A new route for the synthesis of Sn$_3$Sb$_2$S$_6$ thin films by chemical deposition

Una ruta nueva para la síntesis de películas delgadas de Sn$_3$Sb$_2$S$_6$ por depósito químico

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Abstract

In this work, SnS-Sn$_3$Sb$_2$S$_6$ stack films were formed by sequential chemical deposition, and then they were annealed in a nitrogen atmosphere to synthesize Sn$_3$Sb$_2$S$_6$ thin films successfully. The structural and optical properties were studied by X-ray diffraction and transmittance and reflectance. All the samples show a high absorption coefficient of $> 10^5$ cm$^{-1}$ in the visible region. Their optical bandgap and refractive index are between 1.6-1.8 eV and 3.00-2.71, respectively, which decrease with the increase of film thickness. The electrical conductivity is in the range of $10^{-8}$ to $10^{-7}$ Ω$^{-1}$ cm$^{-1}$. The light-generated current density ($J_L$) is presented as a function of Sn$_3$Sb$_2$S$_6$ film thickness when exposed to air mass 1.5 global (AM1.5G) and solar radiation intensity of 1000 W/m$^2$. In short, Sn$_3$Sb$_2$S$_6$ thin films obtained via the proposed new route exhibit appropriate properties for solar cell applications.

Keywords: Sulfosalts thin films, chemical deposition, Sn$_3$Sb$_2$S$_6$, thermal annealing process.

1 Introduction

In the last years, the sulfosalts layers have attracted attention due to their good optoelectronic properties, which give them a great potential to be used in solar cells, sensors, and thermoelectric energy conversions (Boldish and White, 1998; Dittrich et al. 2009; Gassoumi, A., Kanzari, 2011; Musgraves et al., 2011; Ben Rabeh et al., 2015). The sulfosalts can be defined as IV-V-VI ternary compounds. Additionally, quaternary and more complex compounds can be obtained by introducing transition metals through coupled isoelectronic substitution (Dittrich et al., 2007). Among the materials investigated, tin antimony sulfide (TAS) thin films have a significant interest due not only because they have a band gap energy from 1.2 to 2.5 eV with absorption coefficient above $10^7$ cm$^{-1}$, appropriate for photovoltaic and photonic applications, but also for its abundance in nature, non-toxic and low-cost constituents, and reasonably good performance (Khan et al., 2016; Aousgi and Kanzari, 2011; Khemiri et al., 2018; Abdelkader et al., 2014; Drissi et al. 2013; Ali et al., 2017; Tlig et al. 2017).
The TAS materials have prepared by different methods such vacuum evaporation process, thermal evaporation, electro-pyroelectric technique, pulsed laser deposition, electron beam, among others (Abdelkader et al., 2016, Abdelkader et al., 2018; Mellouki et al., 2018; Jebali et al., Chalapathi et al., 2018; 2016; Dittrich et al., 2009; Bennaji, et al. 2019, Gassoumi et al., 2015). However, chemical bath deposition is simple method widely used to obtain semiconductor thin films on substrates due to some advantages, such as large deposition area, reproducibility, low cost equipment and low temperature processes (Lee et al., 2008 and 2009; Hodes, 2003 and 2007). In addition, combinatorial techniques have also been contributed to enhance part to obtain an appropriate semiconductor (Ali et al., 2016; Ovando-Medina et al., 2018).

