

The Development of Crystalline Sb_2S_3 Thin Films as A Component of The Three-Dimensional (3D) Solar Cells

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Abstract. The paper present the research made in order to develop crystalline Sb_2S_3 thin films by Spray Pyrolysis Deposition technique, SPD. The films were developed by varying the deposition parameters: the deposition temperature, the number of sequences and the type of solvents (water and mixed: water-ethanol). The obtained films were analyzed by XRD, AFM, UV-VIS Spectroscopy and current-voltages (I-V) measurements in dark and under illumination. The results prove the formation of crystalline Sb_2S_3 thin films with proper electrical properties to be used as buffer layer and/ or as absorber material in the 3D cells.

Key words: crystalline Sb_2S_3 thin film, Spray Pyrolysis, buffer layer, absorber material, 3D solar cells.

1. Introduction

As the researches shows, in the field of the three (3D) dimensional solar cells, improvements in the solar conversion efficiency can be possible by using buffer layer materials between the n-type transparent semiconductor and the p-type absorber semiconductor, [1, 2, 3] (for a better alignment of the n and p type semiconductor energy bands) and/ or by finding new absorber materials capable to replace the notorial CuInS_2 p-type material. The best efficiency reported until now are 3D cells made from CuInS_2 absorber material in structures as: transparent conducting oxides

(ITO)/ dense TiO_2 (anatase)/ In_2S_3 (buffer layer)/ CuInS_2 / Au, 7%, [4]; transparent conducting oxides/ dense TiO_2 (anatase)/ nanoporous TiO_2 (anatase)/ Al_2O_3 (tunnel layer)/ In_2S_3 (buffer layer)/ CuInS_2 / Au or Pt, 4%, [5].

This paper is proposed to present crystalline Sb_2S_3 thin film material as a possible buffer layer, Fig. 1 and/or as absorber material for the 3D solar cells, Fig. 2.

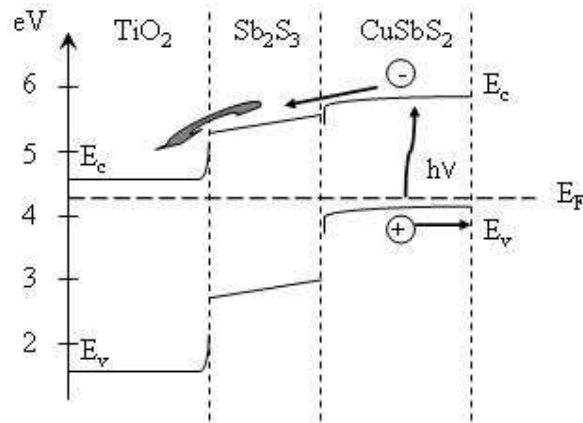


Fig. 1. Energy diagram of a 3D solar cells with Sb_2S_3 as buffer layer and CuSbS_2 as absorber layer.

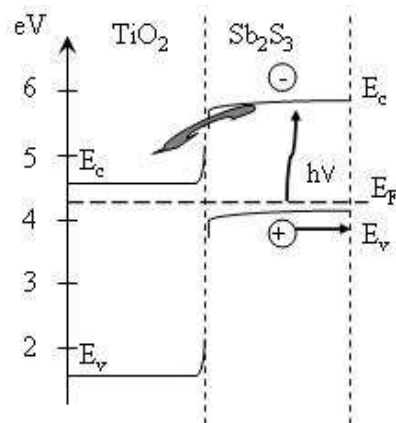


Fig. 2. Energy diagram of a 3D solar cells with Sb_2S_3 as absorber layer.

In the old history of double chalcopyrite materials research, amorphous and crystalline Sb_2S_3 thin films were obtained for different application in: television cameras, microwave devices, switching devices, and optoelectronic devices, using different deposition techniques: electrodeposition, [6], chemical bath deposition, [7, 8], successive ionic layer adsorption and reaction, SILAR, [9], sol-gel process, [10], Thermal evaporation, [11–14], radio frequency sputtering, [15], and spray pyrolysis deposition, [16–20].

