Development and characterization of (Zn,Sn)O thin films for photovoltaic application as buffer layers

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Abstract

In order to study the Zinc Tin Oxide (ZTO) as an alternative buffer layer to replace CdS in thin films solar cells, we synthesized the (Zn,Sn)O material using the vacuum thermal evaporation of the metal compounds, Tin and Zinc, followed by annealing treatments. The obtained samples were divided into two groups according to the amount of Tin used during the thermal evaporation while the amount of Zinc remains invariant. The first group of ZTO thin films was made using a Tin amount of 0.312 g and annealed in air at different temperatures (samples A). The second group was made using a less amount of Tin (0.156 g) (samples B). Part of these samples were annealed in air atmosphere (samples B1) whereas the other sample was annealed under vacuum then annealed in air (sample B2). The annealing process was mainly dedicated to the oxidation treatment of Sn/Zn metallic bilayers but it also plays an important role to improve the crystallinity of the as-deposited films. The structural, optical and electrical properties of the prepared films were characterized using X-ray diffraction, UV–VIS–NIR spectrophotometry and impedance spectroscopy, respectively. We have succeeded to obtain a stable cubic spinel structure of Zn_2SnO_4 phase (sample B2). This thin film showed interesting properties such as a direct band gap energy of value of 3.20 eV, a high optical transmission of 80% and a n-type electrical conductivity.

Keywords ZTO · Thin film · Vacuum thermal evaporation · Annealing process · X-ray diffraction · Band gap energy · Impedance spectroscopy

1 Introduction

Cadmium sulfide CdS synthesized by chemical bath deposition (CBD) is considered to be the most widely used buffer layer in thin-film solar cells based on chalcogenide materials. However, due to environmental risks associated with using toxic cadmium (Cd) and its relatively low band gap (2.4 eV) [1], many research efforts were focused on the replacement of CdS with new Cd-free materials that fulfill the requirements for a non-toxic and effective buffer layer [2]. Recently, a class of thin-film metal oxide semiconductors characterized by high optical transparency and a good electrical conductivity had been applied in several optoelectronic fields: thin film transistors (TFT) [3],

gas sensors [4] and buffer layers in many solar cells, such as CIGS (18.2%) [5], CZTS (12.6%) [6] and CZTSSe (8.6%) [7]. Known as one of the transparent conductive oxides (TCOs) (Zn,Sn)O or ZTO had demonstrated its ability to be a promising alternative as a buffer layer in thin-film solar cell technology as well as a transparent material for bifacial solar cells [8]. M.A. Islam et al. [9] also reported that ZTO could be a better electron transport material (ETM) for high efficiency perovskite solar cells (PSCs) application. They obtained an efficiency of 24.07% by numerical simulation results based on the properties of ZTO synthesized by RF reactive co-sputtering. Indeed, ZTO has interesting properties such as the good thermal and chemical stability, a large band gap without forgetting to mention that

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its elements are inexpensive, non-toxic and earth abundant. The ZTO could be amorphous (α -ZTO) [10] as well as polycrystalline, which showed two different forms: the metastable ZnSnO_3 phase, known as zinc metavanadate, and the zinc orthostannate Zn_2SnO_4 phase with a stable cubic spinel structure under ambient conditions [11]. The ZTO is a n -type semiconductor that has a good electrical conductivity with, generally, a direct band gap values that varies between 2.8 and 3.8 eV [12] depending on the crystalline structure presented by the synthesized films and the experimental parameters in the synthesis process. Actually, the choice of an appropriate deposition technique can be considered as one of the most decisive factors to observe certain physical properties over other methods. In this context, different techniques (physical, as RF sputtering, [13] and chemical, as spray pyrolysis, [14] methods) had been reported for the development of ZTO. The purpose of this work was the synthesis of (Zn,Sn)O thin films by a simple vacuum thermal evaporation followed by an annealing process and the study of the structural, electrical and optical properties of (Zn,Sn)O material as an alternative Cd-free buffer layer of earth abundant solar cells.

