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Electrodeposited ZnO Thin Films: Structural and Optical Characterization for Optoelectronic Applications

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Abstract: Thin film of Zinc Oxide has been fabricated using electrochemical deposition method and its structural and optical properties have been studied in this manuscript. X-ray Diffraction of thin film shows that Zinc Oxide exists in *wurtzite* hexagonal crystalline structure. Absorption spectrum Zinc Oxide thin film shows excitonic characteristic. Further band gap of ZnO thin film was calculated 3.3 eV.
Keywords: Optoelectronics • Photonics • Semiconductor • Potentiostat

Introduction

Growing demand of semiconductor materials in the field of photonics and optoelectronics has attracted Zinc Oxide (ZnO), which has been proved promising material for laser devices, light emitting devices, detectors, solar energy devices etc (Tan et al 2005, Tian et al 2004, Yoshida et al 2004). This potential of ZnO has forced researchers to prepare ZnO nano and meso materials suitable in these fields. Many physical and chemical based techniques to find ZnO materials appropriate in the field of optoelectronics have been utilized (Park et al 2006). What makes ZnO special attraction to present reaserch is not only its wide bandgap semicondcuting nature which is very useful in the field of electronics, medical, optics, sensors but also ZnO is easy to fabricates in various nano shapes such as nanowire, nanodots etc (Sharm et al 2022). Electrochemical deposition is a true bottom-up technique which has been proved very impressive to grow metallic and semiconducting thin films. Physical and chemical as well as photonic properties of ZnO are very much dependent on fabrication environment. Preparation techniques, substrate base and other physical and chemical conditions affect the quality and properties of ZnO. Therefore, to obtain the required devices we need to fabricate ZnO thin films with appropriate conditions (Cai et al 2009, Kayahan 2010, MiZuta et al 2006, Mu et al 2009) . In this work, we have explored electrochemical deposition technique to produce ZnO thin films on conducting substrate of Indium tin oxide (ITO). All samples obtained from these methods were annealed under fixed ambient conditions and characterised by scanning electron microscopy (SEM), X-ray diffraction (XRD) and optical absorption. Properties of Zinc Oxide thin films are highly dependent on its fabrication methods and in case of electrochemical deposition method aqueous solution of electrolytes, deposition voltage and temperatures are the few parameters need to be optimized for the desired properties (Shao et al 2004, Teo and Ramakrishna 2006). After this direct deposition we don't find ZnO thin film directly but $Zn(OH)_2$ thin film. $Zn(OH)_2$ thin film have been further annealed at 400^0C for three hours. Study shows properties of ZnO depend on defects present intrinsically in ZnO. These defects play important role in photonics. ZnO thin films fabricated in present work is cost effective and particularly ease of fabrication could be very helpful for industrial

fabrication of ZnO in the field of electronics, optoelectrnics and photonics.

Methodology

Three electrode potentiostatic method has been utilized to ensure fabrication of high quality Zinc Oxide thin films. This method has advantage over various others deposition methods, such as ease of fabrication and its cost of fabrication among other advantages. Growing optoelectronic and photonic industries are demanding semiconductors which are non toxic and cost effective and among many semiconductors wide band gap ZnO is used to study in present work. Cyclic voltammetry has been studies extensively to find out optimum potential at which thin films have been deposited. Graphical Tauc plot method has been used to calculate band-gap of ZnO thin films. Band-gap is found by extrapolating linear region in absorption versus photon energy plot.

Experimental details

ZnO thin films were fabricated from electrochemical deposition methods. In electrochemical deposition aqueous electrolyte solution of 0.1M $Zn(NO_3)_2$was used on the ITO substrate using a potentiostatic three-electrode method. Thin film obtained from this chemical recipe was in the form of $Zn(OH)_2$ and to obtain ZnO, the sample was annealed at 400^0C for three hours under ambient conditions. ZnO thin film has been prepared using electrolyte made of aqueous solution of 0.1 M Zn $(NO_3)_2$ and pH of the electrolyte is maintained at 5.2. Here we need to apply a threshold potential between electrodes to enable mass transport of Zn^{2+} ions on to substrate. This potential is retrieved by voltammetry of electrolyte, which is also critically dependent on substrate. Voltammetry gives relationship between resultant current and applied potential. There are many methods to perform voltammetry such as linear sweep voltammetry, cyclic voltammetry etc. In the present work, we have performed cyclic voltammetry (CV) to find out the deposition potential. Scan rate of 0.001V/s was used to produce these films for 100 seconds at -1.1V poential.

