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Fabrication of ZnO Based Optical Fibre for Ethanol Sensing Applications

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Abstract: Today, there is an increasing demand for sensors that includes home-security, military, industrial and clinical diagnosis. In the past, Fiber Optic based Sensor (FOS) has made profound impact due to its simple, room temperature detection and large range of detection species. In the present work, a simple optical fiber for sensing ethanol vapour was developed using borosilicate glass rod as core and Successive Ionic Layer Adsorption and Reaction (SILAR) deposited zinc oxide (ZnO) thin film as cladding materials. Structural studies carried out using X-ray Diffraction (XRD) method indicates, the ZnO film was polycrystalline hexagonal structure in nature with preferential orientation along (002) plane. Grain size obtained through line broadening technique was found to be 25 nm. The two ends of the fabricated fiber were connected to a diode laser source of 850 nm and optical power meter of 0.01 dBm accuracy as detector. The transmitted light intensity of the fabricated fiber found to changes in ethanol vapour atmosphere. The response and recovery time towards ethanol at room temperature as a function of cladding thickness and length were analysed and reported.

Key words: SILAR, ZnO, thin film, laser source, atmosphere

INTRODUCTION

Existing sensor for detecting trace amount of volatile vapours in the environment was based on measuring the change in electrical conductivity of semiconducting materials. Also these sensors are operated at elevated temperature for better sensitivity and selectivity (Suryawanshi *et al.*, 2008; Lee and Reedy, 1999). Micro-heaters were used which consume minimum of 35 to 50 mW power (Lim *et al.*, 2001) to operate sensor at elevated temperature. Hence, there is a need of room temperature vapour sensing to minimize the power consumption.

FOS is a good alternative one. Existing FOS are designed either modifying cladding (Vijayan *et al.*, 2008; Grant *et al.*, 2006; El-Sherif *et al.*, 2007) or core of the fiber (Akita *et al.*, 2010; Iga *et al.*, 2003; Liu *et al.*, 2003; Topliss *et al.*, 2010) which makes the design part too complex. Instead, in the present work, the sensing material (cladding) was deposited as thin film over a glass rod (core) of 1 mm diameter. The deposited film will serve as a transducer part which makes the fabrication work simple.

MATERIALS AND METHODS

Fabrication of FOS: Chemical solution methods such as SILAR (Shinde *et al.*, 2007) and Chemical Bath Deposition (CBD) (Khallaf *et al.*, 2009) were used to obtain ZnO films. To prepare core of optical fiber, commercially available borosilicate glass (1.51-1.54 refractive index across the visible range) of 4 mm diameter is dropped into 1 mm. Zinc-ammoniate complex solution ($[Zn(NH_3)_4]^{2+}$) serves as cationic precursor was prepared by adding drop by drop (interval of 2/3 sec) of ammonium hydroxide (40%) in 0.1 M of zinc nitrate solution under constant stirring kept at room temperature. Deionised water kept at 95°C serves as anionic precursor. ZnO thin films were grown heterogeneously by dipping the glass rod on cationic and anionic precursor solution for 3 and 5 sec, respectively. The number of dipping cycles was varied from 25 to 100 insteps of 25 to obtain different film thickness. The as-grown films were annealed at 250°C for 1 h to improve the crystallinity. Figure 1 shows the photograph image of prepared ZnO film on glass rod for different dipping cycle.

Experimental analysis: The grain size and crystalline nature of the prepared ZnO film was investigated using PANalytical X-ray diffractometer (Model D/MAX ULTIMA III). Surface morphology of the ZnO films was investigated by using HITACHI made Scanning Electron Microscope (SEM) of Model S-3000H with 18 kV accelerating potential. The film thickness was measured using Stylus Profilometer (Mitutoyo Stylus Profilometer

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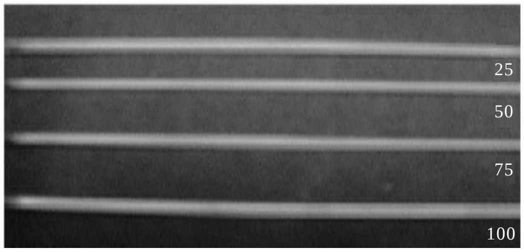

Fig. 1: Photograph image of prepared ZnO film on glass rod for different dipping cycle

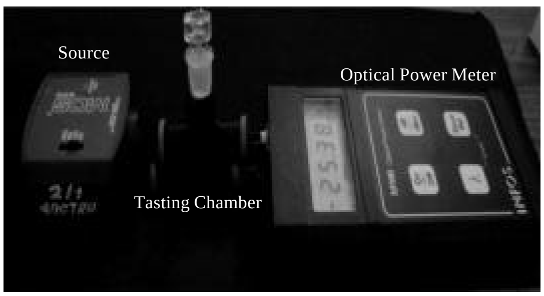

Fig. 2: Photograph image of experimental setup

SJ-301). The optical studies were carried out using ELICO SL 159 UV-Vis Spectrophotometer. The ethanol vapour response measurements of the prepared FOS were carried out in the home made test chamber made. The light source used in the present work is of 850 nm obtained from MOS 850-ST operated at 9V dc. The detector was INFOS-M100 optical power meter. The accuracy of the detector is 0.01 dBm. During testing, the whole chamber is kept in vibration free table and optically made dark. The prepared FOS was connected to source and detector with the help of plastic fiber. Figure 2 shows the photograph image of experimental setup.

