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Fabrication of SILAR deposition system and characterization of ZnO thin films.

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ABSTRACT

An automated SILAR system was fabricated using two D.C. motor which was controlled using NI USB 6212 Data Acquisition board. The control inputs for clockwise and anticlockwise movement of the D.C. motor was made through LabVIEW software. ZnO thin film was deposited on glass substrate using the fabricated system. X-ray diffraction pattern of the prepared ZnO film show polycrystalline nature with hexagonal crystal structure. Scanning electron micrograph shows the formation of nanorods. The ammonia sensing properties was studied by chemiresistive method and the results are reported.

Keywords: SILAR; Data Acquisition; LabVIEW; ZnO; Nanorod;

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INTRODUCTION

Thin films of different nanostructures such as nanorods [1], nanowires [2], nanoplatelets [3,4], nanoparticles [5], nanoflowers [6] and nanoclusters [1] were prepared for different applications due to its large surface area. Among various materials, zinc oxide (ZnO) was one of the widely used materials which offer different multifunctional property. ZnO with different morphology were reported to possess vapour sensing [7], optical [8], magnetic [9] and photocatalytic [9] properties.

Solution phase techniques such as chemical bath deposition [10], Successive ionic layer adsorption and reaction (SILAR) technique [11], spray pyrolysis [12], hydrothermal [13], sol gel [14] were widely reported to obtain different morphology of ZnO thin films. SILAR [15] technique has the advantage of offering desired nanostructures inexpensively. In this technique, the substrate is dipped successively in cationic and anionic solution to form the desired film. To obtain uniform deposition, the dipping speed, withdrawal speed and number of dipping cycles has to be controlled and hence an automated SILAR system is required. In the present work, an automated SILAR system was designed using two D.C. motors which were controlled by Data Acquisition (DAQ) through LabVIEW [16] software. Using the fabricated SILAR system ZnO thin film was prepared under optimized conditions and it's structural, morphological and vapour sensing properties were studied.

EXPERIMENTAL

Fabrication of setup

The home-built SILAR system comprises of two D.C. motor and an arm as shown in Fig. 1. The bottom D.C. motor was controlled to keep the substrate in different beaker position. The top D.C. motor placed above the bottom motor was used to dip and withdraw the substrate for desired speed and duration. The arm connected to top D.C. motor was made up of Aluminium hollow bar with clamp to hold the substrate. The two D.C. motors movement was controlled using LabVIEW program through D.C. motor controller IC L293B. The binary value 01 given to the IC L293B will rotate the motor in clockwise direction and the binary value 10 will rotate the motor in anti – clockwise direction. The time to move in clockwise and in anti clockwise direction was given in the form of delay. The DAQ assistant icon shown in Fig. 2 was used to control the movement of the motor whose output was given to the controller IC. The control values for clockwise and anticlockwise movement of the motor were given in the form of array values with defined delay.

Preparation of ZnO thin film

The glass substrate was chemically cleaned and kept in the sample holder of the home-built SILAR system. To prepare ZnO thin film, a 50 mL of 0.1 M zinc sulphate solution with pH (~ 12.5) adjusted using sodium hydroxide was prepared as cationic solution. The solution was kept at room temperature (32 °C). Deionized water heated to ~ 96 °C was kept as anionic solution.

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Through LabVIEW control, the substrate was successively dipped in cationic solution for 5 s and in anionic solution for 5 s with intermediate steps of rinsing in deionized water. Dipping in all the four beakers together constitute one deposition cycle. Deposition was made for 150 cycles and then utilized for characterization studies.

