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Sol-Gel Derived ZnO Thin Films for Their Potential Applications

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ABSTRACT

Zinc Oxide is a direct wide band gap semiconducting material which is of great interest for numerous technological applications due to its unparalleled properties and the availability of a variety of synthesis approaches. In this paper, we have presented the synthesis of ZnO thin films on micro-glass substrates by sol-gel technique and their structural, morphological and optical properties were investigated. X-Ray Diffraction (XRD) analysis inferred the film's hexagonal wurzite phase with a preferential growth along (101) plane. The mean crystallite size was obtained as 12 nm along (101) plane on the basis of the Deby-Scherer model. A small dislocation density of 6.64×10^{-3} (Line/nm^2) was obtained along (101) plane, showing the presence of few lattice imperfections and good crystallinity which make the films suitable for optoelectronic applications. SEM micrographs inferred the film's granular nature composed of spherical nanoparticles. The absorbance spectra show that the ZnO thin films absorb maximum radiation in the UV region. The refractive index of the thin films has been evaluated using Tripathi relation and a low reflectivity 0.15 obtained with the help of Fresnel's equation which indicates the good absorbance of the films. The optical band gap (3.36 eV) is estimated using UV-Visible spectroscopy. A high absorbance of the thin films was found in ultraviolet region with peaks around 375 nm, indicating that the films have potential applications in making of solar cells.

Keywords: ZnO, XRD, Thin films, SEM, Nanoparticles, ZAD, Spin coating

INTRODUCTION

Nanotechnology is one of the areas of knowledge that attract the attention of researchers worldwide due to innovations created by reducing the size of the materials to nano range. A material is nanometric when its structural components have at least one dimension in the nano regime (1-100 nm). Due to their extraordinary mechanical, electrical, magnetic, optical and chemical properties, ZnO is one of the most studied materials in nanotechnology.

Zinc oxide (ZnO) is an interesting direct wide band gap (~3.32 eV) material of II-VI compound semiconducting family. ZnO has large exciton binding energy (60 meV) and superconducting properties based on oxygen vacancies and the wurzite structured [1-4].

ZnO has become one of the most promising materials and has lots of research interest due to its unique structure and size-dependent electrical, optical and mechanical properties. ZnO is suited for various technological applications such as anti-reflection coatings, transparent electrodes in solar cells [5], piezoelectric devices [6], gas sensors [7], varistors [8], UV and blue light emitters and even thin-film transistors [9]. It is also being considered as a potential candidate in the field of spintronics [10].

Unlike many of its competitors, ZnO is inexpensive, chemically stable, easy to prepare, non-toxic and abundant. It has been used in variety of applications such as conductive films, solar cell windows, photoelectric cells, non-linear optics, bulk and surface acoustic wave devices [11-13] and micro mechanical devices [14,15]. ZnO wide band gap opens the possibility of creating ultraviolet emission diodes [LEDs] and white LEDs with superior color purity. ZnO thin films have good electrical and optical properties and are of lower material cost in comparison to the ITO films [16,17].

Due to large exciton binding energy the exciton remain dominant in optical processes even at room temperature. Due to its vast industrial applications such as electrophotography, electroluminescence phosphorous, pigment in paints, flux in ceramic glazes, fillers for rubber products, coatings for papers, sun screens, medicines and

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cosmetics ZnO is attracting considerable attention in powder as well as thin film form. Its resistance to radiation damages also makes it useful for the applications in space. It is the hardest of the II-VI family of semiconductors having a large shear modulus; its performance is not degraded as easily as the other compounds due to presence of defects. Some of its important properties are listed in **Table 1** [18-24].

Table 1. Properties of zinc oxide.

