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Ag-ZnO Nanowires: An efficient NO_2 sensor

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ABSTRACT

The ability to precisely control the growth, morphology and dimensions has lead to the widespread use of nanowires for sensing application. In the present work, we demonstrate that the modification of ZnO nanowires with an Ag thin layer resulted in a superior NO_2 sensing characteristic. Towards 5 ppm of NO_2 the sensor films with 10 nm layer of Ag exhibited a sensor response (Rg/Ra) of 4.7 with response and recovery time of 60 and 180 s, respectively at 200°C. Our result clearly elucidates that ZnO nanowires with Ag can be looked upon as a potential NO_2 sensor.

Keywords: ZnO, nanowires, hydrothermal, Ag, NO_2, chemiresistive sensor

INTRODUCTION

ZnO nanowires based chemiresistive sensors are gaining importance owing to its ease of synthesis using different precursors, tunable morphology and aspect ratio, tailor made surface reactivity and compatibility with the CMOS processes. Similar to other metal oxide semiconductors (MOS), in its pure form, ZnO also suffers from the drawback of poor selectivity. However, use of sensitizers like Au, Cu, Pd and Pt has been demonstrated an effective way of tailoring the response towards specific gases. In the present work we demonstrate that the modification of nanowires surface with a thin layer of Ag resulted in an improved response towards NO_2. NO_2, is one of the toxic gases whose detection has become of prime importance. Its short term and long term exposure limits are set to 3 5 and 3 ppm, respectively. It is usually released in the atmosphere from vehicular emissions, industrial effluents.

Similar to SO_2, it also recombines with moisture in air forming HNO_3 (nitric acid) causing acid rains. Inhalation of NO_2 causes inflammation in lungs, severe irritation and burning of eyes and difficulty in breathing. Accordingly, it becomes desirable to detect it well below its safety limits.

METHODOLOGY

ZnO nanowires were synthesized using hydrothermal method as reported elsewhere [1-2]. In particular, the growth involves two processes. First the ZnO nanoparticles are synthesized using chemical route and then using the nanoparticles as the seed layer the hydrothermal nanowire growth is carries out. For this, the substrates (glass) containing the seed layer is kept upside down over the aqueous equimolar (25 mM) solution of $Zn(NO_3)_2{:}6H_2O$ and hexamethylene tetraamine (HMTA) in an oven at 95°C for 7 h. After growth the substrate containing nanowires was rinsed (thrice) with DI water and used for further study. Nanowires surface were modified with a thin layer of Ag in the range from 5-50 nm. The samples were then subjected to post deposition annealing at 400°C for 1 h in O_2 environment. Gold layer of ~120 nm thickness was deposited as electrodes.

Gas sensing measurements were investigated in an indigenously developed table top static gas sensing unit as shown in figure 1. It comprises of a stainless steel (SS) test chamber having 7 units to mount 7 sensing elements. Each unit has a pair of heater and reader. The operating temperature of each sensing element can be individually tuned and maintained with the help of potentiometers provided on top of the controller. The PC has a LabVIEW based user interface which records and displays the resistance values as a function of time. In the test chamber, the required concentration of the test gas was realized by inserting the known volume of gas through a syringe. For this commercial grade cylinder having 1000 ppm (± 1%) of NO_2 in N_2 (99.9%) were used. The sensors were let to recover by exposing them to surrounding air. Sensor response was calculated from the response curve using the relation

$$SR=R_g/R_a \quad (1)$$

NO_2 is an oxidizing gas,

Where, R_g is the saturated value of sensor resistance in the test gas and Ra is the resistance in air. Response and recovery times were defined as the time taken by the sensing element to reach 90 and 10% of its saturation and original value upon exposure to test gas and fresh air, respectively.

RESULTS

Hydrothermal growth has been demonstrated to result in ZnO nanowires having diameters in the range from 50 - 200 nm and having length in the range from 1-5 μm [3-4]. Moreover, they have also been shown to exhibit a characteristic hexagonal or wurtzite structure. The associated high surface area to volume ratio is expected to impart high sensitivity in the sensing material. Modification of sensor surface with a thin layer of Ag (10 nm) imparts a high selectivity with improved sensor response towards NO_2.

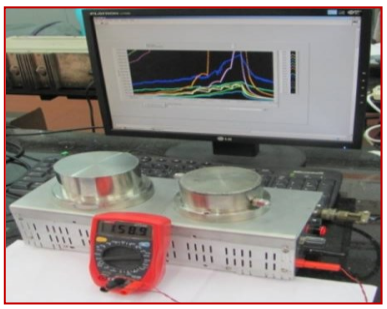

Figure 1. The indigenously developed table top static gas sensing unit.

