

SUBSTRATE TEMPERATURE EFFECT ON THE STRUCTURAL, OPTICAL AND MORPHOLOGICAL PROPERTIES OF COBALT SULFIDE THIN FILMS DEPOSITED USING SPRAY PYROLYSIS

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Article Info	Abstract
Received: 29.01.2024 Accepted: 14.05.2024 Keywords: Cobalt sulfide, Spray-pyrolysis, FTIR, Optical gap, Conductivity.	Thin films of cobalt sulfide were grown on glass substrates at four different temperatures (250 °C, 300 °C, 350 °C and 400 °C) using spray-pyrolysis technique. The precursor solutions were prepared using cobalt chloride and thiourea. The effect of deposition temperature on the structural, morphological, and optical properties of cobalt sulfide thin films was investigated using different experimental techniques such as X-ray diffraction (XRD), and UV-visible spectrophotometry, Fourier Transform Infrared Spectroscopy (FT-IR) and four-probe method. The XRD analysis showed that crystallite size varies from 9.76 to 14.11 nm with increasing the deposition temperature. UV-visible data analysis shows a decrease in the band gap energies with increasing temperature (1.82 eV for 250 °C, 1.76 eV for 300 °C, 1.72 eV for 350 °C, and 1.65 eV for 400 °C). The analysis of the chemical composition by FTIR confirmed the presence of Co, S elements. On the other hand, the electrical conductivity of the cobalt sulfide thin films increased owing to the increase in the crystallite size and reduction of defects density.

1. Introduction

Due to their unique physical and optoelectronic characteristics, metal chalcogenide thin films are very attractive in electrical, optical, and optoelectronic devices [1]. Particularly, the cobalt sulfide is a multiphase crystal due to the presence of cobalt in different oxidation states +2, +3, or +4 [2], yielding the following compounds Co_4S_3 , Co_{1-x}S , Co_2S_3 , CoS , CoS_2 , Co_3S_4 ,

and Co_9S_8 . On the other hand, the cobalt sulfide is a magnetic transition metal chalcogenide. The complex chemical structures of the above phases are of special relevance and they have various applications as super-capacitors [3], water splitter to produce hydrogen [4], catalysts [5], diluted magnetic materials [6], counter electrodes for dye synthesized solar cells [7], anode materials for advanced sodium and lithium ion batteries [8], optical waveguides, thermal sensors, solar selective coatings, and optical filters [9]. Thin films of cobalt sulfide have been deposited using a variety of physical and chemical techniques such as RF sputtering [10], dip-coating [11], chemical bath deposition [12, 13], chemical vapor deposition [7], thermal evaporation [10], molecular beam epitaxy [14], electrochemical deposition [15,16], pulsed laser deposition [17], photochemical deposition [14] and spray technique [18-20]. Among these techniques, the spray-pyrolysis method has many advantages including low cost, easy set-up and it is suitable for large deposition at high temperature. This technique does not use any toxic volatile constituents, could produce binary, ternary, quaternary and pen-ternary metal chalcogenides. In addition, it allows the growth of thin films on various substrates such as indium tin oxide coated glass, microscope glass slide, titanium, mica, stainless steel, and fluorine doped tin oxide coated glass. The properties of these films can be controlled by different parameters such as pH, substrate temperature, solution concentration, complexing agent, and deposition time.

In this article, we report the synthesis and characterization of cobalt sulfide thin films deposited on glass substrates using spray-pyrolysis technique with cobalt dichloride (CoCl_2) and thiourea ($\text{CS}(\text{NH}_2)_2$) aqueous solutions as starting materials. The films were studied using different experimental technique to investigate their potential use as electrode material for application in super-capacitors.

2. Experimental details

2.1. Materials and Methods

The precursor solution was synthesized by dissolving Cobalt dichloride hexahydrate ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$) and thiourea ($\text{CS}(\text{NH}_2)_2$) in 100 ml of ionized water under stirring at 70 °C. To improve the precursor's solubility, a few drops of HCl were added to the solution mentioned above. Using the spray-pyrolysis process, the films were deposited at various substrate temperatures (250, 300, 350, and 400 °C) on glass substrates of $2 \times 7 \text{ cm}^2$. The spray gun nozzle and substrates were spaced by 30 cm. The gas vector consisted of 10 ml/min flow rate of ambient air. A manually driven stepper motor is attached to the nebulizer spray cannon to

regulate the spray location in the plane (X,Y). The films were gradually cooled to room temperature in the presence of ambient air after deposition.

