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A B S T R A C T

An experimental study of the dynamic range maximization with Zinc Oxide (ZnO) nanorods coated glass substrates for humidity and vapor sensing is reported. Growth time of the nanorods and the length of the coated segments were controlled to study the differences between a reference environmental condition (normal humidity or dry condition) and water vapor concentrations. In order to achieve long dynamic range of detection with respect to nanorods coverage, several substrates with triangular patterns of ZnO nanostructures were fabricated by selective hydrothermal growth over different durations of time (5h, 10h and 15h). It was found that maximum dynamic range for the humidity sensing occurs for the combination parameters of normalized length (Z) of 0.23 and normalized scattering coefficient (c) of 0.3. A reduction in transmittance by 38% at humidity levels of 80% with reference point as 50% humidity was observed. The results could be correlated to a first order approximation model that assumes uniform growth and the optimum operating conditions for humidity sensing device. This study provides an option to correlate ZnO growth conditions for different vapor sensing applications which can set a platform for compact sensors where modulation of light intensity is followed.

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1. Introduction

Humidity is the characteristics of water vapor present in gaseous form [1]. The existence of humidity in environment significantly impacts in many areas of life including industrial production processes and health, amongst others [2]. Measurement of humidity level is necessary for many applications such as food production industries, agriculture, chemical, medical, electronics, semiconductors, civil, weather forecasting and home environment [3–7]. Thus, monitoring humidity conditions are important for a better quality of life and to improve operational and manufacturing processes in various industries [8]. Humidity sensors are characterized into two types, namely, relative humidity sensors and absolute humidity sensors. The major difference being in the sensing units is that relative humidity sensor uses the unit of relative humidity (RH) which refers to a function of temperature, while the absolute humidity is determined as a function of pressure with the measurement unit of dew/frost point (D/F PT) [9]. Changes in capacitive, resistive or thermal conductivity properties are generally used for humidity sensing [10]. In conventional electronic humidity sensors, the RH level is determined on the basis of changes in electrical conductivity or capacitance but at high humidity levels it causes electric leakages [11]. Optical sensors based on nanomaterials provide an alternative to conventional electronic transducers. Utilizing optical fibers as waveguides, information can be carried over a long distance and the transmitted signal is resistant to electromagnetic interferences [12]. Nanomaterials have unique optical, magnetic, electrical and mechanical properties which make them attractive choice for various applications such as in gas and chemical sensors, superconductors, photocatalysis, optoelectronic devices, biomedical and agricultural applications, amongst others [13,14]. Zinc oxide (ZnO), tin oxide (SnO₂) and tungsten oxide (WO₃) are commonly used as...
gas sensitive elements due to the changes in conductance upon the adsorption of gas molecules on the surface [15]. ZnO is a n-type semiconductor which has direct and wide band gap energy of 3.37 eV and a large exciton binding energy of 60 meV [16]. It also has a good optical transparency in visible spectrum which makes it useful for short wavelength optoelectronic applications [17,18]. It as well has good chemical stability, electrical conductivity, bio-compatibility, and high electron transfer properties which are being utilized for electronics, optics and biomedical applications [19].

ZnO nanostructures have high surface to volume ratio which enhances the potential for the adsorption of water molecules on its surfaces when used in humidity sensing applications [20]. The optical detection of humidity levels can be based on two main factors. First is the change of the surrounding effective refractive index as well as the change of the electrical conductivity of the material due to the adsorption of water molecules [21,22]. The changes in the optical properties modifies the complex refractive index of the ZnO nanostructures. Combination of these two factors affects the optical scattering behavior of light incident on ZnO nanomaterials [23]. Most of optical humidity sensor studies were done on optical fibers, either silica or polymer type, where one-dimensional ZnO nanostructures such as nanorods were grown on the curved surface of the optical fibers. Growth of nanorods on flat surfaces such as glass [24], silicon wafer [25] or sapphire substrates [26] provides better control during the growth process and thus to an increase in the surface area in a smaller region for humidity sensing [27]. Due to the higher real part of the refractive index of ZnO nanorods (nZnO ~1.9 ~2.1), it leads to the possibility of inducing leakage of the incident light beam when coated on lower refractive index medium, such as glass, due to scattering [28,29]. Optimization of the growth process and the coating area are crucial to enhance the sensitivity, and system performance, which leads to the reduction in the complexity of signal detection and analysis. The growth of ZnO nanostructures have been extensively reported in the literature mainly based on optimizing the synthesis parameters such as growth duration [30,31], growth temperature [32], concentration alterations [33] and solvents variation [34]. Formation of various nanostructures of ZnO like nanorods, nanowires, nanoflowers or tetrapods as well as its dimension (nanorod length, density and optical scattering cross-section etc.) varying the synthesis conditions have been reported in the literature [35,36]. This morphology of the nanostructures would affect the optical response of the coated layer such as scattering and attenuation coefficients.

