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ZnO Nanoparticles Synthesis Using Hydrothermal Method

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Abstract: ***ZnO nanorods were prepared using hydrothermal method and their Structural and Optical properties were studied. ZnO seed layer thin films were prepared by dip coating technique on to well cleaned glass substrate using Zinc acetate as starting material. ZnO nanorods it is grown by hydrothermal method by implementing ZnO seed-coated glass to be substrates in aqueous solution of Hexamethylenetetramine (HMT) and Zinc nitrate it taken different concentrations 1:10, 2:10 & 3:10 for annealed for 5 hours in 500°C. The prepared ZnO nanorods were characterized by X-ray diffraction (XRD) and Scanning electron microscopy (SEM). Improved hydrothermally grown ZnO Nano rods were prepared and investigated using sol-gel dip coating method for Solar Applications.***

Keywords: ZnO Nano Rods, Structural properties, Sol-Gel Method, Dip Coating, Hydrothermal, Solar Cell.

I. INTRODUCTION

The Zinc oxide (ZnO) thin films have wide applications as gas sensors, solar cells, laser systems light emitting diodes (LED's), and transparent electrodes and also have the properties of excellent optical, electrical and structural. Moreover, they can be prepared by different techniques, such as magnetron sputtering, hydrothermal method, atomic layer deposition and thermal evaporation. Among the above methods to prepare ZnO, hydrothermal method is extremely attractive due to its advantages features over other thin film deposition technique, such as its simple low temperature, low cost, easy coating of large surfaces and low evaporation temperature. Obviously, the size of ZnO nanorods play important role in optimizing the optical properties of ZnO nano rod array films. In this paper, ZnO nano rod films were prepared by hydrothermal method at the temperature of 500°C. Effect of the growth temperature of ZnO nano rod films structure and morphology properties were investigated by XRD and SEM. To deposit ZnO films used the method of dip coating for forming on the glass as a seed layers.

II. EXPERIMENTAL DETAILS

A. ZnO Seed layer - Sol-Gel Method

The dip coating method was implemented in the current work to prepare ZnO seed layer on the glass substrates because of its low cost and easy to handle. A coating solution was prepared by dissolving Zinc Acetate Hexahydrate [Zn(NO3)2.6H2O] (Nice, 99.0% purity) and equivalent 20 ml Ethanol [C2H5OH] in 0.25 mol of deionized water. This solution was stirred continuously for 2 hours at room temperature. The resulting solution was used as seed layer and deposited on well cleaned glass substrates by automatic dip coating. All the glass substrates were dipped 5 times at room temperature. Then the 5-layer films were annealed in a furnace at the temperature 200°C for 1 hour. Fig.1 shows the Flow Diagram of the ZnO Growth Layer Nano Rods Thin Film prepared from sol gel process using the dip-coating method.

B. ZnO Growth Layer - Hydrothermal Method

ZnO nanorods were grown on seed coated glass substrates by hydrothermal technique. After uniformly coating the glass substrates with ZnO thin films, hydrothermal growth of ZnO Nano rod was achieved by suspending these ZnO seed-coated glass substrates upside-down in a glass beaker filled with aqueous solution Zinc Nitrate 0.02 mol (Zn(NO3)2.6H2O) (Sigma Aldrich, 98% purity) and Hexamethylenetetramine 0.2 mol (C6H12N4) (Sigma ldrich,99.5% purity) During the growth, the glass beaker was heated with a laboratory oven and maintained at temperature 100°C for 4 hours. At the final stage of the growth period, the substrates were removed from the solution, then immediately to be washing lightly with the deionized water to the remove any kind of residual salt

from the surface, and dried in the room temperature. Thus the above films were annealed in the muffle furnace at temperature level of 500°C for 1 hour.

