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Structural, Morphological and Optical Properties of ZnO Nanorods Prepared by Hydrothermal Method

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***Abstract:** The Chemical Bath Deposition method was used for the preparation of ZnO nano rods and their structural, morphological and optical properties were taken for study. ZnO seed layer thin films were prepared by chemical bath deposition method on well cleaned glass substrates.*

ZnO seed-coated glass substrates were immersed in aqueous solution of zinc nitrate and hexamethylenetetramine (HMT) for 4 hours at low temperature. The above materials were taken in three molar concentrations of 1:10, 2:10 & 3:10 were used for the growth of zinc oxide nano rods.

The growth layer concentration influences the structural, surface morphology and optical properties of the films were examined. The structure of the ZnO nano rod was studied with x-ray diffraction.

The surface morphology was studied with scanning electron microscope. The optical property was studied by UV-Vis spectroscopy. The grown ZnO nano rods were prepared and investigated for gas sensor applications.

***Keywords:** Chemical bath deposition, Hydrothermal, ZnO Nano Rods, Structural, Morphological, optical properties, Gas sensors.*

I. INTRODUCTION

Semiconductor materials are always a research focus in material science due to their unique electronic, optical properties and extensive applications.

Understanding the fundamental properties of these materials will provide opportunities to design advanced materials and to fabricate novel nano-devices for future applications. In these materials, wide and direct band gap semiconductors are of great interest in blue and ultraviolet optical devices such as light-emitting diodes and laser diodes (1).

Zinc oxide (ZnO) is a wide and direct band gap (3.37 eV) semiconductor with a large exciton binding energy (60 meV) (2), has already been widely used in piezoelectric transducers, gas sensors, optical waveguides, transparent conductive films, varistors and solar cell windows, bulk acoustic wave devices (3-7).

Over the past decades, ZnO crystallites had been obtained by several preparation approaches including sol–gel method (8), evaporative decomposition of solution (9), wet chemical synthesis (10), gas-phase reaction (11) and hydrothermal synthesis (12) etc. A variety of morphologies including prismatic forms (13), spheres (14), nano rods (15), and nano wires (16) had been synthesized.

Among the above methods to prepare ZnO, hydrothermal synthesis route, is an important method for wet chemistry that has been attracting material chemists' attention.

The seed layer is prepared by low cost and simple chemical bath deposition method. Employing this method, ZnO nano rods were obtained.

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II. EXPERIMENTAL DETAILS

A. Preparation of ZnO Seed layer - Chemical bath deposition Method

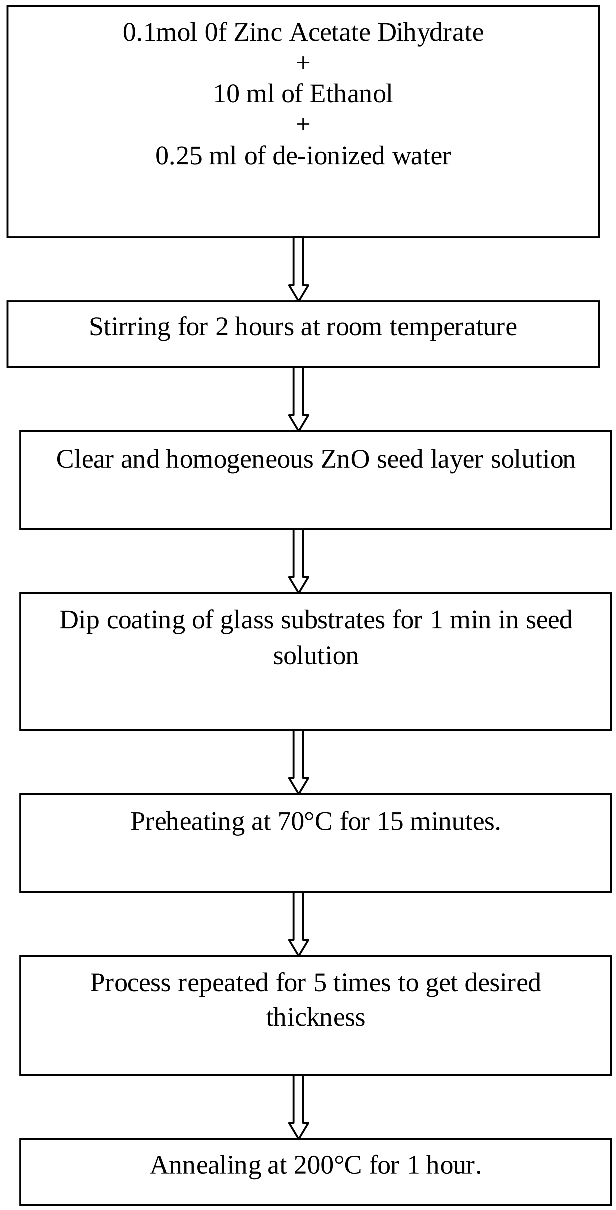

Fig 1. Flow chart depicting the preparation of ZnO seed layer thin film.