The researches published in literature present the development of crystalline Sb₂S₃ thin films with orthorhombic structure, variable band gaps between 1.2 eV and 2.13 eV and n-type semiconductor, [15, 21–25]. In 2005 N. Ticaou *et al.* present the Sb₂S₃ material to be a promising semiconductor material for solar cells due to the high absorption coefficient ($\alpha > 10^3 \text{ cm}^{-1}$) and an optimum band gap (1.8 eV), [11]. The latest researches present Sb₂S₃ as near-intrinsic Sb₂S₃ thin films into a p-i-n solar cell structure: SnO₂:F-(*n*)CdS:In-(*i*)Sb₂S₃-(*p*)CuSbS₂-Ag, [26] and a possible absorber in the development of photovoltaic cells, [27–29]. These results represent a motivation to continue the development of Sb₂S₃ thin films, as important component in 3D cells, using a relative simple and low cost deposition method, suitable for large area thin films deposition and tailoring of structural, morphological and electrical properties as is Spray Pyrolysis Deposition technique, SPD, [30–32].

2. Experimental part

The development of crystalline Sb₂S₃ thin films by SPD have represented and represent a challenge for science; the obtaining of amorphous structures being frequently presented in literature. If until 2002 the depositions of crystalline films were only obtained from non-aqueous solutions, S.R. Gadakh and C.H. Bhosale present for the first time the deposition of crystalline films from aqueous solution rich in complexing agent, [19].

The paper presents the development of crystalline Sb₂S₃ thin films from aqueous and mixed solvent solutions (water-ethanol) using as precursors antimony (III) acetate (CH₃COO)₃Sb, (99.99%, E. Merck, Darmstadt), and thiourea H₂NCSNH₂, (99%, Aldrich Chemical Company, Inc) in weight ratio of 1:2.22. Small amount of HCl are used to increase the solubility of (CH₃COO)₃Sb and to prevent the precipitation of the Sb₂S₃ in aqueous solution by formation of Sb(OH)₃ as white precipitate.

The Sb₂S₃ thin films are deposition on FTO (fluorine doped tin oxide, 5×5 cm² SnO₂:F, TEC 8, Libbey Owens Ford) and FTO/TiO₂ substrates [30], on a heated plate (CERAN 500 ± 1°C), in open atmosphere at the deposition parameters (deposition temperature, T_{dep}(°C), number of sequences, no. sq., and mixed solvent: water-ethanol, H₂O-EtOH) presented in Table 1, having fixed the spray height at 25 cm and the pressure of the carrier gas (N₂) at 1.2 bar.

The Sb₂S₃ thin films deposited on FTO were analyzed using X-Ray Diffraction (XRD, Bruker D8 Advance Diffractometer), Atomic Force Microscopy (AFM, NT-MDT model BL222RNTE), in contact mod, with Si-tip (CSG10, force constant 0.15 N/m, tip radius 10 nm) and UV - VIS Spectroscopy (UV - VIS spectrophotometer Perkin Elmer Lambda 25 UV/VIS).

The Sb₂S₃ thin films deposited on FTO/ TiO₂ structure were analyzed by current-voltage (I-V) measurement recorded in dark and under illumination using an DC Source Meter, Keithley, model 2400 and an calibrated solar simulator SolarConstant 1200 (K.H. Steuernagel Lichttechnik GmbH) as visible light source. Graphite paste (graphite conductive adhesive aqueous based, Alfa Aesar) is used for contacts.

Table 1. The deposition parameters varied in the development of Sb_2S_3 thin films

Test	T_{dep} ($^{\circ}\text{C}$)	No. of sequences	Mixed solvent (H_2O -EtOH)
1	220	16	100% H_2O
2	240		
3	260		
4	260	8	100% H_2O
5		12	
6		16	
7	260	16	10% EtOH
8			20% EtOH

3. Results and discussions

The Sb_2S_3 films were deposited by SPD and varying the deposition parameters in conformity with Table 1. The structural investigations of the films by XRD patterns reveal formation of crystalline, orthorhombic Sb_2S_3 thin films in conformity with JCPDS: 74-1046, Fig. 3 and Fig. 4, and $a = 11.2$, $b = 11.28$, $c = 3.83$.