This paper is structured in different sections. The following section explains the different experimental operations adopted in the preparation of ZTO thin films and the different equipment used to characterize their physical properties. The structural, optical and electrical properties of the different ZTO samples are presented in the Result section. The last section is devoted to discussing these results and comparing them with some previous works.

2 Experimental details

2.1 Synthesis

The first step of this work was the deposition of the Sn/Zn metal bilayers through a successive thermal evaporation of Sn and Zn. This step was performed using an Alcatel vacuum group with adapting the Joule effect as the method of evaporation (resistance). In this case, the procedure took place in a vacuum chamber under a low pressure P of 10^{-5} – 10^{-6} Torr, in order to reduce the density of possible contaminants on the surface of the prepared thin films. To assure a good adhesion of the deposited metals, the glass substrates were previously cleaned by a chemical process. To obtain Sn/Zn metallic bilayers, we used 2 procedures. In the first procedure, we evaporated an amount of 0.312 g of tin on unheated substrates (T_s = ambient temperature) using a molybdenum crucible, followed by a second deposition of 0.597 g of Zinc using a tungsten boat. We obtained Sn/Zn bilayers that we named “Samples A”. In the second procedure, we started by the deposition of 0.156 g of tin on unheated substrates, followed by an evaporation of 0.597 g of Zinc. We obtained Sn/Zn bilayers that we named “Samples B”. In order to obtain (Zn,Sn)O films from Sn/Zn metallic bilayers, we used different annealing processes (Table 1). Indeed, we tried to vary the three parameters that usually characterize the annealing process: the temperature, the duration and the atmosphere. Samples (A) were annealed in air atmosphere for 2 h at different temperatures: 400 °C (A1), 450 °C (A2), 500 °C (A3) and 550 °C (A4). For the samples (B), they were divided into two subgroups: B1 and B2. B1 samples were annealed in air atmosphere at 550 °C for 2 and 3 h. The sample B2 was annealed under vacuum at 500 °C for 2 h, followed by another annealing in air atmosphere at 550 °C for 2 h. The annealing treatments were carried out using a programmable tubular furnace type Nabertherm-Germany

Table 1 Experimental parameters and nomenclature of the prepared samples

Evaporation process of Sn/Zn bilayer	Annealing process of Sn/Zn bilayer	Nomenclature of samples
Samples A		
Pressure: 10^{-5} – 10^{-6} Torr	Annealing in air atmosphere at 400 °C for 2 h	A1
T_s : ambient $m_{\text{zinc}}=0.597$ g $m_{\text{tin}}=0.312$ g	Annealing in air atmosphere at 450 °C for 2 h	A2
	Annealing in air atmosphere at 500 °C for 2 h	A3
	Annealing in air atmosphere at 550 °C for 2 h	A4
Samples B		
Pressure: 10^{-5} – 10^{-6} Torr	Annealing in air atmosphere at 550 °C for 2 h and 3 h	B1
T_s : ambient $m_{\text{zinc}}=0.597$ g $m_{\text{tin}}=0.156$ g	Annealing under vacuum at 500 °C for 2 h + in air at 550 °C for 2 h	B2

equipped with a manual gas supply system allowing the gas and the vacuum operations. This furnace offers a great flexibility in the production process of the different thermal profiles. The experimental conditions of the different annealing processes were listed in Table 1. Photographs of some samples were presented in Fig. 1.

