Available online at sjuoz.uoz.edu.krd

Science Journal of University of Zakho

Vol. 11, No. 4, pp. 600 – 605 October-December, 2023

journals.uoz.edu.krd

p-ISSN: 2663-628X
e-ISSN: 2663-6298

INFLUENCE OF ANNEALING TEMPERATURE ON ZINC OXYSULPHIDE THIN FILMS PROPERTIES DEPOSITED BY THERMAL SPRAY TECHNIQUE

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Received: 18 Aug., 2023 / Accepted: 9 Nov., 2023 / Published: 28 Dec., 2023. https://doi.org/10.25271/sjuoz.2023.11.3.1181

ABSTRACT:

This article studies the effects of annealing temperatures on the stoichiometry, structure, and optical characteristics of Zinc Oxysulphide thin films deposited on glass substrates using the thermal spray method. The annealing was done in the air under a temperature of 150 ºC, 250 ºC and 350 ºC. Field Emission Scanning Electron Microscopy (FESEM), X-ray diffractometry (XRD), and a UV-Vis Spectrophotometer were utilized to examine the thin films' morphological, structure, and optical characteristics as grown and annealed samples. It has been observed that the film thickness decreases as the annealing temperature increases. The XRD pattern demonstrates that as-prepared and annealed samples at 250°C, and 350°C are amorphous, while film annealed at 150 °C is crystalline with the hexagonal phase and orientated along (110) plane. FESEM image of the as-prepared Zinc Oxysulphide thin films shows flower-like sheet nanostructures. However, for the annealed samples, the FESEM images show cone-like with a combination of sheets and aggregate nanoparticles. EDX analysis reveals the presence of Zn, O as well as sulfur. The transmittance of the film decreases from 63% to 37% and the band gap energy reduces slightly from 3.99 to 3.92 eV as the annealing temperature increase.

KEYWORDS: Thermal Spray, Thin Films, morphology, Energy Bandgap, Zinc Oxysulphide

1.1 INTRODUCTION

II-VI chalcogenides and oxides are wide direct bandgap semiconductors with interesting application properties, with zinc sulphide (ZnS) and zinc oxide (ZnO) being two examples [1]. These solid solutions for chemicals have the potential to lead to new applications as well as novel integrations. In ZnS, there are two periods of polymorphism. The hexagonal wurtzite phase is one, and the cubic sphalerite phase is another [2-4]. Because of the large differences in size and electronegativity between O and S (rS/rO = 1.3), [5], it is expected that substituting "O" with "S" in ZnS will impact the material's electrical and optical properties. [6]. Bandgap engineering may also be achievable because ZnS and ZnO have different bandgaps at 300 K [7].

Many optoelectronic devices use Zn-based materials, including ZnO, ZnS, and Zinc Oxysulphide [8]. Because of their compositional and crystal structural tunability, Zn (O, S) thin films [9-11] have recently gained popularity as buffer layers in solar cells, in addition to being employed as phosphor host materials and photocatalysts [12]. Upon varying their composition, the bandgap [13], the offset of the conduction band, and the conductivity can be carefully tuned. Furthermore, Zn (O, S) is a wide bandgap, non-toxic, earth-abundant semiconductor material. As a result, it is one of the most promising alternatives for CdS in the buffer layer of chalcopyrite-based thin-film solar cells [7, 14].

Chemical bath deposition and atomic layer deposition are two efficient pre-processing techniques [15, 16]. Another desirable deposition process is sputtering [17], which has been extensively utilized in the manufacturing of other cell layers. Sputtering of an undoped ZnO layer (i-ZnO) onto the surface of the CdS is necessary for the fabrication of chalcopyrite cells and modules [18]. This eliminates the requirement for a separate buffer layer by allowing the sputtered zinc oxide layer as a modification of the original zinc oxide layer (ZnO) [19]. ZnO and ZnS targets were co-sputtered to create the Zinc Oxysulphide using the spray method which is considered one of the most important techniques to prepare thin films [20-23].

Chemical solution (chemical composition, concentration), the distance between the substrate and atomizer interaction during film deposition, spray temperatures, substrate homogeneity, annealing conditions, and spray rates are some of the major factors influencing the properties of the spray-deposited film [24]. Spraying is an effective approach for the growth of thin film, multilayer film, thick film, and porous film on a low-cost substrate [25].