Recent studies have been focused in the ternary compound Sn$_3$Sb$_2$S$_6$ because its thin films have shown a good transmission, strong absorption coefficients, a band gap energy close to the theoretical optimum for efficient conversion of solar radiation into electrical power, and a resistive hysteresis behavior (Larbi et al., 2014a; Fadhli et al., 2016). These properties confer to this material great importance in photovoltaic, optical storage, and solar applications. Thin films of Sn$_3$Sb$_2$S$_6$ were deposited by vacuum thermal evaporation (Larbi et al., 2014a and 2014b). The authors found that the optical band gap varies from 1.18 to 1.75 eV, and the absorption coefficients were in the range of 10$^4$ and 10$^6$ cm$^{-1}$, as a function of thicknesses and temperature substrates. Also, the films shows preferential growth in the (416) plane. The same authors synthesized Sn$_3$Sb$_2$S$_6$ thin films by an oblique angle deposition method, obtaining a direct band gap in the range of 1.44 eV to 1.66 eV and absorption coefficients from 10$^5$ to 10$^6$ cm$^{-1}$ (Larbi et al., 2016). (SnS)$_{}$ nm(Sb$_2$S$_3$)$_{}$ thin films, including Sn$_3$Sb$_2$S$_6$, were prepared by thermal evaporation using the glancing angle deposition technique (Abdelkader et al., 2018), the Sn$_3$Sb$_2$S$_6$ thin films showed direct band gap of 1.67 eV, absorption coefficients of 10$^5$ cm$^{-1}$ and photocurrent of 25 mA/cm$^2$ for the sample with a thickness of approximately 250 nm. Bennaji et al. (2019) obtained Sn$_3$Sb$_2$S$_6$ thin films via vacuum evaporation process followed by annealing process. They noticed that thermal conductivity and heat capacity values of the thin films increased by increasing annealing temperature, and the conductance was thermally activated, with an activation energy of about 0.813 eV. However, although thermal, electrical, and optical properties of Sn$_3$Sb$_2$S$_6$ thin films prepared by vacuum thermal evaporation and oblique angle deposition method were characterized over six years ago, little attention has been paid to obtain these thin films via CBD technique. For this reason, this work shows a new route for the synthesis of Sn$_3$Sb$_2$S$_6$ thin films using successive chemical deposits of Sb$_2$S$_3$ onto SnS thin films with a subsequent thermal annealing process. The chemical composition and the structural, electrical, and optical properties of these thin films are presented.

2 Materials and methods

2.1 Thin films deposition

Glass/SnS-Sb$_2$S$_3$ thin film stacks were prepared with two successive chemical bath deposition. First, SnS thin films were grown in corning glass following the methodology proposed by Nair et al. (2016). The deposition was carried out during 1 h 30 min, 3 h, and 4 h 30 min at 25 °C, which generates film thicknesses of 30, 50, and 70 nm, respectively. Afterward, the Sb$_2$S$_3$ thin film was stacked by deposition for 1 h 30 min, forming a thickness of this film of 250 nm, according to the procedure described by Rodríguez-Lazcano et al. (2005). Finally, the thin film stacks were treated by thermal annealing process for 30 min in N$_2$ atmosphere at 300 °C and 10 Torr to synthesize the Sn$_3$Sb$_2$S$_6$ thin films.

2.2 Characterization

The chemical composition of Sn$_3$Sb$_2$S$_6$ thin films was characterized in an Oxford X-act energy dispersive X-ray spectrum (EDX) analyzer attached to a Hitachi-SEM SU1510. X-ray diffraction (XRD) patterns were recorded on a Rigaku Ultima IV diffractometer with Cu-Kα (1.5406 Å) radiation at an incidence of 0.5, 1.5, and 2.0° with the sample plane. Such grazing incidence XRD (GIXRD) study helps to assess the structure and composition of the film along with its thickness.

The photocurrent response of the films was examined using a Keithley 230 programmable voltage source and Keithley 619 electrometer. The electrical measurements were performed using two silver-printed-electrodes of 5 mm long at 5 mm separation applied on the film surface and dried at 60 °C for 30 min. The current (I) in the films was recorded at each
0.5 s for the first 20 s in the dark to ensure the sample stabilization, the next 20 s under illumination and for the last 20 s after switching off the illumination, with a bias (V) of 100 V across the electrodes (established previously as ohmic region). A tungsten-halogen ELH lamp provides an intensity of 1000 W/m² on the plane of the film. Electrical conductivity (σ) of the Sn₃Sb₂S₆ thin films was estimated from the current and voltage values, the electrode geometry, and the film thickness.

