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INVESTIGATIONS ON UNDOPED ZINC OXIDE NANOMATERIALS AS HUMIDITY AND GAS SENSOR

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ABSTRACT

In this paper we have reported investigations on humidity sensing and liquefied petroleum gas sensing studies of undoped ZnO (or pure ZnO) pellets. Samples of undoped ZnO were analyzed for humidity sensing studies and the material showed good humidity sensing results. It had also been noticed that the pellet samples with different thicknesses showed different sensitivity values for humidity sensing studies. Pellet samples of undoped ZnO (or pure ZnO) nanomaterial showed the best results with sensitivity of 296 (%) in the 500-5000 ppm range for liquefied petroleum gas sensing. The sensing elements developed from undoped ZnO nanomaterials showed low hysteresis, low aging effects and better sensitivity values

KEYWORDS: Humidity; Gas; Pellets; Sensing Elements, Sensitivity

I. INTRODUCTION

Humidity and gas sensors are widely used in the entire world for specific purposes. These sensors are very important as these are used in various industrial areas as well as in scientific laboratories. Moisture is the water vapor content present in the air. It can make hot temperatures even more unbearable. Humidity is integral to both biological life and automated industrial processes. There is need to develop humidity sensors having applications based on specific needs and desired range. Research laboratories are making best efforts to find the suitable materials with better parameters like good sensitivity over large range of relative humidity, low hysteresis, low response and recovery time, and properties that are stable. For the development of nanodevices (or microdevices) with superb performance an in-depth knowledge of physical, chemical and structural properties of the metal oxide nanomaterials is required. ZnO is a very versatile compound semiconductor with a band gap of about 3.3 eV. Numerous metal oxides (e.g., zinc oxide) have been analyzed for potential applications in the field of gas and humidity sensing [1-11].

Gas sensing materials like SnO_2 have been investigated by different researchers in the entire world [12-13]. For the development of highly sensitive gas sensors the research focus of scientists and engineers is on thick and thin film based sensors [14-19]. The need for monitoring, measuring and controlling the relative humidity (RH) precisely has led to the development of variety of humidity sensors [20–24]. In ceramic sensors, sensitivity and response time are generally governed by the surface morphology; pore volume, shape and size distributions. Ceramic surface features show strong affinity for chemical and physical adsorption of water vapor molecules. Thus, porous metal oxides offer opportunity to develop humidity sensors with added advantage of their chemical and physical stability. Ceramic sensors are generally impedance or resistive type and are in vogue for their quality and low cost. Water adsorption mechanism leads to change in resistance of the oxide surface subsequent to exposure to humidity. Film or pellet sensors with nano-size grains and nano-porous structures offer high surface to volume ratio leading to efficient adsorption of water molecules and thus giving high sensitivity to these sensors. Yawale *et al.* fabricated SnO_2/ZnO with TiO_2/Al_2O_3 films and measured DC-electrical resistance of the films. SnO_2-5Al_2O_3 and ZnO-5Al_2O_3 proved to be better humidity sensing materials. Rutile and hexagonal structures of SnO_2, ZnO and Al_2O_3 and their nano-meter grain size formed nano-sized pores that adsorbed water [25]. Li *et al.* investigated humidity sensors prepared using synthesized inorganic/organic nanocomposites of sodium polystyrenesulfonate and ZnO. Sensor performance based on composite film changed by four orders of magnitude over the humidity range 11–97% RH [26]. The humidity sensing properties of mesoporous ZnO-SiO_2 composites synthesized by sol-gel methods with different Si/Zn