2. 2. Characterization

The structural analysis was carried out using an X-ray diffractometer (DRX; Mini Flex-Rigaku) with a Cu-K α radiation ($\lambda= 1.5418 \text{ \AA}$, 40 kV, 40 mA) in the range of 20 – 60°, using the Brentano-Bragg geometry. A dual-beam UV-visible spectrophotometer (UV-3101; Shimadzu), was used to measure the transmittance and absorbance in the wavelength range of 300 – 1100 nm. The electrical conductivity of the films was measured in a coplanar structure obtained with evaporation of four golden stripes on the film surface. All the analysis were conducted without any post-thermal treatment after deposition. Infrared transmission measurements were performed using FTIR Shimadzu, model IR Affinity-1, in the range of 400 - 4000 cm⁻¹.

3. Results and Discussions

3.1. Film thickness

The film thickness was estimated using the weight method, which yields the thickness according to the formula [21]:

$$t = \frac{m}{\rho \times S} \quad (1)$$

where: t is film thickness of the film, m is the mass deposited on the substrate, S is the area of the film and ρ is the density of cobalt sulfide (5.45 g/cm³ [22]). Its variation with the different substrate temperatures is shown in Figure 1.

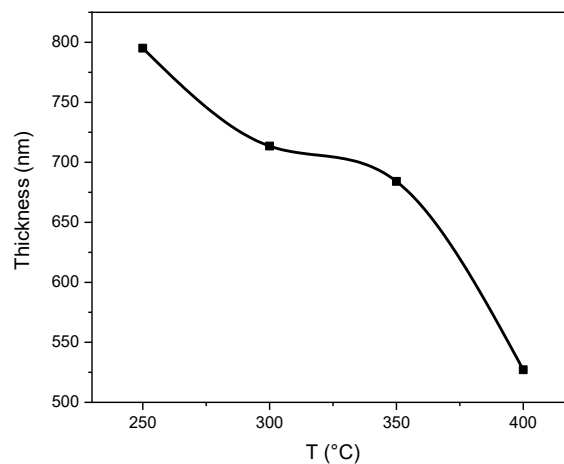


Figure 1. Thickness of cobalt sulfide thin films deposited at different substrate temperatures (250, 300, 350 and 400 °C)

3.2. X-Ray Diffraction analysis

The aim of this study was to highlight the structural characteristics of the deposited thin films at different substrate temperatures (250, 300, 350 and 400 °C). Figure 2 shows the XRD patterns of deposited cobalt sulfide films.

