

Effect of Annealing Temperature on Response of ZnO Sensor Deposited on Glass Substrate

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The fabrication of deposited ZnO thin films by the sol-gel method on a glass substrate as a waveguide sensor is presented in this work. The deposited ZnO films are annealed using different annealing temperatures (350°C, 450°C and 550°C) to enhance their sensitivities. The optical, structural and morphological properties of the ZnO thin films are characterized by UV-VIS Spectrophotometer, X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR). The result of the structural characterization shows that the size, shape and high quality hexagonal wurtzite crystal evolution are achieved after annealing the ZnO thin films. Sharp peaks after the annealing temperature, related to the wurtzite structure, are observed and verified with XRD. Additionally, the sensitivity of the ZnO sensor after annealing is increased to detect different concentrations of glucose solution. Moreover, at a ZnO sensor thickness of 283 nm and annealing temperature of 550°C, the maximum sensitivity of 142% dB/con is achieved. Due to the high sensitivity, the proposed sensors may find important applications in biochemical and biological detection.

1. Introduction

Zinc oxide thin film is an optical transparent semiconducting material in the visible range and direct band gap of 3.36 eV [1]. In recent years, ZnO has attracted considerable attention due to its properties, such as low cost [2], inexpensive and chemically stable [3]. In addition, ZnO has been applied in different fields owing to its important optical and electrical properties, such as high electron Hall mobility [4], humidity sensors [5], photonic, optoelectronic devices [6], solar cells [7] and chemical and thermal stability under operating conditions [8]. Such properties attracted researchers' attention to investigate ZnO as a sensing material [9, 10, 11]. Moreover, ZnO thin films have been synthesized using different techniques, which include chemical vapor deposition (CVD) [12], sputtering and sol-gel [13]. Such films are mostly specified by their morphological surface and structures, which strongly depend on the method of preparation [14]. Sol-gel is one of the most important methods to prepare thin films due to its easy control of the chemical composition, low cost and homogeneity [15]. Besides, the annealing temperature of ZnO thin films has received considerable attention due to their high sensitivity, good linearity, operating stability and excellent absorption. In addition, high temperature treatment and long annealing time are commonly used to obtain high quality ZnO

films, which reduces the defects and improves the crystallinity [16]. Therefore, annealing temperature improves the sensitivity of ZnO thin films compared to un-annealed samples [17, 18].

In this work, thin ZnO films were deposited on a glass substrate using the sol-gel method as waveguide sensors to detect a glucose solution. The thin films were annealed with different annealing temperatures to enhance the sensitivity of the ZnO sensors. Moreover, the optical, crystalline, morphological and sensing properties of the ZnO were studied before and after annealing at different temperatures.

2. ZnO Preparation Method

In this work, the sol-gel method was used to synthesize different thicknesses of ZnO films on a glass substrate as waveguide sensors for glucose solution detection.

A mixture of ethanol-deionized water with a concentration of (10 mL: 90 mL) was used as a precursor to dissolve 0.2195 gm from zinc acetate dehydrate ($Zn(CH_3COO)_2 \cdot HO$) at room temperature. To make the mixture more homogeneous, the mixture was placed on a magnetic stirrer for 15 min. Finally, polyvinylpyrrolidone (PVP) at 0.4 g was added to the mixture to obtain the gelatin form, and again the mixture was placed on a magnetic stirrer for 24 hr to ensure homogeneity of the

solution. Next, the glass substrates were carefully cleaned using deionized water, isopropanol alcohol and ultra-sonicated, which was followed by drying with dust free tissues to remove any impurities on the glass surface. Then, the ZnO produced by the sol-gel method was synthesized on the cleaned glass substrates with different thicknesses. Moreover, the thicknesses of the ZnO layers were calculated using the following equation:

$$t = \frac{m_2 - m_1}{A \cdot \rho} \dots\dots\dots(1)$$

Where t is the thickness of the ZnO, m_1 is the weight of the sample before ZnO deposition, m_2 is the weight of the sample after ZnO deposition, A is the sample area and ρ is the density of the ZnO layer (5.6 gm/cm³).