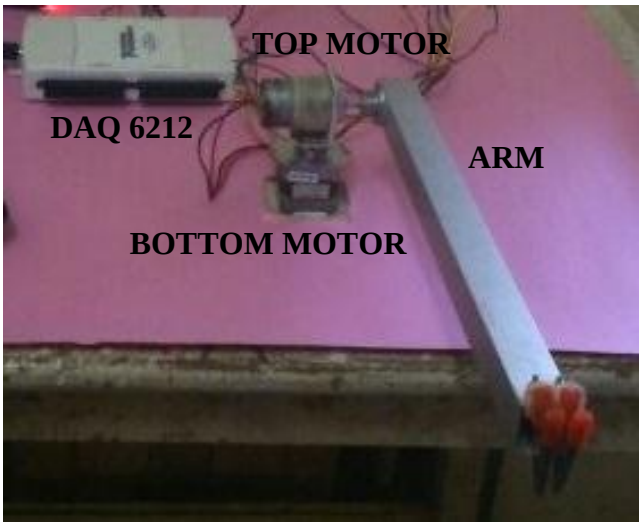

Figure 1: Photograph of the motor arrangement in SILAR deposition system

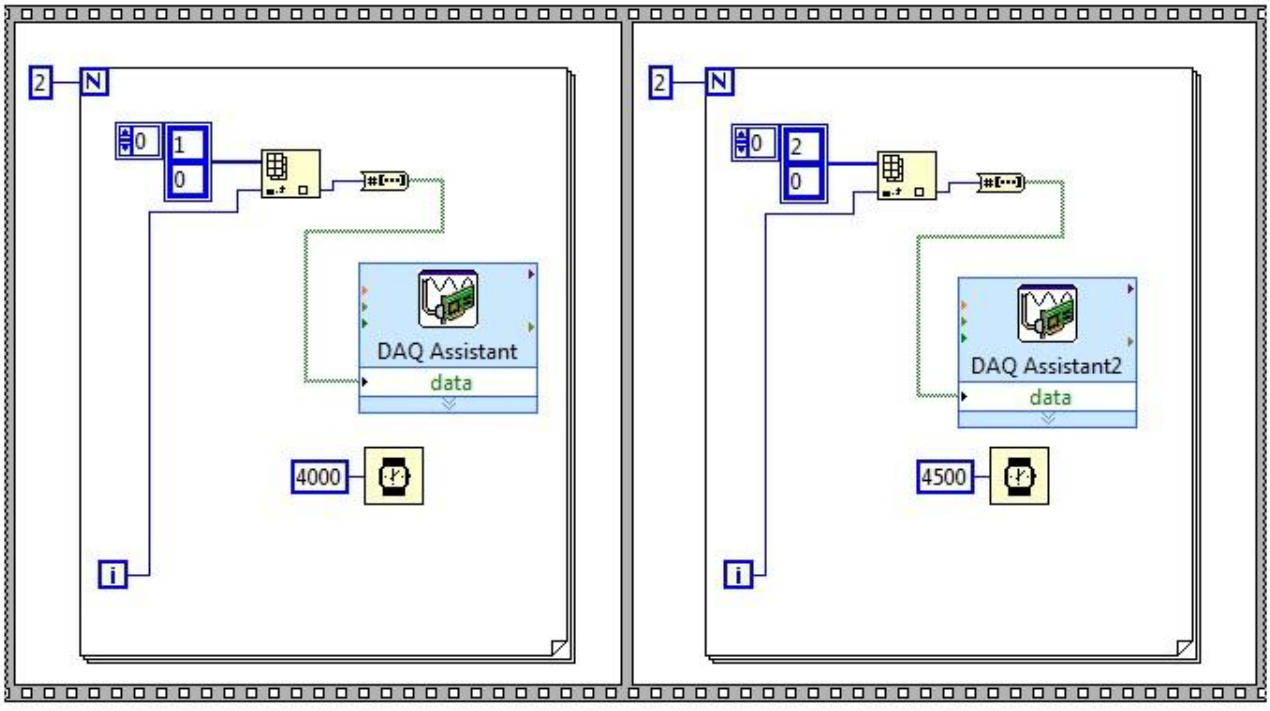

Figure 2: Control panel of LabVIEW

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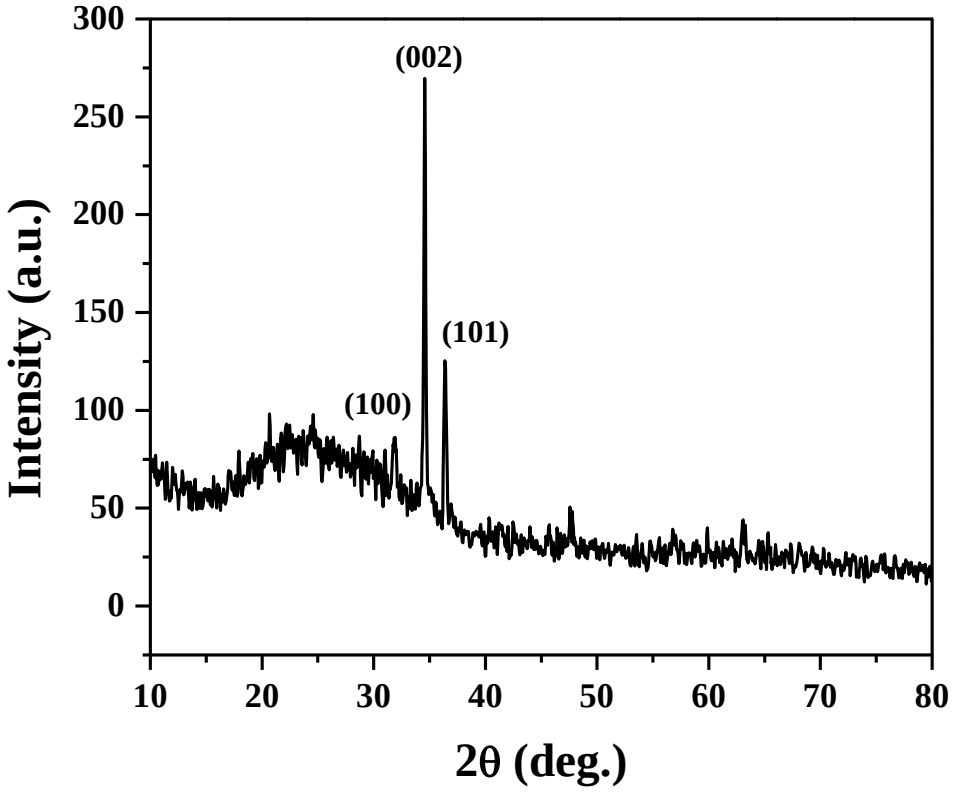