The spray technique has different advantages including no need for a high-quality substrate and vacuum, the thin film can be easily doped in the desired proportion, the film's thickness can be easily controlled, it is operable at different ranges of moderate temperatures, no restriction of substrate materials and its dimensions, it is a reliable, reproducible, and a simple growth method [26, 27].

Several oxides, such as ZnO [28], CdO [29], TiO2[30], SnO2[31], and NiO [32] have been deposited using the spray method. This method includes spraying a water/alcohol solution of metal salts over a heated substrate and then allowing it to break down into an oxide coating. The breakdown reaction produces oxide, which is thermodynamically possible and leaves no residue on other reactants. The temperature of the substrate has a significant impact on the shape of the film. The shape of the film can be changed from fractured to porous by increasing the temperature [33].

Other critical variables that determine the characteristics and structure are the types and concentrations of precursor and additive components [34]. Because of its low crystallinity and the presence of organic residues, the unannealed spray-deposited

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layer exhibits high resistivity, low roughness, and less transparency [35-37]. The properties of the unannealed film can be enhanced due to thermal annealing, plasma treatment, and laser treatment [38]. Thermal annealing is one of the easiest and most successful methods for treating spray-deposited films. The effect of annealing temperature on the structural, optical, morphological, and thickness of the Zn (O, S) has been reported by different groups in the literature [39-41].

Thermal annealing temperatures, time, and gaseous conditions all affect films and structural flaws in materials. Dislocations and other structural defects in the material, adsorption, or breakdown are kept on the surface during the thermal annealing process; as a result, the structure and stoichiometric ratio of the material is altered [42]. Common flaws in deposited ZnO films comprise oxygen interstitials, zinc interstitials, oxygen vacancies, zinc vacancies, and excess oxygen. The most frequent flaws are zinc interstitial and oxygen vacancies [43, 44].

In this study, the effect of annealing on zinc oxidized thin film prepared by spray method, and the stoichiometry of the sprayed material was investigated. The optical properties were examined to calculate the energy band gap of the zinc oxysulfide thin films. Furthermore, the field emission microscope was applied to analyse the films' morphology and (EDX) analysis to confirm the stoichiometry and applicability of the film for future application.

The maximum paper length is restricted to 12 pages. The manuscript should have the following structure:

1.2 Methodology and Characterization

Zinc Oxysulphide thin film nanoparticles were used to prepare thin films at 35˚C which were prepared using chemical bath deposition from zinc acetate and thiourea precursor. It was dried using an oven under 50˚C for one hour without a vacuum, then it was ground to prepare them for deposition. Microscope glass substrates having dimensions of (1×25 ×75 mm3) were coated with thin Zinc Oxysulphide.

The substrates were cleaned with distilled water, acetone, and chromic acid immersion for 24 hours. Before deposition, the substrate was ultrasonically cleaned using ethanol and distilled before being dried in the desiccator to eliminate the last residue. In this research, Zinc Oxysulphide thin films were sprayed on the glass substrate using disposed chemical waste of CBD-ZnS which is the novelty of this work. The residuals were dried and ground, then examined by EDX to find the powder composition. The thickness of Zinc Oxysulphide of thin film was measured by using an optical method.

In the industry, metallic or ceramic materials are dissolved and then coated to a surface using a thermal spray technique [45, 46]. During the annealing process, material's physical and chemical properties are changed and become more suitable for a desired application [47, 48]. Figure 1 shows the thermal spray system utilized for the deposition of Zinc Oxysulphide thin films on the glass substrate. In this study, the solution spray rate was maintained at 3ml/min, and the distance between the glass substrates and the spray gun atomizer was fixed to 15 cm.

Each thermal spray growth cycle lasts for three seconds, followed by a ten-second rest period. The waiting period gives the glass substrate time to reach the necessary temperature for growth before beginning the subsequent growth thermal spray cycle. The sprayed film was annealed under different annealing temperatures (150oC, 250oC and 350oC).

1.3 Results And Discussion

The influence of annealing temperatures on the thickness of Zinc Oxysulphide nanostructured thin film was studied and shown in Fig. (2).