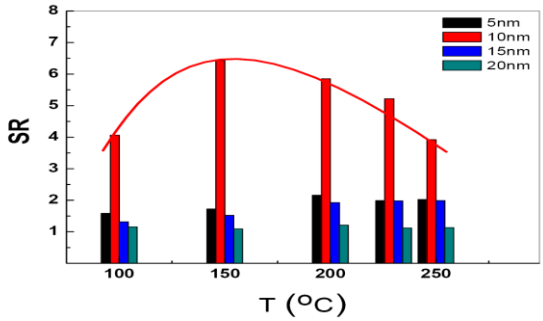

Figure 2. Sensor response as a function of operating temperature and Ag thicknesses.

Figure 2 shows the effect of operating temperature on the sensor response of samples containing different layer thickness of Ag. It is clearly evident that sensor film with 10 nm layer of Ag shows better sensor response at 150°C in comparison to that of sensor films with other Ag thicknesses.

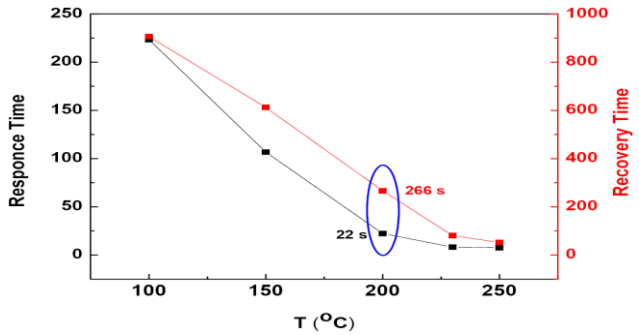

Figure 3. Response and recovery times as a function of operating temperature recorded for sensor film with 10 nm of Ag upon exposure to 5 ppm of NO_2 at 200°C.

Figure 3 shows the effect of operating temperature on response kinetics of the sensor with 10 nm Ag, when subjected to 5 ppm of NO_2 gas. With increase in temperature both response and recovery times were observed to decrease.

However, the sensor response increases with temperature upto 150 and then decreases with further rise in temperature. However, at 200°C there is slight decrease in sensor response but an appreciable improvement in the response kinetics. For example, towards 5 ppm of NO_2, the sensor film exhibited a response and recovery times of 25 and 270 s, respectively. Accordingly, 200°C was chosen as the best sensor temperature for NO_2 detection.

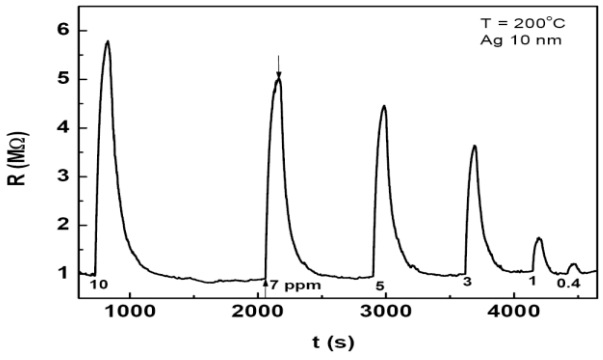

Figure 4 shows the response curve recorded for the sensor film when it is subjected to decreasing concentrations of NO_2.

Figure 4 shows the response curve recorded for the sensor film when it is subjected to decreasing concentrations of NO_2. From figure 5, it is clear that the sensor response increases with concentration upto 10 ppm with lowest detection limit of 0.4 ppm. For all gas concentrations the sensor films exhibited a faster and complete recovery as also shown in figure 2.

Annealing of Ag in oxygen atmosphere leads to the formation of Ag_2O. This leads to the adsorption more oxygen species on the sensor surface. Now the type of adsorbed oxygen species depends on the operating temperature. At 200°C, oxygen gets adsorbed as O^{2-}. Now, NO_2 being an oxidizing gas abstracts electron from the sensor surface thereby causing an increase in the resistance of the sensor films. The exact sensing mechanism however, needs more experimentation like x-ray photoelectron spectroscopy and work function measurements before and after exposure to the test gas and are underway.

CONCLUSION

In conclusion, modification of ZnO nanowires with thin layer of Ag has been demonstrated to result in NO2 sensor with enhanced response characteristics. In particular, the sensor film containing 10 nm layer of Ag demonstrate a sensor response of 4.7 towards 5 ppm of NO_2, with response and recovery time of 60 and 180 s, respectively. The investigation of exact sensing mechanism and long-term stability measurements are underway.

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