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molar ratios were investigated by Yuan et. al [27]. Introduction of ZnO improved humidity sensitivity of composite in the range of 11% to 95% RH and the sample with a Si/Zn ratio of 1:1 showed promising results. Sensor resistance changed 4 times in 11%-95% RH range. Sensor showed response and recovery time of about 50 s and 100 s, respectively. Spin-coated nanorod thin film humidity sensors of aluminium doped zinc oxide were prepared by Sin *et al*. With increase in doping concentration, length of nanorods increased. Sensor that contained 0.6 at% aluminium doped in ZnO exhibited highest sensitivity in 40%–90% RH humidity range [28]. Jeseentharani *et al. tested* composites of CuO-ZnO, CuO-NiO and NiO-ZnO for humidity sensing in the range of 5%–98% RH. CuO-NiO compound showed the maximum sensitivity. Response and recovery times of CuO-NiO composites were 80 s and 650 s, respectively [29]. ZnO-In_2O_3 thin film humidity sensors were fabricated by radio-frequency layer by layer sputtering of ZnO and In_2O_3 precursors. Sample fabricated by applying ZnO two times and In_2O_3 one time showed the best results as total resistance changed by 4 orders in 11%-95% RH range [30]. Yongsheng *et al.* prepared ZnO nanorod and nanobelt films on the Si substrates with comb type Pt electrodes by the vapor-phase transport method. They found that at room temperature, resistance changed by more than four and two orders of magnitude when ZnO nanobelt and nanorod devices were exposed respectively to a moisture pulse of 97% relative humidity [31]. Jayanti *et al.* doped ZnO nanocrystals with impurities of Li, Na, Cu, Pr, and Mg under similar conditions by solid-state reaction method. Their study showed that undoped ZnO, Li and Na doped ZnO showed well-developed nanorods but Cu doped ZnO nanorods were not well-formed, rather they tended to form clusters [32].

II. EXPERIMENTAL PROCESS

Solid-state reaction route was adopted to fabricate pellets from nanomaterial samples of undoped ZnO (or pure ZnO). 10% by weight of glass powder (binder) was added to undoped ZnO (or pure ZnO) nanomaterial. Mixtures were grinded separately till uniformity was achieved. Powders were pressed at room temperature into disc shaped pellets by applying pressure of 260 M Pa by a hydraulic pressure machine. Pellets with thickness 3 mm and diameter 4 mm were formed. Pellet samples formed from undoped ZnO with thickness 3 mm and diameter 4 mm were labeled as Z-0. Pellet samples with thickness 2 mm and diameter 4 mm were also analyzed for comparison purpose. Pellet samples formed from undoped ZnO (or pure ZnO) with thickness 2 mm and diameter 4 mm were labeled as VZ-0.0 [33]. Pellets were annealed in air at different annealing temperatures from 300°C-500°C for 3 hours. The developed pellets were analyzed for humidity sensing and liquefied petroleum gas (LPG) sensing studies.

III. SEM AND XRD STUDIES

Study of surface morphology of the samples was carried out using scanning electron microscope [LEO-430, Cambridge, England]. Figure 1 shows SEM micrograph of undoped ZnO (or pure ZnO) nanomaterial samples. From the micrographs it became clear that ZnO is scattered throughout the whole substrate forming a network of voids and pores. SEM micrographs show porous structure and small crystallites without inside pores but many inter grain pores. These pores are expected to provide sites for humidity and gas adsorption. As a matter of fact, higher porosity increases surface to volume ratio of the materials and therefore, helps in getting good sensitivity. Consequently, SEM image shows that the molecules are agglomerated and equally distributed.

The average grain size for Z-0 samples measured from SEM micrographs was found to be 380 nm. The average grain size for VZ-0.0 samples measured from SEM micrographs was found to be 620 nm [33].

The crystallinity, structural phases and the gross crystal structure of the as synthesized nanomaterials were investigated by powder X-Ray diffractometer XPERT PRO-Analytical XRD system (Netherlands). Wavelength of CuKα source used is 1.54060 Å. X-ray patterns for undoped ZnO (or pure ZnO) samples have been shown in Figure 2. The diffraction peaks were well defined. The XRD shapes of different composition looked similar. The average crystallite size of the sample was calculated using Scherrer's formula (given below).

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Here, D = crystallite size, K = fixed number of 0.9, λ = X-ray wavelength, θ = Bragg angle, B = full width at half maximum of the peak. In case of pure ZnO (undoped ZnO) sample VZ-0.0 there is distribution in size of crystallites [33]. Minimum crystallite size is 447 nm and maximum 534 nm for VZ-0.0 samples [33]. In the present research work, the crystallite size of Z-0 sensing elements ranges from 208 nm to 416 nm.