Several researches have demonstrated optimization of ZnO nanorods synthesis process on optical fiber for chemical vapor detection. Fallah et al. reported the impact of the aqueous growth conditions of the nanorods on light scattering and optical fiber coupling power [37]. Bora et al. showed that 2.2 μm tall ZnO nanorods led to maximum average coupling efficiency of cladding mode light side coupling [38]. The intensity of coupled light was reported to improve upon the exposure to various chemical vapors (methanol, ethanol, toluene and benzene). In another work we have shown that the maximum side coupling of light on plastic optical fiber (POF) could be achieved on spirally-patterned of ZnO coatings [39]. The spiral-patterned coatings was found to provide better side coupling when contrasted with unpatterned coatings and optimization of the width of spiral-patterns led to maximum light side coupling which was applied for the detection of different concentrations of alcohol vapors (methanol, ethanol and isopropanol) [40].

In spite of the optimization of ZnO nanorod coatings on optical fibers successfully presented in the reports as noted above, inconsistencies in terms of optimum growth conditions related to the performance of the fabricated devices exist. Two major factors that influence this uncertainty are uniformity of the coatings and repeatability of coating structures. The sensing surface area is one of imperative factor that influence the sensitivity of a sensing device as discussed earlier. Uniformity of nanomaterial coatings guarantee a consistent sensing response throughout the surface. The reduction of the rough surface due to good uniformity of the coatings make the device suitable for being utilized as transducers, particularly in optoelectronic applications [41]. Despite the fact that a uniform coating is achievable, repeatability of the nanomaterial structures is another factor that affects the consistency of the fabricated sensor performance. Due to the sensitivity of growth processes the physical of nanostructures (e.g. length or cross-sectional area of nanorods) grown under similar growth parameters (concentrations, growth time or temperature) may not necessarily be achievable at all times. Also, the optimization of the effective area of the ZnO coating in combination with growth time may not be possible to be repeated synonymously leading to variations in batch to batch fabrication processes since minor variations in the process could lead to dramatic changes in material properties [42].

Thus, the optimization of ZnO nanostructure growth and effective sensing surface area may not be an effective way to determine the maximum sensing response of sensor devices. In retrospect, the analysis of optimum operating conditions of fabricated sensor device delivers promising sensing device performance regardless of growth conditions or surface coverage. This implies, as long as any fabricated sensor device meets the optimum operating conditions, the device would produce best sensing performance. However, the ZnO nanorods growth time and coverage area are required to be first determined in order to realize the optimum working conditions. Thus the effect of the length of the coated ZnO nanorods depending upon the hydrothermal growth time are studied explicitly to better understand the system response over the desired limits of operation. Maximizing the effective scattering/attenuation coefficient of the nanorods layer does not necessarily result in the best performance particularly when considering intensity modulation. Instead, one needs to maximize the dynamic range of operation between two limits (i.e. dry condition or normal room humidity and maximum achievable level in the case of study) to achieve higher sensitivity.

2. Hypotheses

To realize the analysis, ZnO nanorods were proposed to be grown in triangular shape on glass substrate as shown in Fig. 1. The main purpose of the triangular form is to serve the variation of the ZnO coating length (L) along the propagation medium between the light source (LED) and detector as they move along edges of the glass substrate.

