JOURNAL OF NANO- AND ELECTRONIC PHYSICS
Vol. **15** No 3, 03034(6pp) (2023)
ЖУРНАЛ НАНО- ТА ЕЛЕКТРОННОЇ ФІЗИКИ
Том **15** № 3, 03034(6cc) (2023)

Optical Characterization of SnO_2 Thin Film Using Variable Angle Spectroscopic Ellipsometry for Solar Cell Applications

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(Received 15 March 2023; revised manuscript received 26 June 2023; published online 30 June 2023)

In this work, we prepared a nanostructure of TCO in a thin film of tin oxide using the spray pyrolysis method. The different deposition parameters have been set and optimized to prepare the source solution from Tin chloride dihydrate deposited on a cleaned glass substrate. The characterization of the thin layer of pure tin oxide deposited by spraying was achieved. The X-ray diffractometer shows that the tin oxide film is polycrystalline with a tetragonal structure, which consists mainly of orientations (101), (211) and other less intense. Ultra-violet-spectroscopy approved an excellent transparency of the thin layer of pure SnO_2 with a transmission of 95 % at 600 nm. The optical gap value of the deposited sample is equal to 3.95 eV. The spectroscopic ellipsometry measurements of the Psi and delta parameters were carried out at various angles of incidence 65°, 70° and 75°. The optical constant of the SnO_2 layer was modeled using a *B*-spline model. The goodness of the ellipsometric fitting was found at an incidence angle of 75°, which indicates a minimum MSE equal to 4.022. The SE characterization results of SnO_2 thin film on the glass substrate have shown that the layer thickness, the refractive index and the extinction coefficient are equal to 219.78 nm, 1.41 and 0.123, respectively. The resulting structural and optical parameters confirmed that a thin SnO_2 layer was formed. This layer showed a wide bandwidth and high transparency of a type of TCO semiconductor, which can be used as an anti-reflective layer in solar cell devices.

Keywords: SnO_2 Thin film, Spray pyrolysis, Spectroscopic ellipsometry, Optical constants.

DOI: 10.21272/jnep.15(3).03034 PACS numbers: 78.66. – w, 81.15.Rs, 07.60.Fs

1. INTRODUCTION

Transparent conductive oxides (TCO) are well known for their applications in various fields, such as optoelectronic devices. Tin oxide (SnO_2) material has been well known as a scientific and technologically promising *n*-type semiconductor TCO exhibited high transparency in the visible region of the spectrum, low electrical resistivity, non-toxic and high infrared IR reflectivity [1]. SnO_2 has a wide band gap range of 3.62-4.09 eV [1, 2]. This kind of TCO can be employed in a wide range of applications. In solar cells, the SnO_2 can be used as a transparent electrode or an antireflection film [3]. In microelectronics, the SnO_2 can apply in photocatalysis [4] and photodetection [5] such as the gas and chemical sensors, photodetectors, solid-state high lithium storage devices, gas discharge display, flat and touch control screens, heat reflecting windows, mirrors and electro-chromic windows and for electromagnetic protection. Several methods including chemical vapor deposition [6], sputtering [7], sol-gel [1], spin coating [8], dip coating [9], chemical spray pyrolysis [10], physical vapor deposition (PVD) [11], and others can realize the deposition of the SnO_2 films. The spray pyrolysis technique (SPT) is widely used because of its simplicity, affordability, low cost, the feasibility of mass production, and high purity of deposited products [2]. Generally, tin chloride is the most used precursor for spray depositing SnO_2 thin film oven preheated substrates.

In the literature, many characterization tools have been used to investigate the structural and optical properties of SnO_2 thin film such as X-ray diffractometer (XRD), Ultraviolet-visible (Uv-vis-NIR), Scanning electron microscope (SEM) and others [12]. On the other hand, a few previous studies tacked the SnO_2 thin film using the spectroscopic ellipsometry (SE) technique [13]. For this reason, we have focused in our work on determining the layer thickness and the optical constants of the SnO_2 thin film adopting the variable angle spectroscopic ellipsometry (VASE).