3. Experimental Setup

Figure 1 illustrates the experimental setup of the waveguide sensor synthesized with ZnO. A polarized chromatic light from a semiconductor laser source at 650 nm was focused on the waveguide sensor synthesized with ZnO using the microscope objective. A high quality avalanche photo-detector with spectral response from 100 to 1250 nm was used to collect the light that passed through the waveguide sensor. The laser source, photo-detector, waveguide sensor and lenses were all aligned using 3D micro positioners to ensure the maximum signal received by the photo-detector. The proposed setup was used to detect different glucose concentrations using different thicknesses of ZnO.

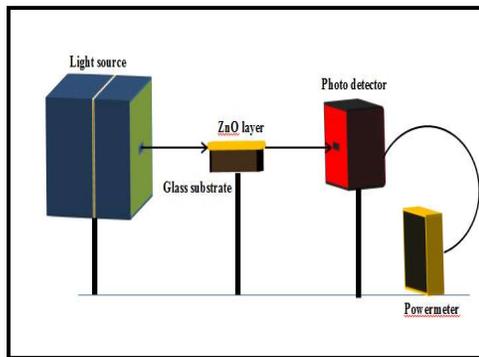


Fig. 1. Experimental setup

The structural properties of the ZnO thin films were investigated using the X-ray diffraction technique. Figure 2 shows the XRD patterns of the zinc oxide films annealed at 350°C, 450°C and 550°C on glass substrates. It is clear that the ZnO thin film was better with a wurtzite hexagonal structure and preferential c-axis orientation at 002, which correspond to $2\theta=34.495^\circ$ of the pre-annealed film. The grain sizes of the ZnO thin films gain enough energy to grow larger after annealing. However, the intensity of the peak position increases with increasing annealing temperature, other peaks are observed at 100, 101, 102 and 110, which correspond to $2\theta = 31.83^\circ, 36.33^\circ, 47.619^\circ$ and 56.661° . Additionally, the new peaks at 103, 200, 112, 201 and 202 belonged to $2\theta = 63.23^\circ, 68.13^\circ$ and 77.19° . It can be seen that the FWHM values decrease linearly with an increase in annealing temperature, which is attributed to the increase in the grain size at higher annealing temperatures, and there are no significant changes to the position of the peaks in the Bragg reflections. Furthermore, higher annealing temperatures lead to a significant increase in the crystallite. Crystallite size (D) can be calculated from the FWHM using the Scherrer formula [19], and this result is in agreement with previous work [20].

$$D = \frac{k\lambda}{B \cos\theta} \dots\dots\dots(2)$$

Where λ is the wavelength of the X-ray beam (0.154606 nm), θ : is the scattering angle, D is the grain size (nm), B is the FWHM and k is a constant ($k=0.89$).

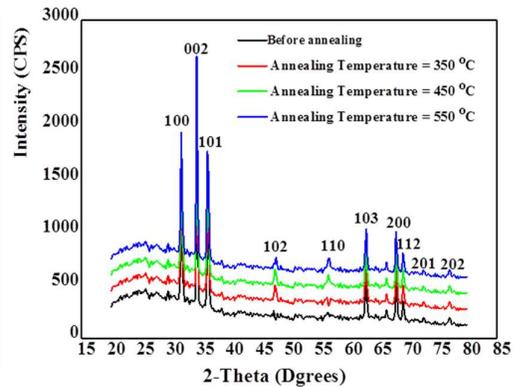


Fig. 2. XRD patterns of ZnO thin films before and after annealing at

4. Results and Discussion

4.1. Structural properties

Table 1. XRD results of ZnO thin films before and after annealing at temperatures of 350°C, 450°C and 550°C