The chemical bath deposition method was implemented in the current work to prepare ZnO seed layer on the glass substrates. A coating solution was prepared by dissolving Zinc Acetate Hexahydrate $[Zn\ (CH_3C0_2)_2.6H_2O]$ (Nice, 99.0% purity) and equivalent 20 ml Ethanol $[C_2H_5OH]$ in 0.25 mol of de-ionized 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 machine. The dipping process was repeated for 5 times to get desired thickness.

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 seed layer thin film preparation by chemical bath deposition.

B. Preparation of ZnO Growth Layer - Hydrothermal Method

0.02mol 0f Zinc Nitrate
+
0.2 mol of Hexamethylenetetramine
+
De-ionized water

⇓

Stirring for 2 hours at room temperature

⇓

ZnO Growth layer solution

⇓

Hydrothermal process at 90°C for 4 hours in hot air oven

⇓

Rinsing with de-ionized water to remove impurities

⇓

Annealing at 500°C for 1 hour.

Fig 2. Flow chart depicting the preparation of ZnO nanorods

ZnO nanorods were grown on seed coated glass substrates by hydrothermal method. 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 of 0.02mol of Zinc Nitrate ($Zn(NO_3)_2$ (Sigma Aldrich, 98% purity) and 0.2 mol of Hexamethylenetetramine ($C_6H_{12}N_4$) (Sigma ldrich,99.5% purity) at 1:10 concentration. 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 de-ionized water to the remove any kind of residual salt from the surface and dried in the room temperature. Then the above films were annealed in the muffle furnace at 500°C for 1 hour. The above process was repeated for other two concentrations of 2:10 and 3:10.
Fig.2 shows the flow diagram of the ZnO growth layer nano rods thin film prepared from sol gel process using hydrothermal method.

III. RESULT AND ANALYSIS

X-ray diffraction (XRD) was carried out on a XPERT-PRO X-ray diffractometer with Cu K_radiation (λ = 1.54060 nm) at a scanning rate of 0.05◦ s−1in the 2θ range from 10◦ to 80◦. Scanning Electron Microscopy (SEM) micrographs were taken on a Scanning Electron Microscope. UV–Vis spectra were recorded on a JASCO Corp., V-570 spectrophotometer.

A. X-Ray Diffraction studies

Fig. 3 shows the XRD patterns of the ZnO nano rods with three different growth layer concentrations of 1:10, 2:10 & 3:10 and seed layer.

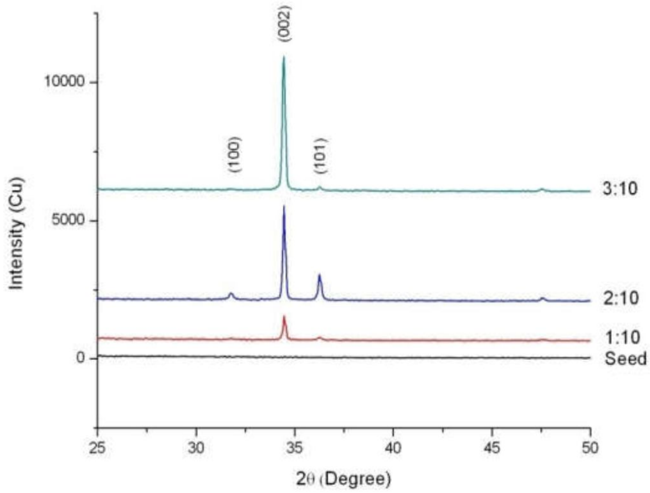

Fig 3 XRD patterns of ZnO nanorods prepared at three different growth concentrations

It reveals that the well-aligned ZnO nano rods have grown with hexagonal wurtzite crystal structure with a c-axis (002) preferential orientation (17). All the diffraction peaks can be indexed within experimental error as hexagonal ZnO phase (Wurtzite-structure) which matches with the JCPDS card no 036-1451. The strong and narrow diffraction peaks indicate that the material has well crystalline and size (18). As growth layer concentration increases the c – axis orientation increases, this is shown clearly by the increase of (002) peak in the graph. At 3:10 the intensity of the (002) peak is high.

B. Scanning Electron Microscope studies (SEM)

The SEM images can be indexed as hexagonal wurtzite-structural ZnO, which is very consistent with the analysis of XRD. The growth rate of the ZnO nano rod is defined along with growing length per growth concentration. Fig.4a shows the image of ZnO seed layer where wurtzite structure can be seen but not clearly because it is in the preliminary stage. Fig.4b shows SEM images of ZnO nanorods grown at 1:10 concentration in which rods have grown in all directions, which reveals the XRD patterns, where all three peaks are moderately reflected [19].