2.2 Characterization

The crystalline structure of the obtained films was carried out using PANalytical X'pert Pro diffractometer equipped with a BRAGG BRENTANO geometry goniometer using 1,54060 Å CuK radiation source. The optical analyses were performed using a Shimadzu UV 3100S spectrophotometer equipped with an integrating sphere LISR 3200 to measure the specular or diffuse reflection of the sample, in order to determine the optical transmittance and reflectance of the thin films in UV–VIS–NIR range (from 300 to 1800 nm). Based on these measurements, we were able to define the band gap energy values. For the Impedance spectroscopy analyses were performed using an Hp-4192 impedance analyzer driven by HP VEE (Hewlett Packard Visual Engineering Environment) software. This analyzer is equipped with a programmable oven to vary the working temperature. We have measured the impedance of ZTO thin films deposited on unheated glass for a frequency range between 5 Hz and 13 MHz. Before any measurement impedance, two electrodes (very thin copper wires) were attached to the surface of the samples to collect surface charges and, finally, the conductivity types of the thin films were determined using the hot probe technique.

3 Results

3.1 Structural properties

Figure 2 displays the XRD patterns of Sn/Zn annealed in the air atmosphere at different temperatures for 2 h (Samples A). As is seen, all samples showed a

Fig. 1 Photographs of thin films of **a** Sn/Zn bilayer **b** sample A4 **c** sample B1 and **d** sample B2

polycrystalline structure. The peaks verified the existence of the metallic elements, Zn (JCPDS #. 00-004-0831) and Sn (JCPDS #. 01-086-2264), with the formation of the ZnO oxide phase with the absence of other possible oxides such as SnO_x or ZnSnO. The intensity of the peaks varied according to the variation of the annealing temperature with no formation of a new phase. The highest intensity peak for films annealed at 400 and 450 °C followed preferentially the principal plane (101) of the ZnO phase. The same results were observed with the existence of Zn, Sn, ZnO and even the SnO in the as-deposited ZTO thin films prepared by R. Ramarajan et al. [15], using the same concept as the current work. However, the films were realized by an alternating thermal evaporation at the substrate temperature 350 °C of Sn and Zn in the sequence Sn10/Zn15/Sn15/Zn15/Sn5.

In order to reduce the influence of the metal in the films, we reduced the amount of Tin used in the synthesis of the Sn/Zn metallic bilayers (Samples B). After annealing in air at 550 °C for 2 h and 3 h (samples B1), the color of these films turned from the grey dark color to white (Fig. 1). This change in the color was explained by the XRD pattern that revealed a different structure from the previous ones. Indeed, Fig. 3 showed the presence of (100), (002), (101), (102) and (110) peaks that belong to the hexagonal wurtzite ZnO oxide phase (JCPDS #00-080-0075). Indeed, Fig. 3 showed the presence of (100), (002), (101), (102) and (110) peaks that belong to the hexagonal wurtzite ZnO oxide phase (JCPDS #00-080-0075). No other phases such as Zn or Sn were detected, even with the increase in the annealing duration from 2 h to 3 h. However, the increase of the annealing time affected the peak intensity by making them more intense (Fig. 3). To better understand this modification in the intensity of the peaks on the microstructure of the ZnO phase, we tried to determine the crystallite size of the highest peak (101) using the Debye–Scherrer formula [10]:

$$D = 0.9\lambda / \beta \cos\theta \quad (1)$$

where D denotes to the crystallite size, β is the FWHM, λ is the X-ray wavelength ($\lambda = 1.54060$ Å) and θ is the position of the diffraction peak considered.

The obtained results showed that the average size of the ZnO crystallites at the surface increases from 31 to 35 nm by increasing the duration of annealing. Yuki Nakanishi et al. [16] fabricated ZTO films by magnetron sputtering using low Sn concentration targets (from 0 to 7 at.%) and had a wurtzite structure of ZnO with an increase in the length of the parameter c of the ZnO. On the contrary, these ZTO films become amorphous when Sn concentrations were increased from 15 to 100 at.%.

