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Time Optimization of Chemically Deposited Cadmium Silver Sulphide (CdAgS) Ternary Thin Films at Room Temperature

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Abstract

Chemical bath method of thin film deposition has being successfully used to deposit thin films of Cadmium Silver Sulphide (CdAgS) at room temperature from the mixture of aqueous solutions of Cadmium chloride hemi(pentahydrate), $CdCl_2$. $2.5H_2O$, Silver nitrate ($AgNO_3$) and Thiourea, they serve as precursors for Cd^{2+}, Ag^{2+} and S^{2-}. Triethanolamine (TEA) was employed as the complexing agent while ammonium solution was used as a pH stabilizer. Deposition time as bath parameter was optimized. Five samples of Cadmium sliver sulphide films were fabricated at various time intervals of 1 hour, 2 hours, 3 hours, 4 hours and 5 hours. Optical properties of the films were studied using VU – VIS – NIR spectrophotometer within VIS and NIR regions. Micrographs of the films were taken using microphotometer. The as – grown films have low absorbance and high transmittance in VIS and NIR regions. Energy band gap of the films ranges from 1.90 eV – 2.40 eV. Micrographs of the films were analyzed using ImageJ, the average particle sizes of 94.54 nm – 723.71 nm was obtained.

Keywords

Thin Films, Chemical Bath Deposition, Optical Properties, Energy Band Gap, Micrographs

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1. Introduction

Thin film forms the basis for today's electronic components. Even complicated device structures are constructed from thin films. According to [1], a material is said to be a thin film when it is built up as a thin layer on a substrate by controlled condensation of the individual atomic, molecular or ionic species directly by a physical process or through a chemical or electrochemical reaction. Thin film as defined by [2], is a crystalline or non-crystalline material developed two dimensionally on a substrate surface by physical or chemical method. A thin film can be binary, ternary, and quaternary, depending on the number of elements that make up the film. Ternary thin films are thin films that contain three different elements [3].

According to [4], preparation and study of physical properties of ternary chalcogenide compounds have increased in recent years. There is considerable interest in the deposition of ternary derivative material, due to the potential of tailoring both the lattice parameters and the band gap by controlling depositions parameters [5]. Ternary thin film compounds are found to be suitable materials for optoelectronic device applications and good material for window layer solar cells [6]. Some of these films have been investigated for use as superionic conducting materials [7]. Ternary compounds had also been studied for efficient solar energy conversion materials [8]. Ternary thin film of cadmium silver sulphide can be viewed as altering the properties of two semiconducting thin films of CdS and AgS to produce a thin film with properties falling between the properties of the two parents films. Some properties and applications of these parents' binary semiconducting films of cadmium silver

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sulphide are reviewed below.

Cadmium sulphide (CdS) belongs to II – VI group compound of semiconducting materials. Its band gap varies between 2.1 eV to 2.45 eV [9]. The CdS thin films have wide range of well perspective applications such as optical coating, optical data storage and sensing [9], field effect transistors, light emitting diodes, photocatalysis and biological sensors [10], transparent conducting semiconductor for optoelectronic devices [11]. Numerous reports are available in the literature on synthetic techniques used for deposition of CdS thin film. Polycrystalline CdS thin film deposited by evaporation has been reported [12], CdS thin films were prepared by chemical spray pyrolysis technique by [13]. CdS thin films were fabricated using chemical bath method of thin film deposition by [14].

Silver Sulphide Ag_2S is an important chalcogenide compound which has been investigated for its numerous applications. Silver sulphide (Ag_2S) belongs to I–VI compound semiconductor materials with monoclinic crystal structure, has promising photoelectric and thermoelectric properties [15]. Silver sulphides (Ag_2S) have been used in IR detectors, photoconductors, photovoltaic cells, electrochemical storage cells, it is also well known as a mixed ionic and electronic conductor at high temperatures above 200℃ [16]. The optical band gap of the films deposited by CBD and SILAR techniques are estimated using the optical absorption measurements and is found to be 1.78 eV and 2.09 eV respectively. Optical properties of Ag_2S thin films using $AgNO_3$, thiourea, EDTA and ammonia has been reported, the film shows high absorbance, low reflectance, moderately high refractive index and band gap of 1.80 eV [17].