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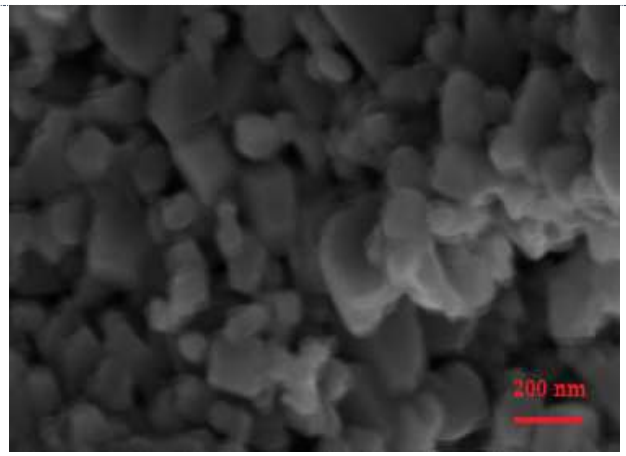

Fig.1. SEM of sample Z-0 annealed at 500°C

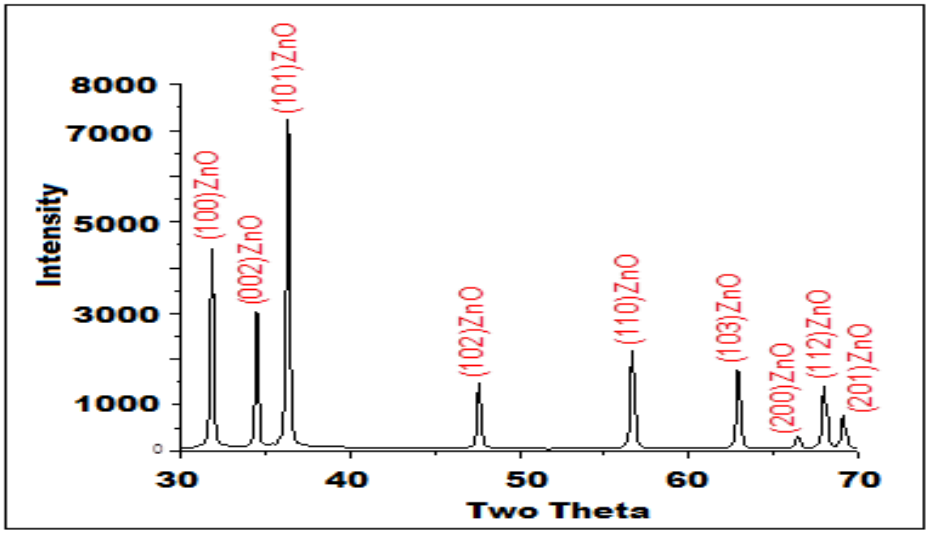

Fig.2. XRD of sample Z-0 annealed at 500°C

IV. EXPERIMENTAL

Humidity and gas control chambers were used for studying humidity and gas sensing applications. The change in values of resistance was recorded corresponding to the change in humidity level. Moreover, the change in values of resistance was recorded corresponding to the change in gas concentration. The pellets (after annealing) were inserted in a humidity control chamber. Copper electrodes were used for measuring resistance normal to the pellet's cross-section. Surface contact area of all sensing elements with electrodes was 113.11 mm^2 and the cylindrical surface area that was exposed to the humidity in the chamber was also 113.11 mm^2. Calibration of the chamber was done by a standard hygrometer (Huger, Germany, ±1% RH) and a thermometer (±1°C). A multi-meter (±0.001 MΩ, model: VC-9808) recorded variation in resistance with change in %RH. Aging and reproducibility studies were done by repeating the process after six months. To check stability, samples were exposed to chamber humidity at fixed values of % RH, and resistance was recorded as a function of time. The stability was within ±3%. Standard solution of potassium sulphate has been used as a humidifier and potassium hydroxide as a de-humidifier. For gas sensing studies sensitivity values of the sensing elements were recorded with the variation in the LPG gas concentration. Sensitivity values of the sensing elements were also recorded with the variation in the humidity level. All the experimental measurements were performed at room temperature.

