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SYNTHESIS AND CHARACTERIZATION OF ZnO SINTERED FILMS

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ABSTRACT
Zinc Oxide (ZnO) is an important multifunctional material which has received great attention during the last few years due to their unique applications in microelectronic and optoelectronic devices, and for self-assembled growth of three-dimensional nanoscale systems. Zinc oxide films have been prepared on to highly cleaned glass substrates in open air atmosphere by screen printing technique followed by sintering process. The ZnO sintered films are characterized by optical spectroscopic measurements, X-ray diffraction pattern and scanning electron microscopy. The spectroscopic measurements of ZnO sintered films deposited on to highly glass substrates have been made by spectrophotometer (Hitachi, model U-3400) at room temperature. Reflection spectra of ZnO sintered films are used to calculate the band gap of the films material by Tauc relation for direct band transitions. The XRD patterns of ZnO sintered films show the hexagonal wurzite structures. The surface morphology of the films has been studied by SEM.
Key words: Zinc oxide; Screen printing; Sintered; Reflection spectra

INTRODUCTION:

ZnO is the II-IV semiconductor material with wide and direct band gap (3.37 eV) and large exaction binding energy (60 MeV). It is an attractive and promising material for many applications such as surface acoustic wave devices (SAW), light emitting diodes, laser diodes, photo detectors, Solar cell windows and gas sensor. Various growth techniques such as chemical vapor deposition, r.f. magnetron sputtering, pulsed laser deposition (PLD), evaporation, spray pyrolysis, photo-atomic layer deposition, metal oxide chemical vapor deposition (MOCVD), molecular beam epitaxy (MBE) and sol–gel process have been used for ZnO film. Currently many research groups are working on this material.

TECHNIQUES OF THIN FILM PREPARATION:

Film synthesis techniques used in the laboratory are based in physical or chemical vapor deposition of thin films ('physical vapour deposition' or PVD and 'chemical vapour deposition' or CVD, respectively).

In both cases, the techniques are based in the formation of vapor of the material to be deposited, so that the vapor is condensed on the substrate surface as a thin film.

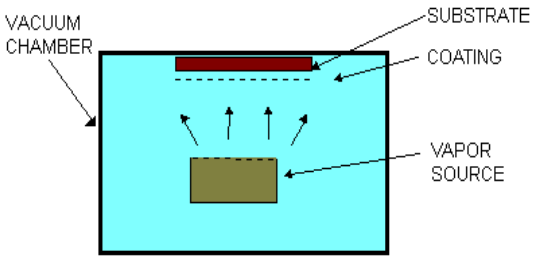

Fig:-1 Thin Film Preparation

Usually the process must be performed in vacuum or in controlled atmosphere, to avoid interaction between vapor and air.

- In physical techniques (PVD) we part from a solid material converted to vapor through heating (evaporation) or energetic ion bombardment. The material in form of vapor finally condenses on the substrate surface as a thin film.
- In chemical techniques (CVD) we part directly from gases which react and give place to a new product that condenses as a thin film on the substrate.
- Other film synthesis techniques include high temperature thermal oxidation and anodic oxidation.

REVIEW OF LITERATURE:

Kumar et.al [2017] have fabricated p-type ZnO epitaxial thin films and studied their physical properties. **Amit Kumar et. al.[2015]** have deposited ZnO thin films by a RF magnetron sputtering technique and studied their nonvolatile resistance memory switching properties. **Amit Kumar et.al[2014]** have synthesized Al-N codoped ZnO thin films deposited on n-Si substrate by RF magnetron sputtering technique and show the induction of p-type conduction in deposited Al-N codoped ZnO thin film. **Amit Jean Paul Mosnier et. al. [2008]** has deposited ZnO films by Pulse Laser Deposition on soda lime glass substrate for the ultraviolet inactivation of Staphylococcus epidermidis bio-films. **Schristoulakis et.al. [2017]** have prepared ZnO nanostructures transparent thin films of different thickness on silicon and coning glass substrate by Pulse laser deposition and show the sensing properties can be controlled by modifying the deposition condition.