The SE method is considered powerful optical spectroscopy, which can provide much information on principally the refractive index (*n*), extinction coefficient (*k*) and layer thickness of the characterized samples. Additionally, the SE also provides the electronic transitions in the solid materials and on the overall structure of the sample including the density and the nanostructure. This technique provides two experimentally ellipsometric parameters (ψ and Δ) which are related to the change in its polarization state. It is important to note that SE can also provide an optical band gap, dielectric constants, and optical conductivity [13].

In this work, we have studied the structural and optical properties of the SnO_2 thin film prepared by a spray deposition process. The VASE method was utilized to determine the best incidence angle, which gives a good sensibility of the parameters required such as *n*, *k*, and thickness of the prepared SnO_2 thin film.

2. EXPERIMENTAL DETAILS

2.1 Preparation of SnO_2 Solution

Tin oxide thin film was prepared onto ultrasonically

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cleaned ordinary glass substrate by using spray pyrolysis. The SnO_2 solution was prepared by dissolving 0.1 M concentration of the precursor tin chloride dihydrate ($SnCl_2 \cdot 2H_2O$). This precursor was solved in a mixture of ultra-pure water and methanol (volume ratio 1:1) for 50 mL volume of the solution. Then, a few drops of HCl were added to get a clear solution. The obtained solution was put on a stirrer and the solution was left for 24 hours to make sure that there are no residues. Before being inserted into the spray pyrolysis chamber, the ordinary glass substrate was rinsed thoroughly with ethanol and acetone then cleaned with distilled water ultrasonically.

2.2 Deposition and Characterization of SnO_2 Sample

The spray pyrolysis technique (SPT) is a simple homemade chemical process experimental setup [2, 10]. This technique is classified as solution-based chemistry on the nature of the deposition. SPT involves spraying solutions of the film on a heated surface [2]. SPT equipment consists of a precursor solution, an atomizer, a substrate heat source, and a temperature controller. The commonly used atomizers are ultrasonic [10]. The equipment used in this work is HOLMARC OPTO-MECHATRONICS PVT LTD model number: H0-TH-04. Thereafter, the prepared chemical solution was deposited on the substrate heated to a temperature of 400 °C. The chemical reagents are selected so that the undesirable products decompose pyrolytically at the deposition temperature [2]. The SPT deposition was carried out using an ultrasonic atomizer for 3 min with scanning on both x and y axes in speed [200, 5], the solution deposition rate was 2 mL/min and the spray nozzle at a height of 8 cm.

The sprayed films were characterized by X-ray diffraction (XRD) measurements using CuKα radiation ($\lambda = 0.1540$ nm), a tube voltage of 40 kV, and a current of 40 mA for angles between $2\theta = 20$ to 70° in 0.02 steps. The optical characterization was measured in the wavelength range 300-800 nm through Uv–visible spectroscopy (Cary 5000 UV–Vis–NIR spectrophotometer version 2.24).

Spectroscopic ellipsometry (SE) is used with Woollam Company's alpha-SE ellipsomères, the details of data collection through measurement operations in the 380-900 nm wavelength range. SE depends on both the system type and individual ellipsometer configuration to determine and analyze optical constants.

3. RESULTS AND DISCUSSION

3.1 SnO_2 Structural Properties

In this section, we investigated the structural properties of the SnO_2 thin film by using XRD techniques. Fig.1 shows the XRD pattern of the sprayed SnO_2 thin film. The dominant peak was detected at around $2\theta = 51.57°$ in this figure. Furthermore, other peaks less intense were recorded at 37.83° and 33.57°. These peaks correspond mainly to orientations (211), (200) and (101) crystal planes. The observed XRD patterns are in accord with ICDD card no 41-1445 of tin oxide. The presence of peaks of differing intensities and widths indicates that the prepared SnO_2 thin film was made of polycrystalline with a tetragonal phase.