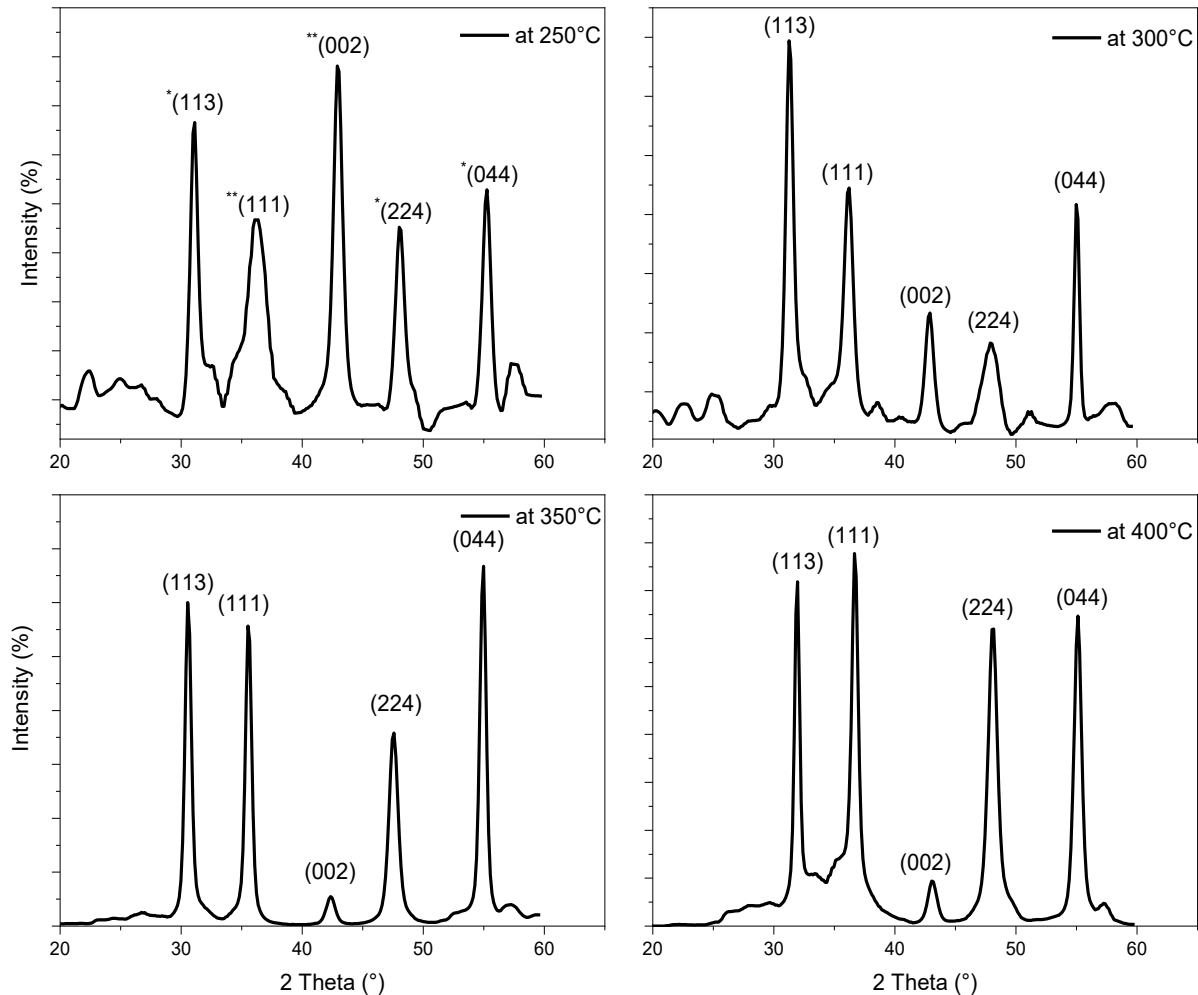


Figure 2 . XRD patterns of cobalt sulfide thin films deposited at 250, 300, 350 and 400 °C

The analysis of the data was done by High Score software. We have found that the films consisted of a mixture of cubic Co_3S_4 (JCPDS 1703-073-01) and cubic CoO (JCPDS -048-00 1719). We have also noticed that the formation of CoO is proportional to the increase of temperature. On the other hand, the grains size increases as summarized in Table 1. The sharp peaks (113), (224) and (044) in the XRD patterns are well match with XRD data in JCPDS file of cubic Co_3S_4 (#1703-073-01) and the peaks (111) and (002) were assigned to CoO cubic structure according to JCPDS file (#1719-048-00). It is worth mentioning that both peaks intensities of Co_3S_4 (113) and CoO (111) increased with the increasing the substrate

temperatures from 250 °C to 400 °C suggesting the enhancement of crystallinity.

The texture coefficient (TC_{hkl}), which is calculated in terms of the intensity of each orientation (I_{hkl}) to the corresponding intensity of the JCPDS card (I_{0hkl}), gives information on the probability of the growth according to an orientation $[hkl]$. The coefficient (TC_{hkl}) is given by the following relation [23]:

$$TC_{hkl} = \frac{I_{hkl}/I_{0hkl}}{N^{-1} \left[\sum_{i=1}^n \frac{I_{hkl}}{I_{0hkl}} \right]} \quad (2)$$

where $I_{(hkl)}$ is the measured relative intensity of (hkl) plan, $I_{0(hkl)}$ is the standard intensity of the same plane taken from the JCPDS No: 1703-073-01 card and N is the reflection number. The calculated $TC_{(hkl)}$ values of the prepared thin films are gathered in Table 1.

Table 1. Different texture coefficient $TC_{(hkl)}$ values of cobalt sulfide sprayed thin films

	at 250 °C	at 300 °C	at 350 °C	at 400 °C
TC_{311}	1.0767	1.6519	0.5540	0.2521
TC_{111}	0.0596	0.3910	0.9010	0.4187
TC_{002}	1.3244	2.0311	1.7612	1.1230
TC_{422}	0.8446	0.6950	1.0198	0.9175
TC_{440}	0.6945	0.2308	0.7637	1.4885

It is observed that the value of the greater texture coefficient corresponds to the plan (002), indicating a preferred orientation. Hence, the peak (002) has the highest intensity when compared to the other peaks and the growth of the films occurs parallel to the substrate.

The crystallites size (D) was estimated for all diffraction peaks using Debye formula [24]:

$$D_{hkl} = \frac{k \times \lambda}{\beta_{1/2} \cos \theta} \quad (3)$$

where: k is a constant ($k = 0.90$), $\beta_{1/2}$ the full width at half the maximum of the diffraction peak, θ the Bragg angle for each plan (hkl) , and $\lambda = 1.5406 \text{ \AA}$ is the wavelength of the X-ray radiation. The obtained values of D_{hkl} for each plan are gathered in Table 2.

