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SYNTHESIS AND CHARACTERIZATION OF ZnO NANOPARTICLE USING ELECTRODE POSITION METHOD

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ABSTRACT

ZnO is a wide band-gap (3.37 eV) II–VI compound semiconductor with hexagonal wurtzite structure. Zinc oxide films were deposited by electrode position, containing very low concentrations of sodium citrate and 30% hydrogen peroxide. Ammonium hydroxide is added to control the reaction. The structural analysis were initially studied with X-ray diffraction (XRD), scanning electron microscope (SEM), energy-dispersive X-ray spectroscopy (EDS). The average grain size is measured to be 48nm. Resistivity and carrier concentration of the as deposited film are observed to be 30 Ω-cm and 4.7×10^{17}carriers/cm^3respectively.

INTRODUCTION

ZnO is a wide band-gap (3.37 eV) II–VI compound semiconductor with hexagonal wurtzite structure. Higher exciton binding energy of zinc oxide (~60 meV) enhances its luminescence efficiency. It has direct band gap energy, which makes its transparency in the visible region and most of the activity in the ultra violet/blue region (Norris *et al*., 2003; Choi *et al*., 2009 and Sun *et al*., 2005). It is an n- type semiconductor material having several applications in optoelectronics, transparent electronics, spintronics, sensor applications and widely used for the fabrication of transistors and FETs, light emitting diodes, dye sensitized quantum dot solar cells (Wang *et al*., 2008 and Kim *et al*., 2009). Several methods have been applied to obtain ZnO thin films, such as sputtering, electron beam evaporation, chemical vapour de position (Willander *et al*., 2009). Drawbacks of using such a technique are high cost of equipment and complexity of operation (Wang *et al*., 2006).

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ZnO is a biofriendly oxide semiconductor and an inexpensive luminescent material. In general, ZnO is considered "generally recognized as safe" (GRAS) but ZnO nanoparticle system may be toxic (Dutta and Ganguly, 2012 and Zhao *et al*., 2010). ZnO nanosystem may be of important relevance in the context of nanomedicine, where targeted treatment of biological systems at molecular level is a necessity. Due to the advanced technological applications, high quality of zinc oxide nanostructures are greatly demanded, which induces worldwide research and development on the synthesis and application of zinc oxide nanostructures (Senthilkumar *et al*., 2009). There are many reports about zinc oxide thin films obtained by different methods and from different precursors. In this work we have synthesized zinc oxide thin films using electro deposition technique because of easy control of the thickness, simplicity, low equipment cost and the possibility of making large - area thin films. This method is useful to make pure material from less pure starting material. The film is characterized by various techniques like XRD, SEM, EDS, etc. The ZnO thin films were deposited from a solution of analytical grade sodium citrate and 30% hydrogen peroxide in an alkaline solution of ammonia and distilled water. Zinc plate used as substrates, were cleaned in ethanol ultrasonically.

The electrode position was carried out using zinc plate as anode and cathode. In 40ml of distilled water 0.1M sodium citrate and 30% hydrogen peroxide were introduced. The pH of the electrolyte was maintained 9-10 by introducing ammonia solution. The electrolyte was stirred moderately using magnetic stirrer. The two Zn plates (5cm × 2cm × 0.25cm) were put into the reaction solution in a parallel configuration with an interelectrode separation of 1 cm. The deposition was carried out at-4V at room temperature. The duration of the deposits was 15 minutes. The as deposited film were whitish grey in colour. The Zn cathode with the deposited products was washed with distilled water for several times, dried at room temperature, and examined in terms of their structural, compositional and electrical properties (Wang and Chen, 2011). The surface morphology for thin films were obtained using scanning electron microscope. Compound formation was studied using X-ray diffractometer fitted with curved position sensitive detector. Radiation of CuKα, glancing angle was kept at 3 degree per minute. Electrical measurements of ZnO thin films were carried out using Four probe setup and Hall effect setup.

RESULTS AND DISCUSSION

Figure 1 shows the SEM photograph of as deposited ZnO film. The grains are spherical, well connected and uniformly distributed. The grains are densely packed, free from pin holes pits and voids as is evidence from the contrast of the film. There is not any effect of charging on the film. Figure 2 shows the compositional analysis of as deposited ZnO film. The peaks of the spectra are quite distinct and shows the presence of zinc and oxygen. The atomic percentage ratio of Zn and O are observed to be 1.22 which is close to unity.

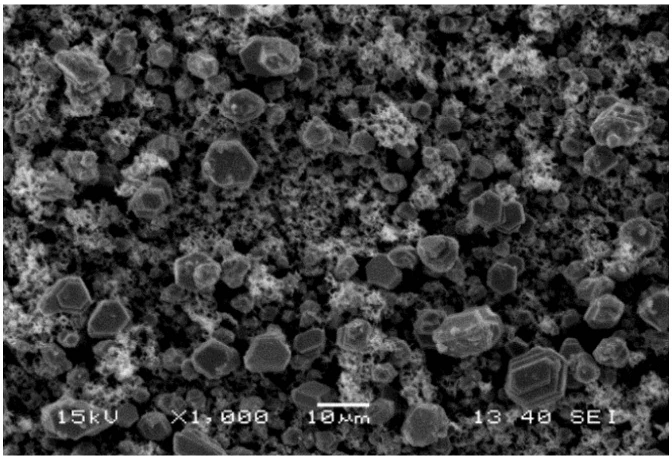

Fig. 1. SEM photographs of as deposited ZnO film

Table 1 shows the atomic and weight percentage of Zn and O. The deviation in the said percentage can be minimized by optimizing the growth conditions.