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V. RESULTS AND DISCUSSION

1. Sensitivity (Humidity Sensing)

Sensitivity of humidity sensor is defined as the change in resistance (ΔR) of sensing element per unit change in relative humidity (Δ%RH). Sensitivity of the sensing elements is given below:

$$\text{Sensitivity} = (\Delta R)/(\Delta\%RH)$$

Variation in resistance with change in %RH for undoped ZnO samples for annealing temperature 500˚C were analyzed. Figure 3 shows variation in resistance with change in the relative humidity (%RH) for increasing cycle, decreasing cycle and for increasing cycle after six months for Z-0 samples. It was found that a decrease in resistance is noticed with increase in the % RH for all sensing elements. For increasing cycle of relative humidity the sensing element VZ-0.0 developed from undoped ZnO (or pure ZnO) nanomaterial showed sensitivity to be 2.65 MΩ/%RH [33]. For decreasing cycle of relative humidity the sensing element VZ-0.0 developed from undoped ZnO nanomaterial (or pure ZnO) showed sensitivity to be 2.95 MΩ/%RH [33]. After six months the experiments were performed again to determine sensitivity for VZ-0.0 sensing elements and it was found to be 2.51 MΩ/%RH for increasing cycle [33]. From the humidity sensing investigations of the Z-0 pellets with thickness 3 mm it was found that the sensitivity was found to be 4.80 MΩ/%RH for increasing cycle of relative humidity. Similarly, it was noticed that for decreasing cycle of relative humidity the Z-0 sensing element showed sensitivity of 4.18 MΩ/%RH. After six months the experiments were performed again and the sensitivity was found to be 3.82 MΩ/%RH for Z-0 sensing elements. So, pellets with different thicknesses show different sensitivity values.

It has been observed that there is a sharp fall for initial range of 35%-55% RH and then it is gradual for 55%-95% RH range. In case of undoped ZnO sample there is distribution in size of crystallites. Undoped ZnO sample has relatively low adsorption capacity for moisture and hence low sensitivity due to uniform distribution of grains, less formation of voids; lower inter-connected voids or capillaries which are important conditions for adsorption of water molecules in the sample. It is possible that multiplicative effect of two or more parameters of nanomaterial may cause higher sensitivity. The mechanism by which a metal atom interacts with the surface of a metal oxide is varied and complex, it is not easy to establish the exact single parameter affecting the sensitivity. Higher porosity increases surface to volume ratio and enhances diffusion rate of water into or out-of the porous structure. This causes sensitivity to increase. At high relative humidity, liquid water condenses in capillary nano-pores, forming a liquid like layer. Resistance may also decrease due to change of grain boundary barrier height in ceramics. Adsorption of water molecules at metal oxide surface penetrated inside the sample decreases height of potential barrier at grain surfaces and also at surface of necks between metal oxide grains. Thus, size of depletion regions in the vicinity of necks in electric field direction gets lowered and conductance increases. The initial chemisorptions on the surface of sensing elements cause hysteresis. It can be minimized through the process of thermal desorption only. To estimate hysteresis, chamber humidity was scaled up from 35% RH to 95% RH and then cycled down to 35% RH. Samples show tolerable hysteresis values. Hysteresis recorded for samples Z-0 samples were found to be very low as compared to VZ-0.0 samples.

2. Sensing principle (for humidity sensing)

Either ionic or electronic type mechanism is responsible for the conduction mechanism in the sensors based on the ceramic materials. Electrons are donated by the water molecules in the electronic type mechanism. These water molecules get chemisorbed and hence in this way the electronic conductivity gets increased or decreased. This conductivity actually depends on the fact whether the material is p-type or n-type semiconductor. If the mechanism is ionic type then the impedance of the sensor decreases with increase in the value of RH (relative humidity) due to the formation of the physisorbed layer and condensation of the water molecules in the capillary on the material‘s surface. As soon as the nanomaterials developed from the ZnO come in contact with humid air, the chemisorption process starts and the water molecules chemisorb on the available sites of the material surface. Firstly, the chemisorbed layer is formed due to the dissociative chemisorption process and thereafter the physisorbed layer is formed. The electrons are accumulated at the surface of ZnO and consequently, the resistance of the sensing element decreases with increase in relative humidity. V_2O_5 nanoparticles doped in ZnO enhance the adsorption and desorption rates of the water molecules and hence helps in increasing the sensitivity of the material. In chemisorption, molecules are adsorbed on the surface by valence bonds and only form monolayer adsorption. Chemisorption is a kind of adsorption which involves a chemical