Fig. 2 XRD diffraction patterns of Sn/Zn annealed in the air atmosphere at different temperatures for 2 h (Samples A)

Fig. 3 XRD diffraction patterns of Sn/Zn annealed in the air atmosphere at 550 °C for 2 h and 3 h (Samples B1)

Fig. 4 XRD diffraction pattern of Zn_2SnO_4 thin film (sample B2)

Figure 4 displays the XRD pattern of Sn/Zn bilayer annealed under vacuum at 500 °C for 2 h and followed by an annealing in air atmosphere at 400 °C for 2 h. As is seen, the peaks can be indexed as those of Zn_2SnO_4 phase with cubic spinel structure according to the Joint Committee on

Powder Diffraction Standards (JCPDS) file No. 01-074-2184. No other crystalline phases such as tin and zinc metals or ZnO were detected by X-ray diffraction, indicating a total

oxidation of the Sn/Zn bilayer. This result is very important because we succeeded to obtain a polycrystalline phase of (Zn,Sn)O or Zn_2SnO_4 thin film. The lattice parameter a of Zn_2SnO_4 thin film was calculated using the following relation [17]:

$$1/(dhkl)^2 = (h^2 + k^2 + l^2)/a^2 \quad (2)$$

where $dhkl$ is the inter-reticular distance determined by Bragg's equation and h , k , l are the Miller indices of the lattice planes. The calculated value was 8.60 Å.

The formation of the (Zn,Sn)O phase depends on the technique and the experimental conditions of the ZTO deposition. Indeed, ZTO films prepared by alternating vacuum thermal evaporation [15] showed the formation of Zn_2SnO_4 phase after an annealing in air at 650 °C for 2 h with the presence other phases as SnO and ZnO oxides. However, a pure phase of Zn_2SnO_4 was obtained by Sung et al. [18] using RF magnetron sputtering for $T_s = 650$ °C and 750 °C.

3.2 Optical properties

Figure 5 displays the transmittance and reflectance spectra of Sn/Zn annealed in the air atmosphere at different temperatures for 2 h (Samples A). The optical transmittance shows a very low response for all the samples. Indeed, the obtained values do not exceed 0.06%. The reflectance of all samples increases by increasing the wavelengths and the values vary between 20% (in the visible region) and 40% (in the near infrared region). The same results were observed by Acherya et al. [19] for the ZTO films prepared by co-evaporation technique.

Fig. 5 Transmittance and reflectance spectra of Sn/Zn annealed in air atmosphere at different temperatures for 2 h (Samples A)

As is seen in Fig. 6, the transmittance of Sn/Zn annealed in the air atmosphere at 550 °C for 2 h and 3 h (Samples B1) was improved compared to the transmittance of samples A. Indeed, in the near infrared region, a value of 60% was achieved. The reflectance reached a maximum of 50% in the visible region (at 450 nm) for the sample annealed for 3 h. The same was observed in Ref. [17].

Figure 7 displays the transmittance and reflectance spectra of Zn_2SnO_4 thin film (sample B2). It is clear that the vacuum annealing improved the transmittance. Indeed, the value was important in the transmittance. Indeed, the value was important in the transparency region (about 80%). This value is higher than the value 40% obtained by Ramarajan et al. [15] and close to the values obtained

Fig. 6 Transmittance and reflectance spectra of Sn/Zn annealed in the air atmosphere at 550 °C for 2 h and 3 h (Samples B1)

Fig. 7 Transmittance and reflectance spectra of Zn_2SnO_4 thin film (sample B2)

by Tang and al. [20]. The optical transmittance of the ZTO films can be enhanced by increasing the annealing temperature as in the case of the ZTO films prepared by co-evaporation method [19]. We note that no interference fringes were observed, which considered to be an opposite result of ZTO films obtained by RF magnetron sputtering [18] or by chemical techniques as MOCVD [21].