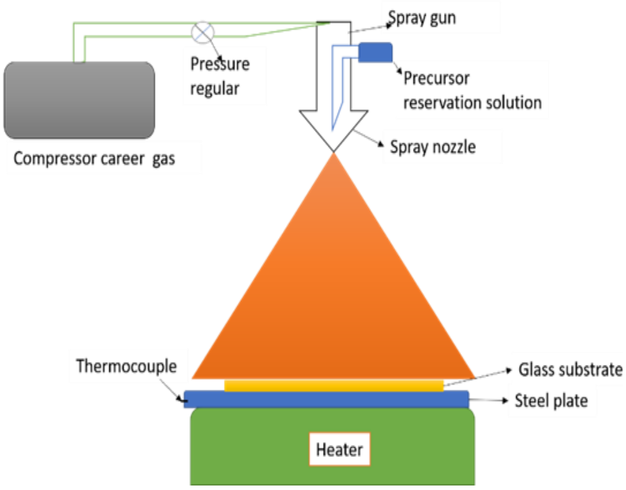

Figure. 1: Thermal spray deposition system used for preparing Zinc Oxysulphide thin film.

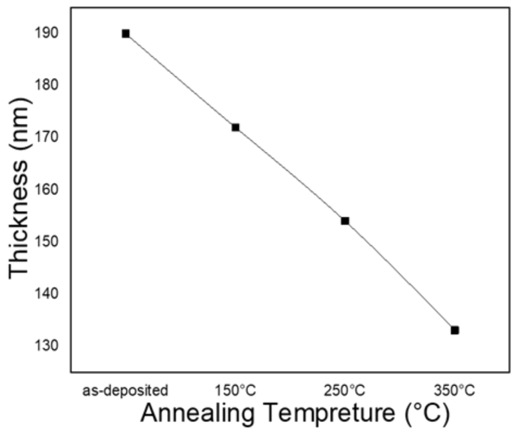

Figure. 2: Zinc Oxysulphide Growth as a function of annealing temperature

In this work, before investigating the influence of annealing temperature on thermally sprayed Zinc Oxysulphide thin film, the growth temperature and amount of sprayed solution were kept at T= 35oC, 3ml/min respectively. In the thermal spray growth method, the substrate temperature performs two primary tasks in addition to providing mechanical support for the film itself. First, it provides the energy for chemical reactions and second, it drives the produced particles in the X-Y plane. As a result, the values of surface roughness and film thickness will decrease. In addition, it might increase the compactness of the thin film [49].

In this work, the thickness of the as-deposited Zinc Oxysulphide film was about 190 nm. The increase in the annealing temperature from 150oC to 350oC resulted in the thickness reduction due to the re-evaporation of the sprayed solution constituents [28]. XRD pattern of the as-deposited and annealed (150, 250, and 350˚C) Zn (O, S) are presented in Fig. 3.

The XRD of the deposited thin films was carried out using PANanalytical X'Pert Pro X-ray diffractometer with CuKα radiation of wavelength, λ = 1.5406 Å, at 35kV/40 mA. The as-deposited and the annealed films are shown to be amorphous [JCPDS (01-079-0205]. There are several studies in the literature reporting the growth of both crystalline and amorphous Zn (O, S) mainly depending on the growth and annealing conditions [50-52].

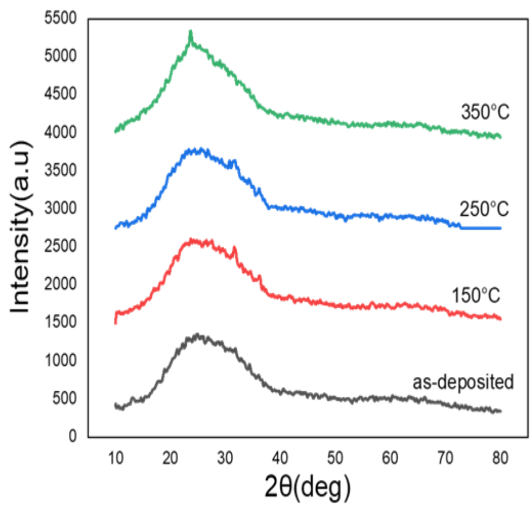

Figure. 3: XRD patterns of Zinc Oxysulphide as-deposited and annealed at 150, 250, and 350˚C.

Figures 4 and 5 show the EDX spectrum and FESEM image of the Zn (O, S) obtained from the ground and dried disposal of CBD-ZnS and then annealed at 50oC for one hour in the furnace. The EDX spectrum elucidates the presence of elemental oxygen, zinc, and sulphur in the deposited thin film. The FESEM of the Zn (O, S) powder shows approximately large spherically-shaped grains with their size between ~ (50-250) nm. Different research groups have also reported the EDX spectra of Zn (O, S) deposited using different growth techniques and the EDX results reported in this work are similar to those reported in the literature [53-55].