Fig. 2 shows the proposed model where uniform ZnO nanorods grown on a glass substrate is considered. The coating length is L and the nanorods forward scattering coefficient is σ. A light source is applied at one end of glass substrate while the output is measured by a detector at the other end as shown in Fig. 2(a). Light guided in the glass substrate is assumed to decay exponentially
due to forward scattering by the ZnO nanorods. The transmittance, $T$ measured by the output detector is expressed as [43],

$$T = \frac{I}{I_0} = \exp(-\alpha L) \tag{1}$$

where $I_0$ is the intensity of the light entering the medium or sample while $I$ is the intensity of the light leaving the medium.

Fig. 2(b) schematically represents the transmittance for two different limits: maximum humidity ($T_{\text{max}}$) and minimum humidity ($T_{\text{min}}$). With rise in humidity levels, the effective index of the medium surrounding the nanorods increases, leading to a more prominent contrast in the refractive index differences between the ZnO rods and the medium. The forward scattering coefficient is expected to increase with a subsequent reduction in transmittance of light.

As shown in Fig. 2(b), the target here is to control the length of the ZnO coating and scattering coefficient in order to maximize $\Delta T$, which is the difference between the optical transmissions, $T_{\text{max}}$ and $T_{\text{min}}$.

$$\Delta T = \exp(-\alpha_1 L) - \exp(-\alpha_2 L) \tag{2}$$

where $\alpha_1$ and $\alpha_2$ are the scattering coefficients of the nanorods for the cases of maximum humidity and minimum humidity levels, respectively. To generalize the analysis, two normalized coefficients are introduced: normalized length, $Z = \alpha_2 L$ and normalized scattering coefficient, $\zeta = \alpha_2/\alpha_1$. Therefore, Eq. (2) can be simplified as

$$\Delta T = \exp\left(-\frac{Z}{\zeta}\right) - \exp(-Z) \tag{3}$$

The normalized length, $Z_{\text{max}}$, which results in a maximum dynamic range ($\Delta T_{\text{max}}$) is derived from the derivative of Eq. (3) with respect to $Z$

$$Z_{\text{max}} = \frac{\zeta \ln(\zeta)}{\zeta - 1} \tag{4}$$

and the maximum dynamic range as a function of $\zeta$ is

$$\Delta T_{\text{max}} = \zeta - \frac{\zeta}{1 + \zeta} \tag{5}$$

The contour plot shown in Fig. 3 depicts $\Delta T$ variation with respect to changes in $Z$ and $\zeta$. The dashed line represents $Z_{\text{max}}$ calculated from Eq. (4). Increasing the value of $\zeta$ (which depends only on scattering of light from the nanorods) both $Z_{\text{max}}$ and $\Delta T_{\text{max}}$ increases. This means that as humidity levels increase, $\zeta$ becomes lower as $\alpha_1$ increases. This results in reduced $\Delta T$ value which can be translated as the loss of light intensity upon exposure to humidity. That does not necessary indicate increase in the length $L$ as $Z$ depends on $\alpha_2$ (scattering at lower humidity limit). Hence, in the experimental part, both ZnO segment length $L$ and scattering are varied.

3. Experimental

3.1. Sample preparation

Standard ground edge microscope glass slides (Sail Brand model no. 7101) with dimensions of 25.4 mm × 76.2 mm × 1 mm were used as substrates. Every glass slide was cut into three pieces of an approximate width of 15 mm each. These were then cleaned successively with hydrochloric acid (HCl), sodium hydroxide (NaOH), soap water, acetone, ethanol and deionized (DI) water in ultrasonic bath respectively for 15 min each, followed by drying in an atmospheric oven for 1 h at 120 °C. Hydrothermal growth of ZnO nanorods are carried out by first seeding the glass substrates with ZnO nanocrystals, followed by liquid phase epitaxy for the nanorod growth following the process described elsewhere...
3.2. ZnO nanocrystal seeding process

1 mM concentration solution of zinc acetate (Zn(CH3COO)2)2 in ethanol was used for the seeding process which has been reported earlier [37]. Briefly, the masked glass substrates were placed on a hot plate at a fixed temperature of 70 °C and 50 μL of zinc acetate solution was drop casted on the exposed surface of each sample and dried. This drop and dry process was repeated ten times before the samples were annealed in a furnace at 250 °C for 5 h. The samples were then cooled down to room temperature in air for subsequent hydrothermal growth of ZnO nanorods.

