Structural, Morphological and Optical Comparison of In-S Films Deposited by CBD and Ultrasonic Pyrolytic Spraying, as a Buffer Layer in CIGS Solar Cells

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ABSTRACT: Indium sulphide (In_2S_3) has positioned itself as an environmentally friendly and efficient option compared to traditional CdS, used as a buffer layer in thin-film solar cells that use Cu(In,Ga)Se_2 (CIGS) as an absorbent material. This study provides a comparative analysis of two techniques for depositing thin films of In_2S_3: chemical bath deposition (CBD) and ultrasonic pyrolytic spraying. Their structural, morphological, compositional, and optical properties were evaluated. The films obtained by pyrolytic spraying, showed adequate crystallinity and uniformity. On the other hand, the films deposited by CBD had better stoichiometry (In2:S3 ≈ 40:60% at.) and higher bandgap values (up to 4.5 eV), which favors optical transmission in the blue region of the solar spectrum. All films produced by both techniques remained within the optimal thickness range (50–100 nm) to function as a buffer layer. In summary, both methods proved to be viable for the fabrication of In_2S_3 layers, with CBD excelling in optical and compositional characteristics, while pyrolytic spraying excels in structural quality and morphology. These findings consolidate In_2S_3 as a promising and sustainable candidate to CdS in the manufacture of thin-film solar cells.

KEYWORDS: Indium sulphide; ultrasonic pyrolytic spraying; chemical bath deposition; CIGS

1 Introduction

Thin-film solar cells using Cu (In,Ga)Se_2 (CIGS) as an absorber have emerged as one of the most promising photovoltaic technologies. It is due to their high conversion efficiency, long-term stability, and ability to be deposited on flexible or low-cost substrates [1]. Traditionally, these cells include a cadmium sulfide (CdS) buffer layer, using a chemical bath deposition (CBD) process. This layer acts as a barrier and helps form an efficient hetero-structural junction between the CIGS absorber and the window layer [2]. However, the use of CdS raises environmental and health concerns due to the toxicity of cadmium, as well as being an optical limitation, since its relatively narrow bandwidth (~2.4 eV) contributes to the absorption of photons in the blue region of the solar spectrum, reducing the current generated [3]. Given these limitations, indium sulfide (In_2S_3) has been the subject of numerous studies as a viable candidate to replace CdS in CIGS cells. In_2S_3 is a semiconductor material with a wider optical bandwidth (greater than 2.7 eV), which allows for better transmission of incident light to the absorber, thus improving short-circuit current (Jsc) performance [4].

In_2S_3 can be found in three different crystalline forms: α (cubic), β (tetragonal and cubic), and γ (hexagonal). Among these phases, β-In_2S_3 in its tetragonal structure stands out for its superior performance, widely used in optoelectronic and photoconductive devices [5]. In addition, In_2S_3 is well-suited to large-scale manufacturing processes, making it a more environmentally sustainable option [6]. Various deposition techniques had used to create thin films of In_2S_3, such as: chemical bath deposition (CBD) [7], ultrasonic pyrolytic spraying [8], thermal evaporation in vacuum [9], low-pressure metal and organic compound chemical vapor deposition (LP-MOCVD) [10], atomic layer deposition (ALD) [11], sputtering [12], and chemical vapor deposition in air (air CVD) [13]; chemical bath deposition (CBD) and pyrolytic spraying

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being two of the most common, due to their simplicity, low cost, and the possibility of scaling up to industrial levels. The CBD method allows for fairly precise control over film thickness and stoichiometry, and is compatible with large substrates at low temperatures [14]. On the other hand, pyrolytic sputtering offers additional advantages, such as the ease of adjusting morphological parameters and the ability to produce uniform films with excellent optical and electrical properties, which are essential for the functionality of the buffer layer [8]. Therefore, replacing CdS with In_2S_3 in CIGS solar cells is not only a key strategy for improving the optical performance of the device, but also responds to the need to move towards cleaner and more sustainable technologies.