Figure 3 presents the XRD patterns of the Sb_2S_3 thin films deposited from aqueous solutions at different temperature and show the formation of more crystalline and single phase films with the increasing of the temperature, confirmed by the increased peaks presented for the sample deposited at temperature of 260°C . At temperature below 260°C crystalline Sb_2S_3 films are formed along with $\text{Sb}_{9,8}\text{S}_{15}$.

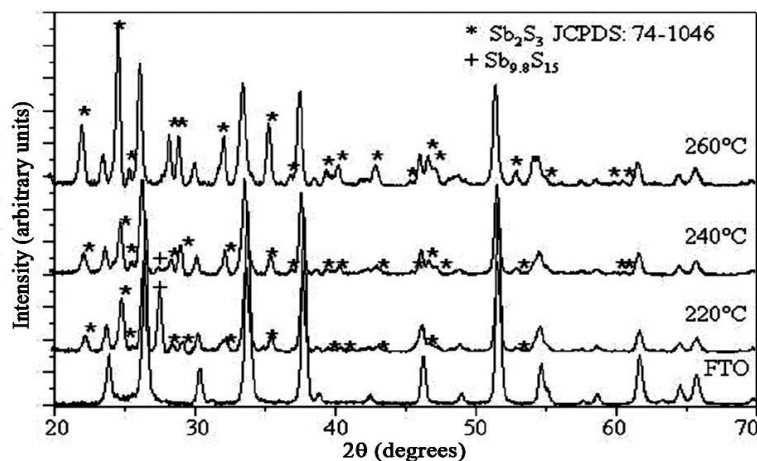


Fig. 3. XRD pattern of Sb_2S_3 thin films deposited on FTO at: a) 220°C ; b) 240°C and c) 260°C .

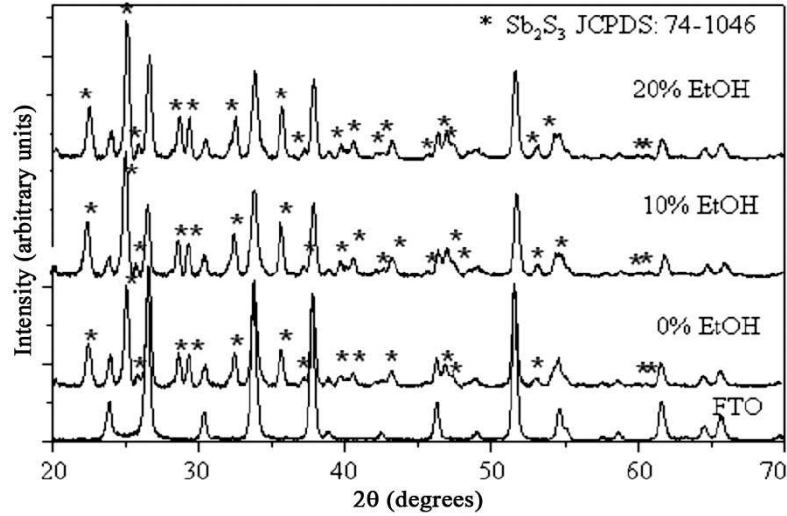


Fig. 4. XRD pattern of Sb₂S₃ thin films deposited at 260°C on FTO from solutions with: a) 0%; b) 10% and c) 20% EtOH.

Figure 4 presents the XRD patterns of the films deposited at 260°C but from sprayed solutions obtained with mixed solvents: water–ethanol. In the development of an efficient 3D cell an important part is the n and absorber semiconductors interface. A good interface led to a good contact on large surfaces and avoids recombination. This can be obtained by tailoring the films morphology and a way to do this is represented by the variation of the alcohol proportion in the aqueous solvent, along to the deposition parameters as was presented in latest research of our group, [30, 31, 33, 34]. Generally, the alcohol in the solvent determines a faster solvent evaporation, a higher reaction rate and formation of denser and homogeneous films as can be seen from the AFM images, Fig. 5 for the Sb₂S₃ films. In the case of Sb₂S₃ thin films the use of mixed water-ethanol solvents in different proportions led to formation of Sb₂S₃ thin films with increased crystallinity, as well as the crystallites size increase and the thickness of the films with the increased of the ethanol proportion in the sprayed solution.