Table 2. Different texture coefficient $D_{(hkl)}$ values of cobalt sulfide sprayed thin films

	at 250 °C	at 300 °C	at 350 °C	at 400 °C
$D_{311}(\text{nm})$	11.752	18.915	14.904	9.586
$D_{111}(\text{nm})$	5.957	14.369	15.099	9.711
$D_{002}(\text{nm})$	10.138	9.778	10.277	9.919
$D_{422}(\text{nm})$	10.328	9.962	10.470	20.527
$D_{440}(\text{nm})$	10.645	12.325	16.196	20.842

The dislocation density (δ_{hkl}), which measures the amount of defects in a crystal is defined as the length of dislocation lines per unit of volume. It was determined using the following formula [25]:

$$\delta_{hkl} = \frac{1}{D_{hkl}^2} \quad (4)$$

The obtained values of δ_{hkl} for each plan are gathered in Table 3.

Table 3. Different texture coefficient $\delta_{(hkl)}$ values of cobalt sulfide sprayed thin films

	at 250 °C	at 300 °C	at 350 °C	at 400 °C
$\delta_{311} (10^{15} \text{ lines/m}^2)$	7.241	2.795	4.502	10.883
$\delta_{111} (10^{15} \text{ lines/m}^2)$	2.817	4.843	4.387	10.603
$\delta_{002} (10^{15} \text{ lines/m}^2)$	9.73	10.459	9.468	10.164
$\delta_{422} (10^{15} \text{ lines/m}^2)$	9.374	10.076	9.123	02.373
$\delta_{444} (10^{15} \text{ lines/m}^2)$	8.824	6.583	3.812	02.302

On the other hand, the microstrain (ϵ_{hkl}), was estimated using the following formula [26]:

$$\epsilon_{hkl} = \frac{\beta_{1/2}}{4 \tan \theta} \quad (5)$$

The obtained values of ϵ_{hkl} for each plan are gathered in Table 4.

Table 4. Different texture coefficient $\epsilon_{(hkl)}$ values of cobalt sulfide sprayed thin films

	at 250 °C	at 300 °C	at 350 °C	at 400 °C
$\epsilon_{311} (10^{-3})$	11.2	6.8	8.6	13.5
$\epsilon_{111} (10^{-3})$	18.6	7.8	7.4	11.5
$\epsilon_{002} (10^{-3})$	9.3	9.7	9.2	9.6
$\epsilon_{422} (10^{-3})$	8.2	8.6	8.1	3.9
$\epsilon_{442} (10^{-3})$	7.1	6.1	4.6	3.6

Finally, the averages of crystallites size $\langle D \rangle$, micro-stress $\langle \epsilon \rangle$ and dislocation density $\langle \delta \rangle$ can be deduced using Williamson- Hall relations [27-29]:

$$\left\{ \begin{aligned} \langle D \rangle &= \frac{\sum TC_{hkl} \times D_{hkl}}{n} \end{aligned} \right. \quad (6)$$

$$\left\{ \begin{aligned} \langle \varepsilon \rangle &= \frac{\sum TC_{hkl} \times \varepsilon_{hkl}}{n} \end{aligned} \right. \quad (7)$$

$$\left\{ \begin{aligned} \langle \delta \rangle &= \frac{\sum TC_{hkl} \times \delta_{hkl}}{\sum TC_{hkl}} \end{aligned} \right. \quad (8)$$

Figure 3 shows the average calculated values of the crystallite size, stress and dislocation density as a function of deposition temperature.

It is evident that the micro-stress and dislocation density decrease with increasing the substrate temperature, indicating the development of high-quality thin films [30]. Furthermore, the increase in the substrate temperature leads to an increase of the crystallites size in the samples, followed by an improvement in their electrical and optical properties, as evidenced in the sections below.