The value of the optical gap energy E_g of Zn_2SnO_4 thin film (sample B2) was determined using the Tauc relation [21]:

$$(\alpha h\nu)^2 = A(h\nu - E_g) \quad (3)$$

In this equation, α is the absorption coefficient, h is the Planck constant, ν is the frequency of the incident beam, A is a constant that depends on the transition probability and E_g is the optical energy gap. Figure 8 shows the curves of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) of the sample B2. The values of E_g are determined by the intercept of the extrapolation to zero absorption with the horizontal photon energy axis. The band gap energy E_g of the Zn_2SnO_4 thin film was 3.20 eV. This value is lower than the value 3.40 eV obtained by Ramarajan et al. [15, 22].

3.3 Electrical properties

In this part, we only focused on the determination of the electrical properties of the Zn_2SnO_4 film (sample B2) by impedance spectroscopy and the hot probe method. Figure 9 shows the complex impedance spectra of the Zn_2SnO_4 thin film in the temperature range of 593 to 633 K. It is also observed that the diameters and the maximum of the circular arcs decrease by increasing the

Fig. 8 Plot of $(\alpha h\nu)^2$ versus $h\nu$ for Zn_2SnO_4 thin film (sample B2)

Fig. 9 Nyquist diagram of Zn_2SnO_4 thin film (sample B2) and its equivalent circuit

temperature. These circular arcs can be modeled by an electrical circuit equivalent to a resistance R in parallel with a capacity C [23].

Figure 10 a shows the variation of the imaginary part (Z'') of the impedance as a function of pulsation at different temperatures. These spectra have a single maximum peak that presents a maximum pulsation (ω_m) which varies with temperature. From this variation, we can determine the activation energy characterizing this semiconductor. Indeed, the activation energy E_a is the energy necessary to move the charge carriers from one level (site) to another. This energy was determined experimentally by studying the variation of $\ln(\omega_m) = f(1000/T)$. This variation leads to a linear function (Fig. 10b) in good agreement with the Arrhenius law [23]:

$$\omega_m = \omega_0 \exp(-E_a/K_B T) \quad (4)$$

where ω_0 is the pulsation constant, K_B is the Boltzmann constant and T is the temperature. The estimated value of the activation energy E_a of the Zn_2SnO_4 film was 1.06 meV. The conductivity type was determined by the hot probe method. Zn_2SnO_4 film showed n -type conductivity. This result reveals that the electrons are the majority carriers in these films as mentioned in the literature [22].

Fig. 10 Electrical properties of Zn_2SnO_4 film (sample B2): **a** variation of Z'' as a function of $\ln(\omega)$ for different temperatures and **b** variation of $\ln(\omega_m)$ as a function of $1000/T$

4 Discussion

Our first goal of this study was the synthesis of earth abundant and non toxic Zn_2SnO_4 film for photovoltaic applications. Starting from Sn/Zn bilayers deposited by sequential thermal evaporation, we tried different annealing processes to achieve our first goal. In the first experiment, we annealed Sn/Zn bilayers in air atmosphere at different temperatures (samples A). According to the XRD results, we obtained a mixture of Sn, Zn and ZnO phases. The presence of ZnO oxide suggested that there was a partial oxidation at the surface of the films, but not a complete oxidation of Sn and Zn metals. This result is confirmed by the optical properties of samples A. Indeed, the almost zero transmittance and the significant reflectance (20–40%) proved that samples A showed a metallic behavior. It is also clear that Zn was more easily oxidized than Sn.

