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Deposition of ZnO thin films with different powers using RF magnetron sputtering method: Structural, electrical and optical study

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ABSTRACT

Zinc Oxide thin films at room temperature with good crystallinity quality have been deposited at different Radio Frequency powers. Magnetron sputtering technique has been carried out on glass and oriented Si(100) substrates. The film structure has been characterized by X-ray Diffraction (XRD) and MicroRaman spectroscopy, which possesses a wurtzite structure with (002) preferential orientation selecting suitable conditions. Scanning electron microscope (SEM) and atomic force microscopy (AFM) have been utilized to determine the films surface morphology. The stoichiometry has been verified by Energy dispersive X-ray spectroscopy (EDX) analysis. The optical behaviors of the deposited films have been characterized by Ultraviolet Visible (UV-Vis) (optical transmittance measurements) as well as by Photoluminance characterization. Electrical properties, Current-Voltage (I-V) and Capacitance-Voltage (C-V) have been studied in details for Zinc Oxide on Silicon film that deposited at different Radio Frequency power. The high transparency, electrical behavior and smooth surface allow to use these Zinc Oxide films in photovoltaic cells and optoelectronics application.

1. Introduction

ZnO attracted attention latest research due to their properties such as semiconductors and transparent, piezoelectric, photoresponses [1,2], and environmentally friend, that characters candidate to its probable device applications (solar cells, biosensor.etc). Also, ZnO has two poly-kinds with cubic structure (zinc blende) and hexagonal structure (wurtzite), the first structure could be grown at high temperature and/or high pressure to be stable while the hexagonal structure does not need a high temperature and it is the most common likes ZnS materials [3,4].

Films of ZnO, ZnS and WO₃ used for gas sensing such as methanol and ethanol [5–7], and H_2O [8]. The nanostructure influences to gas responses [6] as well as the electrical and optical behaviors [5].

Nonlinear properties (NLO) of ZnO have been examined using a Z-scan method to evaluated the absorption factor and band gap 3.2 eV to be utilized in solar cell, photovoltaic technology manufacturing [6,9], photocatalysts and as sensors [10,11].

Different techniques like spray pyrolysis [12,13], molecular beam epitaxy [14], pulsed laser deposition (PLD) [15], thermal evaporation [16,17], electron beam deposition (EBE) [18,19] and magnetron sputtering [4] have been used to obtain semiconductors film [20].

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Fig. 1. SEM images (a) cross section and (b) surface view and AFM images (c) $3 \times 3 \mu m$ 2D and (d) $3 \times 3 \mu m$ 3D for ZnO/Si film deposited at 500 W.

The RF magnetron sputtering with frequently has been used [21] in semiconductors for many applications, in order to obtain interesting quality films with modified morphology and thickness [22,23]. Procedures of deposition like oxygen ratio, heating temperature, pressure of gas and power RF affect the often properties (physical like structural and optical) [24]. Possibility to obtain a good films (at room temperature) using RF magnetron sputtering is the most interesting advantage [25], while another processes need high temperature, which would limit the substrate types like glass and polymers [26].

The magnetron system is carefully chosen in this current paper to deposit Zinc Oxide thin films, since it presents a large flexibility and eases, where both deposition film rate and thickness can be controlled by adjusting the power of the source that can be verified using SEM characterization. EDX analysis has been applied to discover the elements content in the ZnO films. The XRD method has been performed to discover the crystalline properties of the films. Since, ZnO attracts large interest in the almost applications of optoelectronic, the optical properties (band gap and transmittance) of the grown films have been studied by both UV-Vis and PL methods. The power effect on the optical properties and structural of the films have been calculated and related to electrical value (C-V an I-V).

2. Experimental procedures

Magnetron sputtering (RF) method (PLASSYS-MP600S deposition system made in France) has been employed to deposit the ZnO



Fig. 2. EDX spectrum for ZnO/Si film deposited at 500 W.