Many deposition techniques have been successfully employed for the purpose of depositing ternary thin film. These techniques include: Chemical Bath Method [2], Chemical Vapour Deposition (CVD) [18], Successive Ionic Layer and Reaction (SILAR) [19], and Sol-gel Methods [20].Some researchers have deposited ternary transition metal thin films such as CuZnS [21], $CuInS_2$ [22], PbCdS [23], ZnNiS [24] $Cd_{1-x}Pb_xSe$ [25] and $Zn_xCd_{(1-x)}Te$ [26] for various applications. Cadmium silver sulphide has been deposition by some researchers like; [27] who prepared nanosized CdS-Ag_2S core shell nanoparticles using XPS techniques. CdS-Ag_2S nanoparticles have being synthesized using inverse micro-emulsion techniques by [28]. Study on the excitation dynamics of CdS-Ag_2S nanorods with tunable composition was done by [29]. They studied Ag^+ cation exchange to converts CdS nanorods into CdS-Ag_2S heterostructure. According to literatures reviewed, chemical bath deposition method has not been used for the deposition of this ternary thin film.

The optical absorbance spectra of the deposited films were obtained by means of UV/VIS Janway 6405 spectrophotometer. Surface morphology of the thin films deposited on glass substrate was examined using Olympus optical microscope. From the spectrophotometer, the absorbance in arbitrary units was measured. Parameters such as transmittance, reflectance, refractive index and extinction coefficient were then calculated using the relationship explained below.

Transmittance is the fraction of incident radiant power at a specified wavelength that passes through a sample. Transmittance (T) of a specimen is defined as the ratio of the transmitted flux (I_t) to the incident flux (I_0) [30], that is

$$T = \frac{I_t}{I_0}. \quad (1)$$

According to Beer – Lambert Law

$$A = -log_{10}\frac{I_t}{I_0} = -log_{10}T, \quad (2)$$

$$T = 10^{-A}. \quad (3)$$

Reflectance (R) is the fraction of the incident radiant power of a given wavelength that is reflected when it strikes a surface. A relation between transmittance (T), spectral absorbance (A) and spectral reflectance (R), according to the law of conservation of energy is as

$$A + T + R = 1, \quad (4)$$

transforming equation (4), we have that

$$R = 1 - T - A. \quad (5)$$

Optical constants, refractive index (η) and extinction coefficient (k) are most commonly and reliably determined from the simultaneous measurement of transmittance and reflectance of the thin film layer. The boundary between vacuum (or air) and absorbing layer specified by refractive index, extinction coefficient at normal incidence, yields the reflectance in terms of the optical constants of the layer as

$$R = \frac{(\eta-1)^2+k^2}{(\eta+1)^2+k^2}. \quad (6)$$

According to [31], for a semiconductors and insulators, or material in the range of frequencies in which absorption is weak, $k^2 << (n-1)^2$ equation (6) can be reduced to;

$$R = \frac{(\eta-1)^2}{(\eta+1)^2},$$

hence,

$$\eta = \frac{\left((1+\sqrt{R})\right)}{(1-\sqrt{R})}. \quad (7)$$

Absorption coefficient (α) with reference to extinction coefficient (k) is defined by [32] as

$$\alpha = \frac{4\pi k}{\lambda},$$

hence

$$k = \frac{\alpha}{4\pi}. \quad (8)$$

The relation between absorption coefficient and photon energy as expressed in the equation below was given by [33]

$$\alpha = \left(hv - E_g\right)^n, \quad (9)$$

where ν is the frequency, h is the planck's constant, E_g is the energy band gap and n is a number which characterizes the optical processes, $n = 1/2$ is for direct allowed transition and $n = 2$ is for indirect allowed transition.

This research work presents Cadmium Silver Sulphide thin films deposited using chemical bath method, time as a bath parameter was optimized. Optical properties and surface structures of the deposited thin films were investigated.

2. Materials and Experiments

Materials used for the deposition of CdAgS films are; five different 100 mls beaker, glass rod stirrer, reagents, distilled water, substrates (Glass slides), digital weighing balance, thermometer.