Optical transmittance (T) and specular reflectance (R) spectra of the films were recorded for film-side incidence with air and a front aluminized mirror as references on a UV-VIS-NIR V-670 JASCO spectrophotometer. The optical absorption coefficient (α) was calculated with T and R values, considering multiple reflections within the thin films.

3 Results and discussion

3.1 Formation of ternary thin films

The conditions of successive deposition of Sb₂S₃ on SnS thin films previously grown onto a corning glass substrate, both by chemical bath deposition, allowed the formation of Sn₃Sb₂S₆ thin films. This fact was corroborated by EDX and XRD techniques, as shown in the following sections.

Starting from a 250 nm film of Sb₂S₃ and a SnS film of 30, 50 or 70 nm thickness; and from the mass densities and formula mass of SnS (5.13 g/cm³ and 150.75 g/mol), Sb₂S₃ (4.72 g/cm³ and 339.68 g/mol), and of Sn₃Sb₂S₆ (4.93 g/cm³ and 792.02 g/mol) the thickness of the ternary compound film was estimated as 270, 291 and 312 nm, respectively. The basic stoichiometric reaction for the formation of Sn₃Sb₂S₆ from Sb₂S₃ and SnS thin films that takes place during the thermal annealing process is:

\[ 3\text{SnS}^+ + \text{Sb}_2\text{S}_3 \xrightarrow{\Delta} \text{Sn}_3\text{Sb}_2\text{S}_6 \]  

(1)

3.2 Chemical composition

Figure 1 shows the EDX spectra for SnS (30, 50 or 70 nm) - Sb₂S₃ (250 nm) stack films annealed at 300°C for 30 min at 10 Torr in nitrogen atmosphere. EDX analysis show peaks due to Sb-Lα₁ at 3.61 keV, Sn-Lα₁ and Sn-Lβ2 at 3.44 and 3.90 keV, respectively and S-Kα₁ at 2.31 keV. Also, the Na-Kα₁ and Si-Kα₁ at 1.74 keV identified peaks come from the substrate.

The intensity of Si is higher in figure 1a) because it corresponds to the sample with the smallest thickness. The relative intensity between the peaks of Sn, Sb and S was constant for all three samples, indicating that the atomic percentage is similar.

The elemental analysis by EDX software showed the following percentage of Sn, Sb and S: 2.64, 2.31, and 6.05% for sample with 270 nm of film thickness; 2.97, 1.98 and 6.05% for sample with 291 nm of film thickness; and 3.08, 1.87 and 6.05% for sample with 312 nm of film thickness. This suggests that SnS + Sb₂S₃ stack transforms to Sn₃Sb₂S₆ under annealing conditions. This result is corroborated by XRD analysis in the next section.

3.3 Structural properties

The Grazing Incidence X-Ray Diffraction (GIXRD) analysis was conducted by varying the angle of incidence to analyze the phases present throughout the thickness of the developed samples. The sampling depth (SD) is the depth into the film at which the intensity of x-rays entering the film at a grazing angle δ (angle between the beam and the film surface) drops to 1/e = 0.37 of its initial value. The mass absorption coefficient (μmax), linear absorption coefficient (α), penetration depth (PD), and sampling depth (SD) concerning these angles were estimated following standard procedure (McCandless, 2005) using standard data (Hubbell and Seltzer, 2004). The procedure is described as follow:

- Elemental mass absorption coefficients (μmax) for Cu-Kα radiation (0.00824 MeV) are S, 89.1 cm²/g; Sn, 256 cm²/g; Sb, 270 cm²/g.

![Figure 1. EDX spectrum of Sn₃Sb₂S₆ for thickness of a) 270, b) 291, and c) 312 nm.](image)
Fig. 2. XRD patterns of: a) powder di-

The small crystal size obtained is common in films deposited by chemical deposition technique (Hodes, 2003). These results are shown in Table 1.

