Sol–Gel Processed Zinc Oxide Film Deposited on Equilateral Prism As Optoelectronic Humidity Sensor

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الخلاصة

رسب غشاء اوكسيد الزنك على موشور متساوي الاوجه بطريقة الطلاء بالبرم. لُدنت العينة بعد ذلك بدرجة (400)م^0 لساعه واحدة. أكد حيود الاشعه السينية تكون التركيب المتعدد التبلور لأوكسيد الزنك وبمعلمات خلية وحدة a= (0.325) nm و c =(0.52) nm. قيست تغيرات النفاذية والامتصاصية مع الطول الموجي بالمدى من (300) nmالى (1100) nm. حسبت فجوة الطاقة البصرية للغشاء من تحليل الامتصاص البصري. اعتمادا على تغيير القدرة الخارجة لضوء الليزر المنعكس أستقصيت حساسية الغشاءالمرسب للرطوبة بمدى رطوبة نسبية مابين (10)الى (90) ٪. استعملت ثلاث زوايا (45 ، 60 و80) درجة لاسقاط شعاع الليزر. بينت النتائج المستحصلة زيادة القدرة الخارجة للشعاع المنعكس مع الرطوبة. سجلت اعلى قيمة للرطوبة النسبية عند زاوية سقوط (80) درجة.

الكلمات المفتاحية

متحسس رطوبة، اوكسيد الزنك، الطلاء بالبرم، تغيير القدرة والرطوبة النتسبية.

Abstract

Zinc oxide film has been deposited on an equilateral prism using spin coating method. The sample is then annealed at (400) 0C for one hour. X-ray diffraction (XRD) confirms the formation ZnO polycrystalline structure with the unit cell parameters of a= (0.325) nm and c= (0.52) nm .The variations of both the transmittance and absorbance with wavelength in the range from (300) to (1100) nm is measured. The optical energy gap of the film is calculated from analysis of optical absorption. Based on output power modulation of reflected laser light, the humidity sensing of the deposited film is investigated at relative humidity (%RH) in the range (10–90) %. Three angles (45, 60 and 80) degree are used to apply the laser beam. The obtained results showed that the output power of reflected beam is increased with humidity. Maximum relative humidity was recorded at incidence angle Ɵi = (80) degree.

Keywords

Humidity sensor, ZnO, Spin coating, power modulation and relative humidity.

1. Introduction:

Humidity is important a factor of environment drastically affects all organisms. For industrial applications and human life humidity sensors are becoming more important. Measurement of this parameter is needed in large applications such as, civil engineering, electronic processing, agriculture, air conditioning and meteorological services. In general humidity sensors can be classified to two types. The principle of first type is based on the measurement of electrical parameters like resistance, capacitance and impedance [1,2]. Second type measures the variations on optical parameters like frequency shift, refractive index and wavelength variation [3]. The second type is called optical (opto)-electronic humidity sensors. During the current work, attempts are done to measure the output power of reflected laser beam as many workers done [4,5].

Sol-gel spin coating technique has many advantageous over other frequent thin film techniques due to the following: it produces uniform thin film, it needs less equipment, it is relatively less expensive [6] Besides, uniformity of the deposited films and their microstructure can simply be controlled by arranging the preparation condition like fluid density, solution concentration, fluid viscosity, annealing temperature and speed of the spinner [7]. ZnO is a semiconductor material with direct wide band gap, a hexagonal wurtzite structure and binding energy of (60) meV. In fundamental research and due to its potential applications, this material has attracted much interest. ZnO is utilized in large scale of application like solar cells, violet/blue emission devices, acoustic devices and laser diodes [8].

In this contribution, an attempt is done to fabricate optoelectronic humidity sensor, the working principal is depend on measuring the variation of reflected laser output power from the base of prism located inside a cell with variable humidity circumstances.

2. Experimental part:

Microscope glass slide is used as substrate to deposit ZnO film. The dimensions of this substrate were (75) × (25) × (1) mm. The substrate is washed with ethanol and then distilled water. Sol solution was prepared by adding (3.1) g Zinc acetate dihydrate (Zn(CH3COO)2.2H2O) to isopropanol alcohol then (0.86) g mono ethanolamine (MEA) is added to yield a homogeneous and clear solution. The solution is then aged at room temperature for one day [9]. Home made spin coating machine is utilized with speed (2000) rpm. (100) micro liter from prepared sol solution is injected by using micropipette on substrate surface when the speed reaches the mentioned speed. After coating the substrate is heated to (400) 0C under ambient condition for one hour. The same procedure is repeated to deposit ZnO on one prism face and then heated at the same conditions. Shimadzu X-ray diffractometer is used to determine the orientations of ZnO. Bragg angle is ranged from (10) to (80) degree. To characterize the optical properties of the samples UV-Visible 1800 spectrophotometer is used. Transmittance% and absorbance% are measured at ambient conditions " temperature = (30) degree and %RH = 35 ".

The output power of reflected laser beam from prism is measured by spectrometer type HR4000CG-UV-NIR as a function to %RH inside this chamber. The used laser has output power equals to (100) mW. Two saturated salts are used to increase and decrease humidity inside chamber, these salts is tabulated in Table (1).