SAMPLES PREPRATION:

ZnO is a II-VI group semiconductor material with a large direct band. Zinc oxide film has been prepared by screen printing and pulsed laser deposition (PLD) techniques on to highly cleaned glass substrate. The as-prepared film is characterized by optical absorption spectra and transmission spectra, X-Ray Diffraction (XRD) pattern and Atomic Force Microscopy (AFM). The absorption spectra and transmission spectra of the ZnO film have been taken from UV-VIS-NIR Spectrophotometer at room temperature.

RESULTS AND DISCUSSION:

Optical properties

Reflection spectra of sintered ZnO films is taken at room temperature with the help of a Hitachi Spectrophotometer model U-3400.In this model the prism/grating double monochromatic system is used, the lenses used in conventional monochromator are replaced with mirrors. So, the image deviation due to chromatic aberration is eliminated. Its wavelength range is 187- 2600nm.The lead sulphide detector (PbS) is used for the detection of infrared rays. The visible wavelength light source is long life WL lamp. The optical band gaps of these films are determined with the help of reflection spectra. Almost all the II-VI Compounds are direct band gap semiconductors. According to Tauc relation, the absorption coefficient for direct band gap material is given by $\alpha h\nu = A (h\nu - Eg)^n$, Where $h\nu$ is photon energy, A is constant, Eg is the band gap, and n is equal ½ for direct band gap material. To measure energy band gap from reflection spectra, a graph between $(\alpha h\nu)^2$ Vs. $(h\nu)$ is plotted. Absorption coefficient α is proportional to Ln [(Rmax – Rmin) / (R – Rmin)], where reflectance falls from Rmax to Rmin due to absorption by the material and R is the reflectance for any intermediate energy photons. So α is used in terms of reflectance as Ln [$h\nu$ (Rmax – Rmin) / (R – Rmin)] and extrapolation of straight line to $(\alpha h\nu)^2 = 0$ axis give the value of energy band gap of film material. Fig. 2 shows the reflection spectra of sintered ZnO by above described Hitachi spectrophotometer model U-3400.

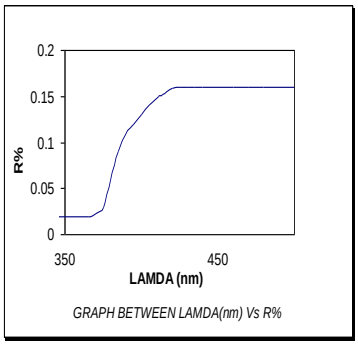

Fig.2. Reflection spectra of ZnO sintered film.

The mode of optical transition in these films is of band to band direct type. This has also been confirmed by plotting Ln (α hν) Vs [Ln (hν -Eg)] for direct allowed type transitions, therefore we are using here the Tauc relation for direct band gap material in which we plot a graph between (α hν)2 Vs. (hν) . Fig. 3 shows a plot between [hν Ln {(Rmax - Rmin) / (R –Rmin)}]2 Vs hν for sintered ZnS film. The extrapolation of straight line to (α hν)2 =0 gives the value of direct band gap. From this graph the value of energy band gap comes out to be 3.50eV.The band gap does not show noticeable change with sintering temperature the band gap of 3.30eV of ZnO film prepared by screen printing technique and have reported the and gap 3.30 eV of ZnO films sintered at 650°C for 10 min in nitrogen atmosphere

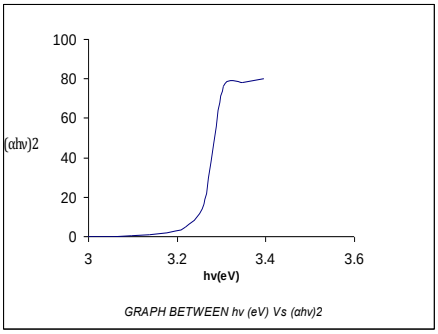

Fig. 3. Energy Band gap Determination of ZnO Sintered from Reflectance Measurement

EXPERIMENTAL DETAILS:

Zinc Oxide films have been prepared by pulsed laser deposition (PLD) technique onto highly cleaned glass substrates. The target of ZnO was prepared using 99.99% pure ZnO powder. This powder was grinded for 6 hr and then calcined at 450 ^{0}C for 10 hr. The calcined powder was regrinded for 8 h and was then pressed into pellets of 15 mm in diameter and 2 mm thickness under the pressure of 60 MPa. Then, the pallets were sintered at 800 ^{o}C . Glass was cleaned with distilled water and acetone. ZnO thin film has been deposited on Glass substrate using pulsed laser deposition (PLD) technique employing a KrF laser source (λ =248 nm). Various parameters used in pulsed laser deposition technique are shown in **Table-1**. We rotated the target at 2 rpm to avoid texturing of the target surface. The thickness of the grown film is typically ~250 nm and buffer layer thickness is ~50 nm.