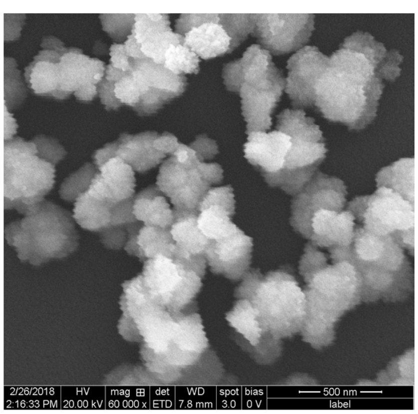

Fig.4(a) ZnO Seed layer

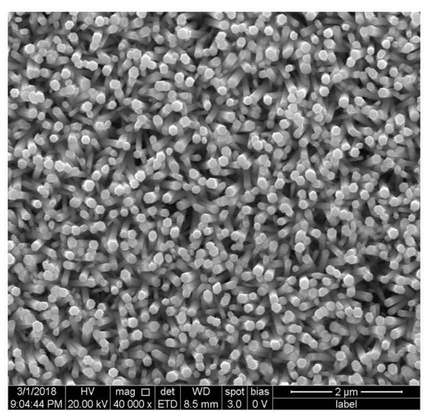

Fig.4(b) ZnO Growth Layer 1:10

Fig.4c shows SEM image of ZnO nanorod 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. Fig 4d shows the SEM image of ZnO nanorod grown at 3:10. The rods clearly show it is well aligned in C-axis orientation with better length and size when compared to all other growth concentrations.

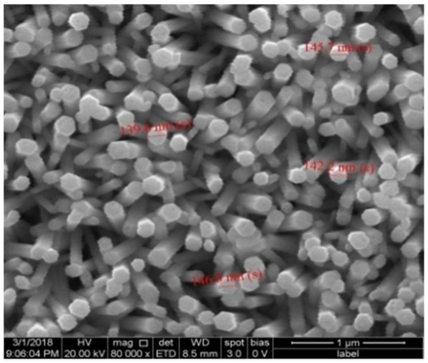

Fig.4(c) ZnO Growth Layer 2:10

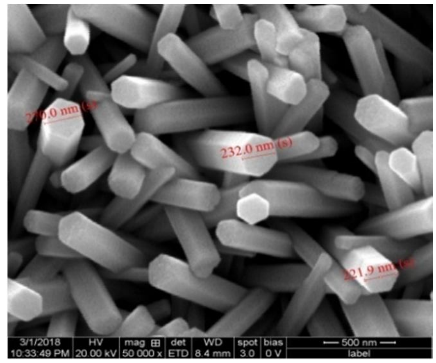

Fig.4(d) ZnO Growth Layer 3:10

C. Optical Studies (UV-Absorption and Transmission)

The UV-Visible spectrum of ZnO comprises of Absorption and Transmittance and the relationship between the two depends strongly on the preparation method and post-preparation treatment. The optical absorption spectrum is shown in Fig.5 (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.5 (b) shows the optical transmittance spectra from samples with three different growth solution concentrations of 1:10, 2:10 and 3:10. 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 decrease in the average transmission was observed when there is an increase in the growth layer concentration and it is attributed to the surface roughness ie., it becomes 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.

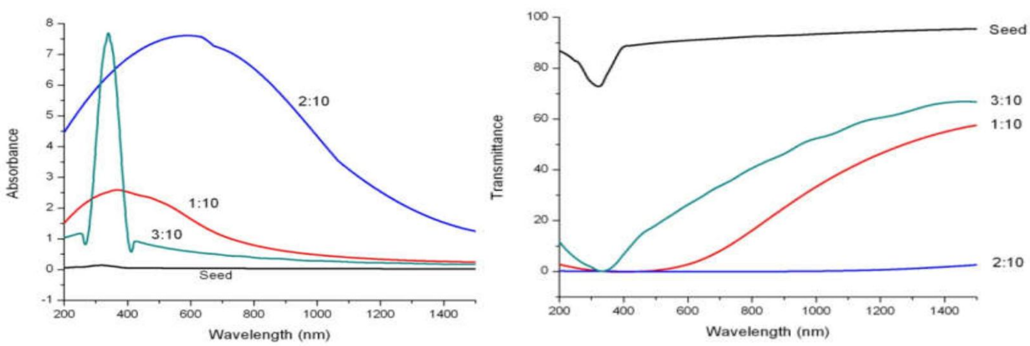

Fig.5 (a&b) Absorption and Transmittance spectra at three different growth concentrations

IV. CONCLUSION

ZnO nano rods had been successfully synthesized in a simple Chemical bath deposition method at low growth temperature of 90°C for 4 hours via the hydrothermal method three different growth concentrations of 1;10, 2:10 and 3:10 and annealed at 500°C. From the results of XRD and SEM, it was clearly indicated that the high growth layer concentration of 3:10 of the film leads to fast growth rate through size on the nano rods. The intensity of the absorption spectra increases considerably as growth solution concentration increases from 1:10 to 3:10.. This leads to the improvement in the crystalline quality of the films. A slight decrease in average transmission was observed with the increase in annealing temperature and those results are attributed to the increase of surface roughness. Experiments showed that the different growth solution molar concentration would influence morphologies and optical properties of the prepared ZnO nano rods. These prepared ZnO nanorods can be used for gas sensing properties.

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