Observation and Discussion

The Cyclic voltammetry (CV) of 0.1 M $Zn(NO_3)_2$ on ITO substrate, recorded over the range of 0 to -1.5V. When potential goes from 0 to -1.5V, reduction of reactant happens and this is called reduction cycle (Fig 1).

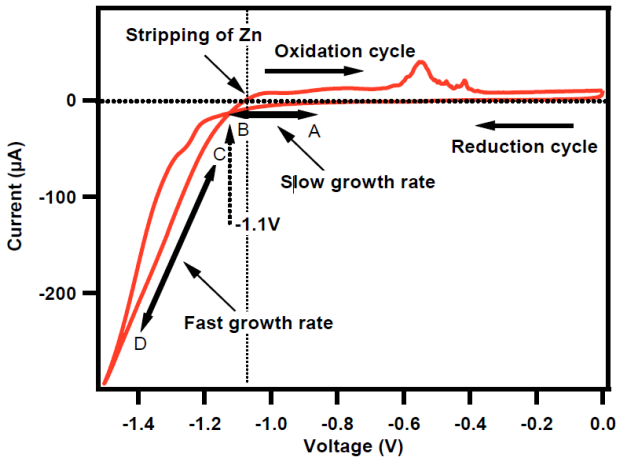

Fig 1. Cyclic voltammetry of aqueous solution of $Zn(NO_3)_2$ on ITO.

At -0.9V, current goes below zero i.e. cathodic current flows in cell and deposition of Zn^{2+} occurs. Upto -1.1V deposition is slow but after that there is a steep increase in cathodic current which shows a fast deposition rate for Zn^{2+}. However, in the reverse direction i.e. from -1.5V to 0V, after a certain value of voltage (-1.06 V), current value goes to

positive i.e. cathodic current turns into anodic current. After this point stripping of metallic Zn^{2+} occurs. As we can see in the CV plot stripping of Zn happens in very less amount as compared to deposition, therefore, even after full oxidation cycle deposition of Zn will remain on the ITO. In the reduction cycle, ZnO can be deposited from -0.9 V. No deposition has been observed below this value. We have to choose a compromising potential between slow and fast growth to achieve a satisfactory deposition rate. A potential of -1.1V was found to be suitable to deposit good quality stoichiometric ZnO thin films. Hence, films deposited at this potential were used for several characterizations. In the first step of deposition process, nitrate (NO_3^-) ions (Zn^{2+} and NO_3^- ions lives in ionic form in the polar solution of deionised water) react with water molecules and a reduction reaction takes place with the formation of hydroxyl ions. Zinc ions react with these hydroxyl ions and form $Zn(OH)_2$ thin film on ITO. To obtain desired ZnO films the $Zn(OH)_2$ films should be de-hydrated ($Zn(OH)_2 \rightarrow ZnO + H_2O$), and for that the samples were annealed at 400^0C for three hours under ambient conditions.

Structural and Optical characterization of ZnO thin film

ZnO thin films obtained from this method show following structural and optical properties. **Fig 2a** shows a typical X-ray diffraction pattern (XRD) of ZnO thin film electrodeposited on ITO at -1.1 V using the electrolyte solution of zinc nitrate. The three most intense peaks are at 2theta values of 31.77^0, 34.42^0 and 36.27^0 corresponding to (100), (002) and (101) planes, which confirms that ZnO exists in *wurtzite* hexagonal crystalline structure (3-6). Diffraction peaks obtained from sample are identified with JCPDF file no. 80-0075. Unlike few other cases no preferential growth is observed and appearance of multiple diffraction peaks reveal polycrystalline nature of the film. No other peak (including substrate) is identified which implies deposition of dense and defect-free ZnO thin film.

Fig 2b shows the SEM image of the same ZnO thin film prepared at potential -1.1V which is annealed at 400^oC for three hours. SEM image of electrodeposited film shows dense and island type morphology, which can be further improved by controlling the deposition potential, substrate treatment, temperature and pH of the solvent.