Table (1): Humidifier and dehumidifier salts.

Salt	RH range%	Function
K_2SO_4	to 95 10	Humidifier
KOH	to 10 95	Dehumidifier

Illumination of the first face of prism is achieved by laser diode with wavelength (531) nm. After reflection from the second face (coated with ZnO and inserted inside the chamber); the laser beam exists from third face to be measured by spectrometer, as shown in Fig. (1).

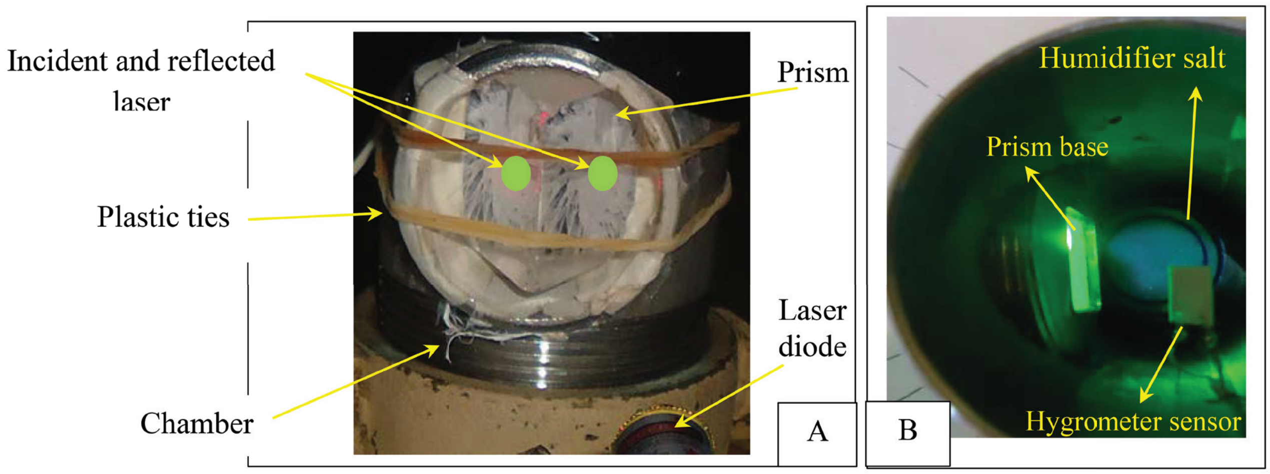

Fig. (1): A- The fixed prism on humidity chamber.
B-The details inside the chamber.

To measure the response and recovery time, humidity is stabilized at extreme %RH and then slowly exposed to atmosphere to achieve atmospheric %RH.

3. Results and discussion:

Fig. (2) shows the indexed XRD pattern of ZnO film. This pattern indicates that the film is polycrystalline. All appeared diffraction peaks are corresponded to the ZnO wurtzite structure (JCPDS Card, No. 36-1451). Fig. (2) illustrates enhanced intensities of the peaks corresponding to plane (002), indicating preferential orientation along the c-axis [10]. Debye Scherrer equation [11]:

Crystallite size(ξ) = 0.94λ / βcosθ

is used to calculate average crystallite size of the ZnO. Where λ is x-ray wavelength, β is the broadening of the hkl diffraction peak measured at half of its maximum intensity (in radians) and θ is Bragg diffraction angle. Table (2) illustrates some parameters calculated from Fig. (2).

Table (2): Calculated parameters from XRD pattern.

Crystallite size(nm)	Unit cell parameters (nm)	
	a	c
2.73	0.324982	0.52066

The peaks' sharpness indicates to a good crystallinity of the deposit. Lower intensities than that of (002) peak are observed for (102), (110), (103) and (112) peaks. The agglomeration of particles produces bigger crystallite size.

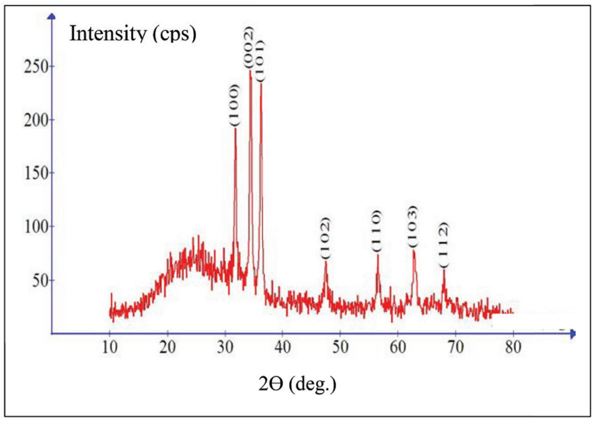

Fig. (2) : XRD pattern of ZnO film.

Fig. (3) illustrates the variations of both the transmittance% and absorbance with wavelength in the wavelength range from (300) to (1100) nm. Low relatively transmittance values of the film may be due to one or all the following: (1) increasing photons scattering due to crystal defects, (2) its comparatively high thickness (2) μm and (3) absorption of photons by free carriers [12].