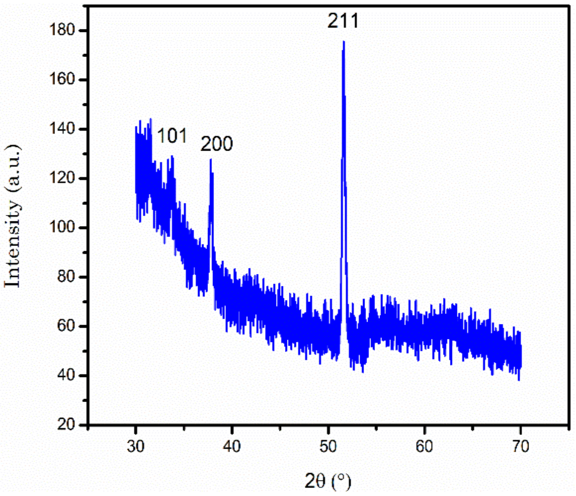

Fig. 1 – X-ray diffraction pattern of SnO_2 thin film prepared by spray method

The structural properties of SnO_2 thin film are obtained at 400 °C. The average crystallite size D of the film is calculated using Scherrer's formula [14]:

$$D = \frac{k\,\lambda}{\beta \cos \Theta} \tag{1}$$

where k is the shape factor with value $k = 0.94$, λ is the wavelength of the X-ray used (here, $\lambda = 1.540$ Å), β is the full width at half maximum (FWHM) of the corresponding peak and θ is the Bragg's angle as shown in Table 1. The crystallite size was calculated for the planes, where the calculated value of the average grain size D equals 41.95 nm. It is found that the intermediate crystals are of relatively small size (see Table 1).

The lattice constants a, and c for tetragonal structure, are determined by the following equation [15]:

$$\frac{1}{d^2{}_{hkl}} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \tag{2}$$

where, d_{hkl} is the interplanar distance and h, k, l, are Miller indices. The calculated d-spacing, grain size, a, and c values are almost similar to those given in ICDD card no. 41-1445 and those from the literature [16].

3.2 SnO_2 Optical Properties

The optical transmittance of SnO_2 thin film was investigated in the wavelength range of 300-800 nm by using UV–Vis–NIR spectroscopy and was used to take transmittance spectra. In addition, the band gap of the prepared SnO_2 film is defined by using the absorbance data.

Fig.2 shows the transmission spectrum of SnO_2 thin film prepared using SPT. This figure indicates that the prepared sample has high transmittance of visible light, with an average value over 95 %. Obvious interference phenomena can be observed in the transmission spectrum. Therefore, we have concluded that the prepared sample has a good feature in terms the optical transmission in the visible region. This feature plays an important role in the final performance of a p-n junction composed from the n-type SnO_2 thin film.

Table 1 – Values of crystallite size in tetragonal SnO_2

(*hkl*) plane	Standard 2θ [°]	Observed 2θ [°]	Standard *d*-spacing [Å]	Calculated *d*-spacing [Å]	FWHM [°]	Grain size *D* (nm)	Standard parameters $a = b$; c (Å)	Calculated parameters $a = b$; c (Å)
(211)	51.78	51.57	1.764	1.771	0.15744	31.95	4.738; 3.187	4.752; 3.196
(200)	37.95	37.83	2.364	2.376	0.31488	81.27		
(101)	33.89	33.57	2.642	2.667	0.75571	12.61		

