350 ^{0}c	
2.82050	2.82125	100
2.60620	2.60618	002
2.48060	2.48062	101
	For AZO @ 400 ^{0}c	
2.81370	2.81371	100
2.61450	2.61452	002
2.47780	2.47778	101
	For AZO @450 ^{0}c	
2.81680	2.81682	100
2.60550	2.60552	002
2.47800	2.47797	101
	For ZnO@400 ^{0}c	
2.82740	2.82741	100
2.60970	2.60972	002
2.48610	2.48607	101

Table 3. VARIATION OF AVERAGE PARTICLE SIZE WITH SUBSTRATE TEMPERATURE

Temperature	Average Particle size (nm)	Dislocation density δ $(nm)^{-2}$
	For AZO	
300 ^{0}c	35.83	5.41×10^{-6}
350 ^{0}c	73.87	1.83×10^{-4}
400 ^{0}c	83.36	1.44×10^{-4}
450 ^{0}c	95.34	1.100×10^{-4}
	For Zno	
400 ^{0}c	70.06	2.03×10^{-4}

Table 4. Summary of Structural parameters of ZnO and AZO at different substrate temperature.

Hkl	d (A^0)	FWHM (β)	2Θ	D(nm)	$\delta(nm)^{-2}$	$\varepsilon \times 10^{-3}$
		For AZO @ 300 ^{0}c				
100	2.82741	0.0192	31.619	35.83		0.2959
		For AZO @350 ^{0}c				
100	2.82125	0.2519	31.699	32.78		3.8720
002	2.60618	0.0551	34.383	150.91		0.7772
101	2.48062	0.2204	36.182	37.91		2.9442
		For AZO @ 400 ^{0}c				
100	2.81371	0.1889	31.777	43.72		2.8961
002	2.61452	0.0480	34.270	173.17		0.6794
101	2.47778	0.2519	36.225	33.18		3.3608
		For AZO @450 ^{0}c				
100	2.81682	0.0945	31.741	87.38		1.4506
002	2.60552	0.1260	34.392	65.99		1.7767
101	2.47797	0.0630	36.222	132.65		0.8406
		For ZnO@400 ^{0}c				
100	2.82741	0.2519	31.619	32.77		3.8823
002	2.60972	0.0551	34.335	150.89		0.7783
101	2.48607	0.3149	36.100	26.53		4.2168

Table 5. Comparison of observed and standard Lattice Parameters of ZnO and AZO at different substrate temperature.

STANDARD a(A^0)	CALCULATED a(A^0)	STANDARD c(A^0)	CALCULATED c(A^0)	hkl
	For AZO @ 300 ^{0}c			
3.2648	3.2648	5.2194	5.6548	100
	For AZO @350 ^{0}c			
3.2568	3.2568	5.2125	5.6409	100
3.2568	3.0094	5.2125	5.2124	002
3.2568	2.8644	5.2125	4.9612	101
	For AZO @ 400 ^{0}c			
3.2490	3.2491	5.2290	5.6274	100
3.2490	3.0190	5.2290	5.2290	002
3.2490	2.8611	5.2290	4.9555	101
	For AZO @450 ^{0}c			
3.2525	3.2526	5.2110	5.6274	100
3.2525	3.0086	5.2290	5.2290	002
3.2525	2.8613	5.2290	4.9555	101
	For ZnO@400 ^{0}c			
3.2648	3.2648	5.2194	5.6548	100
3.2648	3.0134	5.2194	5.2194	002
3.2648	2.8707	5.2194	4.9721	101