The crystallites size was calculated using Scherrer formula: $D = \frac{k\lambda}{\beta \cos \theta} = \frac{0.9\lambda}{\beta \cos \theta}$ (1), where: λ – the wavelength ($\lambda = 1.54060 \text{ \AA}$), k – shape coefficient (usually is 0.9), β - the full-width at half-maximum of the peak in radian, and θ - the Bragg angle; and the thickness of the films from the UV-VIS Spectroscopy analysis.

The calculation of Sb₂S₃ thin films thicknesses was made from absorption date using the following relation: $a = p \frac{\lambda_1 \cdot \lambda_2}{\lambda_1 - \lambda_2} \cdot \frac{1}{2n_d}$ (2), where λ_1 and λ_2 represent the wavelengths where the absorption has a maximum or a minimum, n_d is the reflective index (for Sb₂S₃ the reflective index is 3), “p” is the number of the waves (between two adjacent maxima, $p = 1$), and “a” is the thickness of the film. The values of crystallites size and film thickness are presented in Table 2 and Table 4.

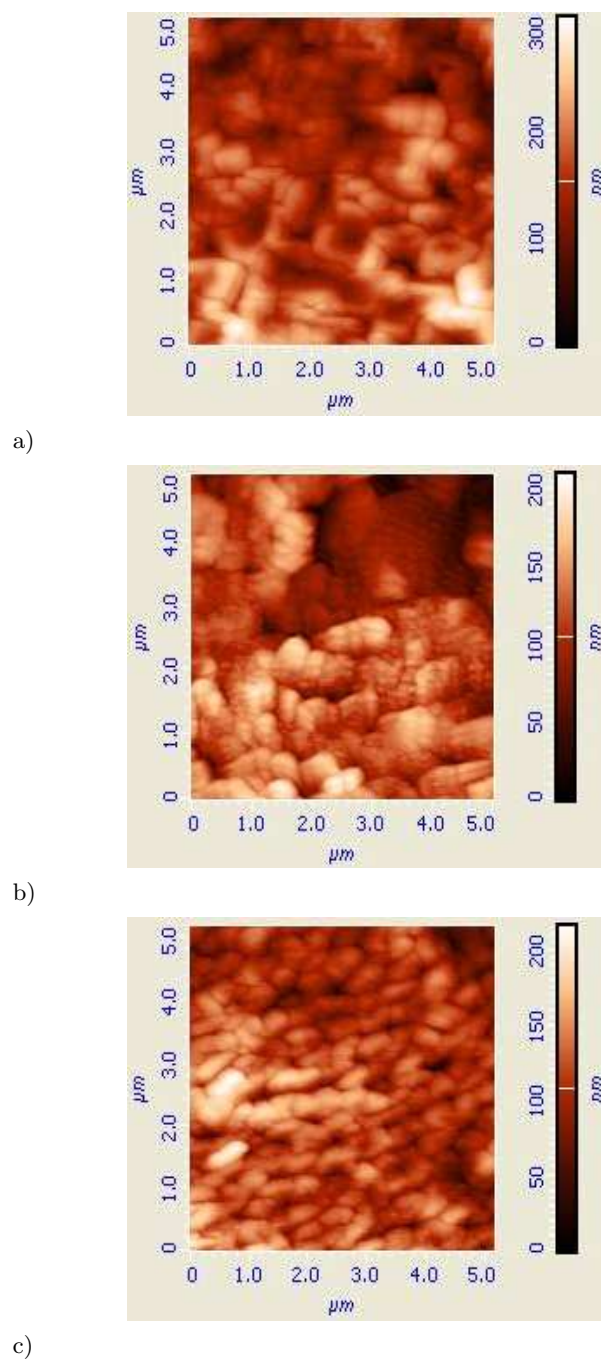


Fig. 5. AFM image of Sb_2S_3 films deposited at 260°C from solutions with: a) 0% EtOH; b) 10% EtOH and c) 20% EtOH.