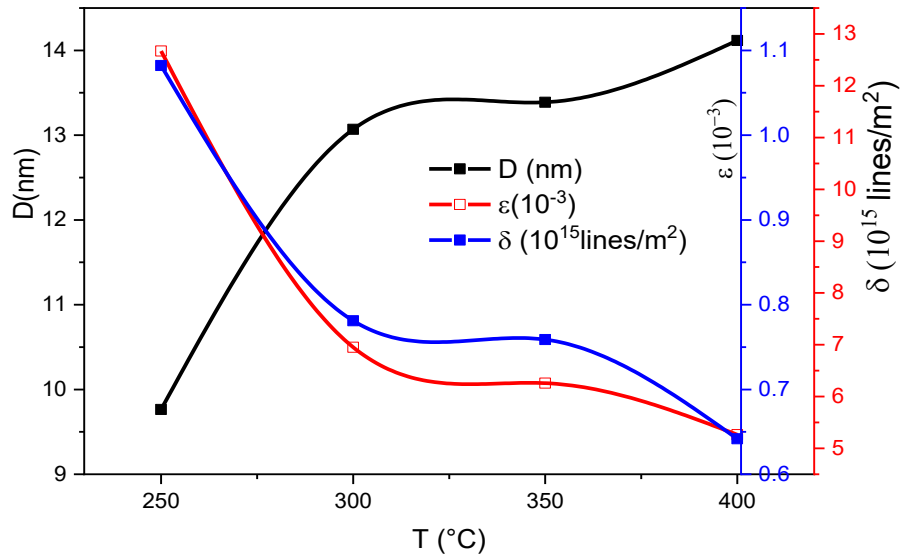


Figure 3. Variation of the average values D, ε and δ of thin films of cobalt sulfide as a function of deposition temperature

3.3. Optical analysis

The optical properties of nanocrystalline cobalt sulfide deposited on glass substrate were analyzed at room temperature using UV–visible spectrophotometry. Figure 4 shows the recorded spectra of the films.

It is observed that the transmittance increases in the visible range and it is enhanced with the increase of substrate temperature. This enhancement is related to both crystallites' growth and reduced films thickness. On the other hand, the reduced optical losses suggest an

improvement in the film's quality and reduction of defects. We have found that the average optical transmittance for the deposited films is 0.51, 0.70, 0.77 and 1.78% for the films prepared at 250, 300, 350. and 400 °C, respectively.

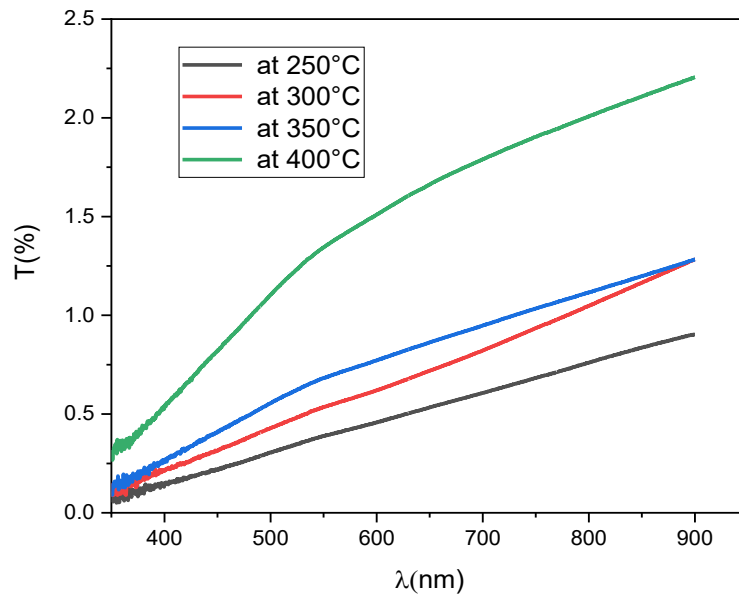


Figure 4. Transmittance spectra of cobalt sulfide thin films deposited at 250, 300, 350 and 400 °C

The absorption coefficient (α), is given by the following formula [21]:

$$\alpha = \frac{1}{d} \ln \left(\frac{100}{T} \right) \quad (9)$$

where: d is the thickness and T the transmittance. Figure. 5 shows the variation of the absorption coefficient (α) as a function of the wavelength (λ). The average absorption coefficient is $8.217 \times 10^4 \text{ cm}^{-1}$, $7.284 \times 10^4 \text{ cm}^{-1}$, $7.212 \times 10^4 \text{ cm}^{-1}$ and $6.891 \times 10^4 \text{ cm}^{-1}$ for the films prepared at 250 °C, 300 °C, 350 °C and 400 °C, respectively. The observed decrease when increasing the deposition temperature is due to the improvement of the crystallinity of the films, removal of defects, and the reduction of the grain boundary density. In addition, the absorption coefficient values are close to those values reported in the literature [31].