This paper focuses on a comparative analysis between two deposition techniques for the manufacture of indium sulfide thin films: (i)—the modified chemical bath deposition (MCBD), which includes rinsing with deionized water every 5 min for 5 s to improve adhesion, and (ii) ultrasonic pyrolytic spraying. The objective is to evaluate the viability of these films as a buffer layer in thin-film solar cells with CIGS as the absorbing material. The results obtained allow us to identify the advantages and limitations of each method, considering their structural, morphological, compositional, and optical properties.

2 Experimental

2.1 In-S deposition by Chemical Bath Deposition (CBD) and Modified Chemical Bath Deposition (MCBD)

The chemical solution used for the preparation of In-S films, employing the modified chemical bath deposition (MCBD) technique, consisted of: 0.1 M indium chloride (III) (Sigma Aldrich 99.999%), 1 M thioacetamide (Sigma Aldrich 99%), 0.5 M acetic acid (J.T. Baker 99.7%), and 0.5 M sulfuric acid (J.T. Baker 97.9%). The modified chemical deposition technique consists of immersing and removing the glass substrates from the chemical solution, alternating these times. The purpose of removing the substrates temporarily is to improve the adhesion of the film. In unmodified chemical bath deposition, the substrates are only immersed and removed until the agreed deposition time. The glass substrates were immersed in 100 mL beakers, with commercial glass substrates having the following dimensions: 2.5 cm wide by 5 cm long. In order to control the temperature, these beakers were immersed in a Science Tech model C130 recirculating bath with temperature control, as shown in Fig. 1.

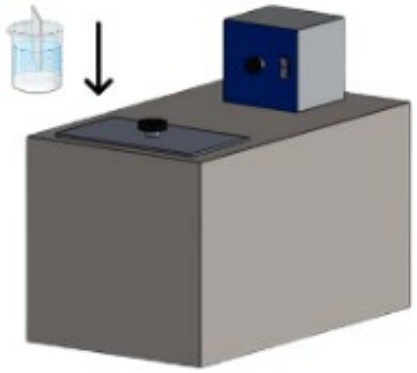


Figure 1: Schematic representation of chemical bath deposition.

The deposition conditions for each of the films are in Table 1.

Table 1: Experimental conditions for the preparation of In-S films using the modified CBD technique.

Sample	Bath Temperature (°C)	Deposition Time (min)	Rinse Every 5 min for 5 s
A	80	35	Not
B	80	35	Yes
C	80	60	Yes

2.2 Deposition of In-S by Ultrasonic Pyrolytic Vaporization

The ultrasonic pyrolytic sputtering technique uses a solution of 0.1 M indium (III) chloride (Sigma-Aldrich 99.999%) and 0.1 M thiourea (Sigma-Aldrich 99.0%) for the preparation of In-S films. The pyrolytic sputtering equipment was manufactured in-house and consists of a Yuehua wh-802 nebulizer and a Velp Scientifica SP311 peristaltic pump, as it shows in Fig. 2. The experimental deposition conditions are in Table 2.

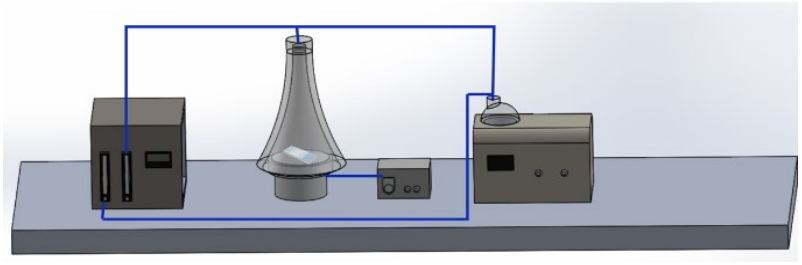


Figure 2: Schematic representation of the ultrasonic pyrolytic spray equipment used.

Table 2: Experimental conditions for the preparation of In_2S_3 thin films using the ultrasonic pyrolytic spray technique.

Sample	Nitrogen Flow Rate (mL/min)	Substrate Temperature (°C)	Time (min)
D	20	500	10
E	20	500	15
F	20	500	25

With the ultrasonic pyrolytic spray technique, the use of temperatures above 450°C favors the formation of the tetragonal phase of β-In_2S_3, which is widely used in optoelectronic applications due to its superior photosensitive capacity [5]. According to the literature, it is more common to obtain the α or β phases in its cubic structure [16–18].