3.3. ZnO nanorods growth process via hydrothermal method

ZnO nanorod growth has been studied in great details by our group and reported extensively in the literature [46–49]. Briefly, prior to the growth process, an equimolar solution containing 10 mM of zinc nitrate hexahydrate (Zn(NO3)2·6H2O) and hexamethylenetetramine or HMT ((C2H5)6N4) was prepared and used as a precursor for the ZnO nanorods growth. The seeded samples were placed on a petri dish where the seeded area of each samples were facing downwards with some gap from the bottom surface of petri dish. The samples were dipped in the precursor solution and placed inside a growth chamber at 90 °C for the varied growth durations of 5 h, 10 h and 15 h. The growth durations were selected to cover all optimum growth conditions based on literature survey [37–40]. The solution was replaced with a new precursor solution every 5 h in order to maintain a constant growth rate of ZnO nanorods [48]. At the end of the process, the masks were carefully removed from the samples which were then thoroughly rinsed with DI water and dried in an atmospheric furnace kept at 120 °C. Following this, the coated samples were annealed in the furnace at 350 °C for 1 h to remove any contaminations and defects from the surface of ZnO nanorods [50]. The microstructure of the ZnO nanorods were characterized by scanning electron microscopy (SEM, Hitachi SU5000 FE-SEM) operating at 20 KV.

3.4. Optical characterization of the samples towards humidity sensing for dynamic range maximization

Fig. 4 shows an illustration of the top view of the setup for humidity measurements with the fabricated samples. A humidity chamber with the dimension of 0.35 m x 0.22 m x 0.23 m was used for all the measurements. The ZnO nanorod coated glass substrates were placed on an automated translation stage controlled by Thorlabs APT-DC Servo Controller (Model no.: TDC001) and connected to a PC unit through graphical user interface (GUI). Thorlabs LED with 530 nm wavelength (Model no.: MS30PI) was used as a light source which was controlled by Thorlabs LED driver (Model no.: LED11B). Green LED light source (530 nm) was chosen based on previous study by the group [40]. In this study, three channels were tested (red, green and blue) and the effect of forward scattering for vapor sensing showed that green source produced the best sensing performance. The LED source was set as close as possible to the edge of the substrates that was aligned with a detector placed at the other end of the sample. Thorlabs power meter (Model no.: PM100USB) with an input adapter (Model no.: S150C) ranging between 350–1100 nm with maximum power of 5 mW was connected through a polymer optical fiber (POF). The power meter was connected to a PC unit for data recording purposes. A hygrometer was mounted on the wall of the chamber as a reference to determine the actual humidity level inside the chamber during the experiments.

Before conducting humidity sensing measurements, the light scattering behavior of the fabricated samples were first analyzed with respect to the variation of ZnO coating lengths on different samples dependent on the growth conditions. The measurement was done at standard level of humidity and temperature in the measurement room. As LED and detector were stationary and aligned at both sides of the sample, the output intensity power was measured and recorded as the automated translation stage moved horizontally between the LED and the detector. As the automated translation stage moved, the sample was scanned throughout the edge in such a way that the length of the ZnO coating on the light propagation between the LED and the detector varied from minimum to maximum as schematically represented in Fig. 1. The scattering coefficient of each sample was analyzed from the output intensity power with respect to the input incident power.

For dynamic range analysis, the samples were tested with a simple experimental setup. In this experiment, the humidity level of the chamber was increased from 50% (room level) to 80% at constant temperature of 25 °C by placing a mixture of sodium hydroxide (NaOH) and water inside the chamber. The lid of the chamber was then closed and the humidity level of the chamber was monitored. After the humidity level in the chamber reached 80%, the output intensity power of the fabricated samples were recorded by similar scanning process throughout the edge of the samples and repeated for all the samples grown with different processing times. The sensing response for each samples were then analyzed by comparing the power of the output intensity between 50% and 80% humidity levels.