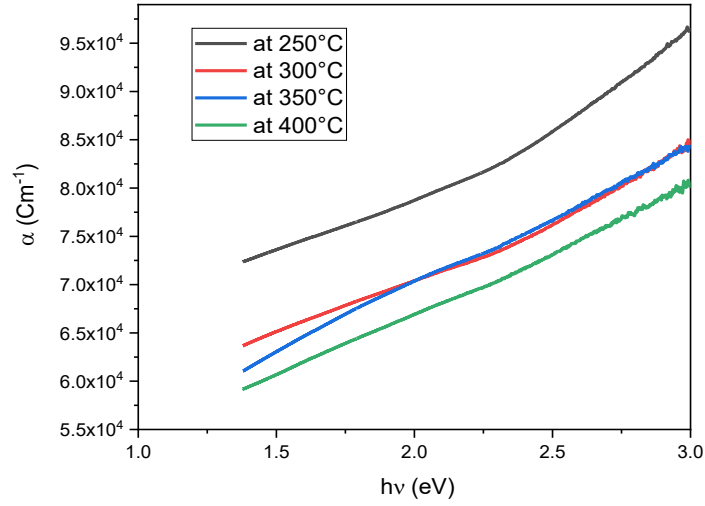


Figure 5. Absorption coefficient (α) vs. $h\nu$ of cobalt sulfide thin films

The optical band gap of the material was determined using the following formula [26]:

$$\alpha h\nu = B(Bh\nu - E_g)^p \quad (10)$$

where B is a constant, $h\nu$ the photon energy, p is an integer that takes the value 1/2 or 2 for direct or indirect transitions, respectively. Figure 6 shows the plots $(\alpha h\nu)^2$ vs. $(h\nu)$ of cobalt sulfide films deposited at different substrate temperatures.

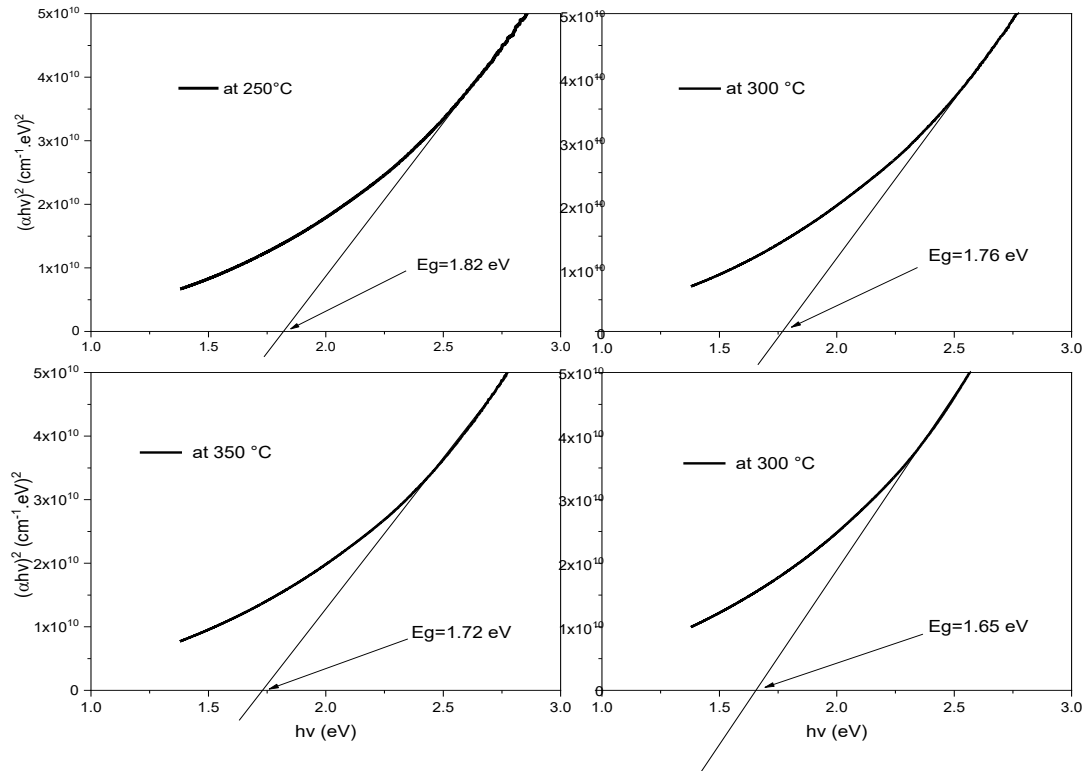


Figure 6. Tauc plot of $(\alpha h\nu)^2$ vs. $h\nu$ of cobalt sulfide thin films

The linear extrapolation of the curve yields the intersection point with the abscissa axis, which corresponds to the energy of the optical transition. The optical band gap of the film was found to be 1.82, 1.76, 1.72, and 1.65 eV for the films deposited at 250 °C, 300 °C, 350 °C, and 400 °C, respectively. Hence, a decrease is witnessed when increasing substrate temperature of deposition. Therefore, the optical band gap of cobalt sulfide thin films decreases from 1.82 eV to 1.65 eV with the increase of the substrate temperature from 250 °C to 400 °C, respectively (Figure 7). On the other hand, the crystallites size increases as confirmed from the XRD measurements.