2.3 Characterization

The structural characterization of the materials was performed by X-ray diffraction (XRD) using the powder technique on a RIGAKU Ultima IV model device, with a Cu Kα radiation source (λ = 1.54 Å) and a secondary graphite monochromator. The diffractograms were recorded in the 2θ range from 10° to 80°, with a scanning speed of 0.5°/s. The average crystal size was estimated using the Scherrer equation [8] (Eq. (1)):

$$D = \frac{K\lambda}{\beta \cos\theta} \tag{1}$$

where D is the crystal size, K is the Scherrer constant, λ is the radiation wavelength, β is the full width at half maximum (FWHM) in radians, and θ is the Bragg angle.

The specular reflectance and transmittance spectra were obtained using a Jasco V-670 UV-VIS-NIR spectrophotometer in the range of 2500 to 250 nm. The bandgap energies were calculated using Tauc's equation [19,20] (Eq. (2)):

$$\alpha h\nu = \mathrm{A}(h\nu - \mathrm{Eg})^n \tag{2}$$

where α is the absorption coefficient, $h\nu$ is the photon energy, A is an arbitrary constant, and n = 1/2 for allowed direct transitions. The surface morphology of the films was analyzed by scanning electron microscopy (SEM) and the atomic composition of the films by EDS, using a Hitachi S-500 microscope with a secondary electron detector and a Broker XFlash 5010 energy dispersive X-ray detector. The thin film thickness was measured using an Alpha-step 100 profilometer.

3 Results

3.1 X-Ray Diffraction (XRD)

Fig. 3 shows the X-ray diffractograms for the indium sulfide films for samples A, B, and C, produced by CBD and MCBD, as well as by ultrasonic pyrolytic spray (D, E and F).

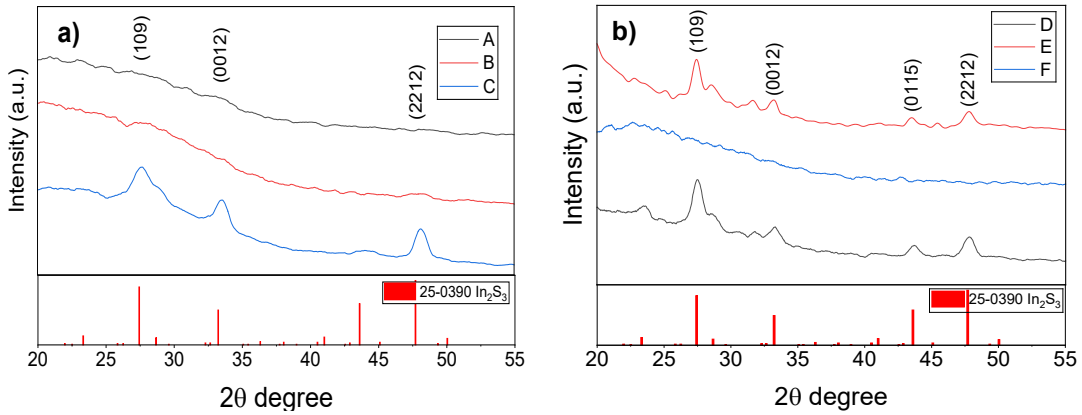


Figure 3: Diffractograms of In-S films deposited by the technique of (**a**) MCBD and CBD and (**b**) ultrasonic pyrolysis spray.

Fig. 3a shows that sample A, produced by the CBD technique, is completely amorphous, as is sample B, which was produced by MCBD using the same deposition time. However, sample C, produced by the MCBD technique but using almost twice the deposition time, shows diffraction peaks associated with PDF 25-0390, which an improvement in crystallinity with the following diffraction peaks: at 2θ = 27°, attributed to the reflection of the (109) plane; another peak at 2θ = 33°, corresponding to the (0012) plane; and another peak at 2θ = 48°, attributed to the (2212) plane. According to PDF 25-0390, the tetragonal structure, the peaks are observed to be broad, which indicates that the material is semi-crystalline. This condition could be improved with the application of heat treatment. Fig. 3b shows the diffractograms for indium sulfide films fabricated by the ultrasonic pyrolytic spray technique. The differences between these samples lie in the deposition time, as shown in Table 2.