Sample	(hkl)	2θ	FWHM	Lattice constant	D
ZnO		(deg.)	(deg.)	(nm)	(nm)
Without annealing	100	31.83	0.452	0.280	14.32
	002	34.49	0.503	0.247	14.14
	101	36.33	0.478	0.259	15.03
	102	47.619	0.424	0.190	14.77
	110	56.661	0.461	0.162	14.22
Annealing at 350°C	100	31.78	0.448	0.276	17.87
	002	34.34	0.491	0.242	17.51
	101	36.13	0.463	0.251	18.03
	102	47.57	0.411	0.183	18.12
	110	56.05	0.452	0.154	18.34
Annealing at 450°C	100	31.27	0.432	0.271	18.55
	002	34.14	0.487	0.230	18.77
	101	36.06	0.451	0.247	18.86
	102	47.41	0.376	0.177	18.93
	110	55.86	0.432	0.146	18.96
Annealing at 550°C	100	31.02	0.428	0.263	19.02
	002	34.12	0.467	0.228	19.31
	101	36.04	0.441	0.210	19.43
	102	47.35	0.362	0.164	19.56
	110	55.72	0.415	0.124	19.88

Table 2. shows the crystallite sizes increasing from 14.32 nm to 19.88 nm with increases in the annealing temperature.

Table 2. Crystallite sizes at various annealing temperatures

Annealing temperature °C	Grain Size (nm)
Without annealing	14.32
350°C	17.87
450°C	18.55
550°C	19.88

4.2. Morphological properties

Figure 3 shows the surface morphology (SEM) of the ZnO thin films annealed at 350°C, 450°C and 550°C. It can be seen that the nucleation of the ZnO thin film was observed at room temperature due to impurities and structural defects, such as oxygen vacancies and zinc interstitial. In addition, the grain size increases with increasing annealing temperature, lead to a higher formation of particles as a result of a large number of nuclei and cluster like structure at 350°C and 450°C, and this leads to an improvement in the crystallization process on the ZnO surface at 550°C [21]. Thus, the surface of the ZnO thin film has a homogeneous structure with an increase gradually with annealing temperature [22]. Our results are in good agreement with other work [23,24].

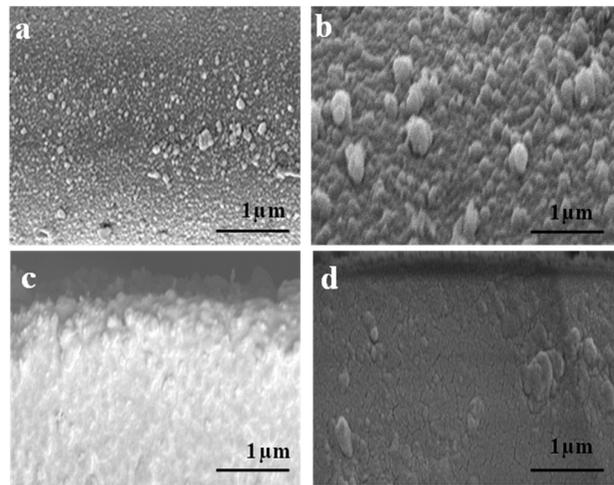


Fig. 3. SEM images of ZnO thin films before and after annealing: (a) without annealing, (b) at 350°C, (c) at 450°C and (d) at 550°C

4.3 Optical properties

The absorbance spectra from 300-800 nm for ZnO thin films annealed at 350°C, 450°C and 550°C are illustrated in Figure 4. It can be seen that the absorbance spectra increases with an increase in the annealing temperature due to the scattering of light at the boundaries [25]. The absorption edges of the annealed samples are located at 350 - 400 nm, and this is at a slightly longer wavelength as the annealing temperature increases. Figure 5 shows the optical transmittance spectra of the ZnO layers that have a high transmittance in the visible region and decreases with an increase in annealing temperature.