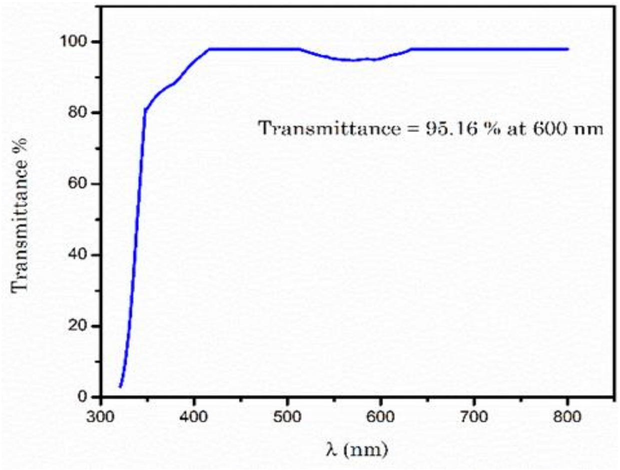

Fig. 2 – Evolution of the transmission spectrum of SnO_2 thin film as a function of wavelength

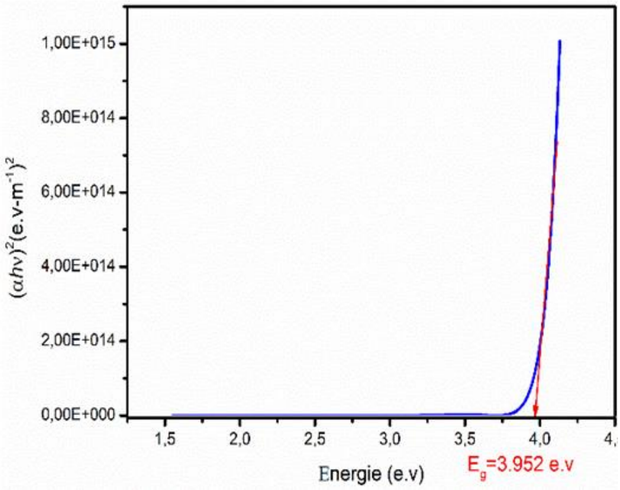

Fig. 3 – Plot of $(\alpha h\nu)^2$ versus the photon energy ($h\nu$) of SnO_2 thin film

In order to calculate the optical band gap energy (E_g) of the prepared SnO_2 thin film, the optical absorbance data has been carried out. Fig. 3 represents the evolution absorbance data of the SnO_2 thin film as a function of the photon energy. From this curve, we have utilized equation (3) to determine the value of the E_g parameter [17]:

$$\alpha h\nu = A(Eg - h\nu)^n \tag{3}$$

where α is the absorption coefficient, E_g is the band gap, $h\nu$ is photon energy, A is constant and n assumes values of 1/2, 2, 3/2 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions, respectively. The correct band gap value is equal to 3.95 eV determined by tauc-plot technique [12].

These results of high transmittance and band gap are in accordance with the literature [12, 13].

3.3 Spectroscopic Ellipsometry Characterization

The experimental ellipsometric SE spectra measure only the ellipsometric parameters Psi (Ψ) and Delta (Δ) versus the wavelength of thin film material, to determine the sample geo-optical properties, such as layer thickness and optical constants.

These ellipsometric angles Ψ and Δ are linked to the complex reflection coefficients, R of the polarized light. The coefficients R_p and R_s define respectively the polarization of parallel and perpendicular light to the plane of incidence through the following relation [13]:

$$\rho = \frac{R_P}{R_S} = \tan\psi \exp^{j\Delta} \tag{4}$$

However, the process of ellipsometric characterization has several stages such as measurement, modeling, fitness, and results. In this study, at first, the various measurements are achieved to characterize the prepared thin film *n*-type TCO on the glass transparent substrate. The measurements were carried out at various incidence angles of the SE (VASE), namely: 65°, 70° and 75°. The collected experimental data of the SE parameters (Psi and Delta) for the SnO_2 sample have two spectrum curves are given in Fig. 5 and Fig. 6 respectively. Secondly, the modeling stage for each sample is analyzed using a model. B-spline and Cauchy models modeled the structure of the SnO_2 thin film and the glass substrate, respectively. The roughness was modeled using the Bruggeman approach. The diagram of the modeling structure is illustrated schematically in Fig. 4.