Sample F does not show any peaks associated with the formation of In_2S_3, i.e., it is amorphous. However, samples D and E have the following diffraction peaks: at 2θ = 27°, attributed to the reflection of the (109) plane; another at 2θ = 33°, corresponding to the (0012) plane, a peak at 2θ = 43° corresponding to the (0115) plane, and another peak at 2θ = 48°, attributed to the (2212) plane, according to PDF 25-0390, with a tetragonal structure, obtaining β-In_2S_3. Similarly, sample D shows a peak at 2θ = 23°, corresponding to the (116) plane. The crystal size of sample D was 3.85 nm, and that of sample E was 3.08 nm.

Comparing the diffractograms of the In_2S_3 material of the films deposited by the CBD and pyrolytic spray techniques, it is possible to say that pyrolytic spray deposition produces more crystalline films with better-defined peaks. The pyrolytic spray technique produces tetragonal β-In_2S_3, which is a good candidate for use as an n-type film in solar cells using CIGS as the absorber.

3.2 Scanning Electron Microscopy (SEM)

Fig. 4 presents micrographs of In-S films deposited using Chemical Bath Deposition (CBD) and Modified Chemical Bath Deposition (MCBD) techniques (samples A, B, and C), as well as those created by ultrasonic pyrolytic spray (samples D, E, and F). In Fig. 4a, the morphology achieved through the CBD technique is shown, revealing a smooth surface for sample A.