Figure 3: XRD pattern of ZnO thin film

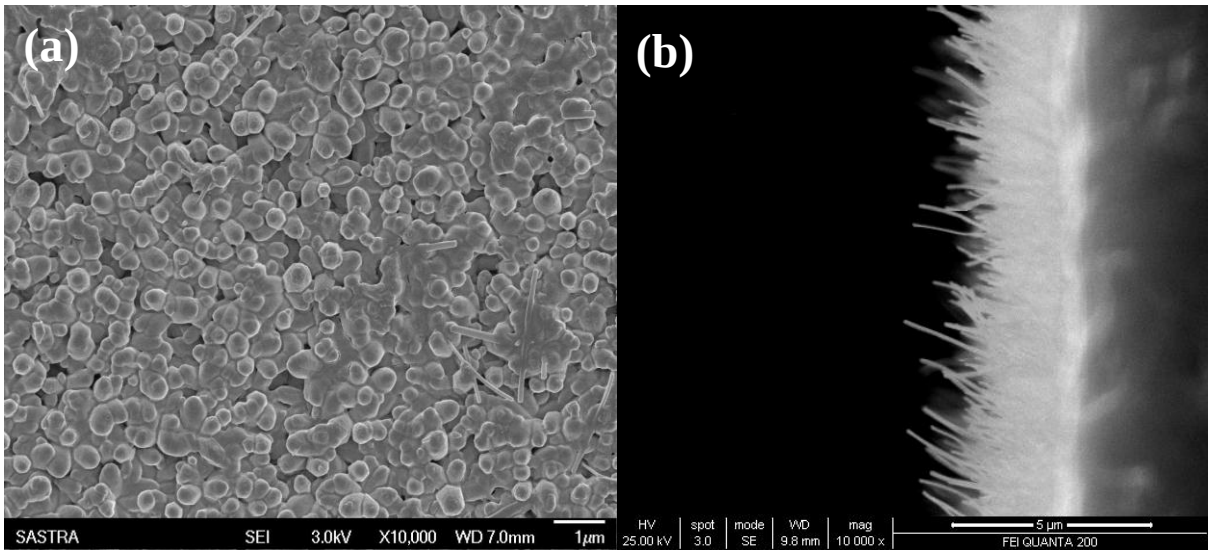

Figure 4: (a) FE-SEM and (b) cross sectional SEM of ZnO thin film

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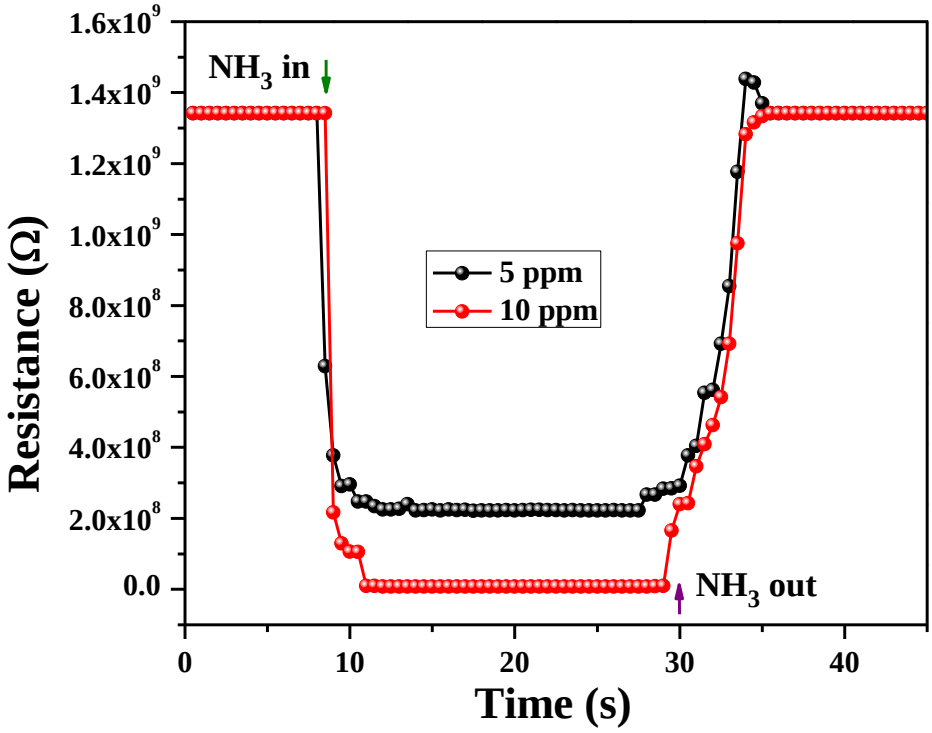