Table 2. Crystallite size of the crystalline Sb₂S₃ thin films deposited at the varied parameters

T(°C)/ mixed solvent (H ₂ O: EtOH)	(hkl)	D (nm)
220 / 100% H ₂ O	(130)	43.43
240 / 100% H ₂ O	(130)	58.36
260 / 100% H ₂ O	(130)	49.21
260/ 10% EtOH	(130)	43.32
260/ 20% EtOH	(130)	42.18

In order to identify the electrical properties of the obtained Sb₂S₃ films and the photovoltaic response of the developed cells at the varied parameters the current-voltage measurements in dark and under illumination were performed.

The I–V curves recorded in dark show formation of semiconductor Sb₂S₃ films without pinholes and with the best conductivity for the films deposited at 260°C, 16 numbers of sprayings, and aqueous solutions, as can be seen in Fig. 6, Fig. 7 and Fig. 8. This film will be used for next researches in the development of a 3D solar cell with Sb₂S₃ films as buffer layer and CuSbS₂ as absorber layer.

The I–V curves recorded in dark and under illumination for the obtained cells: FTO/ TiO₂ (n-type semiconductor)/Sb₂S₃ (absorber layer, deposited at the varied parameters)/ graphite, show the diode behaviours for the all cells, without shunts and with the best cell characteristics for the cell developed with Sb₂S₃ films at 260°C, 16 numbers of sprayings, with 20% ethanol in the sprayed solution, Fig. 9: $V_{oc} = 264.22$ mV, $I_{sc} = 2.63 \cdot 10^{-6}$ A, $V_{max} = 163.31$ mV, $I_{max} = 1.63 \cdot 10^{-6}$ A and FF = 0.383. The cell parameters for the all cells are presented in Table 3.

In order to find responses in the values of the photovoltaic characteristics, obtained for the developed cells, the band gaps values of the Sb₂S₃ thin films were investigated.

Table 3. Cells characteristics V_{oc} , I_{sc} , V_{max} , I_{max} and FF

T (°C)/ No. of sprays/ mixed solvent	V_{oc} (mV)	I_{sc} (A)	V_{max} (mV)	I_{max} (A)	FF
220 / 16/ 100% water	347.7	$1.49 \cdot 10^{-7}$	203	$8.73 \cdot 10^{-8}$	0.342
240 / 16/ 100% water	216.34	$4.65 \cdot 10^{-7}$	128	$2.84 \cdot 10^{-7}$	0.361
260 / 16/ 100% water	224	$1.69 \cdot 10^{-6}$	133	$1.02 \cdot 10^{-6}$	0.358
260/ 8 / 100% water	174.61	$1.45 \cdot 10^{-7}$	90	$7.83 \cdot 10^{-7}$	0.278
260/ 12 / 100% water	216.46	$4.77 \cdot 10^{-7}$	130	$2.81 \cdot 10^{-7}$	0.353
260/ 16 / 100% water	224	$1.69 \cdot 10^{-6}$	133	$1.02 \cdot 10^{-6}$	0.358
260/ 16/ 10% Ethanol	268.85	$1.99 \cdot 10^{-6}$	165.47	$1.18 \cdot 10^{-6}$	0.365
260/ 16/ 20% Ethanol	26 4.22	$2.63 \cdot 10^{-6}$	163.31	$1.63 \cdot 10^{-6}$	0.383

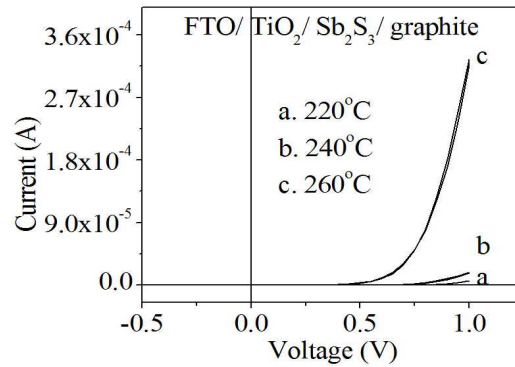


Fig. 6. I-V curves measured in dark of FTO/ TiO₂/ Sb₂S₃/ Graphite structures for Sb₂S₃ films deposited at different temperatures: a) 220°C, b) 240°C and c) 260°C.