In figures 2, 3, and 4 the XRD patterns recorded at 0.5, 1.5, and 2.0 degrees for SnSbS6Sn film of 270 nm recorded at 0.5, 1.5, and 2.0 degrees, respectively. On the other hand, in figures 2d), 3d) and 4d), the contribution of the glass substrate for different incidence angles were: \( S D (0.5^\circ) = 80 \text{ nm}, \ S D (1.5^\circ) = 242 \text{ nm}, \text{ and } S D (2.0^\circ) = 322 \text{ nm.} \)

Refractive index of the substance for the X-ray is approximated to 1 whereas it is 0.9999, mentioned for common semiconductors (McCandless, 2005). This requires \( \delta > 0.3^\circ \) for compound semiconductors to avoid total internal reflection of X-rays at the air-to-

The calculated sampling depths for SnSbS6Sn films at different incidence angles were: \( SD (0.5^\circ) = 80 \text{ nm}, \ S D (1.5^\circ) = 242 \text{ nm}, \text{ and } S D (2.0^\circ) = 322 \text{ nm.} \)

The small crystal size obtained is common in films deposited by chemical deposition technique (Hodes, 2003). These results are shown in Table 1.
Table 1. Grain diameter and texture coefficient for Sn$_3$Sb$_2$S$_6$ thin films.

<table>
<thead>
<tr>
<th>Sample (nm)</th>
<th>Texture coefficient (TC)</th>
<th>Grain diameter (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.5°</td>
<td>1.5°</td>
</tr>
<tr>
<td>270 nm</td>
<td>1.88</td>
<td>1.76</td>
</tr>
<tr>
<td>291 nm</td>
<td>1.73</td>
<td>1.65</td>
</tr>
<tr>
<td>312 nm</td>
<td>1.90</td>
<td>1.75</td>
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</table>

3.4 Electrical properties

Figure 5 displays the photoconductivity response curves of the Sn$_3$Sb$_2$S$_6$ thin films. The thin films were slightly photoconductive. Electrical conductivity ($\sigma$) was estimated from the current and voltage values, the electrode geometry and the film thickness. The dark conductivity calculated were $0.11 \times 10^{-7}$, $0.55 \times 10^{-7}$ and $1.77 \times 10^{-7}$ (\text{\Omega} \text{cm})^{-1} for 270, 291 and 312 nm of film thickness, respectively. The increase in the electrical conductivity values with the thickness is related with the atomic percent of Sn in the chemical composition of Sn$_3$Sb$_2$S$_6$ thin film, as seen in the analysis of the chemical composition in section 3.2. According to our bibliographic search, there are no reports available on regarding the electrical conductivity of Sn$_3$Sb$_2$S$_6$ thin film. The electrical conductivity obtained in this work is hoped because it drops between the reported values for the binary starting materials, $10^{-9}$-$10^{-8}$ and $10^{-6}$ (\text{\Omega} \text{cm})^{-1} for Sb$_2$S$_3$ and SnS, respectively (Rodríguez-Lazcano et al., 2005 and Nair et al., 2016).
3.5 Optical properties

The optical properties were recorded in the range between 500 and 2500 nm by measuring at normal incidence the transmittance \((T)\) and reflectance \((R)\). Figures 6 to 8 depict \(T\), \(R\) and \(T+R\) spectra and optical absorption coefficient \((\alpha)\) versus photon energy \((h\nu)\) and \((ahv)^{2/3}\) versus \(h\nu\) of Sn\(_3\)Sb\(_2\)S\(_6\) thin film developed. The value of \(T+R\) at long wavelength was near 90-100\% in the three samples, indicating no significant energy loss due to reflection. The average value of the reflectance toward the long wavelength for these films was 24.9, 23.6, and 21.0\%, respectively (Fig. 6a), 7a), and 8a)).