Table 1: Various Parameters Used In Pulse Laser Deposition Technique

Laser source	:	KrF eximer source
Laser wavelength	:	248 nm
Laser energy	:	300 mJ
Laser fluence	:	2-3 J-cm^{-2}

Repetition rate	:	10 Hz
Target used	:	ZnO,
Base pressure	:	2×10^{-6} Torr
Gas used	:	High purity oxygen (99.7%)
Deposition pressure	:	50 m Torr
Substrate used	:	Glass
Substrate temperature	:	500 ^{o}C
Target to substrate distance	:	30 mm

DETERMINATION OF OPTICAL CONSTANTS:

The optical constants (Refractive index n and the extinction Coefficient k,) of ZnO film have been determined from transmittance measurement by using Manifacier's envelope method. The transmission Spectra of ZnO film in the spectral range 300 to 1600 nm were used to determined the refractive index (n) and extinction coefficient (k).

The refractive index (n), extinction coefficient (k) and ε were calculated using the formula

$$n = [N + (N^2 + n_0{}^2 n_1{}^2)^{1/2}]^{1/2}$$

Where n_0 is refractive index of air ; $n_1$is the refractive index of substrate and the number N is given By

$$N = \frac{n_0{}^2 + n_1{}^2}{2} + \frac{2n_0 n_1 (T_{max} - T_{min})}{T_{max} T_{min}}$$

Where T_{max} is upper extreme transmission point and T_{min} is the lower extreme transmission point of particular wavelength.

The extinction coefficient k is given by

$$k = \left(-\frac{\lambda}{4\pi t}\right) lnP$$

Where t is the thickness of the film and calculated by formula

$$t = \frac{M\lambda_1\lambda_2}{2[n(\lambda_1)\lambda_2 - n(\lambda_2)\lambda_1]}$$

Where M= no of two consecutive max or min

And

$$P = \frac{(n+n_0)(n_1+n)}{(n-n_0)(n_1-n)} \times \frac{(1-T_{max}/T_{min})}{(1+T_{max}/T_{min})}$$

Table 2 Shows the variation of optical constant (n,& k) with wavelength for pulse laser deposited ZnO film.

Table 2: Variation of Optical constant on Glass versus λ

S. N.	□ (nm)	Energy (eV)	T_{max} (%)	T_{min} (%)	N	n	k
1	400	3.0975	72.8	66.4	1.656	1.973	0.504031
2	450	2.753	81.6	70.4	1.672	1.979	0.498375

3	500	2.478	88	72.8	1.682	1.984	0.513297
4	550	2.253	88	73.6	1.678	1.982	0.575271
5	600	2.065	88.8	75.2	1.674	1.980	0.641725
6	650	1.906	89.6	76.8	1.670	1.978	0.711189
7	700	1.77	90.4	76.8	1.672	1.979	0.75319
8	750	1.652	90.4	77.6	1.669	1.978	0.822989
9	800	1.549	91.2	77.6	1.671	1.979	0.863327
10	850	1.458	91.2	77.6	1.671	1.979	0.917285
11	900	1.377	91.2	77.6	1.671	1.979	0.971243
12	950	1.304	92	78.4	1.670	1.979	1.028184

CONCLUSION

Thin films are extensively studied due to their potential technical importance and scientific curiosity. The behaviors of two dimensional solids have been responsible for the interest in study of thin films in science and technology. Thin films have directly or indirectly advanced is many new field of research in solid state Physics and chemistry which are based on the phenomena a uniquely characteristic of the thickness geometry and structure of the films. The order of the thickness of thin films is few hundreds microns. If the thickness is greater than this order, the film will be thick not thin so thin films can be defined as “It is not the thickness that is important in defining a film, but rather the way it is created with the consequential effects on the micro structure and properties.

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