4. Results and discussion

4.1. Characterization of the ZnO nanorods growth

The scanning electron micrographs (SEM) obtained from typical ZnO coatings shows the hexagonal wurtzite ZnO nanorods growth (Fig. 5). Fig. 5(a)–(c) show the cross-sectional SEM image of the ZnO nanorods which were grown hydrothermally for 5 h, 10 h and 15 h respectively. The nanorods were observed to be better oriented when grown for longer durations (15 h) compared to 5 h and 10 h growth times. The average length of nanorods were found to be 0.85 μm, 1.62 μm and 2.26 μm for the respective growth times as shown in Fig. 5(d). It was observed that the length of the nanorods, the width also increases with the growth duration which are shown as insets in Fig. 5(a)–(c). In Fig. 5(e) the trend of the nanorods width with growth time which increased from ~43 nm to ~78 nm for 5 h to 15 h growth times, is shown. It was also found that with increasing diameter of the nanorods, the density or number of nanorods per unit area decreases subsequently from 1.4 × 10^14 nanorods/m^2 to 5.7 × 10^13 nanorods/m^2 for respective growth times as shown in Fig. 5(f). Fig. 6 shows the X-ray diffraction (XRD) pattern which confirms that the ZnO nanorods are highly crystalline and exhibit hexagonal wurtzite structure confirmed with the powder diffraction standards (JCPDS) card no. 01-070-8070. The diffraction angle (2θ) of 34.4° was observed for the maximum XRD peak intensity which corresponds to the (002) plane of ZnO. This demonstrates that the grown ZnO nanorods are well aligned in c-axis and the preferential growth is along the [0002] direction.

4.2. Optical characterization and dynamic range analysis

As we observed above, the growth time affects the nanorods physical structures such as length, density and cross-sectional.
a first order scattering model is considered [37] where total scattering is considered as a superposition of scattering from each rod. Hence, over a distance $dz$ of propagation the intensity is reduced as

$$\frac{dl}{dz} = -C_{sc} \rho_v l$$

where $C_{sc}$ is the scattering cross section and $\rho_v$ is the rods density per unit volume, $\rho_v = \frac{\rho}{\rho_v A}$, and $l$ is the average rods length. The scattering cross section of one rod depends strongly on the rods diameter, shape and length.

It is also worth mentioning that both forward scattering as well as backward scattering influences the intensity losses. Forward scattering contributes to increasing light leakage while backward
scattering lowers the leakage of light. With higher rod density and longer rods backward scattering would dominate and thus improved response/trend can be observed compared to lower density of nanorods or lower nanorod lengths.

The responses of the scattering coefficient with respect to the ZnO nanorod coating lengths are shown in Fig. 7. The effect of the ZnO coating length on the scattering coefficient ($\alpha$) can be derived from Eq. (1) as

$$\alpha = -\frac{\ln(I/I_0)}{L} \quad (6)$$

We observe from Eq. (6) that $\alpha$ is inversely proportional to the length of the ZnO coating, $L$, which indicates that $\alpha$ reduces as the length of ZnO coating increases in the light transmission path. However, the output intensity ($I$) also reduces as the length of ZnO coating increases due to higher leakage by forward scattering of nanorods. Therefore the value of $\alpha$ is expected to be constant throughout the respective length of coating. Fig. 6 shows the optical characterization for the fabricated samples of 5 h, 10 h and 15 h. The inset images depict the optical images of respective samples where the ZnO nanorods layer were grown in triangular shapes on glass substrate. Fig. 7(a) shows the scattering coefficient of the fabricated samples at different coating lengths. At the beginning of the ZnO nanorod coated substrate, the value of $\alpha$ for all the samples were at the peak which indicates the highest leakage of light due to the starting of forward scattering by nanorods. As the length of coating varies, the scattering coefficient responses on all samples were slowly stable and consistent towards the end of the coated layer. There were some fluctuations in the coefficient values observed at certain length of coatings on the samples due to non-uniformities in the coating. The values of $\alpha$ on all samples begin to be consistent from the middle towards the end of coating. Therefore, the average $\alpha$ for each growth time were calculated to be at 0.05, 0.1 and 0.08 for the growth time of 5 h, 10 h and 15 h correspondingly, as shown in Fig. 7(b).