To display the report from the effect of three different concentrations on the grown of ZnO Nano rods with constant annealing the temperature. Concentrations of 0.02mol of Zinc Nitrate Hexahydrate [Zn(NO3)2.6H2O] and 0.2 mol hexamethylenetetramine (HMT) C6H12N4, were taken in the ratio of 1:10. The other two ratios were 2:10 and 3:10. ZnO Nano rods were grown in above three concentrations, annealed at same temperature of 500°C for 1 hour. The properties of grown ZnO Nano rods were investigated by X-ray diffraction (XRD) on XPERT-PRO X-ray diffractometer with Cu K_radiation ($\lambda = 1.54060$ nm) at a scanning rate of 0.05◦ s−1in the 2θ range from 10◦ to 80◦. To investigate the optical properties, the absorption and transmittance spectra were recorded by UV–Vis spectra on a JASCO Corp., V-570 spectrophotometer at room temperature.

C. The following Steps involved in the ZnO Growth Layer Nano Rods Thin Film

1) Zinc Acetate Dihydrate
2) Ethanol + De ionized Water
3) Stirring for 2 Hours at Room Temperature
4) Glass Substrates Dipped 5 times in Clear and Homogeneous Solution
5) Annealing at Temperature 200°C for 1 Hour
6) Seed Layer dipped in below Solution in three different Concentrations 1:10, 2:10 & 3:10 at Temperature 100°C for 4 Hours
7) Zinc Nitrate Hexahydrate + Hexamethylenetetramine + De ionized Water
8) Annealing at Temperature 500°C for 1 Hour
9) ZnO Growth Layer Nano Rods Thin Film

III. RESULTS AND DISCUSSION

A. Structural Analysis

The Fig. 1 shows the XRD patterns of the product of the thin films with growth solution Zinc nitrate and Hexamethylenetetramine (HMT), with different Concentrations 1:10, 2:10 & 3:10 and annealed at 500°C. It reveals that the well-aligned Nano rods have a hexagonal wurtzite crystal structure with a c-axis (002) preferential orientation. All the diffraction peaks can be indexed within experimental error as hexagonal ZnO phase (Wurtzite-structure) which matches with the JCPDS. The strong and narrow diffraction peaks indicate that the material has a good crystalline and size. The XRD patterns of the films concentrations of 1.10 and 2.10 for 5 hours annealed at 500°C shows that, when concentration of 1:10 and 2:10 is compared, the 2:10 concentration is better than 1:10

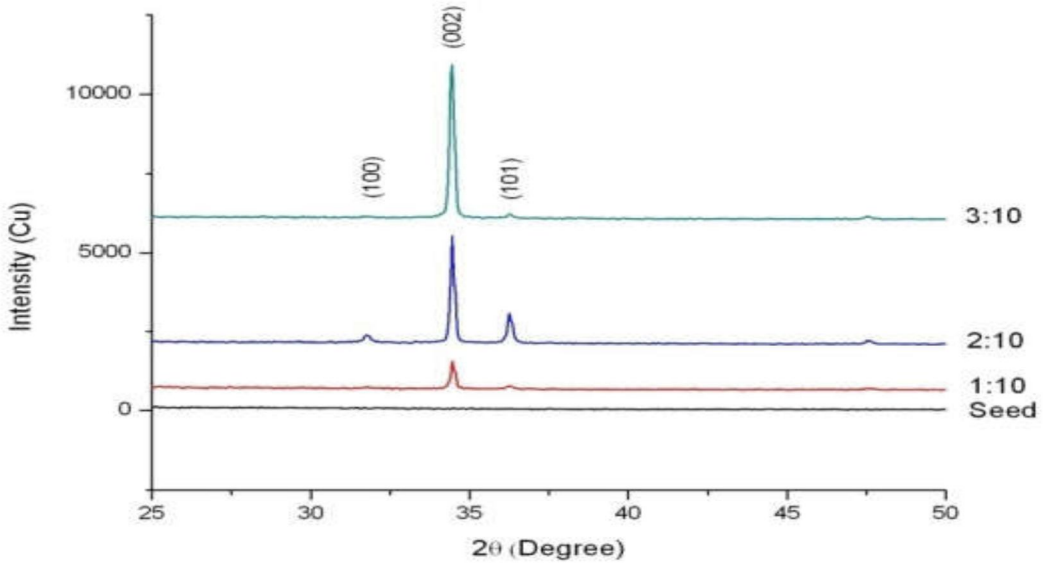

Fig.1 XRD pattern of ZnO growth layers on different growth solution concentrations at 500°C with Seed Layer

concentration, which is seen in the XRD pattern, where (002) peak is stronger. The XRD pattern growth concentration of 3.10 for 5 hours annealed at 500°C has a very strong (002) peak. The above result of XRD patterns it is clearly to seen that the growth concentration level in diffraction peaks were oriented to strongly along with the (002) peak.