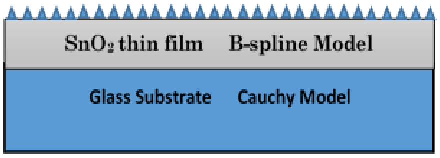

Fig. 4 – Diagram of modeling SnO_2 sample

To model SnO_2/Glass substrate, we used Complete EASE software to apply the B-spline and Cauchy models to describe the refractive index spectra of the glass substrate and SnO_2 layers, respectively. The B-spline function is a mathematical approach used to represent the optical function of the SnO_2 thin film. It is based on a distance resolution equal to 0.3 eV related to the wavelength of the different nodes or splines. This approach was used to analyze the VASE data of the SnO_2 sample. The fitting and results step were used to minimize the mean squared error (MSE) defined as [18].

$$MSE = \frac{1}{2N - M}\sum_{i=1}^{N}\left[\left(\frac{\psi_i^{mod} - \psi_i^{exp}}{\sigma_{\psi,i}^{exp}}\right)^2 + \left(\frac{\Delta_i^{mod} \ - \Delta_i^{exp}}{\sigma_{\Delta,i}^{exp}}\right)^2\right] \qquad (5)$$

where N is the number of Psi and Delta (ψ, Δ), M is the number of fitted parameters in the model and σ are standard deviations of experimental data points, the model data $(\psi_i^{mod}, \Delta_i^{mod})$, and the experimental data $(\psi_i^{exp}, \Delta_i^{exp})$. The MSE was used to quantify the difference between the experimental and the model generated data [19].

In this section, the results of the fitting have been plotted in Fig. 5, Fig. 6 and Fig. 7. These figures show the fit SE data (Psi and Delta), refractive index and extinction coefficient of the prepared SnO_2 thin film as a function of wavelength at different incidence angles (VASE), namely: 65°, 70° and 75°.

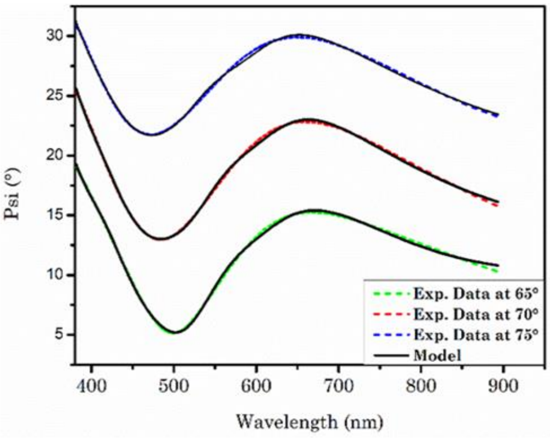

Fig. 5 – The Psi (Ψ) variation versus wavelength of SnO_2 film

Fig.5 shows the superposition of the experimental and model generated of the Psi (Ψ) spectra of the thin film of SnO_2 prepared by the spray pyrolysis method for different angles of incidence. From this figure, it is clearly shown that the magnitude of the Psi value changed between 5.14° and 30.95° when the angle of incidence varied from 65° to 75°. Fig. 6 depicts the superposition of the Delta (Δ) spectra of the thin film of SnO_2 obtained by experiment and model taken at various incidence angles changed from 65° to 75°. We observed that the value of the Delta changed between 0° and 71.85° as the incidence angle modified from 65° to 75°. Fig. 5 and Fig. 6 reveal that the Psi and Delta spectra have a good fit between the experimental and modeling data for all three angles of incidence, with a minimum of the MSE (see Table 2). This implies the validity of the proposed model to describe the structure of the prepared SnO_2 thin film. It should be noted that the high delta value indicates that the nature of this sample is an absorbent. Fig. 5 and Fig. 6 shows that the most acceptable and appropriate MSE value is 4.022 at 75° corresponds to the minimum squared error as shown in Table 2.

The above method of SE allows us to study the sample of the thin layer of SnO_2 using the different angles of incidence of SE (VASE).