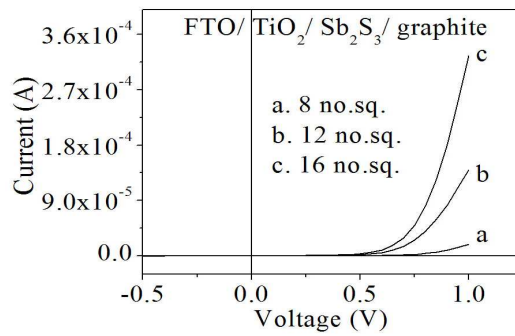


Fig. 7. I-V curves measured in dark of FTO/ TiO₂/ Sb₂S₃/ Graphite structures for Sb₂S₃ films deposited with different number of spraying: a) 8, b) 12 and c) 16 no. sq.

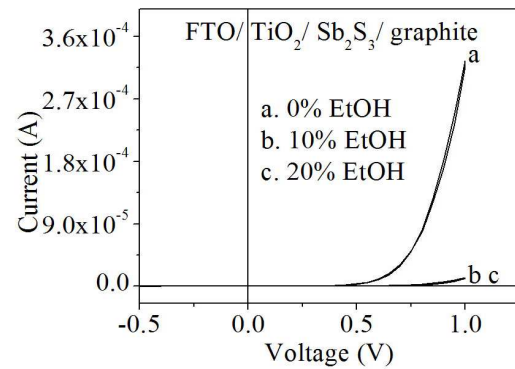


Fig. 8. I-V curves measured in dark of FTO/ TiO₂/ Sb₂S₃/ Graphite structures for Sb₂S₃ films deposited at 260°C from solutions with: a) 0% EtOH; b) 10% EtOH and c) 20% EtOH.

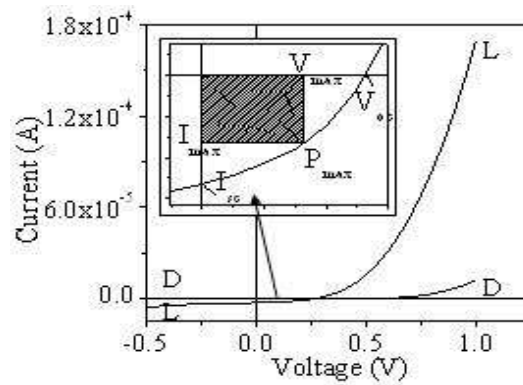


Fig. 9. I-V curves measured in dark and under illumination of FTO/ TiO_2 / Sb_2S_3 (240°C, 16 no.sq., 20% EtOH in the solution)/ graphite.

The band gaps values were determinate from the absorption data, Fig. 10 and the values obtained for the films deposited at 260°C, 16 numbers of spraying from aqueous solution and mixed solvent are presented in Table 4.