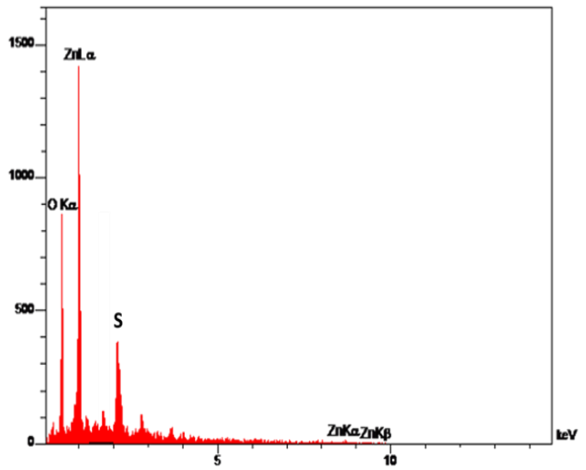

Figure. 4: EDX spectrum of as-deposited zinc oxysulfide thin film.

The effect of annealing temperature on the morphology of thermally sprayed zinc oxysulfide thin film has been studied and is shown in Figure 6 (b-d). For the as-deposited Zn (O, S), the FESEM shows a flower-like morphology with different nanoparticle sizes as shown in Figure 6a. When the film was annealed for one hour under the temperature of 150˚C, a cone-like and leaf-like morphology with different nanoparticle sizes and small cracks appeared, as displayed in Fig. 6b. Also, nanoparticles were non-uniformly distributed on the surface of the substrate having tiny gaps in between the agglomerated nanoparticles.

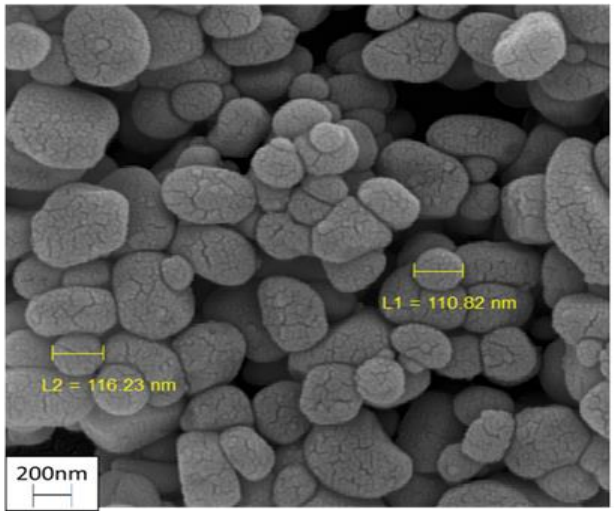

Figure 5: The FESEM image of the prepared Zn (O, S) powder annealed at 50oC for one hour in the furnace.

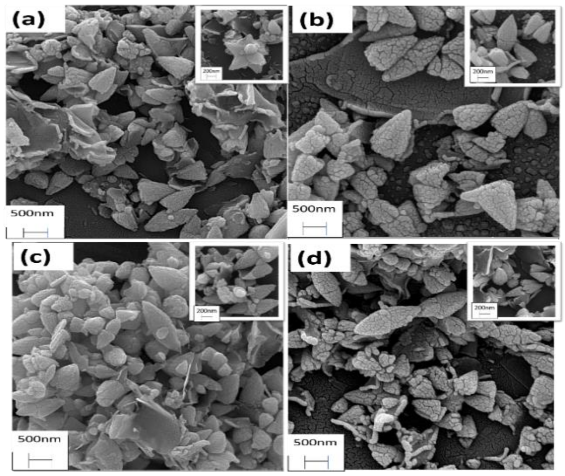

Figure. 6: Effect of annealing temperatures on spray-deposited Zinc Oxysulphide thin film a) as-deposited, at b)150°C, c)250°C, and d)350°C.

By increasing the annealing temperature to 250˚C the cracks in the surface of the nanoparticles disappeared, the cone-like nanoparticle morphology became more compact and the gaps in between the agglomerated nanoparticles were reduced as shown in Figure 6c. When the annealing temperature was increased to 350oC, the gaps between the nanoparticle clusters were increased, the compactness of the film morphology was reduced and the cracks on the surface of the cone-liked agglomerated nanoparticle became more visible (see Fig. 6d). From the observation of this work and comparison of the other works on the Zn (O, S) the annealing temperature has prominent effect on the morphology, shape and size of the nanoparticles [56-58].