So, in the second experiment, we diminished the amount of tin in the Sn/Zn bilayer and we carried out an annealing in air atmosphere at 550 °C for 2 h and 3 h (samples B1). The obtained films showed the presence of the ZnO phase without any detection of un-oxidized element like Sn or Zn and provided a better transmittance due to a better and total oxidation of Zn on the surface of the films (the color turn from dark to white). In an attempt to obtain a better result, we increased the duration of annealing temperature. We remarked an insignificant effect on transmittance with an enhancement in the crystalline quality of the film confirmed by the increase of the crystallite size of ZnO oxide. According to the literature, the absence of SnO or any structure of ZnSnO compound can be explained by the fact that these ZTO films may suffer from the insufficient temperature applied on the film or the substrate, since the films were deposited on unheated substrates, this may be the reason to prevent the growth of ZnSnO_3 or Zn_2SnO_4 films [16]. The presence of only the ZnO phase may be due to the migration of zinc atoms to the surface of the films while the Sn atoms remain at the bottom (near the substrate) which favors the oxidation of Zn instead of Sn. In addition, the ionization energy of Zn is less than Sn, thus, Zn is more easily oxidized than Sn [24]. Based on the previous experiments, we concluded that we need to enhance the inter-diffusion of the Sn/Zn bilayer in order to form a homogeneous mixture of Sn/Zn which can facilitate the formation of $(\text{Zn},\text{Sn})\text{O}$ film. In consequence, we started by annealed the bilayer Sn/Zn in vacuum at 500 °C for 2 h before the oxidation. As a result, we succeeded to synthesis a pure phase of Zn_2SnO_4 . This result leads to an important conclusion: the annealing in vacuum of the bilayer Sn/Zn, before the oxidation in air atmosphere is necessary to form pure Zn_2SnO_4 films. The formation of the phase Zn_2SnO_4 instead of ZnSnO_3 , may be due to the thermal stability of each phase. Indeed, Sung et al. [18] explained that the Zn_2SnO_4 phase is more thermally stable than ZnSnO_3 . We note that the coexistence of the two phases, ZnSnO_3 and Zn_2SnO_4 , was reported in [25].

The second goal of this study was the study of the properties of earth abundant and non toxic Zn_2SnO_4 film as an alternative buffer layer for solar cells. High optical transmittance and adequate optical band gap (between 3 and 3.25 eV) are the most important criteria to consider in choosing a buffer layer. Indeed, we must minimize the short wavelength light absorption by letting a maximum amount of light passing through and thereby reducing the blue absorption losses [26]. This will increase the number of incident photons absorbed by the absorber layer thereby improving the short circuit current and the quantum efficiency of the solar cell [27]. In this work, we obtained a transmittance of 80% and an energy band gap of 3.20 eV. These values match well with the buffer layer

requirements. The absence of interference fringes in the optical spectrum may due to the inhomogeneity of the obtained surfaces.

Many authors [10, 19] studied the electrical properties of ZTO films. However, studying the electrical properties of the Zn_2SnO_4 film using the impedance spectroscopy was done for the first time in this work. The complex impedance spectra obtained have simple circular arcs referring that the conduction process in Zn_2SnO_4 film was carried out by the grains. We also found that the resistance decreases with increasing the temperature, meaning that the conduction mechanism in the film is thermally activated [23] and the film has a semiconducting behavior. Zn_2SnO_4 film showed n -type conductivity which is primordial property because buffer layers must have n -type electrical conductivity to form the p - n junction with the p -type absorber [28]. In conclusion, we can conclude that Zn_2SnO_4 film has a high potential to substitute the most used buffer layer CdS.

5 Conclusion

Our two goals in this work were the synthesis and the study of Zn_2SnO_4 thin film as a possible candidate as buffer layer in thin film solar cells. We successfully developed the zinc orthostannate phase Zn_2SnO_4 by a sequential vacuum thermal evaporation of Zn and Sn metallic elements followed by an annealing process under vacuum and in air atmosphere. Our study highlighted the promising prospects and the potential of the Zn_2SnO_4 material. Indeed, this material based on abundant and non-toxic elements, showed a high optical transmission (about 80%), a wide direct band gap energy of 3.20 eV and n -type conductivity. These properties represent the most important properties in choosing a compound to be a suitable buffer layer in solar cells. In forthcoming work, we will further investigate Zn_2SnO_4 films (morphological, mechanical and compositional properties) and we will try to enhance the crystalline quality of Zn_2SnO_4 by optimizing the experimental conditions.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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