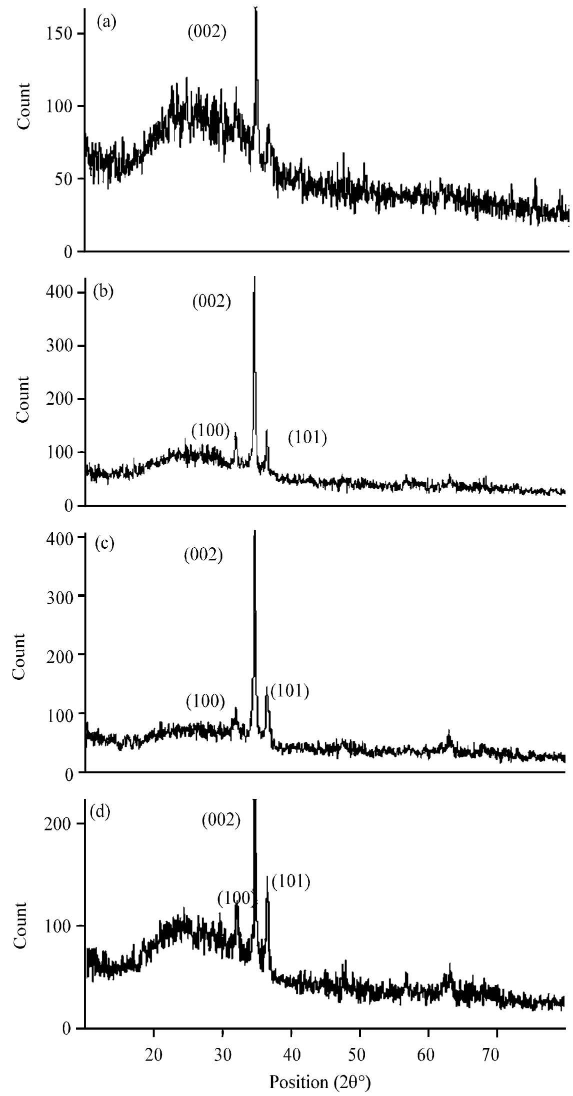

Fig. 3(a-d): XRD pattern of ZnO films prepared by SILAR technique different dipping cycles; (a) 25, (b) 50, (c) 75 and (d) 100

RESULTS AND DISCUSSION

Structural studies: Figure 3 shows the X-ray diffraction pattern of SILAR grown ZnO film deposited at different dipping cycles. All the pattern shows a preferential diffraction peaks at 2θ of 34.6 which corresponds to (002) orientation of hexagonal structure of ZnO (JCPDS card [05-0640]). Peaks such as (100) (101) were also observed and indicating polycrystalline nature of film.

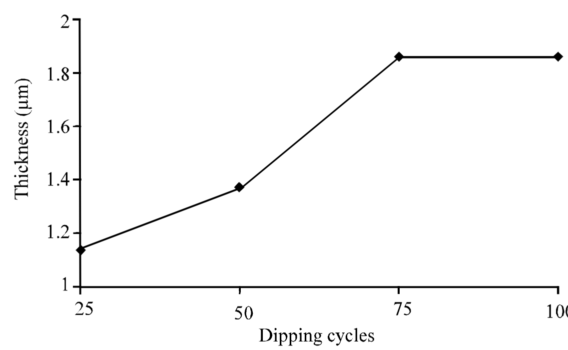

Fig. 4: Variation of film thickness with respect to dipping cycles

Film thickness: Figure 4 shows that the film thickness increases as dipping cycle increased and attains saturation above 75 dipping cycle. Hence, films were deposited up to 100 dipping cycle.

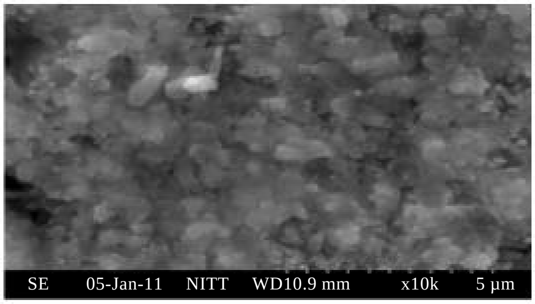

Fig. 5: Scanning Electron micrograph of ZnO thin film

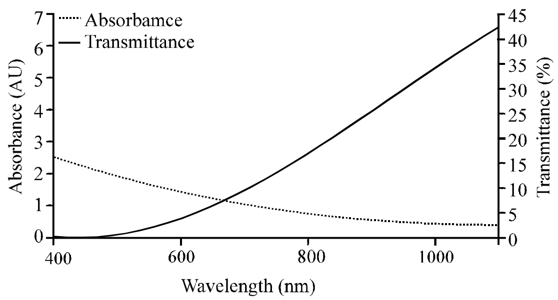

Fig. 6: Optical absorbance and transmittance of ZnO thin film

SEM analysis: Figure 5 shows the Scanning electron micrograph of ZnO film annealed at 250°C. The presence of discrete grain confirms the polycrystalline nature of prepared ZnO film. Also each grain can be indexed to hexagonal structure.