Table 1. The weight % and atomic % of as deposited ZnO film

Element	Weight %	Atomic %
Zinc	85.55	50.17
Oxygen	14.45	40.83

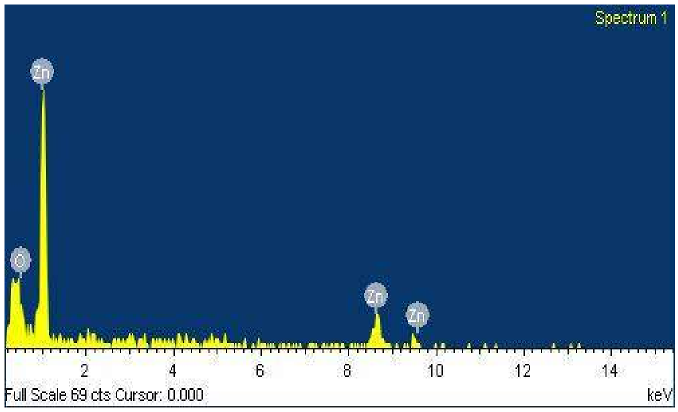

Fig. 2. EDS analysis of as deposited ZnO film

Figure 3 shows the XRD spectra of as deposited ZnO deposited on zinc substrate.The structural analysis clearly indicates the diffracted prominent peak at (101) plane. The film exhibits good crystallinity and the peak are indexed for a hexagonal ZnO lattice. The strong peaks are assigned to the pure hexagonal phase of wurtzite ZnO which are in good agreement with the reported standard values (JCPDS file No. 361451). The small peak widths indicate a good grain size. All the peaks of ZnO and Zn are identified. This indicates that the electrode position method could also be useful for the preparation of crystalline ZnO thin films. The average crystallite size has been calculated with Scherrer relation by using the full width at half maximum (FWHM) values of the (101) peak line.

$D = 0.94\lambda / \beta \cos \theta$,

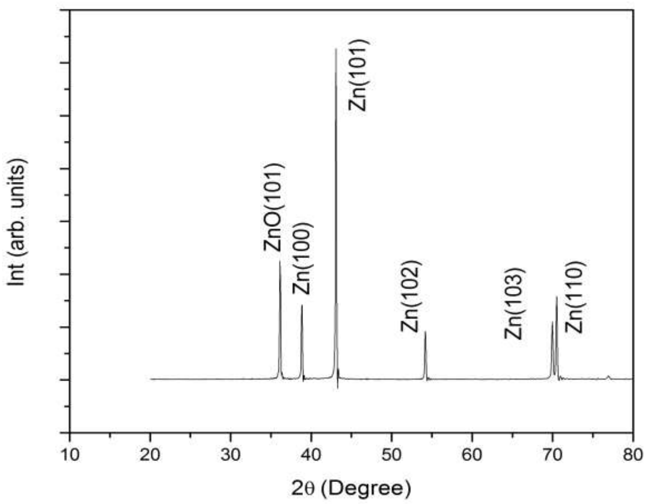

Fig. 3. XRD spectra of as deposited ZnO film

where D is the mean crystallite size, β is the full width at half maxima of the diffraction line, θ is diffraction angle, and λ is the wavelength of the X - ray radiation (Nohavica Dušan and Gladkov Petar Nanocon, 2010). The average grain size using the Debye- Scherrer's formula is observed to be 48 nm. The resistivity of as deposited ZnO film is measured using Four probe method. The resistivity of the film using formula

$\rho = V/I \times 2\pi S$

Where, V is voltage (mV) applied to the specimen, I is current (mA) flowing through the specimen, S is distance between spacing of the probes. The resistivity of the film is observed to be 30 Ω-cm. The carrier concentration of the film is determined using Hall effect set-up. The Hall coefficient is given by

$$R_H = V_H Z/IH$$

Where, V_H is Hall voltage, I is current flowing through the specimen, H is magnetic field applied perpendicular to the specimen and Z is width of sample. The carrier concentration of the ZnO film is observed to be 4.7×10^{17}carriers/cm^3.

Conclusions

The hexagonal shaped ZnO thin film is successfully deposited on zinc substrate using electrode position technique. The films are characterized for structural, compositional and electrical properties. The hexagonal phase ZnO is observed by XRD analysis. The crystallite size of ZnO particle are observed to be 48 nm. The SEM images show uniform, compact, void-free surfaces, and varying orientation of film. The resistivity and carrier concentration of the film are observed to be 30Ω-cm and 4.7×10^{17}carriers/cm^3 respectively. ZnO film presented have good characteristic to be used as for solar cell, optoelectronics, sensor applications.

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