Parameters	Nature/Values
Crystal Structure	Rock Salt, Zinc Blende and Wurzite
Density	5.606 g/cm^3
Nature	Semiconducting
Exciton Binding Energy	60 meV
Electron Mobility	2.5-300 cm^2 Vs^{-1} (Bulk ZnO)
Electron Effective Mass (m_e)	0.26
Boiling Point	2360°C
Melting Point	1975°C
Refractive Index	2.0041
Energy Band Gap	3.2-3.3 eV

ZnO thin films have been made by a variety of techniques among which there can be mentioned reactive sputtering [25], spray pyrolysis [26], zinc oxidation [27], electro deposition [28], pulsed laser deposition (PLD) [29], chemical vapor deposition (CVD) [30], metal organic chemical vapor deposition (MOCVD) [31], plasma enhanced chemical vapor deposition (PECVD) [32], low pressure sputtering [33], chemical bath deposition (CBD) [34] and sol gel route [35]. The synthesis of ZnO nanostructures can be accomplished by chemical and physical routes. However chemical methods are more suitable and cost effective for the production of ZnO on the industrial scale [36].

The sol gel method has emerged as one of the most promising processing route as it is particularly efficient in processing thin transparent, homogeneous, multi components oxide films of many compositions on various substrates at low cost and it allows the tuning of thickness of the film by varying synthesis parameters.

In this work, we have investigated the structural, morphological and optical properties of undoped ZnO thin films spin coated on glass substrates.

BACKGROUND

Sol gel process

A sol is a dispersion of the solid particles (~0.1-1 μm) (~0.1-1 μm) in a liquid where only the Brownian motions suspend the particles. A gel is a state where both liquid and solid are dispersed in each other, which presents a solid network containing liquid components. The sol-gel coating process usually consists of four steps:

Steps:

i. The desired colloidal particles once dispersed in a liquid to form a sol.

ii. The deposition of sol-solution produces the coatings on the substrates by spraying, dipping or spinning.

iii. The particles in sol are polymerized through the removal of the stabilizing components and produce a gel in the state of a continuous network.

iv. The final heat treatments pyrolyze the remaining organic or inorganic components and form an amorphous or crystalline coating [40].

Advantages of sol-gel technique:

- Can produce thin bond-coating to provide excellent adhesion between the substrate and the top coat.
- Can produce thick coating to provide corrosion protection performance.
- Can easily shape material into complex geometries in a gel state.
- Can produce high purity products.
- Can have low temperature sintering capability, usually 200-600°C.
- Can provide a simple, economic and effective method to produce high quality coatings.

MATERIALS AND METHODS

Cleaning of the substrates

The microscope glass substrates were cleaned ultrasonically. Initially the ultrasonic bath was filled with distilled water and then quartz substrates placed in the beaker filled with acetone. The beaker placed in the ultrasonic bath for cleaning purpose of the substrates at 60°C for 15 min. After cleaning the substrates are carried out from the beaker and dried at room temperature for 10 min by keeping the substrates vertically.

Synthesis of thin films

Zinc oxide thin films were prepared by sol-gel process [24]. As a precursor material, (ZAD) zinc acetate dehydrate [Zn (CH_3COO) $2.2H_2O$] was dissolved in isopropyl alcohol with molarities of 0.65 mol L-1and 0.45 mol L^{-1} with the help of monoethanolomine (MEA) as a catalyst. The solution was stirred thoroughly on a magnetic stirrer for 1 h at 60°C temperature. As prepared solution is filtered using filter paper. The filtered precursor solution was deposited on quartz substrates by spin coating (3500 rpm, 120 s). As synthesized films were dried for 30 min at nearly 60°C temperature. The films were subsequently annealed up to 500°C for one hour in order to obtain crystallized ZnO.

Characterization

The crystal structure and orientation of the ZnO films were studied by X-ray diffractometery (XRD) using radiation CuKα (λ=1.5418 Å) operating at 30 kV and 25 mA of electric current. The diffractometer reflection was recorded at room temperature. The morphology of the synthesized ZnO thin films were analyzed by scanning electron microscopy using a JEOL JSM6380 microscope operating at 80 kV. The optical parameters of the ZnO thin films analyzed by a UV-Visible spectrophotometer in the spectral range of 200-1000 nm at normal incidence.

RESULTS AND DISCUSSION

Film thickness measurement

The thickness of the sol gel derived ZnO film is estimated approximately 315 nm by equation (1) given below [41].

$$t=\frac{w_2-w_1}{A\rho}\times 10^4\ \mu m \qquad (1)$$

Where w_1 and w_2 are the weights of the glass substrate before and after the film deposition in grams; A is the area in cm^2 of the deposition of the film and ρ is the density of ZnO (5.6 g/cm^3).