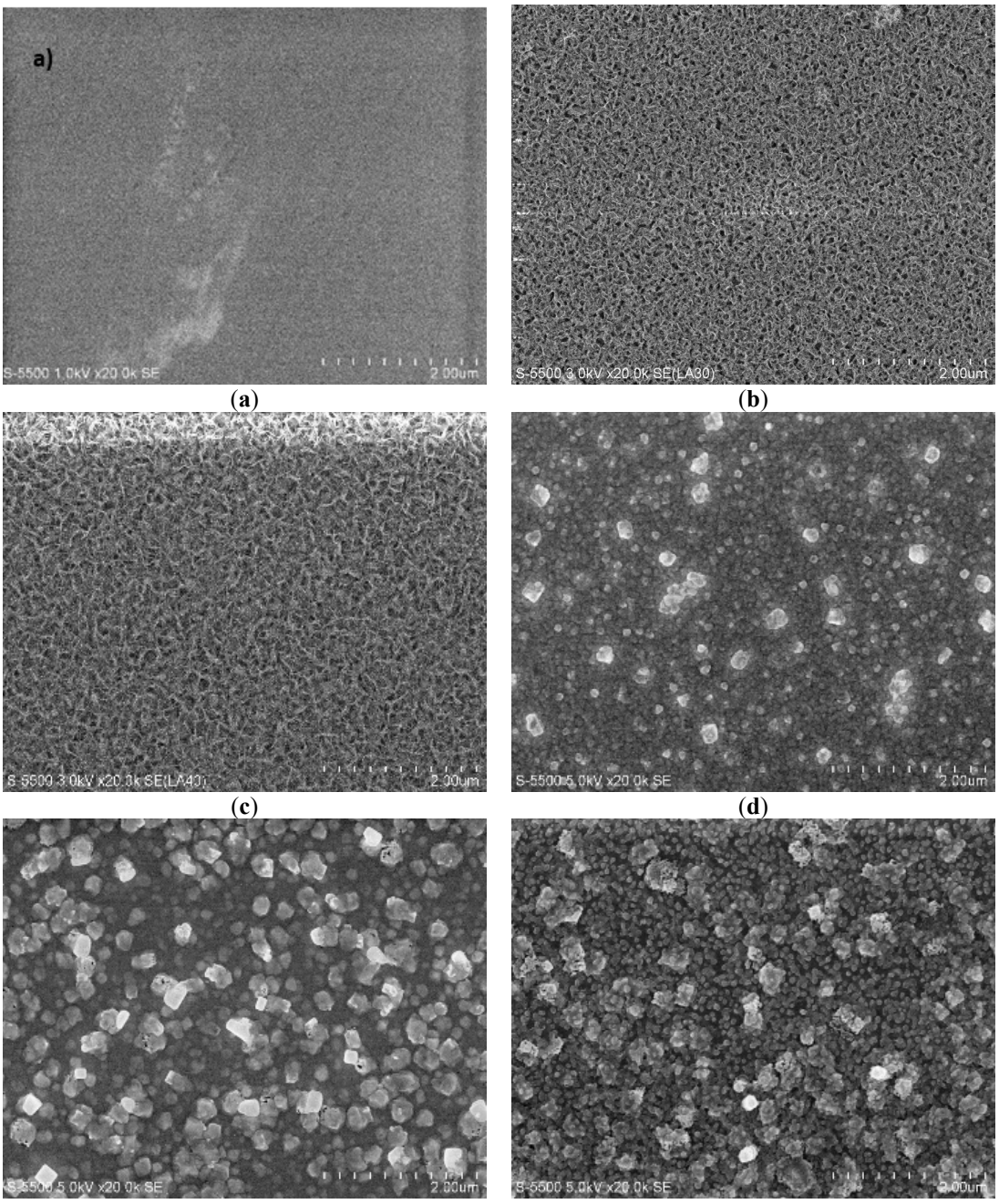


Figure 4: Micrograph of In-S films magnified to 2 μm, deposited by CBD A) and MCBD (B and C), deposited by ultrasonic pyrolytic spray (D, E and F).

In contrast, sample B, obtained via the MCBD technique, displays a rougher texture, indicating evidence of film growth. Sample C (Fig. 4c) exhibits a morphology similar to that of sample B, as previously reported [21]. Both of these samples demonstrate adequate adhesion. For the pyrolytic sputtering samples D, E, and F (Fig. 4d–f), the morphology appears granular with a consistent formation pattern. The average grain size for sample D was measured at 194 nm, featuring separate and dispersed grains. Sample E displayed a greater presence of round grains with an average size of 203 nm, which were more homogeneously distributed, whereas sample F had the largest grain size at an average of 220 nm. This granular morphology aligns with observations made in prior studies [17]. Overall, the studies indicate that the morphology and

adhesion qualities of films produced by pyrolytic sputtering are superior to those obtained through CBD and MCBD techniques.

3.3 Atomic Composition (EDS)

Table 3 highlights the atomic composition of films deposited by Chemical Bath Deposition (CBD) and Modified Chemical Bath Deposition (MCBD) (samples A, B, and C), as well as those produced through the ultrasonic pyrolytic spray technique (samples D, E, and F), analyzed via Energy Dispersive Spectroscopy (EDS). Additionally, the thickness of each film was precisely measured using a profilometer. Remarkably, sample C exhibits an atomic composition that approximates the ideal stoichiometry for Indium Sulfide (In_2S_3), positioning it as an excellent choice for a buffer layer. The stoichiometric ratio of Indium (40% atomic) to Sulfur (60% atomic) not only facilitates superior band alignment with Copper Indium Gallium Selenide (CIGS) but also significantly reduces recombination at the interface, enhancing overall optical-electronic performance. Moreover, sample F, produced through pyrolytic spraying, demonstrates equally favorable atomic percentage values.

Table 3: Atomic composition of In-S films deposited by CBD, MCBD (A,B, and C), and by ultrasonic pyrolytic spray (D, E, and F) and their thicknesses.

Sample	Atomic Percentage (%)		Thickness (nm)
	In	S	
A	64.2	35.8	60
B	49.5	50.5	51
C	*39.9*	*60.1*	55
D	51.7	48.3	62
E	50.2	49.8	75
F	28.4	71.6	87

In contrast, samples A, D, and E reveal a higher indium content compared to Sulfur, while sample B achieves a balanced 50:50 ratio. These differences linked to the diverse experimental conditions employed during the deposition process. Furthermore, Table 3 illustrates the thicknesses of the samples, showing that films deposited by CBD are thinner than those from pyrolytic spraying. Notably, samples D, E, and F exhibited increased thickness with longer deposition times. Importantly, all samples achieve the ideal thickness range of 50–100 nm, making them perfectly suited for application as buffer layers in thin-film solar cells that utilize CIGS as the absorber.