B. Optical Analysis

The Optical properties of ZnO Nano rods are important for many of their technological applications. In most cases the UV-Vis spectra of ZnO comprised of Absorption and Transmittance and the relationship between the two depends strongly on the preparation method and post-preparation treatment. The UV spectra in ZnO Nano rods are well accepted as the near-band-edge emission.

The optical absorption spectrum is shown in Fig.2 (a) is clearly indicates that, as growth solution concentration increases the optical absorption edge shift to a higher wavelength. The intensity of the absorption spectra increases considerably as growth solution concentration increases from 1:10 to 3:10. It is well known that the optical absorption determines the optical band gap and ZnO films have a direct band gap. The optical band gap of ZnO films was found to decrease as growth solution concentration increases from 1:10, 2:10 and 3:10 respectively. The decrease in band gap of ZnO films may be attributed to the improvement in the crystalline quality of the films and increase of grain size.

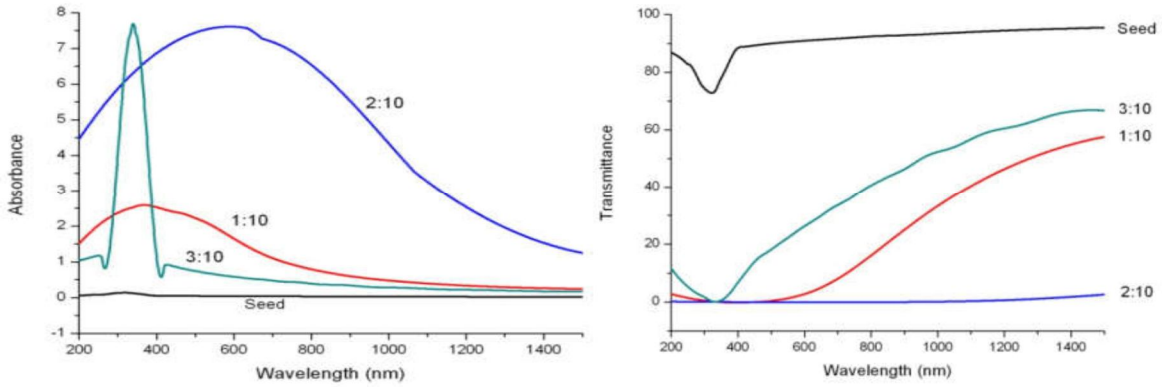

Fig.2 shows the Absorption and Transmittance spectra at three different growth solution concentrations

Fig.2 (b) shows the optical transmittance spectra from samples with three different growth solution mol concentrations of 1:10, 2:10 and 3:10, annealed at constant temperature of 500°C for 1 hour, which was obtained on a spectrophotometer. The transmittance spectra is in the visible range nearer to infrared wavelength region that is at 83%, which reveals the superior optical properties in the ZnO thin films produced by novel sol-gel method. The effect of change in the Nano rod molar concentration on the optical transmittance for samples was investigated.

A slight to the decrease average transmission of the above observed to become increase the growth layer of molar concentration and it is attributed to the different of surface it become uneven or irregular. The optical transmittance of ZnO films was found to different from 58%, 05%, and 63% with the increase of growth layer molar concentration. The results indicated high optical quality ZnO Nano rods were successfully achieved via this low temperature chemical approach.