Prior to deposition of cadmium silver sulphide, the substrates were degreased using nitric acids for 24 hours, washed with detergent and rinsed with distilled water and allowed to dry in an open air partially free from dust particles. The bath constituents for the deposition of cadmium silver sulphide contain $CdCl_2.2.5H_2O$, $AgNO_3$, Thiourea, TEA, ammonium solution and distilled water.

The experiments for the deposition of these films were conducted at room temperature of 300 K. Five 100 ml beakers were used for the deposition. In each beaker, 5 mls of 0.5 M of $CdCl_2.2.5H_2O$ was added to the empty beaker followed with 5 mls of 0.5 M of $AgNO_3$, a white liquid was formed. 2 mls of TEA was added to the mixture and stirred for 3 minutes. On adding thiourea, a white suspension liquid was formed. 5 mls of ammonium solution was added to the mixture which turned the mixture to black. The final mixture was stirred for 2 minutes; the volume was made up with 60 mls of distilled water and stirred for another 2 minutes to have a homogenous mixture. A degreased substrate was dipped inside the beaker and held vertically with the help of synthetic foam. The synthetic foam also served as a protective cover to the reaction bath to reduce inflow of dust particles and other environmental impurities that are air borne. Four more similar beakers were set - up in same way. The five substrates were allowed to stay in the beakers for hourly intervals of 1 hour, 2 hours, 3 hours, 4 hours and 5 hours. At the end of each time, the substrate were removed, rinsed with distilled water and allowed to dry in an open air. Substrates used for the deposition of these films were labeled CA_1, CA_2, CA_3, CA_4 and CA_5 as shown in the table below.

Table 1. Time Optimization of Deposited Cadmium Silver Sulphide Ternary Thin Films.

Baths	Time (hrs)	Cd^{2+}(mls)	Ag^{2+}(mls)	S^{2-} (mls)	$NH_4(OH)$ (mls)	TEA (mls)	H_2O (mls)
CA_1	1.00	5.00	5.00	3.00	5.00	2.00	60.00
CA_2	2.00	5.00	5.00	3.00	5.00	2.00	60.00
CA_3	3.00	5.00	5.00	3.00	5.00	2.00	60.00
CA_4	4.00	5.00	5.00	3.00	5.00	2.00	60.00
CA_5	5.00	5.00	5.00	3.00	5.00	2.00	60.00

The proposed chemical equations for the formation of Cadmium Silver Sulphide Thin films on glass substrate at room are shown below.

$$CdCl_2.2.5H_2O + TEA \rightarrow [Cd(TEA)]^{2+} + 2Cl^-$$

$$[Cd(TEA)]^{2+} \rightarrow Cd^{2+} + TEA$$

3. Results and Discussions

3.1. Optical Analysis

The optical properties of deposited films are studied within the wavelength range of 400 nm to 1100 nm which represents VIS and NIR regions of electromagnetic spectrum. Optical properties studied in this work are absorbance which was gotten from UV – VIS – NIR spectrophotometer, transmittance, reflectance, refractive index, extinction coefficient and band gap energy determined using equations 3, 5, 7, 8 and 9 above.

3.1.1. Absorbance

Absorbance of the grown thin films is shown in Fig. 2. The absorbance in VIS and NIR regions is low. It decreases as wavelength increases and increases as time of deposition increases. Films grown at 5 hours has the highest absorbing power of 0.089 at 400 nm that decreases to 0.032 at 1100 nm while film deposited at 1 hour has the lowest absorbing power of 0.024 at 400 nm that decreases to 0.003 at 1100 nm. These results suggest that CdAgS films with better absorbing power could be fabricated if the films were left for longer time in the bath solution.

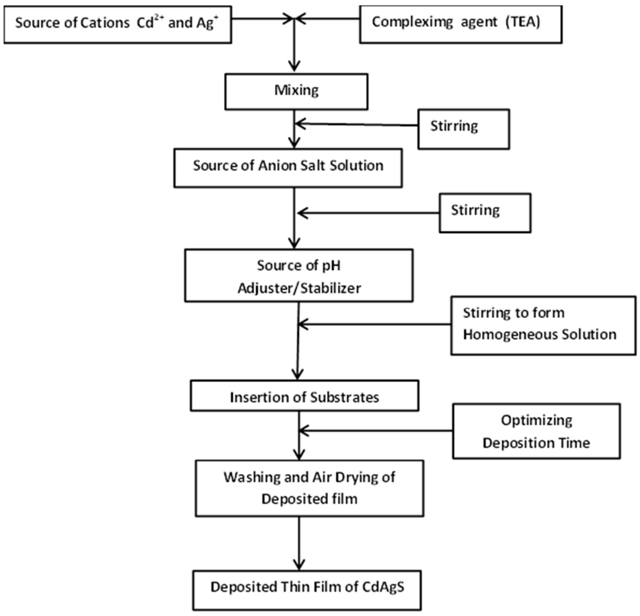

Figure 1. Flowchart Representation of the Growth Processes involve in the Deposition of CdAgS Thin Film.