To estimate the effective refractive index \((n)\) of the films, the average optical reflectance in the long wavelength region, away from the region of band-to-band optical absorption was taken (Smith, 1978):

\[
n = \left(1 + R^{1/2}\right) \left(1 - R^{1/2}\right)
\]  

(6)

An estimate for the film thickness was calculated by the wavelengths \(\lambda_1\) and \(\lambda_2\) corresponding to the adjacent crest and valley in the reflectance spectrum (Smith, 1978):

\[
d = \frac{\lambda_1 \lambda_2}{2n(\lambda_1 - \lambda_2)}
\]  

(7)

This estimation assumes near-uniform film composition and, therefore, nearly constant refractive index along the thickness of the films, which is expected due to the homogeneity of the Sn\(_3\)Sb\(_2\)S\(_6\) phase. The refractive index decreased as increasing the film thickness from 3.0 to 2.71 (reported in Table 3). This fact may be attributed to the content of Sb and Sn present in the films, i.e. the atomic percent of Sb is higher in the thinnest film. The reported values for the high-frequency relative dielectric permittivity \((\varepsilon_r)\) are 8.35 and 14.0 for Sb\(_2\)S\(_3\) and SnS, respectively (Madelung, 1992). Hence, the \(n\) values calculated \((n = \varepsilon_r^{1/2})\) for the binary starting materials are 2.89 for Sb\(_2\)S\(_3\) and 3.74 for SnS; this implies that the estimated values would be acceptable. The estimated thicknesses from equation (7) were 224, 245, and 265 nm. These values are comparable with 270, 291, and 312 nm obtained from the chemical equations in section 3.1 and cited as film thickness in this work. These values differ by less than 17%.
The following equation (Schroder, 1990): 

\[
\alpha = \frac{1}{d} \ln \left\{ \frac{(1 - R)^2}{2T} + \left[ \left( \frac{(1 - R)^2}{2T} \right)^2 + R^2 \right]^{1/2} \right\}
\]  

(8)

Values of absorption coefficient higher than \(10^5 \text{ cm}^{-1}\) were obtained in the visible region, as can be seen in Fig. (6b), (7b) and (8b). This value is a good result for application in solar cells. The interpolation of these plots toward \(\alpha \rightarrow 0\) in the photon energy \((h\nu)\) axis gives the optical band gap \((E_g)\). To obtain the type of electronic transition which occurs during the optical absorption, \((ah\nu)\) is plotted versus \(h\nu\) as shown in Fig. (6b), (7b) and (8b). The best fit is obtained for \((ah\nu)^{2/3}\) versus \(h\nu\) with a correlation factor of 0.99. From the linear regression analysis, the \(E_g\) values were calculated, which are in the range of 1.63 and 1.80 eV (see Table 2). These values are in agreement with the reported in the literature (Larbi et al., 2014b and Abdelkader et al., 2018). Plots of \((ah\nu)^2\) or \((ah\nu)^{1/2}\) versus \(h\nu\) brings down the correlation factor to 0.96 or less, making the straight-line fit unacceptable, i.e., the linear fit establishes that the direct forbidden electronic transitions take place during the optical absorption (Smith, 1978).

The decrease in the values of \(E_g\) depends on the SnS thickness in the SnSbS6 thin film formation, as seen in the analysis of the chemical composition (see section 3.2), which causes the formation of localized states in the gap of the samples. On the other hand, the slight increase in grain size as the function of the thickness, could be contributing to the reduction of the optical band gap.

### 3.6 Light-generated current density

The absorption coefficient, type and values of \(E_g\) have a direct consequence in the light-generated current density \((J_L)\), which sets the upper limit for the short-circuit current density \((J_{sc})\) when a thin film of a particular semiconductor is used as an absorbent element in a solar cell, the current density generated was calculated by the following equation (Nelson, 2003 and Becerra, 2011):

\[
J_L(\text{mA/cm}^2) = 0.1q \int_{E_{g1}}^{\infty} N_{ph}(h)(1 - e^{-\alpha_1 d}) dE \quad (9)
\]

The optical absorber film in the solar-cell structure sees the incident solar radiation (assumed for calculation as the air-mass 1.5 global spectra: AM1.5G of intensity 1000 W/m2) as a source of photons with its flux density \((N_{ph})\) distributed over the wavelength as in Fig. 9a). Such data were obtained from the Standard Global AM1.5 spectrum Tables (Würfel, 2005), assuming that the thickness of the film is large.