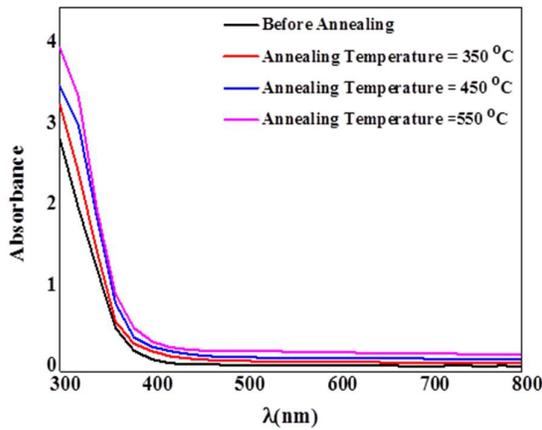


Fig. 4. Absorbance versus wavelength for annealing temperature

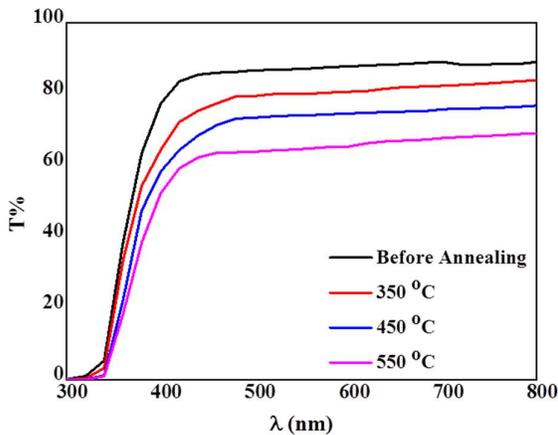


Fig. 5. Transmittance versus wavelength for annealing temperature

The values of the energy gap for the annealed ZnO thin films are illustrated in Figure 6. The energy gap decreases with increasing annealing temperature in the range (3.310 - 3.278 eV). The decrease in the energy gap behavior may be

attributed to the improved crystalline quality of the ZnO films [26]. Additionally, the optical band gap of the ZnO is determined by applying the Tauc model in the high absorption region using equation 3 [27].

$$\alpha h\nu = B(h\nu - E_g)^r \quad \dots\dots\dots (3)$$

where B is a constant, α is the absorption coefficient, $h\nu$ is the incident photon energy and r is a constant, which is equal to 1/2 for direct transition and equal to 2 for indirect transition.

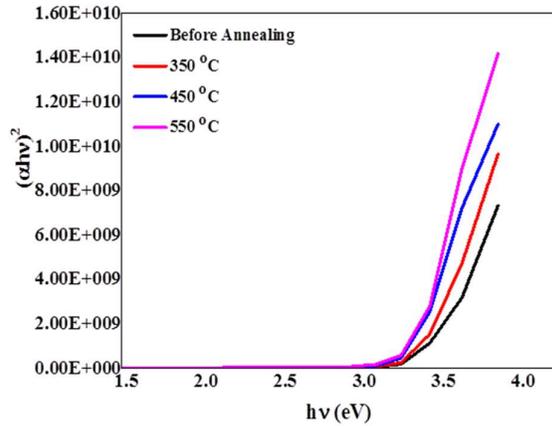


Fig. 6. Relationship between $(\alpha h\nu)^2$ and $(h\nu)$ for annealing temperature

Figure 7 shows the extinction coefficient with energy gap, we note that the extinction coefficient increases with increasing photon energy, and this is attributed to the photons absorbed at the surface. The extinction coefficient (k_o) is determined by equation 4 [28]:

$$k_o = \frac{\alpha \lambda}{4\pi} \quad \dots\dots\dots (4)$$

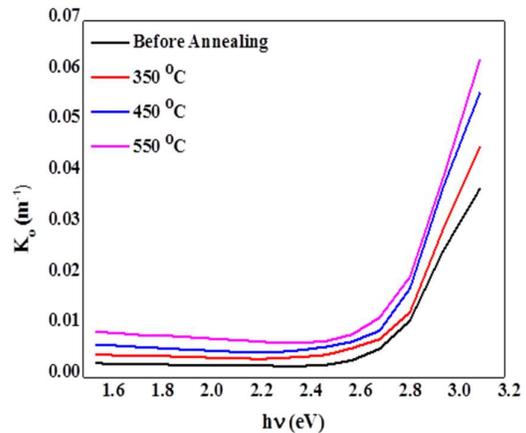


Fig. 7. Extinction coefficient versus $(h\nu)$ for annealing temperature

Figure 8 illustrates the variation of (ϵ_r) as a function of photon energy. It is clear that (ϵ_r) decreases slightly with an increase in the photon energy as well as with annealing temperature. Figure 9 illustrates the change in (ϵ_i) as a function of photon energy. Furthermore, (ϵ_i) increases with an increase in annealing temperature [29]. The dielectric constants (ϵ_r) and (ϵ_i) can be calculated using the following equations:

$$\epsilon_r = n_o^2 - k_o^2 \quad \dots\dots (5)$$

$$\epsilon_i = 2n_o k_o \quad \dots\dots (6)$$

(ϵ_r) and (ϵ_i) are the real and imaginary parts, respectively.

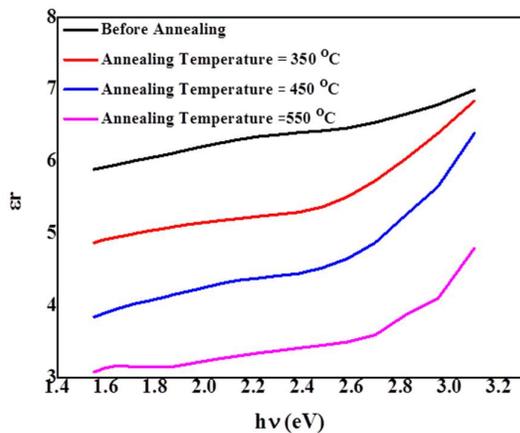


Fig. 8. Real part ϵ_r versus ($h\nu$) for annealing temperature

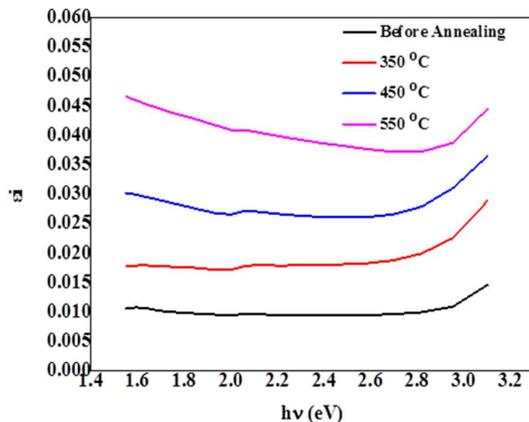


Fig. 9. Imaginary part ϵ_i versus ($h\nu$) for annealing temperature

4.4 Chemical Properties of ZnO Thin Film (FTIR)

FTIR spectroscopy is presented as a non-destructive analytical tool to determine the

chemical bonds in a molecule in the zinc oxide thin film, and different annealing temperatures were used to improve the vibrational properties. The absorbance FTIR spectra of the ZnO samples in the range 400-1750 cm^{-1} are shown in Figure 10. Where for the FTIR spectrum of the ZnO thin film before annealing, the bond (425.54cm^{-1}) corresponded to the Zn-O stretching vibration for a tetrahedral surrounding of the zinc atoms and the other bonds (481.27 cm^{-1}), (520.28 cm^{-1}), (1521.8 cm^{-1}) and (1681cm^{-1}) that correspond to the ZnOH bending mode and C- H stretching mode C=C and C=O, respectively. The occurrence of these modes indicates the presence of organic residues from the fabrication and dangling bounds during the deposition process. The effect of the annealing temperature's evolution shows an increase in the intensity of these peaks with an increase in the annealing temperature due to the crystallization process of the ZnO surface [30, 31].