From Fig. (3) it can be inferred that the reflection of the two used wavelengths (630.6) and (532) nm are more than the transmittance or absorption. This reflection's values are useful in current application because the variations of humidity inside the chamber are measured as a function to reflected laser from active ZnO layer.

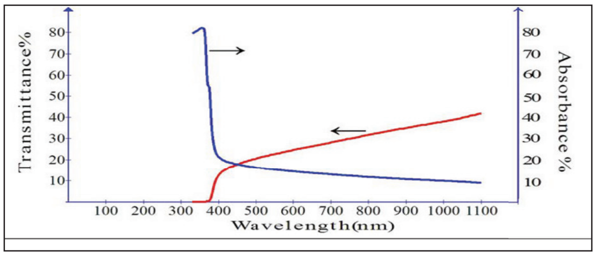

Fig. (3): Transmittance% and Absorbance versus wavelength.

By using extrapolation method the value of optical energy gap Eg is calculated. This method is done by plotting the square of (αhν) versus photon energy, as Fig. (4) shows. Where α is absorption coefficient and ν is the frequency of the light. The Eg value is (3.26) eV in good agreement with others [13].

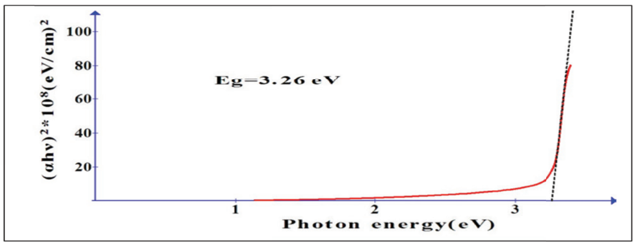

Fig. (4): Estimation of optical energy gap.

Fig. (5) shows the output power variations as a function to %RH. The sensitivities of the sensor are listed in Table (3) which it show slow increasing in reflected laser output powers for the three incident angles in the range (10-60) %RH and then the increasing becomes fast at high %RH. This Table shows also the increasing of average sensitivities with Ɵi. After the total internal reflection from ZnO layer at incident angles (45, 60 and 80) degree; the laser beam exist outside the surface of the prism. The variation of ZnO refraction index due to adsorption of water vapor (with increasing the humidity) produces output power changing of reflect laser. Inside the chamber humid air (water vapor) replaces dry air with increasing of humidity.

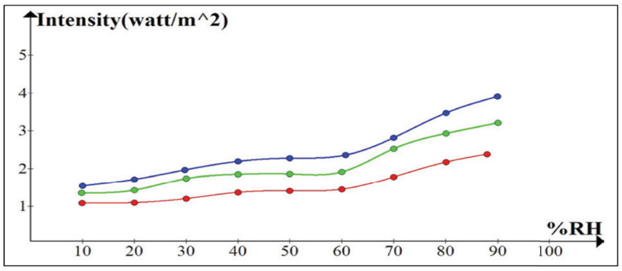

Fig. (5): Variation of reflected laser output power versus %RH.

Table (3): Sensitivity of the sensor as a function to incidence angle.

Incidence angle Θ_i (degree) at the prism-film interface	Average sensitivity (10-50%RH) ((Watt/m²/%RH	Average sensitivity (50-90%RH) (Watt/(m²/%RH
45	30	136
60	48	171
80	68	215

As a result, the pores of ZnO layer are filled with this humid air and water's condensation inside these pores increases the film refractive index [5]. ZnO layer density (5.67) g /cc will be changed as result to adsorption of water; its density (1.004) g/cc [14].

Response and recovery times are measured when %RH is stabilized at (10) and (90) respectively and then slowly subjected to room atmosphere. Fig. (6) shows the variation of output power with time for Ɵi = (60) degree. Recovery time and response time are (10 and 30) s. respectively. These values are close to that measured by Yadav et al [4] who fabricated opto-electronic humidity sensor from (Mg–Zn–Ti) oxide nanocomposite film.

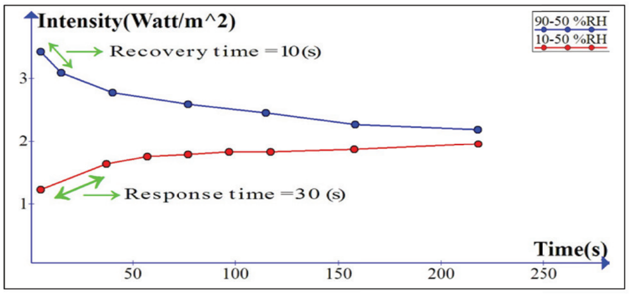

Fig. (6): Response and recovery times of the sensor for ϴi = (60) degree.

4. Conclusions:

The obtained results allow us to draw the following conclusions:

1. Sol gel processed ZnO thin film is successfully deposited on an equilateral prism using spin coating technique.
2. Structural analysis indicates the formation of ZnO thin film with the average crystallite size about (2.73) nm.
3. The measured optical energy gap of thin film was in a good agreement with published values.
4. The present study explored utilizing ZnO thin film deposited on a prism as humidity sensor at room temperature, the output power of reflected laser light increases with increasing of water vapor adsorption.

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