C. Scanning Electron Microscopy (SEM)

The images can be indexed as hexagonal Wurtzite-structural ZnO, which is very consistent with the analysis of XRD. The XRD patterns to be started at the SEM images of the ZnO nano rods that indicate the length of the ZnO nano rods is maximum, when the growth concentration is at 2.10.

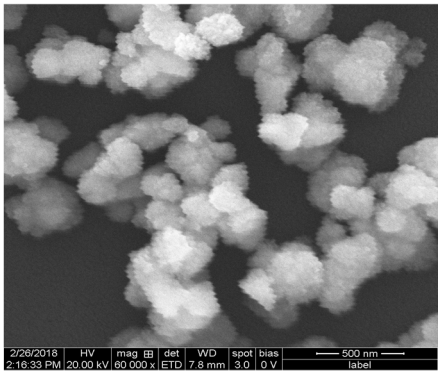

Fig.3 (a) ZnO Seed layer

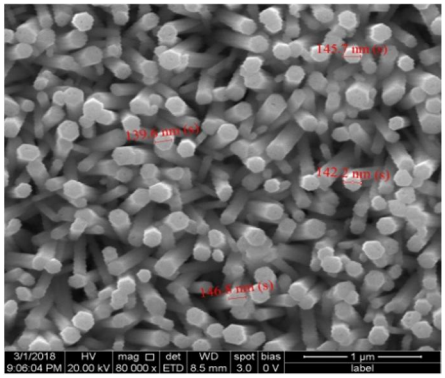

Fig.3 (b) ZnO Growth Layer 1:10

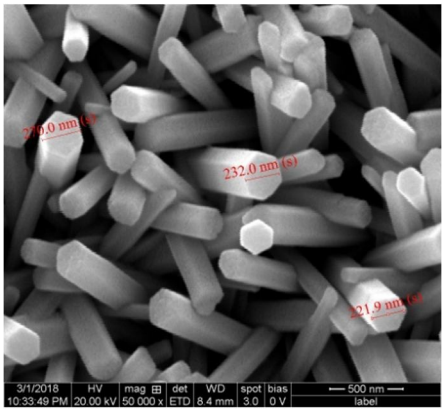

Fig.3 (c) ZnO Growth Layer 2:10

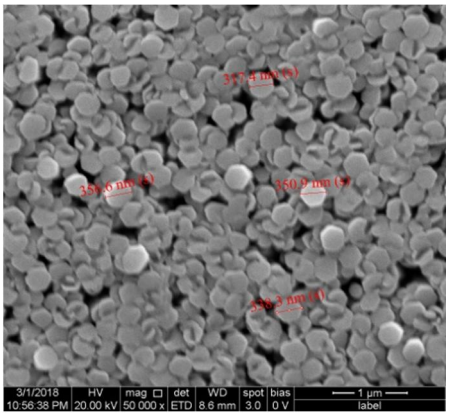

Fig.3 (d) ZnO Growth Layer 3:10

Fig 3 .SEM image of ZnO nanorods

It indicates that the samples grown in same solution experienced different growth rate of nanorods at growth concentration 1:10 and 3:10. The growth rate is defined along with growing length per growth concentration. Fig.3a and Fig.3c shows SEM images of ZnO nanorods grown at 1:10 and 3:10 in which rods have grown in all directions, which reveals the XRD patterns, where all three peaks are moderately reflected. Fig.3b shows SEM image of ZnO nano rod grown at 2:10. These rods show hexagonal structure with increase in diameter and its length towards c-axis orientation. This result relates with the peaks as indicated in the XRD pattern.

IV. CONCLUSION

ZnO nano rods had been successfully synthesized in a hydrothermal method growth concentration of 2:10 for 5 hours and annealed at 500°C. The prepared ZnO nanorods were characterized by X-ray diffraction (XRD), UV and Scanning electron microscopy (SEM). From the XRD results, it is clearly seen that, at the growth concentration of 2:10 the diffraction peaks were oriented strongly along the (002) peak. The grain size of the nano particles are found to be increasing as growth concentration increases. SEM results clearly show that nanorods grown at 2:10 have hexagonal structure with increase in diameter and length towards c-axis orientation when compared to 1:10 and 3:10. Experiments showed that the different growth concentration would influence structure and morphology of the prepared ZnO nano rods.