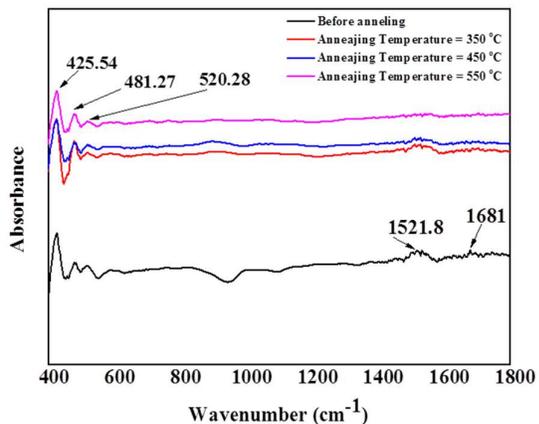


Fig. 10. FTIR of ZnO thin films before and after annealing at temperatures of 350°C, 450°C and 550°C

4.5 Influence of ZnO thickness on ability to detect glucose solution

To investigate the sensitivity of ZnO, four different thicknesses (211, 243, 268 and 283 nm) of ZnO films were synthesized on the glass substrate to detect the different concentrations of a glucose solution (0.1 – 0.5 %). Figure 11 illustrates the normalized intensity (in dB) versus concentration for the glucose solution. It can be seen that the ZnO sensor with different thicknesses has the ability to detect the glucose. For all the ZnO thicknesses, the response to the glucose solution is linear. The slopes of the lines give the sensitivity for each ZnO thickness. Furthermore, the sensitivity of the ZnO increases to detect the glucose as the thickness of the ZnO increased. The maximum

value of the sensitivity for the thickest ZnO film (283 nm) was (1.1 dB/con.), while the minimum values of the sensitivity were for the thinnest ZnO films (211, 243 and 268 dB/con.) and found to be (0.82, 0.87 and 1.04), respectively.

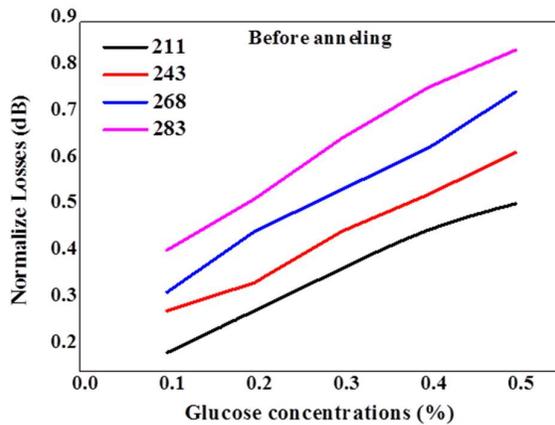


Fig. 11. Normalized losses (in dB) versus glucose concentration for waveguide sensors that were coated with ZnO at different thicknesses before annealing

The linear fitting of the ZnO sensitivity with various ZnO thicknesses is shown in Figure 12. The R-square value for the linear line fitting is 0.98221, which indicates a good linearity. The proposed ZnO sensor can be implemented for glucose detection due to the good line fitting characteristics.

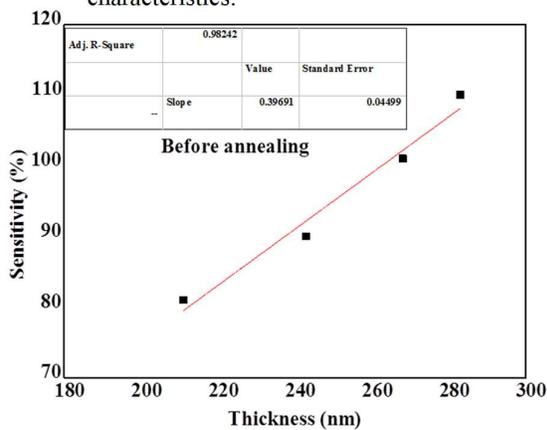


Fig. 12. Sensitivity % versus thickness of waveguide sensors for ZnO before annealing