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reaction between the surface and adsorbate. New chemical bonds are generated at the adsorbant surface. The strong interaction between the adsorbate and the substrate surface creates new type of electronic bonds. The layer formed due to chemisorption process is known as chemisorbed layer. Physisorption is also known as physical adsorption. It is a process in which the electronic structure of the atom or molecule is barely perturbed upon adsorption. For physisorption water molecules can form multilayer adsorption. The layers formed due to physisorption process are known as physisorbed layers. Physisorbed layers are easily desorbed but chemisorbed layer can be thermally desorbed only. In case of physisorption typical binding energy is about 10-100 meV while that in case of chemisorption typical binding energy is in the range of 1-10 eV. So, the energy required for the removal of physisorbed layer is very less as compared to the energy required for the removal of chemisorbed layer.

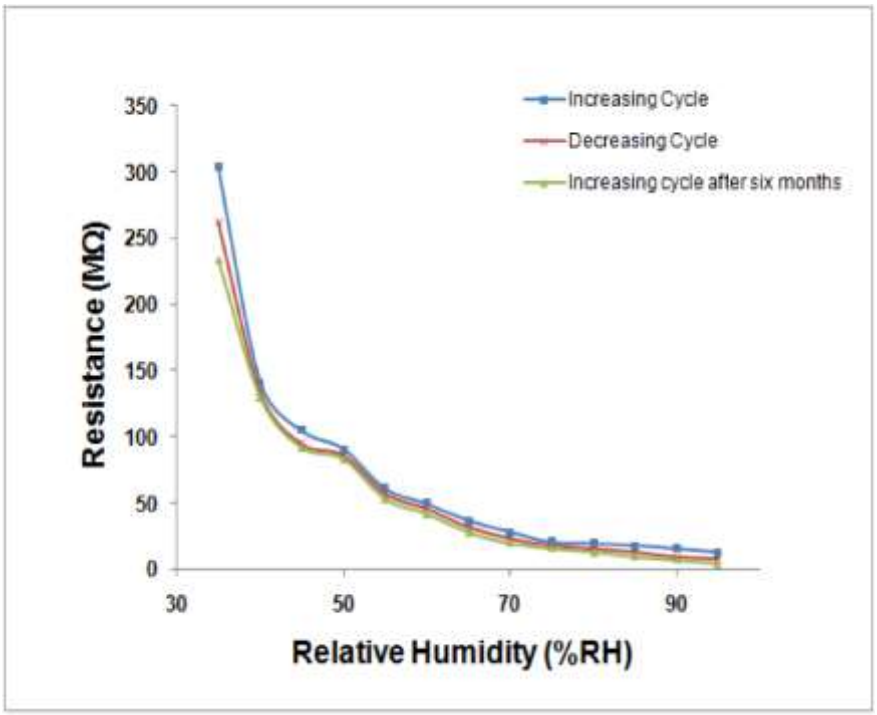

Fig.3. Variation of resistance with change in %RH for the sensing elements Z-0 in case of increasing cycle, decreasing cycle and increasing cycle after six months

3. Sensitivity (Gas Sensing)

The sensitivity of a semiconductor gas sensor at a specific temperature is given by,

$$\text{Sensitivity (\%)} = \left(\frac{R_{air}}{R_{gas}}\right) \times 100$$

Here, R_{air} = Resistance of the sensing element in air; R_{gas} = Resistance of the sensing element in gas.

Figure 4 shows variation of sensitivity with gas concentration for the sensing elements (pellets) annealed at different temperatures. Undoped ZnO (or pure ZnO) sensing elements annealed at 500ºC showed the best results with sensitivity of 296 (%) in the 500-5000 ppm range. When an n-type semiconductor interacts with a reducing gas then the conductivity increases and hence resistance decreases. The texture and grain size distribution are highly affected by increase in annealing temperature. More sites are created during the annealing process and are available for adsorption and desorption of gas molecules.