Fig. 3. X-ray diffraction pattern of films ZnO on (a) silicon and (b) glass substrates prepared at different power.

films at different powers of 500 w, 600 w, 700 w and 800 w without heating. High purity target ZnO (99.99%) has been utilized at diameter of 15 cm, and the distance between the substrate holder and target was been at 6 cm. The residual pressure was larger than 2 $\times 10^{-7}$ Torr. The films have been synthesized on two kinds of substrates glass and oriented silicon (100). The films quality and textured



Fig. 4. Grain size of (002) orientation for films deposited on silicon (black color) and glass (red color) substrate as a function of the power.

ZnO films have been adjusted by choosing the appropriate factors of deposition procedure (distance and pressure), that can be improved to high gain. Thickness has been measured in in-situ and ex-sit using microbalance equipped with the plasma chamber and the SEM (model TSCAN Vega\XMU), respectively. The thickness was constant and equal to 100 nm for all ZnO films. The surface roughness and morphology have been studied by AFM (Autoprobe CP, Park Scientific Instruments). EDX has been employed to control the elementary synthetization of the prepared films. The crystallography of the films has been studied by XRD (Stoe Stadi P). Raman analysis using wavelength 633 nm of He-Ne laser has been utilized to describe synthesized ZnO films. He-Cd laser (325 nm) has been studied for the PL measurements. The optical properties have been calculated via the UV-Vis Spectrophotometer to determine the transmittance of the synthesized films.

3. Results and discussion

3.1. SEM, AFM and EDX study

The thickness of the ZnO/Si deposited film at 500 W has been established from cross section SEM as shown in Fig. 1-a, where the thickness was about 100 nm with dense structure (the deposition rate of the prepared films has been adjusted via microbalance in-situ in the chamber). Fig. 1-b shows the surface top view for the film 50 k magnification, where the morphology of film was smooth.

AFM images characterize the ZnO film which deposited at 500 W at $3 \times 3 \mu m$ area (1-c) for 2D and (1-d) for 3D. AFM images show small grains and highly textured structure (spherical form) with diameter varied between 50 nm and 75 nm. The ZnO film was smooth and had Root Mean Square roughness (RMS) less than 2 nm at $3 \times 3 \mu m$ area.

The composition of the ZnO film at 500 W has been observed firstly by EDX technique (Fig. 2). The EDX presents that the films have been made up mostly of Zn and O with percentage of 49.08 % and 50.92 %, respectively (EDX is semi-quantitative method). However, the C contamination is so little and the ratio O/Zn was about 1, so the film is stoichiometric. All the films were nearly similar due to the chosen conditions. Also, in recent work with Al-Khawaja et al. [6] have been deposited the ZnO films with different deposition time and using the same technique (RF sputtering), the films were stoichiometric. While, the power and pressure effects on structure and optical properties of ZnO film using another set-up [27], and it has been studied the growth mechanism of ZnO on AlN substrates [28], where ZnO films growth was epitaxial on AlN substrates which deposited at low temperature by magnetron sputtering.

3.2. XRD study

Fig. 3-a shows the XRD pattern of ZnO films deposited on Si(100) substrate have preferential orientation (002) peak at about 33.8°, which their intensity decrease with increasing the power from 500 W to 800 W because of slightly decrease of the grain size as well as the crystallinity. The increase of full width at half maximum (FWHM) would significantly decrease the quality of crystalline.

The peak at 71.5° corresponds to the (004) orientation, which associated to the second order of (002), has been confirmed high texture of films. All these peaks are harmonious with wurtzite structure (JCPDS 043-0002) [29]. Similarly, ZnO films deposited on glass substrate have the same behavior (preferential orientation (002) Fig. 3-b. It was found the (002) preferential orientation in previous works, using the same set-up PLASSYS-MP600S but using other conditions [9]. The reduce of the intensity with the increase of the power could be due to increase of the ionic bombardment from 700 W.

The grain size of (002) orientation for films deposited on two substrates as shown in Fig. 4. It has been designed from Scherrer equation, the average grain size ZnO/Si(100) was about 17 nm and quiz-constant with growing the power from 500 W to 700 W, after that it decreases to about 9 nm with increase the power to 800 W. Similarly, the grain size of the ZnO films on glass substrate has the



Fig. 5. (a) UV-Vis spectra and (b) the band gap for films deposited on glass substrate at different RF powers.