4.6 Influence of Annealing Temperature and ZnO Thickness on Sensitivity to Detect Glucose Solution

The other investigated parameter for enhancing the sensitivity toward the glucose solution

detection is the annealing of ZnO at various temperatures. All thicknesses (211, 243, 268 and 283 nm) of the deposited ZnO on the glass substrate were annealed at different annealing temperatures of (350, 450 and 550°C). Figures 13, 14 and 15 show the normalize intensity (in dB) versus the glucose concentration at the annealing temperatures of 350, 450 and 550°C for ZnO, respectively. It can be seen that the responses of all ZnO thicknesses increased linearly. In addition, all the annealed samples have enhanced sensitivities once the annealing temperature was increased. The results show that the response of ZnO increased as the annealing temperature is increased for all thicknesses, and the maximum sensitivity was found to be at the highest annealing temperature (550°C), which could be due to an improved crystallization process on the ZnO surface with increased annealing temperature [32].

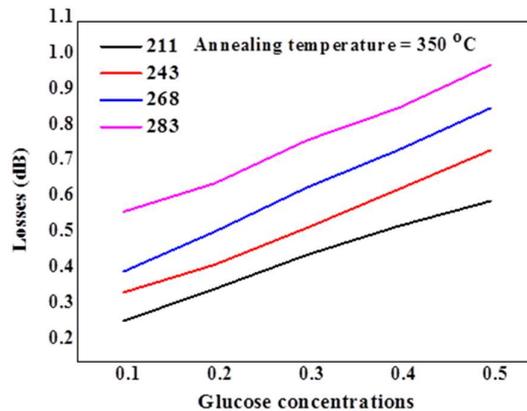


Fig. 13. Power losses (in dB) versus glucose concentration for waveguide sensors coated with different thickness of ZnO after annealing at 350°C

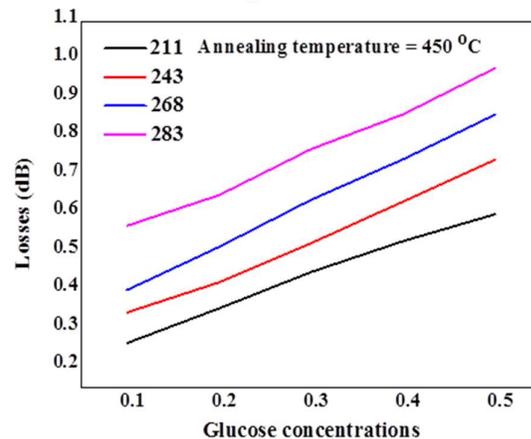


Fig. 14. Power losses (in dB) versus glucose concentration for waveguide sensors coated with different thickness of ZnO after annealing at 450°C

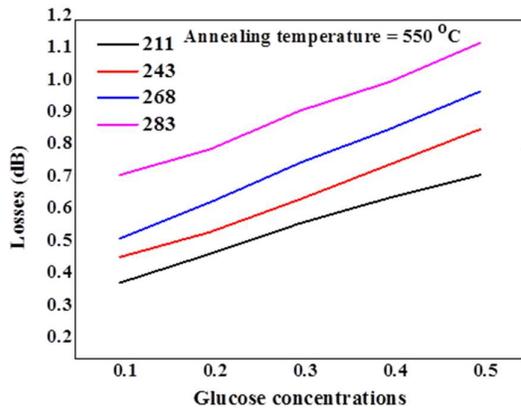


Fig. 15. Power losses (in dB) versus glucose concentration for waveguide sensors coated with different thickness of ZnO after annealing at 550°C

Figure 16 shows a comparison of the sensitivity of ZnO before and after annealing versus ZnO thickness. As can be seen from this Figure, the sensitivity increases as the thickness of ZnO increases. The results show that the thickest ZnO layer (283 nm) and highest annealing temperature (550°C) lead to the highest sensitivity, which was found to be (142% dB/con.) compared to the sensitivities of the thinner thicknesses (211, 243 and 268 nm) of ZnO that were 99%, 114% and 128%, respectively. Due to the grain size increasing with increasing annealing temperature, this lead to an improved crystallization process on the ZnO surface at 550°C [33]. Therefore, the proposed ZnO deposited on the glass substrate can be effectively used to detect the glucose solution.