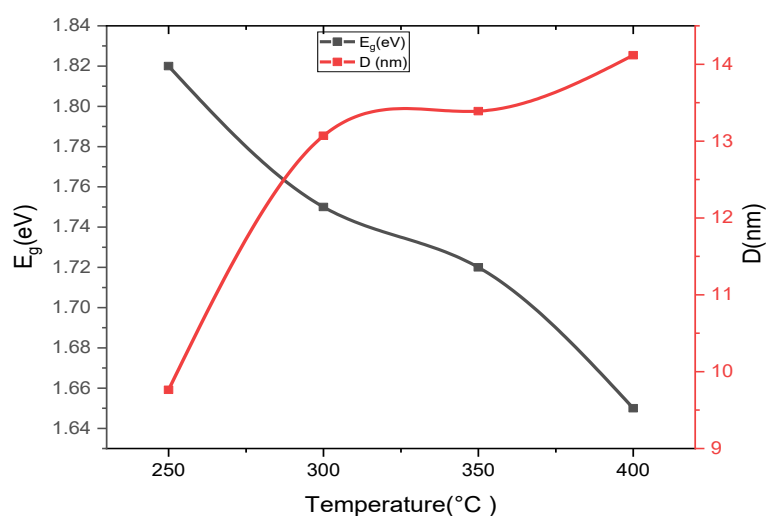


Figure 7. Variation of the optical gap (E_g) and the crystallites size (D) as a function of the substrate temperature

3.4. FTIR analysis

FTIR is a technique used to determine the functional groups and chemical composition of materials. For guidance, the cobalt sulfide is a semiconducting material for which the composition can be affected by factors such as deposition time, annealing temperature, and acid concentration. FTIR can be used to detect the presence of groups such as O-H, C-O-H, C-O, double sulfide and Co-S in thin films. These groups appear in different regions of the infrared spectrum, depending on their corresponding wavelengths and frequencies. The molecular structure of both organic and inorganic compounds can be determined with great accuracy using FTIR spectroscopy. Figure 8 depicts the FTIR spectrum of cobalt sulfide thin films deposited at 400 °C.

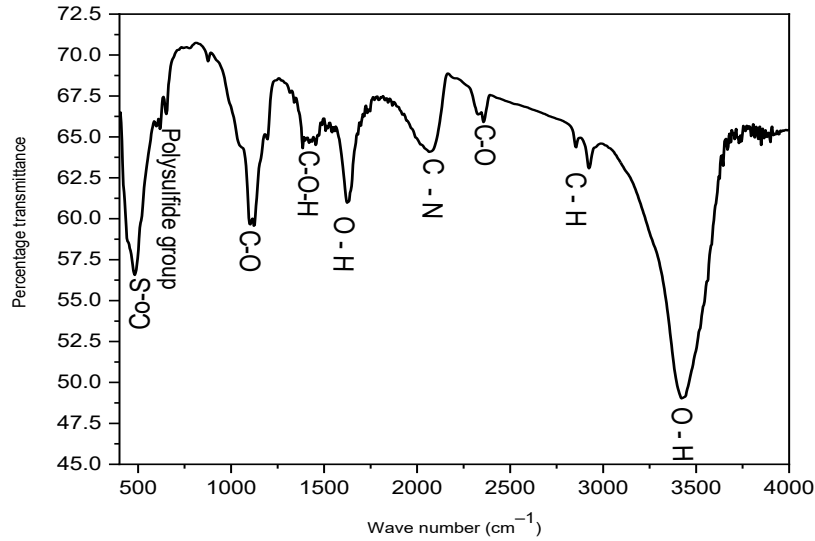


Figure 8. FTIR spectrum of cobalt sulfide thin films deposited at 400 °C

The peaks at 3421.21 and 1624.06 cm^{-1} correspond to the stretching vibration and bending vibration of OH groups of H_2O molecules; thus, indicating the absorption of moisture [32]. The carbon-containing compounds might be either from the starting reagents since all of them are carbon-containing chemicals or from atmospheric CO_2 . Therefore, the absorption peaks at 2924.31 and 1122.51 cm^{-1} were assigned to C-O-H and C-O stretching vibrations [34], respectively. The peak at 651.41 cm^{-1} is a distinctive peak of Co-S stretching vibration mode and the polysulfide bond group, while the peaks at 775.28 and 875.45 cm^{-1} correspond to the bending vibration of sulfonated groups in CoS , Co_3S_4 , and Co_9S_8 phases [35, 36].