3.4 Optical Characterization and Band Gap

The figure shows the transmittance and specular reflectance spectra of In-S films deposited on glass using the CBD, MCBD, and ultrasonic pyrolytic spray techniques. Fig. 5a shows the transmittance values for sample A are slightly higher than those for sample B in the visible region, which is probably due to the lower thickness of sample A, likely associated with the deposition technique. However, sample C behaves similarly to sample B, and the transmittance values are lower for the entire spectrum, indicating a greater thickness in the latter sample. Fig. 5c shows the transmittance values for samples D, E, and F synthesized by ultrasonic pyrolytic spray. These values show similar behavior across the entire spectrum and decrease as the deposition time increases. Similarly, as the deposition time increases, the specular reflectance values shift to the right, allowing for a decrease in the visible region. Fig. 6 shows the different graphs obtained by applying the Tauc method to the transmittance and specular reflectance spectra for each of the samples, produced by CBD, MCBD, and ultrasonic pyrolytic spray deposition.

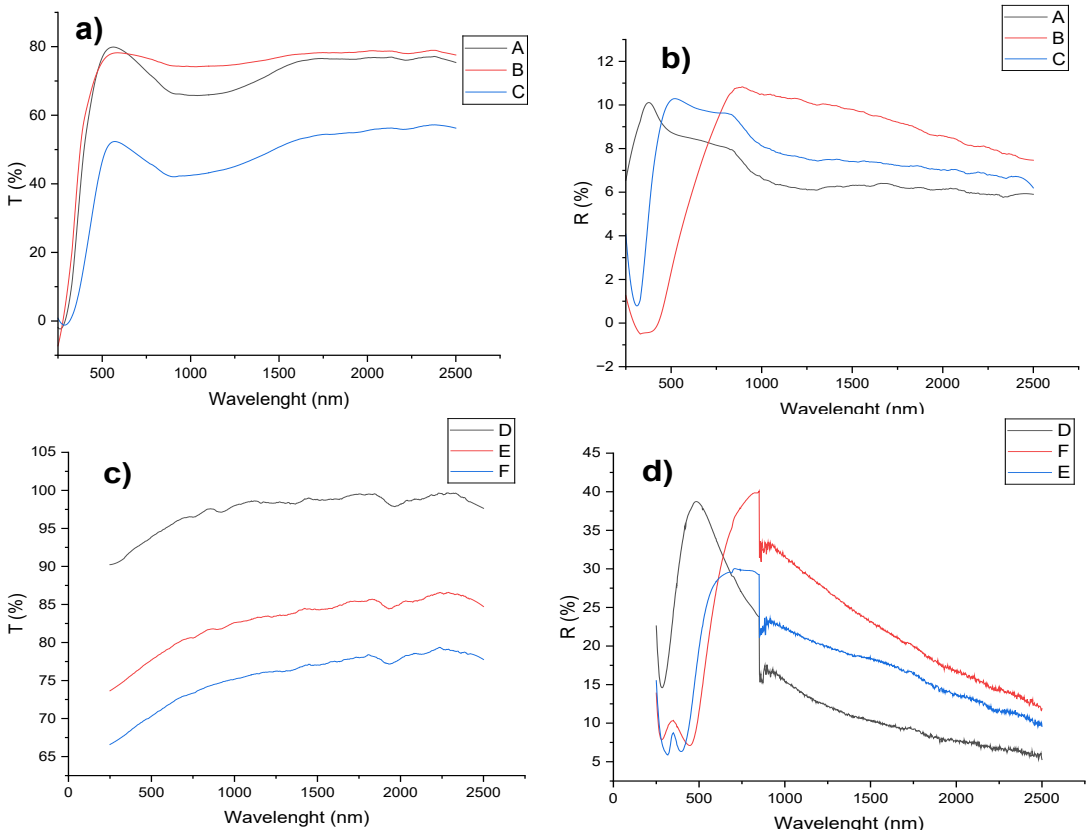
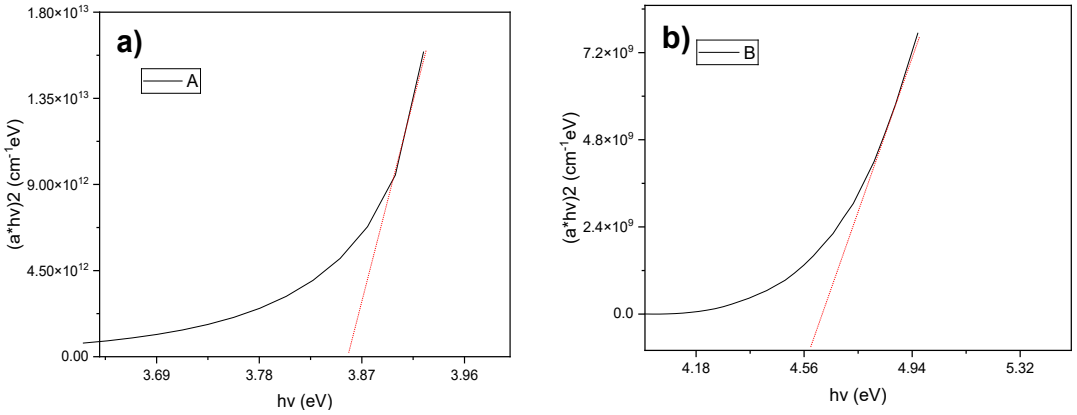