The result of the above SE characterization required the use of a model. Therefore, the modeling by the B-Spline model was chosen according to the result obtained which implies that our layer is absorbent. The choice of better angles of incidence to relate with the minimum MSE helped to extract the spectrum of the index of refraction (n) and the coefficient of extinction (k) represented in the Fig. 7.

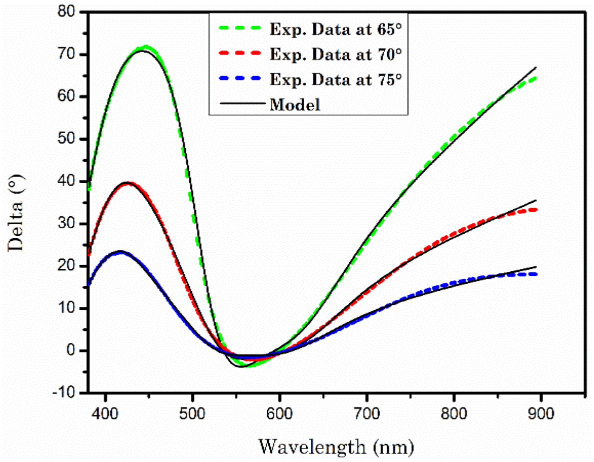

Fig. 6 – The delta (Δ) variation versus wavelength of SnO_2 film

In this part of our work, we have studied the resulting influence of SE angles of incidence on the refractive index and extinction coefficient of a sprayed SnO_2 thin film. Fig. 7 shows the refractive index (n) spectra of the SnO_2 thin film for various angles of incidence varying from 65° to 75°. At 632.8 nm, the refractive indices are equal to 1.49, 1.46 and 1.41 at angles of incidence 65°, 70° and 75° respectively. The extinction coefficient is a measure of the fraction of light lost due to scattering and absorption per unit distance from the average material permeability. Fig.7 shows also the resulting extinction coefficient spectra of the SnO_2 thin film for different angles of incidence changing from 65° to 75°. The found values of the extinction coefficient are equal to 0.14, 0.13 and 0.12 at 632.8 nm of the SnO_2 sample for the incidence angles of SE 65°, 70° and 75°, respectively. Fig. 7 show a maximum variation of the refractive index and the extinction coefficient when the incidence angle of the SE changes between 65°,70° and 75° for the wavelength in the visible spectrum was estimated at about 0.8 and 0.3 respectively.

A considerable variation of Psi (Ψ) and Delta (Δ) parameters as a function of wavelength was found. SE characterization obtained the film thickness for different incidence angles is equal to 206.52 ± 2.558 nm, 214.34 ± 2.985 nm and 219.78 ± 4.15 nm of the above samples at different incidence angles 65°, 70° and 75°, respectively.

Finally, this study allows us to have an accurate result of the three geo-optical parameters in research related with the minimum MSE criterion, which is, the refractive index which is equal to 1.41, an extinction coefficient which is equal to 0.123 and the thickness of the SnO_2 thin film, which is equal to 219.78 nm. The best angle of occurrence is 75 degrees corresponding to the minimum MSE of fitting, which is supported by the previous study. As a result, the obtained thin film has an absorbent film and acceptable transparency. That means that the absorption causes are due to free electrons absorbing wave energy by semiconductors and resulting in a conductivity that can make a good candidate for the anti-reflective layer in solar

Table 2 – Values of optical properties and thickness of SnO_2

Angle of incidence (°)	MSE	Thickness (nm)	Refractive index at 632.8 nm	Extinction coefficient at 632.8 nm
65	4.266	206.52 ± 2.558	1.49107	0.14781
70	4.222	214.34 ± 2.985	1.46045	0.13117
75	4.022	219.78 ± 4.158	1.41539	0.12316