X-ray diffraction (XRD) analysis

The XRD pattern of synthesized ZnO thin films by sol-gel spin coating technique on quartz substrates is shown in **Figure 1**. All the recorded peaks correspond to the peaks of standard ZnO (JCPDS36-1451). For the synthesized ZnO films, different diffraction peaks are recorded in XRD pattern showing in the growth of ZnO along different directions. Strong preferential growth is observed along the (101) planes [42]. The mean crystallite size (τ) was estimated using the Deby-Scherer formula shown in equation (2):

$$\tau=\frac{k\lambda}{\beta_{2\theta}cos\theta} \qquad (2)$$

Where k is 0.94 is a constant and λ=X-ray wavelength (1.5418 Å), $\beta_{2\theta}$=full width at half maximum in radian (FWHM) and θ is the Bragg's angle obtained from 2θ values.

The dislocation density (δ) defined as the length of dislocation lines per unit volume and strain (ε) have been estimated by the equation (3) and (4) given below [42,43]:

$$\delta=\frac{1}{D^2} \qquad (3)$$

$$\varepsilon=\frac{\beta\cos\theta}{4} \qquad (4)$$

Lattice constant 'a' and 'c' of the wurzite structure of ZnO are calculated using relations [42,44,45]:

$$a=\frac{\lambda}{\sqrt{3}sin\theta_{100}} \qquad (5)$$

$$c=\frac{\lambda}{sin\theta_{002}} \qquad (6)$$

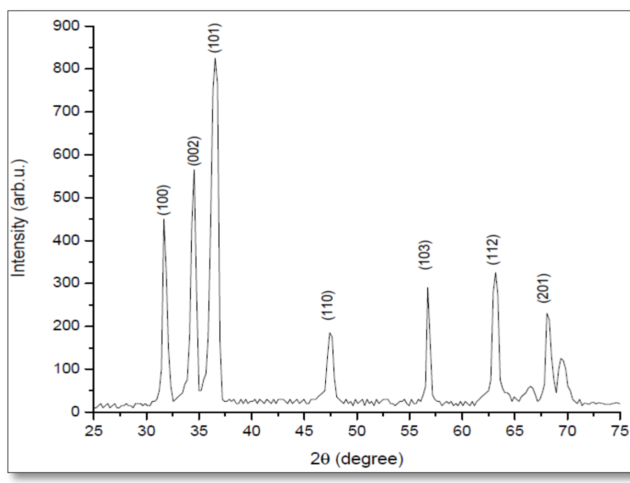

Figure 1. XRD Pattern of ZnO Thin Films deposited on glass substrates.

The estimated structural parameters of sol gel derived ZnO films are presented in **Table 2** and the calculated lattice parameters are presented in **Table 3**. The estimated lattice parameters of ZnO films deposited on glass substrate from recorded XRD data are in agreement with standards reported in (JCPDS 36-1451).

Table 2. Estimated structural parameters of sol-gel derived thin films on glass substrate.

Planes	Inter Planar spacing (Å)	FWHM β (radian)	Grain size τ (nm)	Dislocation density $\delta \times 10^{-3}$ (Line/nm^2)	Strain ε
(100)	2.89	0.3563	18.16	3.030	0.0851
(002)	2.60	0.475	18.30	2.986	0.1127
(101)	2.53	0.7126	12.27	6.642	0.1710

Table 3. Lattice parameters of sol-gel derived thin films on glass substrate.

a (Å)		c (Å)	
Calculated	**Standard**	**Calculated**	**Standard**
3.34	3.25	5.20	5.21

The bond length L of nanostructured ZnO films is found 2.01 Å using equation (7) and (8) [46,52]:

$$L=\sqrt{\left[\frac{a^2}{3}+\left(\frac{1}{2}-u\right)^2 c^2\right]} \qquad (7)$$

$$u=\frac{a^2}{3c^2}+\frac{1}{4} \qquad (8)$$

This bond length is slightly higher than the standard value1.9767 Å for bulk ZnO which is due to strain produced during synthesis of the thin films [47]. The ratio c/a is found 1.6 which confirms the wurzite structure of the prepared thin films.