Optical studies: Figure 6 shows the optical absorbance and transmittance of ZnO film. It shows a smooth increase in transmission above 400 nm. The maximum transmission found to be 43% at 1100 nm and less than 25% at 850 nm which is much preferable for sensing application since, the working of the sensor is based on the transmittance changes with respect to the ethanol concentration.

Sensing properties: The amount of light gets attenuated depends on the ZnO cladding length, thickness and the ethanol vapour concentration. Therefore, sensitivity studies have been carried out for four FOS prepared from different dipping cycle (labeled as S_{25}, S_{50}, S_{75} and S_{100}) with four different lengths (10, 20, 30, 40 mm). Ethanol vapour concentration was fixed to 100 ppm. Appreciable response was observed for S_{50}, S_{75}. Figure 7 shows the ethanol response of S_{50} and S_{75} as a function of sensing length. The lesser response of S_{25} and S_{100} may be due to the presence of voids and inhomogeneity. Therefore, S_{50} and S_{75} were tested for dynamic studies to find response and recovery time.

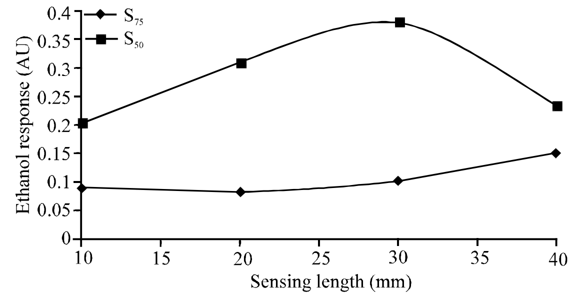

Fig. 7: Sensitivity of S_{50} and S_{75} as a function of sensing length

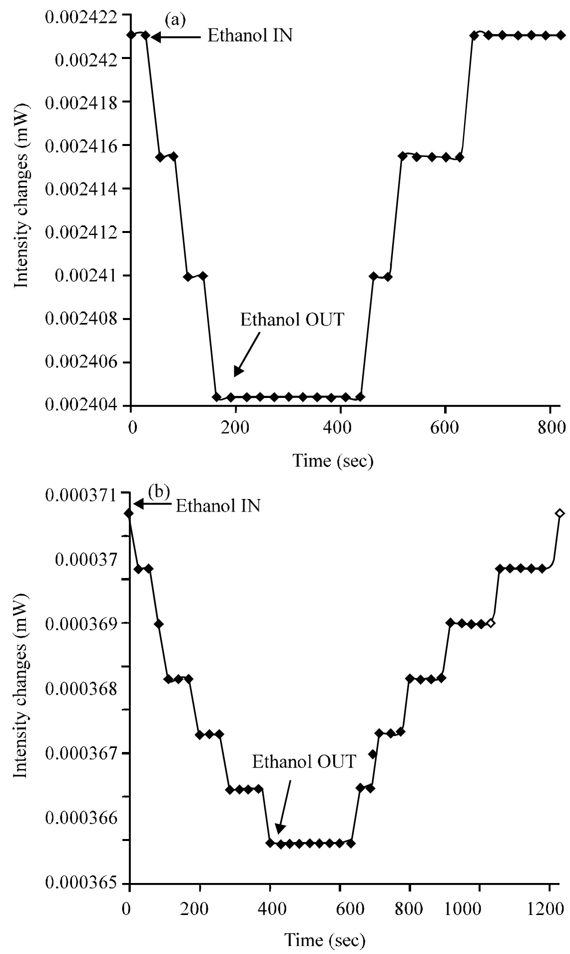

Fig. 8(a-b): Dynamic response of (a) S_{50} and (b) S_{75} FOS towards ethanol vapour

Response and recovery time studies: The response and recovery trend of the fabricated FOS for ethanol vapour was shown in Fig. 8. The stair-case like changes indicates a slow adsorption and desorption of ethanol vapour. The response and recovery time were found to be 200 and 400 sec, respectively. Also it shows S_{50} is better than S_{75} due to lesser ZnO film thickness.

CONCLUSION

Preliminary results of ZnO thin film coated glass rod as optical based ethanol sensor at room temperature were studied. Experiment shows that the transmitted light intensity decreases when ethanol of 100 ppm is injected. For the same ZnO thin film length, it was observed that as thickness increases, sensitivity found to decreases. In conclusion, the reported technique for ethanol sensing at room temperature is encouraging and can be tuned to reduce response and recovery time.

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