Figure 5: Transmittance and reflectance spectra of In_2S_3 films, deposited by CBD and MCBD (**a,b**), respectively and by ultrasonic pyrolytic spray (**c**,**d**), respectively.



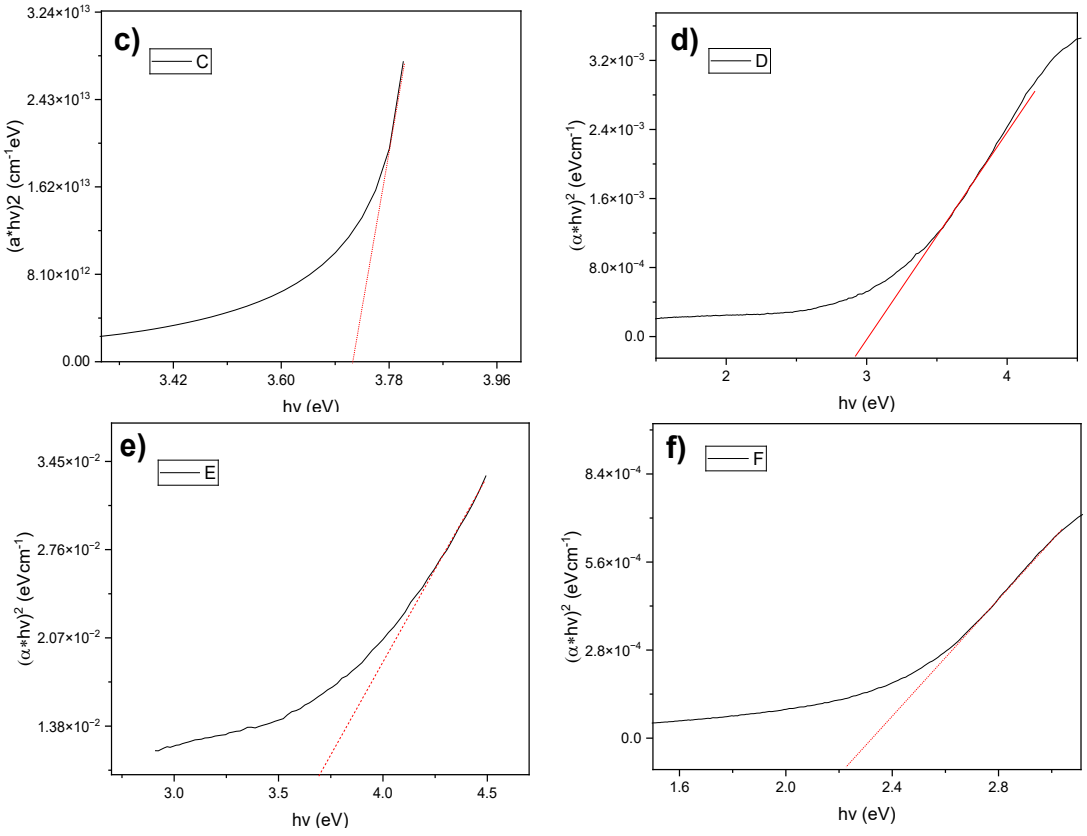


Figure 6: Graphs obtained by the Tauc method to obtain the band width of the In-S films, deposited by CBD and MCBD (**a**–**c**) and by ultrasonic pyrolytic spray (**d**–**f**).