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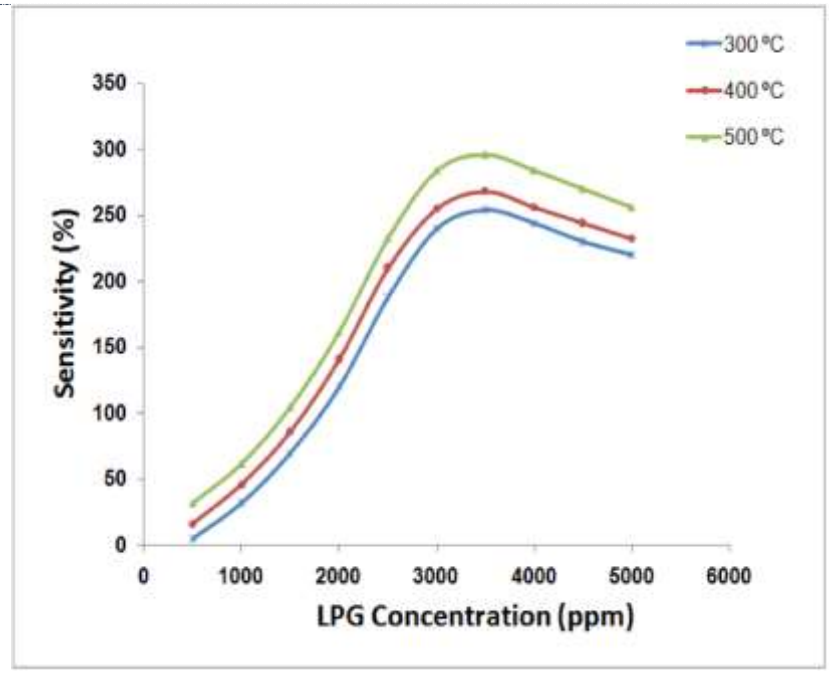

Fig.4. Variation of sensitivity with change in gas concentration for the sensing elements Z-0 annealed at different temperatures

4. Hysteresis And Aging Effect

Figure 5 shows hysteresis behavior of sample Z-0 annealed at 500°C for gas sensing studies. Hysteresis is also an important aspect as far as sensor is concerned. In the present research work all sensing elements have acceptable hysteresis values. Hysteresis was found to decrease with increase in the annealing temperature for all the VZ-0.0 sensing elements for humidity sensing studies [33]. Similarly, it was observed that hysteresis decreased with increase in the annealing temperature for all the Z-0 sensing elements for humidity sensing studies. Moreover, Z-0 samples had low hysteresis as compared to VZ-0.0 samples in the case of humidity sensing. To estimate hysteresis, chamber humidity was scaled up from 35% RH to 95% RH and then cycled down to 35% RH. VZ-0.0 samples show tolerable hysteresis values. Minimum hysteresis recorded for samples VZ-0.0 was ±5.26 [33]. Minimum hysteresis recorded for samples Z-0 was found to be ±3.64. Hence, hysteresis has decreased for sample Z-0 as compared to sample VZ-0.0 in case of humidity sensing studies.

Figure 6 shows aging effect behavior of sample Z-0 annealed at 500°C for gas sensing studies. For analysing the effect of aging, sensing properties of these elements were examined again in the gas control chamber after six months and variation of resistance with change in gas concentration was recorded. Variation of resistance of all the sensing elements with change in gas concentration after six months was analyzed. For all the sensing elements annealed at 500°C, values were generally repeatable within ±8.00% in the 500 ppm to 5000 ppm range after six months. In the case of humidity sensing sensitivity values were reproducible within ±5.28% for samples VZ-0.0 [33]. Sensitivity values were reproducible within ±3.72% for samples Z-0 for humidity sensing studies. Hence, good reproducibility has been achieved for sample Z-0 as compared to sample VZ-0.0. So, in case of humidity sensing studies aging effect over six months was within ±6% for VZ-0.0 sensing elements (i.e. for undoped ZnO); hysteresis within ±6% for VZ-0.0 sensing elements [33]. For Z-0 sensing elements aging effect was within ±4%; hysteresis within ±4% for Z-0 sensing elements. Hence, Z-0 sensing elements showed low hysteresis and low aging effects as compared to VZ-0.0 sensing elements for humidity sensing studies.