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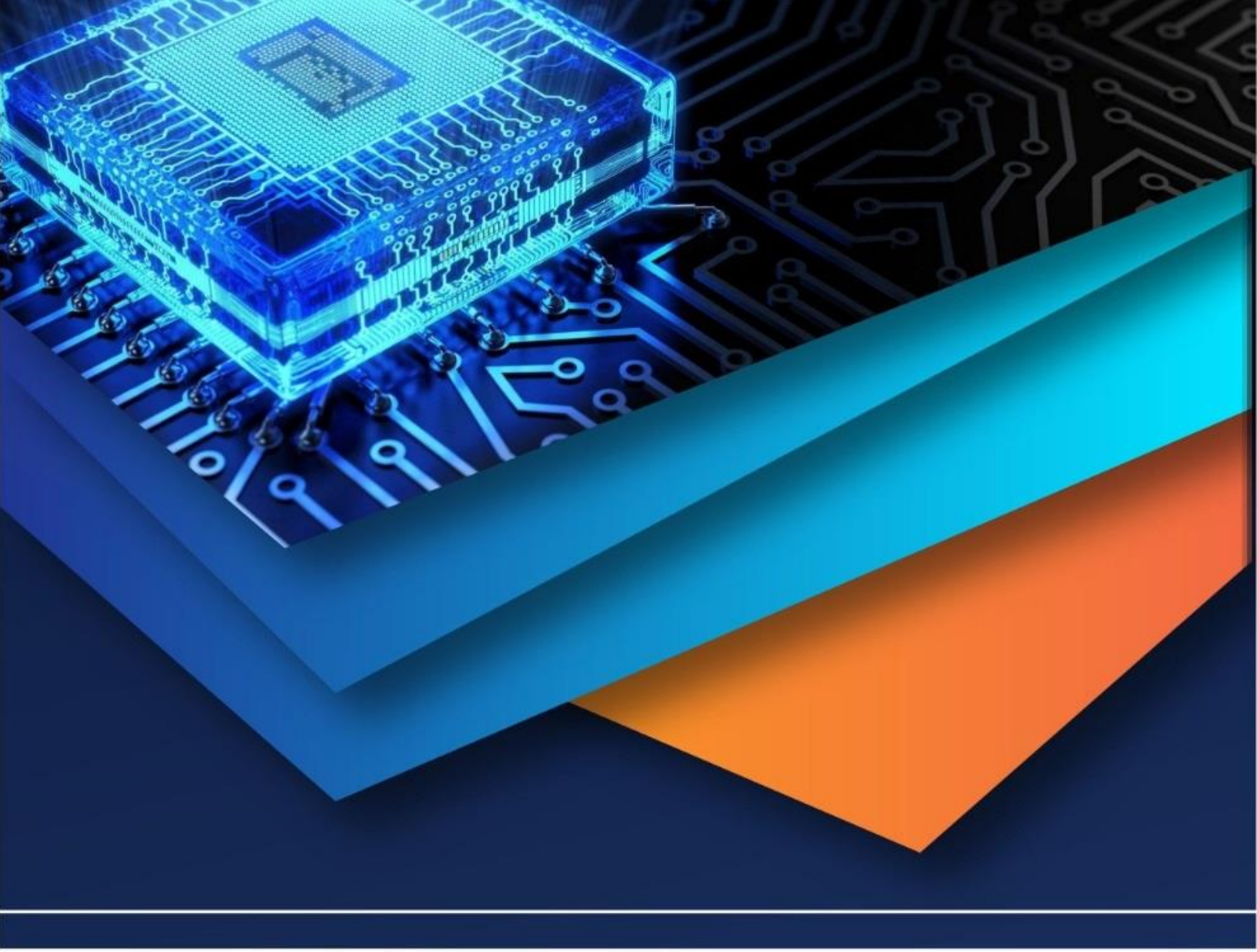

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