The refractive index of the ZnO thin films is estimated 2.28 using Tripathi relation [49] given in equation (9) below:

$$\eta=\eta_0[1+\alpha e^{-\beta E_g}] \qquad (9)$$

Where α=1.9017, η_0=1.73 and β=0.539/eV. The calculated value is found to be slightly greater than the refractive index of bulk ZnO. The high refractive index consisted by ZnO thin films make them suitable for use as anti-reflection coatings. The reflectivity of the ZnO films for air and ZnO interface is calculated using Fresnel's equation [49]:

$$R=\left[\frac{\eta-1}{\eta+1}\right]^2 \qquad (10)$$

Where η is the refractive index of synthesized Zno film; the reflectivity is found 0.152 which shows the good absorbance of the ZnO films suitable for optoelectronic applications. This low value of reflectivity of ZnO thin films makes them suitable for anti-reflection coating ion solar cells.

Morphological analysis

The morphology of the synthesized ZnO thin films was investigated using scanning electron microscopy. **Figure 2** shows the surface morphology of the prepared films in our report. The results recorded by SEM are found to be are very good with stoichiometric formation of ZnO nano crystal of spherical shape and demonstrated aggregation of ZnO nano particles. The aggregation of ZnO nanoparticles occurred probably during the drying process [37,38]. The surface morphology of ZnO thin films indicates high density of grains implying nucleation on all sites of the substrates.

Optical characterization

The absorption spectra of ZnO thin films are recorded in the spectral range of 200-1200 nm using UV-spectrophotometer. The band gap energy of ZnO thin films have been calculated using Tauc's plot.

Tauc's equation (11) is given by Kashyout et al. [39]:

$$\alpha h\upsilon = A(h\upsilon - Eg)^n \qquad (11)$$

Where α is the absorption coefficient, hυ is the photon energy and A is the constant, E_g is the band gap energy of the sample. The value of n is 1/2 or 2 depending upon whether the transition from valence band to conduction band is direct or indirect. The presence of a single slope in the plot suggests that the films have direct and allowed transition. Since ZnO has a direct band gap material, the value of n is taken 1/2 in this case. The absorbance curves of the sol-gel derived ZnO thin films are recorded in the range 200-1200 nm shown in **Figures 3-5**.