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**Table 2. Estimated thicknesses from reflectance spectra, refractive index \((n)\) and bandgap energy \((E_g)\) for SnSbS6 thin films.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Estimated thickness (nm)</th>
<th>Refractive index ((n))</th>
<th>(E_g) (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>270 nm</td>
<td>224</td>
<td>3.00</td>
<td>1.80</td>
</tr>
<tr>
<td>291 nm</td>
<td>245</td>
<td>2.89</td>
<td>1.64</td>
</tr>
<tr>
<td>312 nm</td>
<td>265</td>
<td>2.71</td>
<td>1.63</td>
</tr>
</tbody>
</table>
Fig. 9. a) Spectral distribution of photon flux density ($N_{ph}$) with wavelength ($\lambda$); b) maximum light-generated current density ($J_{L_{max}}$) and c) light-generated current density ($J_L$) as a function of film thickness.

Hence, depending on the $E_g$, the photon flux density for the wavelength indicated in the shaded region for the semiconductor would produce a current density arising from an electron-hole generation for each absorbed photon of energy-$E_g$. It is also assumed that the electron-hole pairs are separated across the absorber without recombination; multiple exciton generation is ignored. These assumptions help place the $J_{L_{max}}$ values in Fig. 9b) for solar-cell absorbers of Sn$_3$Sb$_2$S$_6$ thin films of 270 nm ($E_g=1.8$ eV, Fig. 6b), 291 nm ($E_g=1.64$ eV, Fig. 7b), and 312 nm ($E_g=1.63$ eV, Fig. 8b) in thickness. Values of $J_{L_{max}}$ of for thickness to 270 nm, 291 nm, and 312 nm of Sn$_3$Sb$_2$S$_6$ are 19.60, 24.13, and 24.43 mA/cm$^2$, respectively, as indicated in Fig. 9b). Fig. 9c) shows the estimates values of $J_L$ for Sn$_3$Sb$_2$S$_6$ thin films developed as a function of absorber film thickness, obtained using $\alpha$ versus $h\nu$ plots of Fig. 6b), 7b), and 8b). Variations of $E_g$ and $\alpha$ with thickness, or of reflection losses at interfaces is not considered. The values for $J_L$ are 13.24, 16.82, and 16.50 mA/cm$^2$ for 270, 291, and 312 nm in thickness, respectively. These results are good since the values are close to those of $J_{max}$ estimated. The use of this material as an absorber in solar cells could generate good short circuit current ($J_{SC}$) values.

Conclusions

It can be concluded that Sn$_3$Sb$_2$S$_6$ thin films were successfully synthesized by chemical deposition route. The films were uniform and had good adherence to the substrate. Only a homogeneous Sn$_3$Sb$_2$S$_6$ phase is formed along with the thickness of the samples, which was corroborated by EDX and XRD analysis. The polycrystalline films show crystal size in the range of 13-15 nm with preferential orientation in the (605) plane, and they have a high absorption coefficient >10$^5$ cm$^{-1}$ in the visible region. The optical bandgap of Sn$_3$Sb$_2$S$_6$ thin films is 1.80, 1.64, and 1.63 for 270, 291, and 312 nm in thickness. The refractive index decreases from 3.00 to 2.71 depending on the content of Sb and Sn present in the films. The electrical conductivity of these films is in the range of 10$^{-8}$ - 10$^{-7}$ $\Omega^{-1}$ cm$^{-1}$. Estimates values of $J_L$ were 12.50, 15.02, and 16.98 mA/cm$^2$ for 270, 291, and 312 nm in thickness, respectively. Therefore, these results confirm that the chemical deposition route is a simple synthesis method for controlling the chemical composition and structure of Sn$_3$Sb$_2$S$_6$ with electrical and optical properties appropriate for solar cell applications.
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