Figure 5: Transient plot of ZnO thin film for 5 and 10 ppm concentration of ammonia

Characterization

As-deposited ZnO thin film was structurally analyzed using X-ray diffractometer (D8 Focus from Bruker, Germany) with 1.540 Å CuKα_1 radiations. The morphological study was carried out using field-emission scanning electron microscopy (JEOL-6701F from Japan). The cross sectional micrograph was taken using scanning electron microscopy (FEI Quanta 300, Icon analytical). For vapour sensing studies, electrical contacts were made on the film using Al foil and pressure contact electrode. The ammonia sensing characteristics was studied using home-built sensing system [17] at ambient temperature. To measure the electrical current of the film, a pico-ammeter (Scientific equipment, India) was connected in series and was interfaced to computer through DAQ (NI USB-6212). The readings were recorded through LabVIEW software.

RESULTS AND DISCUSSION

Structural studies

Fig. 3 show the X-ray diffraction pattern of ZnO coated glass substrate obtained from 150 deposition cycles. With respect to standard JCPDS card No. 34 – 1451,the diffraction peaks at 31.93 °, 34.61 ° and 36.39 ° were indexed to (1 0 0), (0 0 2) and (1 0 1) planes of hexagonal crystal structure. The observed diffraction peaks also indicates the polycrystalline nature of the

deposited film. The broad bump found at 20° was due to the substrate effect. The texture coefficient was estimated using the relation (1)

$$TC = \frac{I_{(hkl)} / I_{o(hkl)}}{(1/N)\left[\sum_{N} I_{(hkl)} / I_{o(hkl)}\right]} \quad \text{---------- (1)}$$

Where $I_{(hkl)}$ is the observed intensity, $I_{o(hkl)}$ is the standard JCPDS intensity and N denotes the number of peaks. The maximum texture coefficient value was found to be 2.41 for (0 0 2) plane and indicates the preferred plane of orientation. The average crystallite size was found to be 40 nm which was calculated using Scherer formula.

Morphological studies

The field-emission scanning electron micrograph and the cross sectional scanning electron micrographs of the as-deposited ZnO thin film were shown in Fig. 4. The FE-SEM shows the size of ZnO grain in the order of nanometer. Also from the cross sectional SEM, ZnO nanorods were clearly seen and are aligned perpendicular to the substrate with uniform diameter. The diameter of the rod was found to be approximately 70 nm and the length of the rods ranges from 1 to 5 µm.

Sensing studies

ZnO coated substrate was kept inside the test chamber at ambient temperature and the initial baseline resistance (R_o) was measured and was found to be 1.35 GΩ. The high baseline resistance was due to adsorption of oxygen ion species on the ZnO surface. When ammonia was injected inside the chamber, it will interact with the adsorbed oxygen species and desorbs the oxygen species by donating the electron back to the conduction band of ZnO. This results in the decrease of electrical resistance of the ZnO as shown in Fig. 5. The saturated resistance after injecting ammonia was noted as R_g. The ammonia was exhausted using vacuum pump and air is admitted inside the chamber. The sensing study was performed for 5 ppm and 10 ppm concentration of ammonia and their response and recovery time was estimated. The time taken to reach 90 % of the saturation resistance from the initial baseline resistance is called as response time and the time taken to reach 90 % from the saturation resistance is called recovery time. It was found that the response time was 3 s and 2 s for 5 ppm and 10 ppm respectively and the recovery time was found to be 7 s and 9 s respectively. The observed fast response and recovery may be due to the nanorod formation of ZnO thin film.

CONCLUSION

A simple and cost effective method was implemented to design an automated SILAR system using two D.C. motors controlled via LabVIEW software. ZnO thin film was formed using

the fabricated SILAR system and characterized for its structure and morphology. The formation of ZnO in hexagonal crystal structure was confirmed from XRD studies with a crystallite size of 40 nm. The formation of nanorods was observed from FE-SEM. The sensing studies showed good response and recovery behaviour towards ammonia vapour at ambient temperature.

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