As mentioned above, one of the purposes of this article is to prepare and characterize buffer layer films for the possible construction of cells whose absorber could be $Cu(In,Ga)Se_2$. As is well known, the most commonly used buffer layer in this type of cell is CdS (2.4 eV); however, it is important to replace it in order to reduce toxicity and environmental effects. Therefore, In_2S_3 is one of the most suitable materials, as it is possible to improve optical transmission at short wavelengths, favoring the alignment of the conduction band between CIGS and In_2S_3 [17]. According to the band gap results shown in Table 4, almost all samples, except for sample F, have values higher than those of CdS, achieving the desired result. The samples deposited by the CBD technique (A, B, and C) showed higher values than those deposited by pyrolytic spraying (D, E, and F).

Table 4: Band gap of In-S films deposited by CBD and MCBD (A, B and C) and by pyrolytic spray. (D, E and F).

Sample	Band Gap (eV)
A	3.8
B	4.5
C	3.7
D	2.8
E	3.7
F	2.2

The differences in the observed band widths are likely due to the different thicknesses and atomic compositions obtained with each of the three techniques used. However, it is worth mentioning that it was possible to obtain some films with suitable values for use as a buffer layer.

4 Conclusions

A comparison of three techniques for manufacturing indium sulfide (In_2S_3) films: chemical bath deposition (CBD), modified chemical bath deposition (MCBD), and ultrasonic pyrolytic spray deposition. The films produced through pyrolytic sputtering, particularly samples D and E, exhibited superior crystallinity and more defined diffraction peaks compared to those produced by CBD (sample A) and MCBD (samples B and C). Enhanced structural integrity makes the ultrasonic pyrolytic spray films more suitable for use as buffer layers. From a morphological perspective, the films produced by these deposition techniques displayed characteristics consistent with existing literature. However, the films deposited via ultrasonic pyrolytic spray showed greater uniformity and progressive granular growth over time, positively influencing their electronic properties. In terms of stoichiometry, sample C (deposited by MCBD) was closest to the ideal atomic ratio of 40:60, followed by sample F (obtained through ultrasonic pyrolytic spray. Achieving a balanced composition is crucial to ensure proper alignment of the conduction and valence bands with those of copper indium gallium selenide (CIGS), thereby reducing recombination at the interface. All films exhibited a thickness within the range of 50–100 nm, making them suitable for use as buffer layers. The optical behavior, the samples deposited by CBD showed energy gap values exceeding 3.7 eV, which is significantly higher than the ~2.4 eV observed for cadmium sulfide (CdS). This wider band gap enhances light transmission in the blue region of the solar spectrum. Although the films created by ultrasonic pyrolytic spray also demonstrated acceptable band gap values, these were slightly lower, with samples D and F standing out in particular.

In summary, these results indicate that both deposition methods can be effectively applied in the manufacture of In_2S_3 buffer layers for thin-film solar cells utilizing CIGS as the absorber. However, the MCBD method offers substantial advantages in terms of band gap and stoichiometry, with sample C being the most optimal. On the other hand, ultrasonic pyrolytic spray ultrasonic pyrolytic deposition excels in producing morphologically more homogeneous films with adequate crystallinity, which could enhance device functionality. Ultimately, the choice of deposition technique should consider the balance of optical, structural, and industrial scalability properties. Based on the findings presented, In_2S_3 is positioned as a promising and more sustainable alternative to CdS for future photovoltaic technologies.

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Author Contributions: **A. Ledesma-Juárez[1]**: study conception and design, methodology, editing Investigation, draft manuscript preparation**, J. F. Quintero-Guerrero[2]**: data collection**,** experimentation, **A. M. Fernández[1,*]** analysis and interpretation of results, draft manuscript preparation, writing– review & editing, visualization, data curation

Availability of Data and Materials: Data available on request from the authors.

The data that support the findings of this study are available from the corresponding author, [author initials], upon reasonable request.

Ethics Approval: "Not applicable." for studies not involving humans or animals.

Conflicts of Interest: The author declares no conflicts of interest to report regarding the present study."

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