Fig. 6. Raman spectroscopy for ZnO/glass substrate prepared at different powers.

almost same behavior (the grain increases slightly from 20 nm to 24 nm with increase the power up to 700 W), after that it decreases to 17 nm for power 800 W. The shift (in the size of grain) to higher value could be because of the difference between the glass and Si(100) substrates in nature (amorphous for glass and single crystal for Si) and the chemical composition. Also, the mismatch between Si(100) substrate and ZnO film is important, where the two materials were crystalline. The quality crystalline for ZnO/glass film is better than ZnO/Si film could be due to the glass (SiO₂) like the ZnO films to chemical composition (both were oxide), this behavior accorded with study of Shan et al. [30] using PLD method.

The reduce of the grain size at 800 W power to ZnO film (deposited on Si as well as glass substrates) could be due to resputtering the film due to increase the ionic bombardment (high value of deposition rate at 800 W) using the same methods of synthesis [31].



Fig. 7. Photoluminescence spectra for ZnO films on silicon substrate deposited at different RF powers.

3.3. Optical properties

3.3.1. UV-vis and Raman analysis

Fig. 5-a presents the transmittance UV-Vis spectra of the three films in the range of 200–1000 nm, where the transmittance has been found between 72 % and 82%. The variation of average value of the transmittance could be due to the grain size and/or the roughness of the films. Using Tauc law for the semiconductor materials and helps us to give the band gap of ZnO films [32] which as shown in Fig. 5-b.

Fig. 5-b shows a little difference in the values of the band gaps, where the lowest value (Eg = 3.178 eV) corresponds to the largest grain size (deduced from XRD study for films deposited on glass) for film deposited at 700 W power, while the highest value (3.229 eV) has been found for bad quality crystallin (small grain size) corresponding to films deposited at 500 W and 800 W.

In this case, the grain size increases with increasing the power (quality enhancement) from 500 W to 700 W after that it decreases with increasing the power (800 W). Similar to these results [33], it has been found that the optical band gap increasing with the thickness.

Raman spectroscopy has been utilized to investigate the properties of the ZnO films, on glass substrate, prepared at different power as shown in Fig. 6, where it presents the Raman of these films at the range of 50–1400 cm⁻¹. The peak at 575 cm⁻¹ corresponded $E_1(LO)$ has been more pronounced for film deposited at 800 W, indicating less quality of the this prepared films), where the $E_1(LO)$ mode is related with defects like vacancy of oxygen, interstitial of zinc, or their complexes [34]. Also, the film at 800 W has smaller grain size deduced by XRD (Fig. 3), so it has an according between XRD and Raman characterizations.

3.4. Pl characterization

Photoluminescence (PL) emission spectra of ZnO films at room temperature are shown in Fig. 7. The optical band gap (3.2 eV) corresponds with the peak at 375 nm for ZnO films. Emission peak accorded with band gap, and this value was fewer than get by UV-Vis study. Taunk et al. [35], studied PL characterization ZnO materials. PL characteristics about UV emission corresponding to a radiative recombination of free excitons, where the visible emission is associated with structural defects (such as vacancies oxygen and zinc vacancies(Vo and VZn), oxygen and zinc interstitials (Oi and Zni)) [24]. It can relate the PL about UV emission with the crystallin quality as well as strain/stress, where general behavior could due to change grain size calculation (XRD) with RF power.

There have been many reviews on the PL spectra of massive (bulk) ZnO, but virtually fewer determination has been used on PL studies ZnO nanoparticles. Also, Fig. 7 shows the peak at 440 nm (2.82 eV) that due to defects in the deposited film. It is known that for nanocrystals, as the crystalline size is decreased, the peak of photoluminescence shift towards shorter wavelength.



Fig. 8. I-V characteristics of aluminum/n- zinc oxide/p-silicon heterostructure below forward bias for diverse film power.



Fig. 9. C-V at two frequencies; (a) low (100 kHz) and (b) high (1 MHz) for the films at different powers.