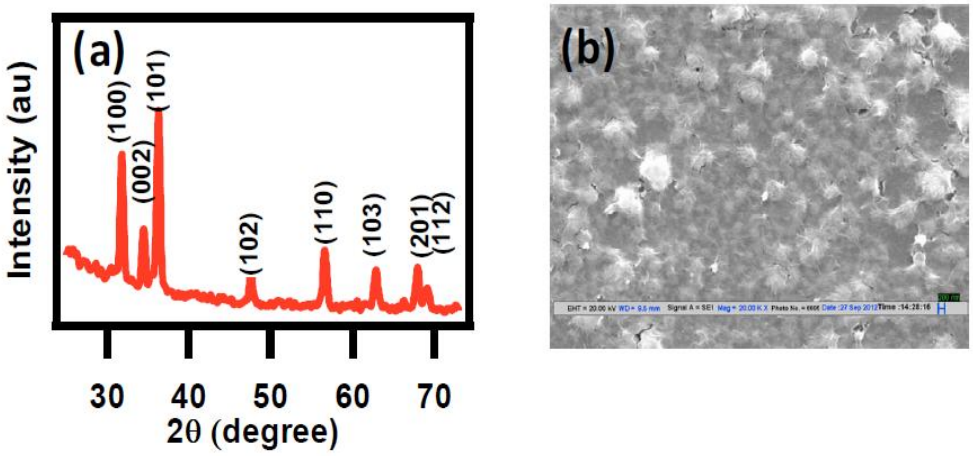

Fig 2a: X-ray diffraction spectra and (b) SEM image for the ZnO thin film electrodeposited at -1.1V on ITO using nitrate solution.

Optical Characterization-

It has been known that ZnO prepared from different methods show marked variation in optical absorption and emission properties. Specifically, three different processes: band to band transition, excitons and deep level

defects are responsible for the optical properties of ZnO. **Fig 3a** shows the absorption spectrum of ZnO thin film obtained from electrodeposition from $Zn(NO_3)_2$ solutions on ITO substrate. These films how dominant excitonic related absorption at 363 nm along with the band to band direct bandgap absorption

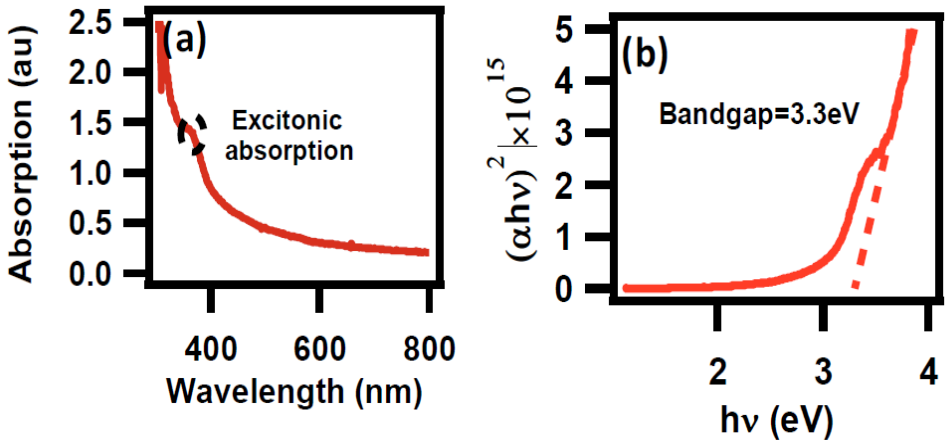

Fig 3a: Absorption spectrum and (b) Tauc plot for the ZnO thin film electrodeposited at -1.1V on ITO using nitrate solutions.

ZnO is recognised as a potential II-VI photonic semiconductor material owing to its wide direct band gap (~3.37 eV) and high exciton binding energy (~60 meV) (4-8). Due to such large binding energies, these excitons (Mott type) are visible even at room temperature. Excitonic absorption and high absorption in UV region make ZnO one of the most important materials for blue and UV optoelectronics. Optical Band gap of ZnO thin film has been calculated using well-known Tauc plot (Figure 3b). Since ZnO is a direct band gap semiconductor, the absorption coefficient can be related to photon energy as,

$$(\alpha h\nu)^2 = A(h\nu - E_g)$$

Where A is proportionality constant, $h\nu$ is the photon energy of the incident light, and E_g is the optical band gap. Extrapolating the tangential line to the x axis i.e. photon energy in the plot between $(\alpha h\nu)^2$ and $h\nu$ gives the band gap. In this case band gap is found to be 3.3 eV which is in good agreement with other reported ZnO prepared by electrodeposition as well as by other fabrication techniques.

Conclusion

Wide band gap of 3.3 eV for the electrodeposited ZnO thin film has been achieved and optimized using Tauc plot. Absorption spectrum shows exciton formation in the thin film prepared using this method. Island type morphology is achieved and XRD pattern shows wurtzite hexagonal crystalline structure of ZnO thin film.

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