EFFECTS OF SUBSTRATE TEMPERATURE ON THE STRUCTURAL PROPERTIES OF DOPED AND UNDOPED ZnO THIN FILM

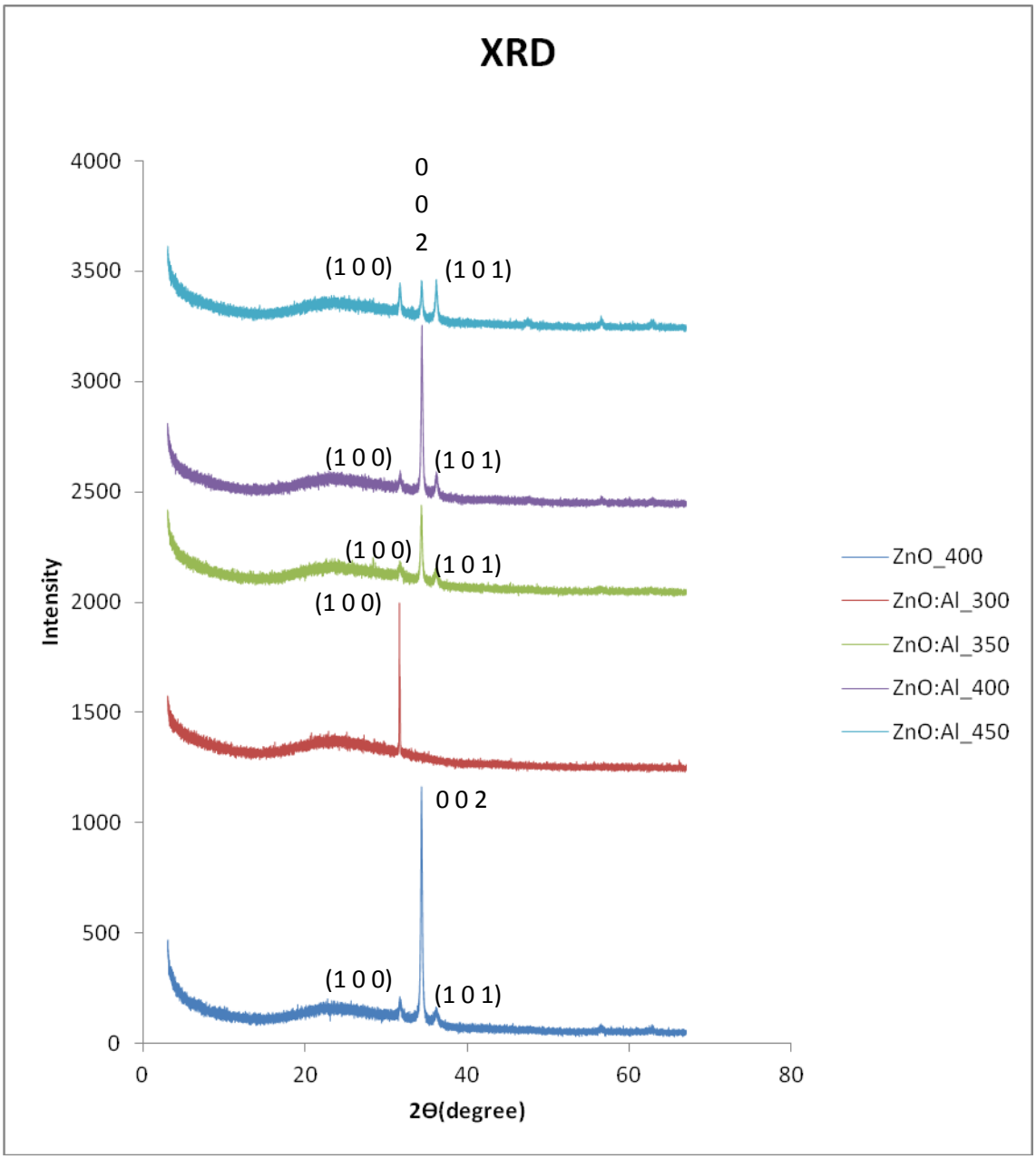


Fig.1. X-ray diffractommeter

The crystalline quality and the orientation of ZnO and AZO thin films were investigated by means of X-rays diffraction (XRD). Figure 1 shows the XRD spectra of ZnO and AZO as a function of substrate temperature (300, 350, 400 & 450 ^{o}C). It can seen that the films were polycrystalline in nature and all peaks (100), (002), (101) were indexed to a hexagonal structural (Space group P63mc) (Albertsson, Abraham, Kvick, 1989)

Doping of ZnO thin films with Al significantly affected the structural properties of the films as shown in figure 1. The preferential orientation of all the films was found to be along the (002) crystal plane. It can be clearly seen that the undoped ZnO films have a strong (002) peak intensity which is in agreement with the report of, Brenier and Ortega, 2004. This intense and sharp peaks in XRD pattern of undoped ZnO reveal the good crystallinity of the films and also confirm the stoichiometric nature of ZnO thin films. However, the AZO films have a reduced orientation along the (002) crystal plane. This is because substituting the differently sized atoms in the lattice causes some lattice defects and the distortion of the lattice (Brenier and Ortega, 2004).