3.5. Electrical analysis

The electrical conductivity of cobalt sulfide thin films was determined by four-points probe method, it is based on the measured sheet resistance of the films as expressed by the following formula [25]:

$$\left\{ \begin{array}{l} R_{sh} = \frac{\pi}{\ln 2} \times \frac{V}{I} \end{array} \right. \quad (11)$$

$$\left\{ \begin{array}{l} \sigma = \frac{1}{\rho} = \frac{1}{e \times R_{sh}} \end{array} \right. \quad (12)$$

where ρ is the electrical resistivity, e the film thickness, I is the applied current = 1 nA and V is the measurement voltage. Figure 9 shows the changes in the measured values versus the crystallites size, and band gap as a function of the deposition substrate temperature.

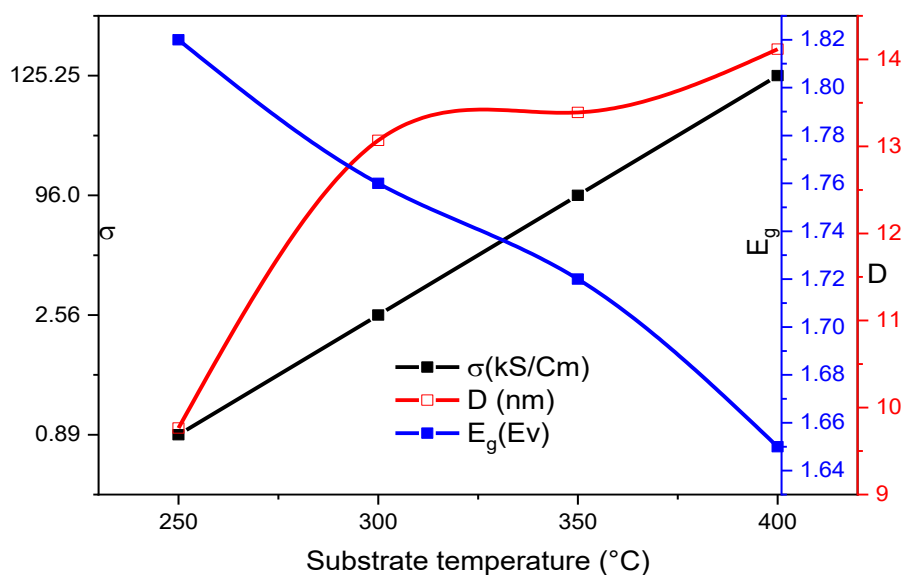


Figure 9. Variation of the electrical conductivity (σ), optical gap (E_g) and the grain size (D) as a function of substrate temperature

From the results shown on Figure 9, the measured values for the films deposited at 250 °C, 300 °C, 350 °C and 400 °C were 0.89×10^3 , 2.56×10^3 , 9.61×10^4 and 1.25×10^5 (S/cm), respectively. We can see that the values of the electrical conductivity are very high and increase with increasing deposition temperature, which indicates that the prepared layers have a semi-metallic behavior. Furthermore, these results are close to the results reported in literature [37, 38]. The increase in conductivity can be attributed to the increase in crystallites size and reductions of structural imperfections. Indeed, narrow band gap films become more conductive as electrons can easily be transferred from the valence band to the conduction band by polarization at room temperature. The above results showed that the cobalt sulfide prepared in this study is highly attractive as an electrode material for application in supercapacitors.

4. Conclusion

Cobalt sulfide thin films were successfully deposited by spray pyrolysis technique on glass substrate. The effect of substrate temperature on the cobalt sulfide thin films structural, optical and electrical properties was studied. It was found that increasing the substrate temperature decreased the energy band gap of the film (1.82, 1.76, 1.72, and 1.65 eV for the films deposited at 250, 300, 350, and 400 °C, respectively). The FTIR spectra of the films indicated the presence of OH, C-O-H, C-O, double sulfide, and Co-S groups. The conductivity of the films increases from 0.89×10^3 to 1.25×10^5 (S/cm) with increasing the substrate temperature from 250 to 400 °C, respectively. From the results obtained, the cobalt sulfide thin

films prepared in this study are considered as good candidates for many applications such as super-capacitors and solar cells.

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