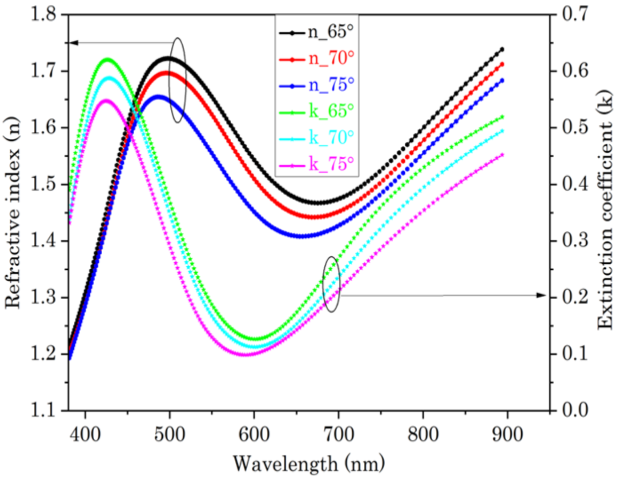

Fig. 7 – The refractive index and the extinction coefficient variation versus wavelength of SnO_2 thin film

cell devices. Therefore, the result is similar to that obtained in the literature [20].

4. CONCLUSION

The SnO_2 film was deposited using a spray pyrolysis technique, from the $SnCl_2.2H_2O$ precursor onto an ultrasonically cleaned substrate at 400 °C. X-ray diffraction analysis revealed a tetragonal structure, with preferred orientations of (211), (200), and (101). The optical study showed that the deposited film exhibited very high transparency with a wide energy band gap of around 3.95 eV. In the SE study, using B-spline to model the SnO_2 thin film and Cauchy for the glass substrate, gave the best fit between the experimental and theoretical SE data for various incidence angles ranging from 65° to 75°, with the best fit occurring at an angle of 75° and a minimum MSE value of 4.022. At this angle, the values of VASE is suitable method to determine the thickness, the real optical constants n, and k were 219.78 nm, 1.41, and 0.123 respectively. We also demonstrated that the structural and optical features confirmed that the SnO_2 film can be a good candidate for use in solar cell devices.

AKNOWLEDGEMENTS

The LRPPS laboratory, university of Ouargla, Algeria is acknowledged for supporting this work.

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Оптичні характеристики тонких плівок SnO_2 як сонячних елементів, досліджених методом кутової спектроскопічної еліпсометрії

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У даній роботі методом розпилювального піролізу отримано наноструктуру TCO в тонкій плівці оксиду олова. Різні параметри осадження були встановлені та оптимізовані для приготування вихідного розчину з дигідрату хлориду олова, нанесеного на очищену скляну підкладку. Була досягнута характеристика тонкого шару чистого оксиду олова, нанесеного методом конденсації. Методом рентгенівської дифрактометрії показано, що плівка оксиду олова полікристалічна з тетрагональною структурою, яка складається в основному з орієнтацій (101), (211) та інших менш інтенсивних. Ультрафіолетова спектроскопія підтвердила високу прозорість тонкого шару чистого SnO_2 з пропусканням 95 % при 600 нм. Величина оптичної щілини осадженого зразка дорівнює 3,95 еВ. Спектроскопічні еліпсометричні вимірювання параметрів Psi і delta були проведені під різними кутами падіння 65°, 70° і 75°. Оптичну постійну шару SnO_2 було змодельовано за допомогою B-сплайнової моделі. Достовірність еліпсометричного підгонки визначена при куті падіння 75°, що вказує на мінімальний MSE, рівний 4,022. Результати SE характеристики тонкої плівки SnO_2 на скляній підкладці показали, що товщина шару, показник заломлення та коефіцієнт екстинкції дорівнюють 219,78 нм, 1,41 та 0,123 відповідно. Отримані структурні та оптичні параметри підтвердили, що утворився тонкий шар SnO_2. Цей шар продемонстрував широку смугу пропускання та високу прозорість типу напівпровідника TCO, який можна використовувати як антибліковий шар в сонячних елементах.

Ключові слова: Тонка плівка SnO_2, Спрей-піроліз, Спектроскопічна еліпсометрія, Оптичні коефіцієнти.