All the films showed a strong peak at 2Θ near 34^{0} which were identified as the (002) peak of the doped and undoped ZnO. The diffraction pattern and the lattice parameters showed that all the films exhibit a hexagonal structure, which indicates that Al^{3+} ions substituting Zn^{2+} ions do not change the hexagonal structural of ZnO films. The diffraction peaks position of 2Θ for all samples shifts towards higher angle compared to bulk ZnO value (34.335^{0}) for all samples which indicate a slight lattice shrink attribute to the smaller atomic radius of Al^{3+} (0.054nm) than that of Zn^{2+} (0.074nm) and the formation of stress induced by ion size difference (Babu, Maldonado, Velumani and Asomoza,2010).

At substrate temperature of 300 ^{0}C the spray falling on the substrate undergoes incomplete thermal decomposition giving rise to a foggy film with a low level of transmittance and has just a peak whose plane is not in the direction of the preferred orientation and tend to decrease with increase in temperature. Similar report has been given by Ceviz, Ozdemir, Bedir and Oztas (2013).

When the substrate temperature increases from 350 to 400 ^{0}c, the (002) orientation of films increases evidently. The high intensity of the (002) diffraction peaks with others being weak indicates the orientational growth of the film and

good crystallinity. The improvement in crystallinity signifies that the dislocations and density of grain boundaries will decrease leading to a decrease of donor sites trapped at the dislocations and grain boundaries which is consistent with the report of Shigesto, Takaki and Haranoh (1991). The kinetic energy of the deposited atoms is often believed to be majorly determined by the substrate temperature when the incident energy is fixed (Biju and wen, 2012). At a relatively high substrate temperature the atoms on the film surface can move quickly to look for a site of lowest energy to form a low energy structure and the (002) plane is therefore considered to be the low surface energy. As a result, the surface of the ZnO thin film deposited at high substrate temperature is smooth and uniform.

However with further increase in the substrate temperature the intensity of ZnO:Al (002) peak decreases inversely. As the adsorption atoms decomposed and re-evaporated from the surface and the ZnO:Al film becomes thermodynamically unstable as rightly observed in Figure 1. Similar phenomenon has being reported by Zhue, Sunb, Zhaob and Su (2007); Biju and wen (2012).

The observed (calculated) d- spacings of the 100, 002 and 101 planes of the ZnO and AZO films along with the standard d spacing values are listed in Table 2. The interplanar distance for the 100, 002 and 101 of all the substrates temperatures are similar to the standard values even though a negligible difference occurs which likely indicates contraction of unit cell volume that in turn reveals the presence of strain.

Table 5 shows the calculated lattice parameter values for all the planes. The lattice constants (a and c) are found to be in close agreement with standard values, but in each case it was observed that one of the parameter is same with standard values while the other varied insignificantly with the standard value, thus conforming the strain present in the samples. This deviation of the lattice parameters by the presence of strain may be due to the presence of residuals of the reaction components.

Micro strain ε and dislocation density δ which are crystal defect parameters are also shown in Table 4, They exhibits a decreasing trend with increase in substrate temperature and particle size. Such change in the strain may be due to the recrystallization process in the polycrystalline films. Also strain is an inherent and natural component of Nano grained materials due to the large number of grain boundaries and the short spacing between them. Increase in particle size with substrate temperature causes increase in surface energy which causes the varying magnitude of the strain. The larger values of δ obtained for the film at 300 compared to the one at 400 ^{0}C and 450 ^{0}C showed that the film has comparatively lesser degree of crystallinity since dislocation density is the measure of the defects in the crystalline structure. At higher substrate temperature, both the micro strain and the dislocation density are minimum, which reveals the reduction in the concentration of the lattice imperfections leading to preferred orientations. This is consistent with the report of Kissinger, Suthagar, Saravana Kumar, Balasubramaniam and Perumal (2010).

4. CONCLUSIONS

ZnO and ZnO doped Al thin films were prepared on soda lime glass substrate by ESD at different substrate temperatures. The effects of substrate temperature on the structural properties of the deposited thin films were investigated. The XRD patterns of the films shows that the films were polycrystalline in nature with hexagonal structure and a preferential orientation along the 002 plane. The average crystalline size increases with increase in substrate temperature indicating better crystallinity of films deposited at higher substrate temperature as the films become more uniform and dense.

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