3.5. I-V and C-V measurements

The I–V properties of Al/ZnO/Si have been collected for films for deposition power values 500 W, 600 W, 700 W and 800 W, as established in Fig. 8. The common direction of the curves is appearing a characteristic n-ZnO/Si current conduction of the hetero-structure semiconductor. In the small voltage region (0 and 0.5 V) recompound current is produced as the excitation electrons (the



Fig. 10. C-V up and down at frequency 100 kHz and 1 MHz for ZnO film deposited at 700 W power.

valence to the conduction bands) recombines with existing holes at the valence band. Further than 0.5 V tunneling current gets part and then one could observe an exponential increasement in the amount of the current (diffusion) at upper forward where the conduction is controlled via the diffusion. It can also be noticed that the current in reverse bias apart is prominent and sharp for all films.

The slope in I-V curve is related to defects difference or to the resistivity between the films which deposited at different power, where the film at 500 W was sharpest. It is as well remarkable that the enhancement in crystalline quality (larger grain) is related with protuberant and needle I–V typical as shown in previous study [4].

Fig. 9-a shows four marked C–V curves of ZnO films get at 100 kHz functional frequency matching to power values: 400 W, 600 W, 700 W and 800 W, as denoted by the legends. The features were confirmed in an extensive bias voltage (5 V and -15 V), the device of film at 700 W shows characteristic flat band capacitance and has the uppermost capacitance value 0.0013 F, though at lower power value which deposited at 400 w has value of 0.0008 F. It can see noticeably conclusive accumulation, inversion and depletion layers demonstrating n-type carriers' conduction. The flat band voltage (VFB), it is a little shifted to negative values for two films at the 700 W and 400 W (\sim -1 V) indicating to the being of positive fixed dielectric charges either at the equal of little quantity of fixed oxide charges or the ZnO–Si interface within the bulk of the film, this phenomena has been observed in previous work [4]. Fig. 9-b shows C-V measurement at 1 MHz, it presents the same evolution with difference value compared to 100 kHz functional frequency.

The difference in capacitance value (C-V measurement) is due to the presence of the defects in the semiconductors film [4] and the higher value, which can be associate to the quality crystalline, where the film deposited at 700 W has larger grain size (XRD study in Fig. 2), while the film at 800 W has smaller grain size. The enhancement in films quality happens with improving thickness and from the C–V measurements for ZnO film, it can be consider that the films display suitably and comparatively enhanced conductivity at upper thickness [36].

Fig. 10 shows the C-V measurement with up and down at frequency 100 kHz and 1 MHz for ZnO films deposited at 700 W. The film deposited has small hysteresis (the difference between up and down measurement) in the two frequencies 100 kHz (Fig. 10-a) and 1 MHz (Fig. 10-b), the film has larger grain size (deduced by XRD characterization due to defect in the film). The C-V merriments with up and down can indicate to density of defects in semiconductors film [4] as well as in dielectrics films [37,38]. It reveals good properties (less hysteresis) corresponding to the higher quality crystalline film (larger grain size) for film deposited at 700 W. In general view, the measurement (up-down) exposes no noticed hysteresis in capacitance manner indicating there is no charge carriers within the film being trapped or de-trapped through the measurement procedure.

4. Conclusion

In summary, the ZnO thin films have been synthesized at RF powers (from 400 W to 800 W) using magnetron sputtering technique. The films have been synthesized on two substrates (Si (100) and glass) at room temperature have 100 nm thickness. AFM has been justified the growth as nanocrystalline and smooth which suitable to solar sell application. EDX technique has been verified that the ZnO films were stoichiometry. All films have (002) preferred orientation for, it indicates that there is an effect of power on structure. XRD and MicroRaman study have accorded the improvement of the crystalline quality with improving the power. The effect of power on the electrical behavior (I-V and C-V) at two frequency 100 kHz and 1 MHz have been studied as well as the optical and structural properties. Optical band gap slightly varied form 3.178 eV–3.229 eV with enhancing the deposited film power from 500 W to 800 W. Photoluminescence and UV-Vis characterizations (band gap and transparency) for ZnO films have been confirmed good candidate for optoelectronic applications.

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Data availability

No data were used for the research described in the article.

CRediT authorship contribution statement

Bassam Abdallah: Writing – review & editing, Supervision, Investigation. Walaa Zetoun: Formal analysis, Data curation. Ahamad Tello: Software.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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