The optical properties of all the samples were investigated using UV-Vis spectrophotometer model 6850 Jenway with a scanning range of (190-1100) nm. The transmittance values of the thermally sprayed Zn (O, S) thin films were determined and plotted as shown in Fig. 7a. The transmittance of the as-deposited and annealed Zn (O, S) films was approximately similar and their transmittance percentages were in the range of (62 to 65) %. When the annealing temperature increased to 150 and 250oC, the transmittance percentage was reduced to the range of (45-47) % and (32-35) %, respectively. To calculate the energy band gap a relationship between (αhυ)2 and photon energy was plotted, as exhibited in Fig. 7b.

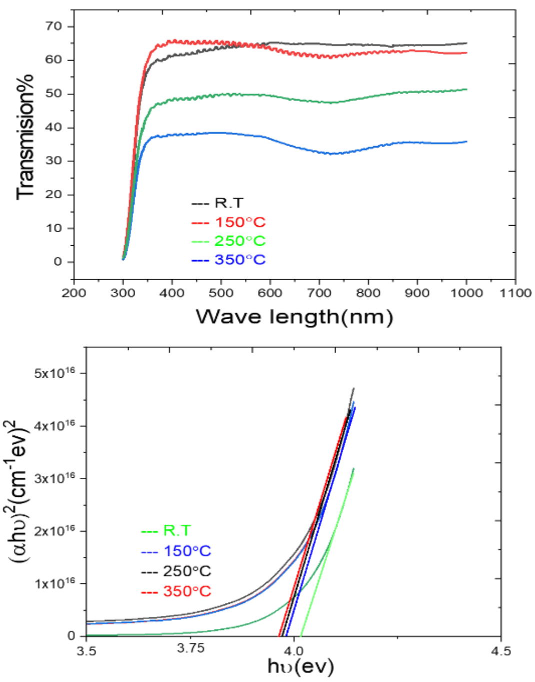

Figure. 7: a) Optical transmittance against wavelength and b) (αhυ)2 vs photon energy for the as-deposited, and annealed Zn (O, S) at 150, 250, and 350 °C, respectively.

Figure. 7: shows the effect of various annealing temperatures on the energy bandgap of Zn (O, S) thin film.

For the as-deposited film, the measured bandgap value was approximately 3.99 eV. When the annealing temperature was increased to, 150, 250, and 350oC, the bandgap values were reduced to 3.98, 3.95, and 3.92 eV, respectively [See table 1]. The reduction in the bandgap can be attributed to the increase in the nanoparticle size with increasing annealing temperature. The observed high bandgap values of the as-deposited and annealed Zn (O, S) can be related to the nanoparticle nature of the deposited and annealed Zn (O, S) films and the quantum confinement effect[59-62] .It is worth mentioning that the high value of obtained band energy can also be related to the FESEM graph [see Fig. 6 (a-b)], as it can be seen that the deposited nanoparticles are non-uniformly distributed on the glass substrate forming gaps in between grains. These gaps open up the pass for light to pass through during optical absorption measurement which can result in higher bandgap value observation and slight measurement error in the bandgap values should be considered.

Table 1. Variation of energy gaps as a function of annealing temperatures

Annealing Temp.	Eg (±0.02) eV
As-depo	3.99
150	3.98
250	3.95
350	3.92

1.5. Conclusion

Zn (O, S) were successfully prepared from the disposed CBD-ZnS and deposited on the glass substrate using the thermal spray growth method. The XRD pattern elucidates the amorphous nature of the deposited and annealed film. The EDX spectrum confirms the presence of elemental oxygen, zinc, and sulfur in the deposited thin film. The FESEM of the deposited and annealed Zn (O, S) film shows cone-like flower morphology with different nanoparticle shapes and sizes. The annealing temperature prominently affects the particle distribution and compactness of the nanoparticles. The optical absorption measurement indicated a bandgap reduction with increasing

Annealing temperature due to nanoparticle size growth, nature, and quantum confinement effect. Based on the XRD, EDX, FESEM, and optical properties of the deposited Zn (O, S) using the thermal spray method in this study, the prepared films can be used for wide bandgap optoelectronic applications.

ACKNOWLEDGEMENTS (OPTIONAL)

Authers would like to thank University of Duhok College of Science Department of Physics for their support to this research.

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