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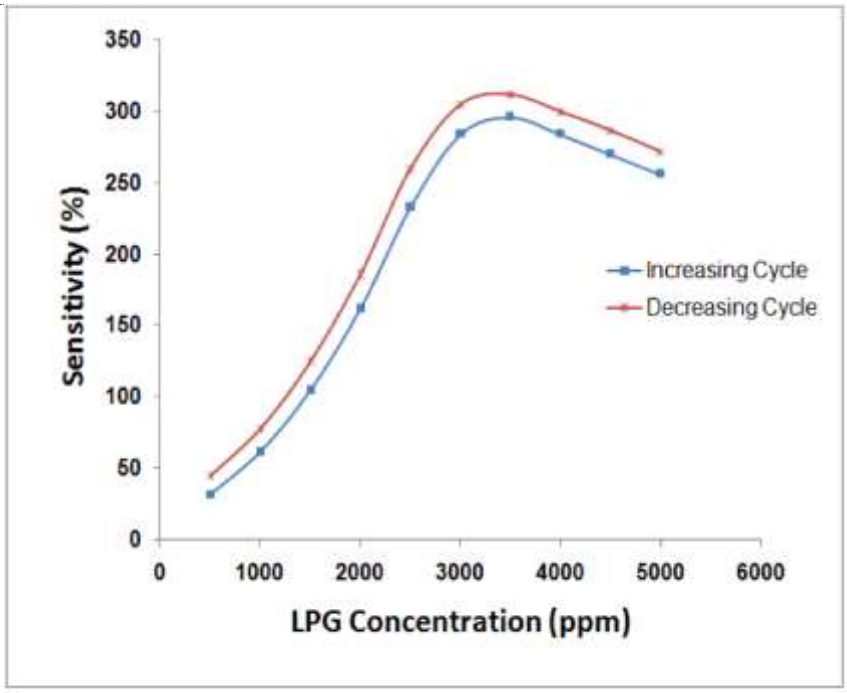

Fig.5. Hysteresis behavior of sample Z-0 annealed at 500°C.

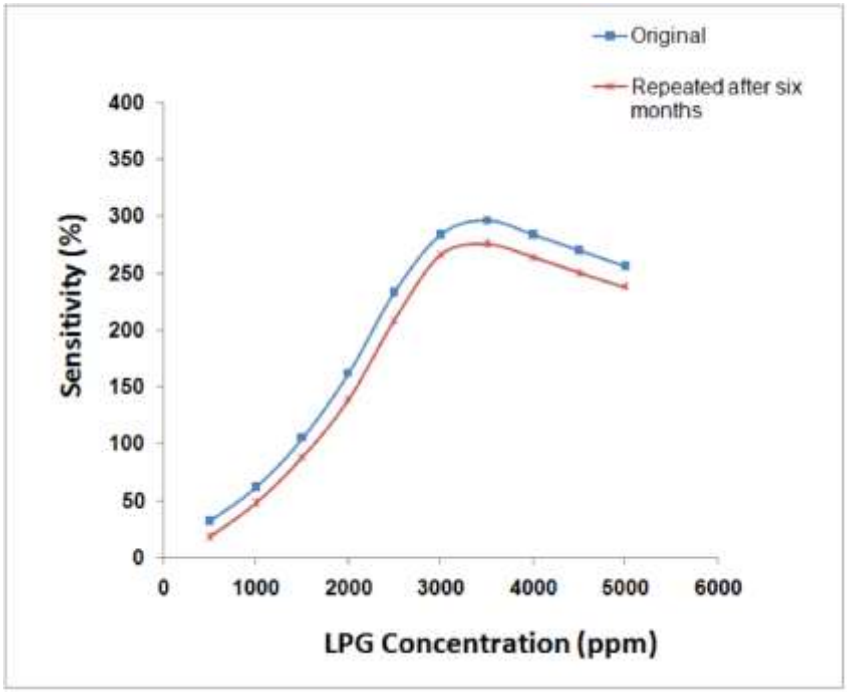

Fig.6. Aging effect behavior of sample Z-0 annealed at 500°C.

VI. CONCLUSION

The undoped ZnO (or pure ZnO) nanomaterials have been prepared and analyzed for humidity sensing and for LPG sensing analysis. The sensors developed from undoped ZnO nanomaterial annealed at 500°C showed the best sensing behaviors for both humidity sensing and LPG sensing. The sensing elements developed from undoped ZnO (or pure ZnO) nanomaterials annealed at 500°C, has properties like low hysteresis, low effect of aging and high reproducibility. Therefore, undoped ZnO sensors, annealed at 500°C, proved to be promising practical humidity and LPG sensors.

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