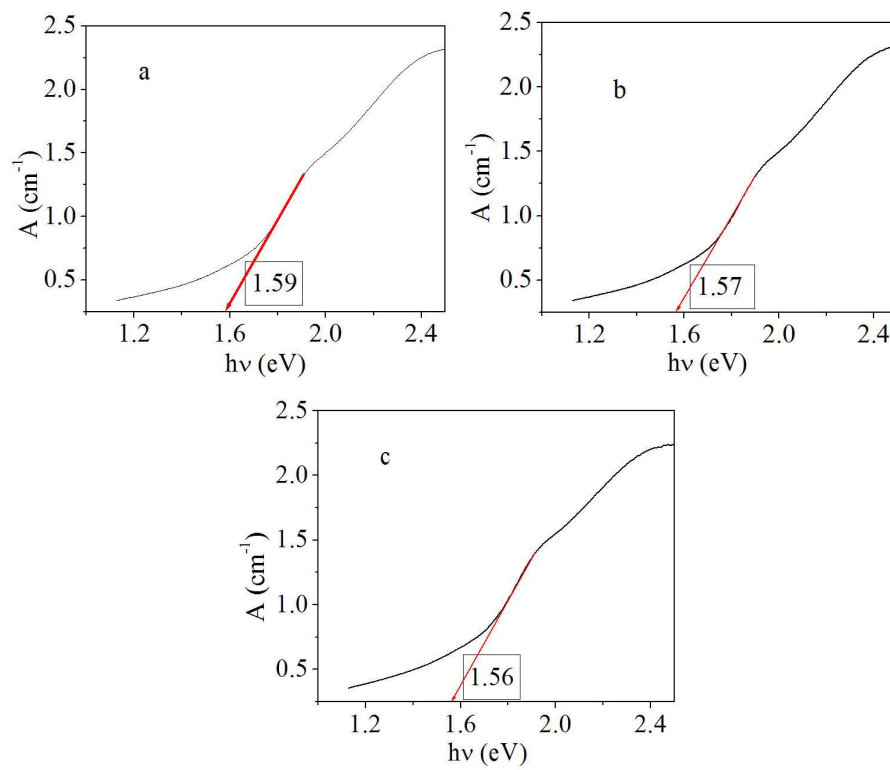


Fig. 10. Band gap energy of Sb_2S_3 thin films deposited at 260°C from solutions with: a) 0% EtOH; b) 10% EtOH and c) 20% EtOH.

Table 4. The crystallite size, the thickness and the band gaps of the crystalline Sb_2S_3 thin films deposited at different ethanol proportion

EtOH (%)	(hkl)	D (nm)	G (nm)	E_g (eV)
0	(130)	49.21	397.56	1.59
10	(130)	43.32	417.77	1.57
20	(130)	42.18	427.45	1.56

The best photovoltaic response obtained for the sample deposited from the sprayed solution with 20% EtOH is the result of a denser film (better $\text{TiO}_2/\text{Sb}_2\text{S}_3$ interface) and of a lower value of the band gap 1.56 eV (value close to the photovoltaic requirement for the absorber materials) that provide a low recombination process and a better injection of the electrons from the conduction band of the Sb_2S_3 semiconductor in the conduction band of the TiO_2 (anatase).

In Fig. 11 is presented a possible energetic diagram for the 3D cell $\text{TiO}_2/\text{Sb}_2\text{S}_3$.

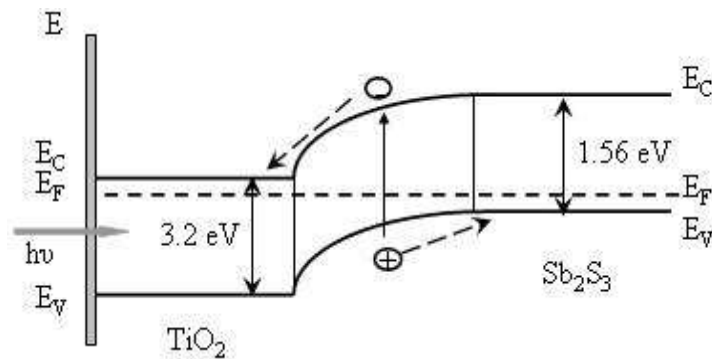


Fig. 11. A possible band diagram alignment for the FTO/ TiO_2 / Sb_2S_3 / Grafit cell.

4. Conclusions

The deposition of crystalline Sb_2S_3 thin films by an easy and cheap technique represents a challenge for the chemistry and material science researchers. This paper has succeeded in presenting a new easy and cheap technique SPD and its capability in development of crystalline Sb_2S_3 thin films from aqueous solution and mixed solvent: water-ethanol solutions using as precursors antimony (III) acetate $(\text{CH}_3\text{COO})_3\text{Sb}$, 99.99%, and thiourea H_2NCSNH_2 , 99% in weight ratio of 1: 2.22. The structural, morphological and electrical properties of the crystalline Sb_2S_3 thin films deposited at the varied parameters proves that this material can be used as possible buffer layer or absorber in the development of a 3D solar cell.

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