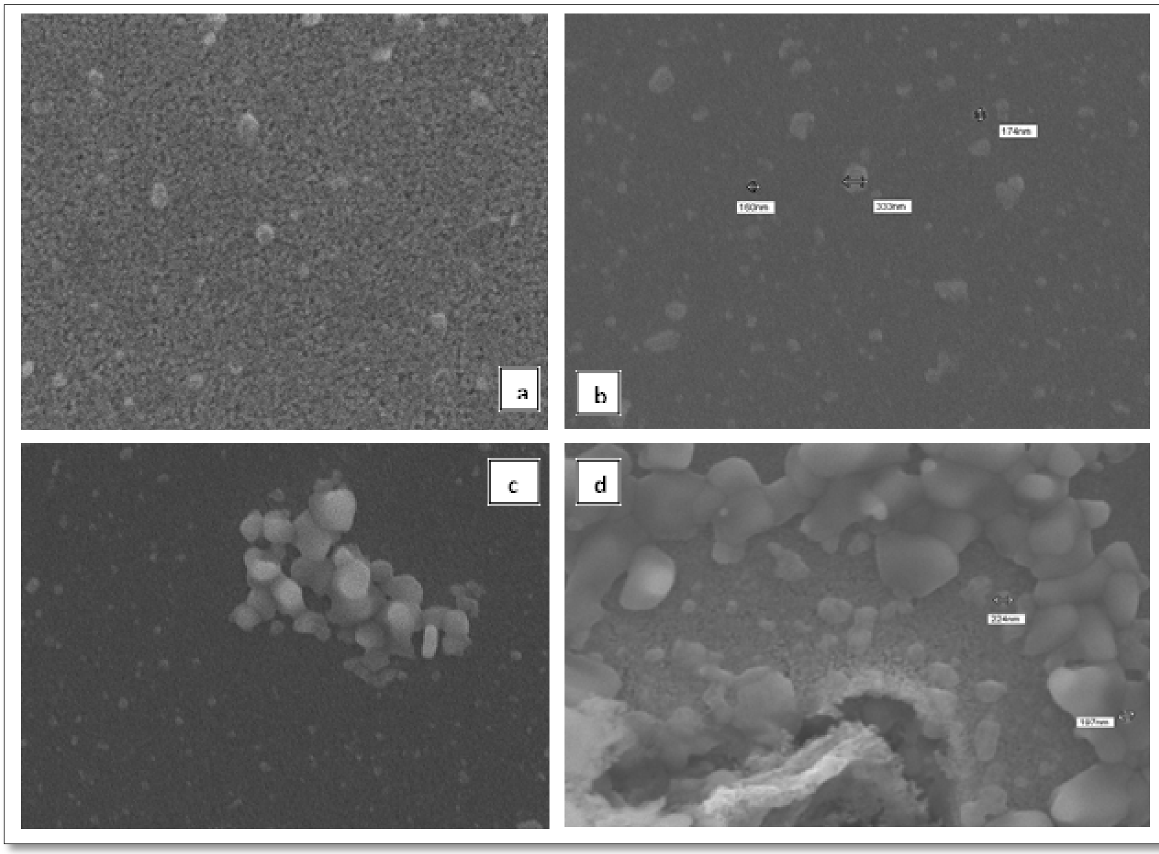

Figure 2. ZnO nanostructures grown on glass substrate using zinc acetate as a precursor material by sol gel technique.

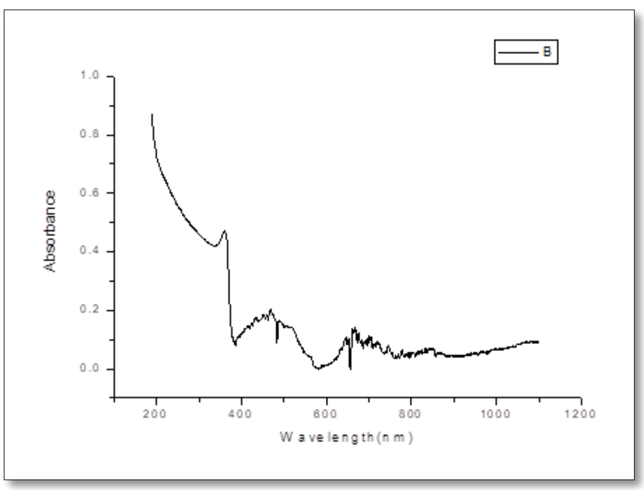

Figure 3. Absorbance spectra of ZnO nanostructure grown on glass substrate using sol-gel spin technique taken zinc acetate as a precursor material (molarity=0.45).

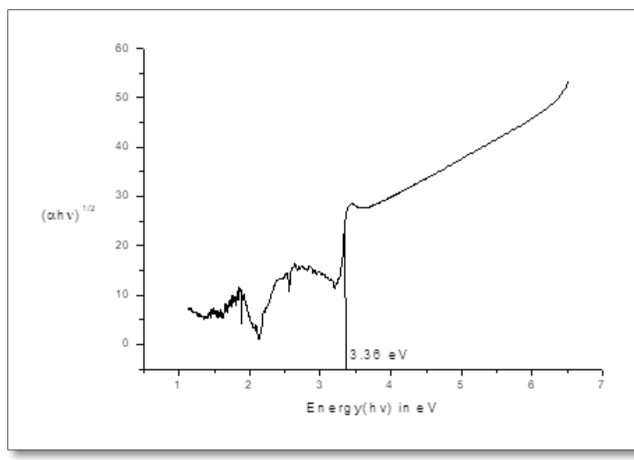

Figure 4. Estimation of optical band gap of ZnO nanostructure grown on glass substrate using sol-gel spin technique taken zinc acetate as a precursor material (molarity=0.45).