The values of $\alpha$ with respect to $L$ were utilized in the dynamic range investigation of the fabricated device for humidity sensing experiments. $\alpha$ in the previous equation (Eq. (6)) represents $\alpha_2$ in the hypothetical model (Eq. (2)), and thus the values of normalized length ($Z$) can be calculated and introduced as one of new scattering coefficients as discussed earlier in Eq. (3). In the humidity determination experiments, it was observed that the optical transmittance ($T$) was reduced as the humidity level increased from 50% to 80%. The incremental water molecules around the ZnO nanorods increases the refractive index of the medium surrounding the nanorods inducing higher light leakage, which is used to determine the response to humidity levels. Thus, lower power intensities are obtained at higher humidity levels that leads to an increase in $\alpha$. Thus $\alpha$ value at maximum humidity level represents $\alpha_1$, in theoretical model; thus, the second scattering coefficient of the model which is the normalized scattering coefficient ($\zeta$) can be determined. Since $\zeta$ is the ratio of $\alpha_2$ to $\alpha_1$, therefore the value of $\zeta$ reduces as the humidity level increases. Henceforth, these two new scattering coefficients were used in subsequent analysis as discussed in the hypothetical model.

Fig. 8 depicts the contour plot for the dynamic range investigation of the fabricated samples. The contour plot demonstrates the
relationship between the normalized length ($Z$) and normalized scattering coefficient ($\zeta$) used to achieve the maximum dynamic range ($\Delta T$) of the fabricated devices. Darker blue and darker red regions indicate the lowest and highest values of $\Delta T$, respectively. It was found that the optimum $\zeta$ for the highest achievable humidity level is $\sim 0.3$ which demonstrates that the scattering coefficient of ZnO nanorods increases by $\sim 23\%$ when the humidity level changed from 50% to 80%. The maximum response of the fabricated samples towards humidity sensing for the combination of growth conditions (growth time and length of coating segment) appears to form a valley running from $Z$ value of $\sim 1.42$ (50% humidity level) to $\sim 0.23$ (80% humidity level) of the graph where the most noteworthy $\Delta T$ was observed to be at $\sim 0.38$. This number represents the reduction of the transmittance by $\sim 38\%$ in response to 80% of humidity level with respect to 50%. There are some incomplete data in the graph (darker red region) which was not obtained throughout the measurements. However, the data in the graph was good to show the formation of valley line which represents an ideal operating condition for fabricated sensor device towards humidity sensing. This implies that the optimum response for humidity sensing device can be achieved without being dependent to a particular growth time and coating area but as long as the combinations of these growth conditions are tuned and meet the optimum point of operating condition, the device should be able to perform the sensing at its best.

In comparison to the hypothetical model presented earlier, the value of $\Delta T$ in Fig. 3 was observed to be in between $-0.3$ and $-0.4$ matching very well to the experimentally determined value of $\sim 0.38$. The trend line of optimum working condition demonstrates the $Z$ value from $\sim 1$ (50% humidity level) to $\sim 0.8$ (60% humidity level) for the hypothetical model which shows the comparative trend of response between theoretical predictions and experimental observations. The slight discrepancies between theoretical and experimentally observed sensing characteristics are expected since the theoretical model was derived based on first principles approximation which considers uniform growth of ZnO nanorods, scattering coefficient of ZnO nanorods ($\alpha$) and length of ZnO coating ($L$). There are several other parameters that should be considered to refine future analysis as a second order approximation model such as incident angle and wavelength of light source could be accommodated.

5. Conclusions

The present study demonstrated the maximization of dynamic range for ZnO nanorods coated on glass slide for humidity and vapor sensing applications with controlled growth duration and length of the ZnO coating. From the experiment, it was found that the scattering coefficient of ZnO nanorods increased by $\sim 23\%$ at 80% humidity compared to 50% humidity conditions. The normalized scattering coefficient ($\zeta$) thus reduced from 1 to 0.3. The maximum dynamic range was observed to be at normalized length ($Z$) of 0.23 with 38% reduction of transmittance upon a change in humidity level from 50% to 80%. This is the optimum operating condition for the fabricated device to work at the best sensing performance. The experimental results were then matched to the first order approximation model which correlated reasonably well. This investigation revealed that the best performance of any fabricated sensing device can be accomplished by achieving the optimum operating condition, independent of nanorod growth parameters or coated area. This study can be a standard technique for designing a compact humidity or vapor sensing devices.

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