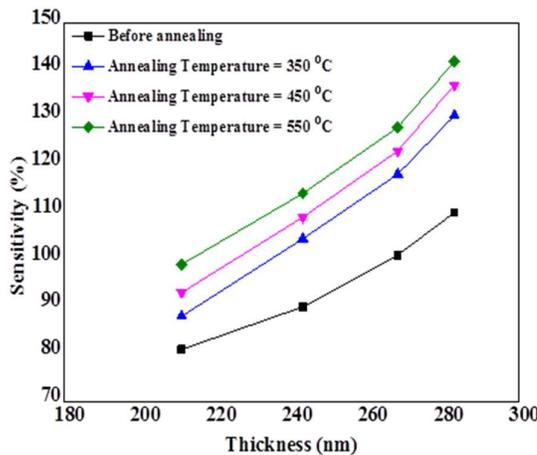


Figure 16. Sensitivity % versus different thickness for waveguide sensors of ZnO before and after annealing

Figure 17 shows a comparison for the sensitivity at a fixed ZnO thickness of 283 nm before and after annealing at a 550 °C temperature. As illustrated in the Figure, the sensitivity gradually increases with an increase in the glucose concentration before and after annealing at a 550 °C temperature. Furthermore, the annealing temperature results in a significant improvement in the sensitivity. In conclusion, the annealing temperature has an influence on the response of the ZnO sensors when compared with the response of the non-annealed ZnO films.

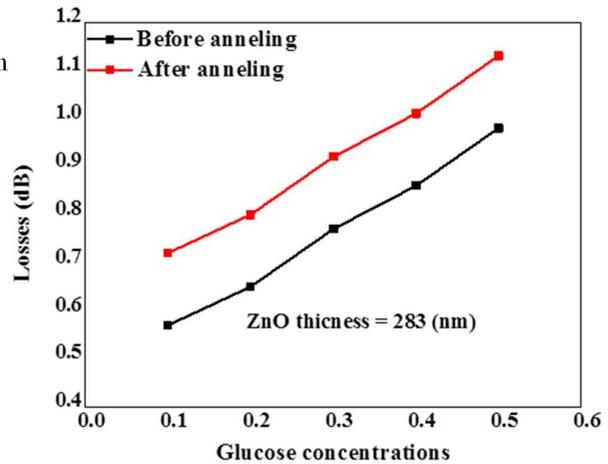


Fig. 17. Power losses (in dB) versus glucose concentration for waveguide sensors coated with ZnO at thickness of 283 nm before and after annealing at a set temperature

5. Conclusion

A waveguide sensor based on ZnO for the detection of different glucose concentrations has been fabricated and characterized in this study. Different thicknesses of ZnO were chemically synthesized via the sol-gel method. The sensor possesses a linear sensitivity in the range 0.1- 0.5 for glucose concentrations. Additionally, the deposited ZnO was annealed through thermal annealing ranging from 350°C to 550°C to enhance the sensitivity of the waveguide sensor. The annealed ZnO thin film is improved and becomes a quasi-spherical hexagonal wurtzite shape as observed by the XRD, SEM, FTIR and optical characterization. The highest sensitivity for the waveguide sensor is 142% dB/con., which is obtained at a ZnO thickness of 283 nm and annealing temperature of 550°C. The results showed that the sol-gel technique can produce ZnO thin films with a

short fabrication time and low cost. According to the obtained results, the waveguide sensor may find applications in biochemical and biological detection.

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