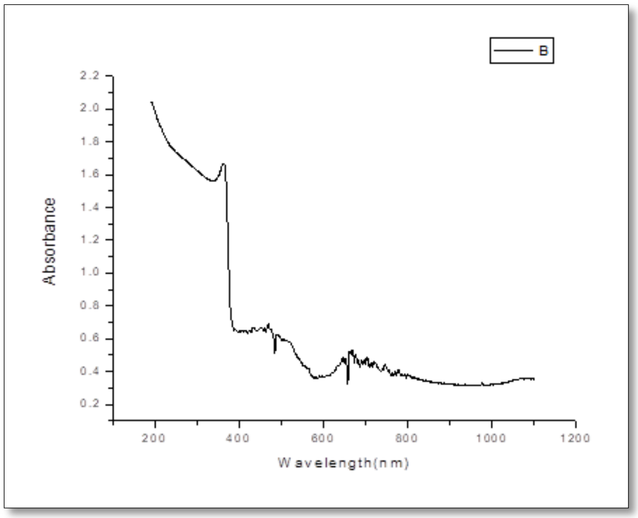

Figure 5. Absorbance spectra of ZnO nanostructure grown on glass substrate using sol-gel spin technique taken zinc acetate as a precursor material (molarity=0.65).

The graphs show that ZnO thin films grown on glass substrates absorb light in ultra violet region of the electromagnetic spectrum. The results are in accordance with the band gap value of the bulk ZnO (3.37 eV) according to which ZnO absorbs light in UV.

The maximum absorbance occurs at wavelengths approx 350-375 nm which indicates that the films have potential application in fabrication of solar cells.

The energy band gap is evaluated by extrapolating the straight line portion of the plot to zero absorption coefficient in strong absorption spectral region when $(\alpha h\upsilon)^{1/2}$ varies dramatically **(Figure 6)**.

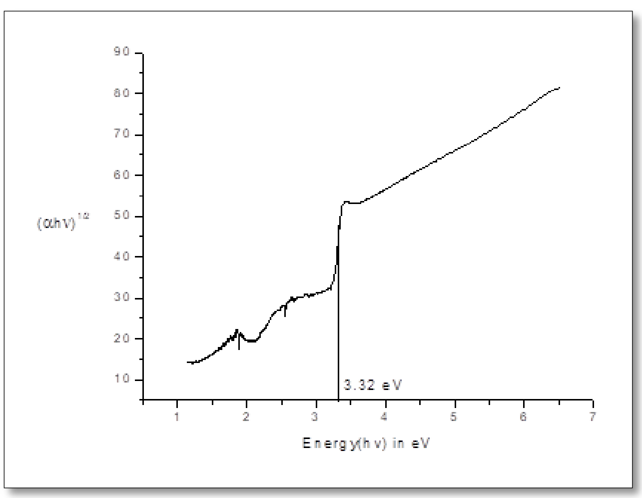

Figure 6. Estimation of optical band gap of ZnO nanostructure grown on quartz substrate using sol-gel spin process taken zinc acetate as a precursor material (molarity=0.65).

The band gap energy is calculated using Tauc's plot in **Figures 4 and 6** which comes out to be 3.36 eV for M=0.45 of ZAD and 3.32 eV for M=0.65 of ZAD, which suggest that E_g slightly decreases with increasing of molarities of the precursor. These values are in good agreement with the values reported by other researchers [28].

CONCLUSION

ZnO thin films having nanostructure were synthesized via sol-gel technique. The morphological, structural and optical features of sol-gel deposited ZnO thin films were characterized. The XRD analysis revealed hexagonal wurzite structure with preferred orientation along (101) plane. SEM images of the ZnO thin films showed ZnO nano particles which coalesced and make clusters on the surface of the film. The surface morphology exhibits that the quality of the film is well adapted to the considered optoelectronic applications. The high refractive index of sol-gel derived ZnO thin films make them suitable for the usage of anti-reflection coatings. The low reflectivity of the ZnO films for air and ZnO interface make them suitable in the fabrication of solar cells. Hence, the sol-gel technique has proved to be effective in the contribution of thin films for optoelectronic applications.

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