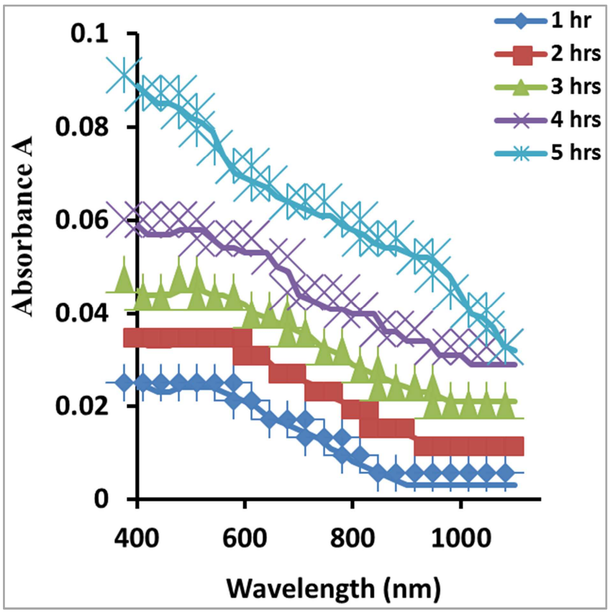

Figure 2. Plot of Absorbance against Wavelength of the As-grown Thin Films of CdAgS.

3.1.2. Transmittance

Transmittance of the films is presented in Fig. 3. Generally, these grown films have high transmittance values. Transmittance of these films increases as wavelength increases and decreases as time of deposition increases. Film grown at 1 hour has the highest transmittance of 94.62% at 400 nm which increases to 99.31% at 1100 nm while film grown at 5 hours has the least values of transmittance of 81.47% at 400 nm to 92.90% at 1100 nm. The high transmittance of these films in VIS and NIR regions make them suitable as spectrally selective window coating in cold climate [34].

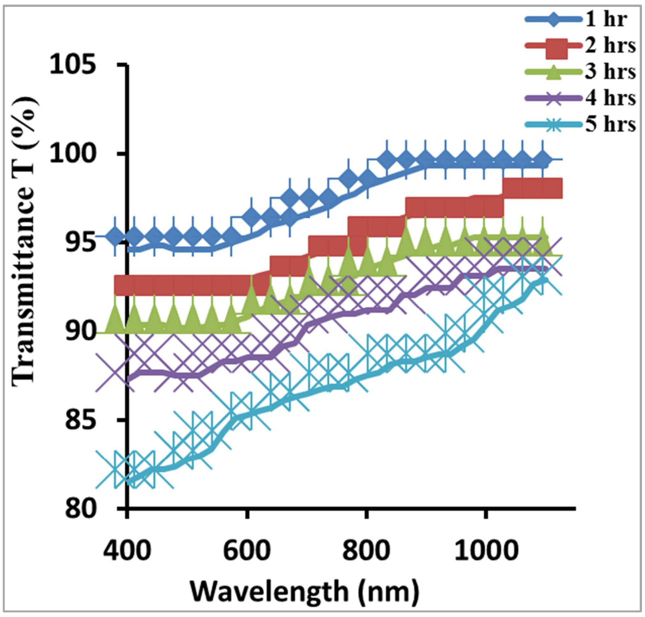

Figure 3. Plot of Transmittance against Wavelength of the As-grown Thin Films of CdAgS.

3.1.3. Reflectance

Reflectance of the grown films is presented in Fig. 4, highest value of reflectance of 9.64% is attained by film grown at 5 hours. From Fig. 4, it can be seen that reflectance increases as the time of deposition increases and decreases as the wavelength increases.

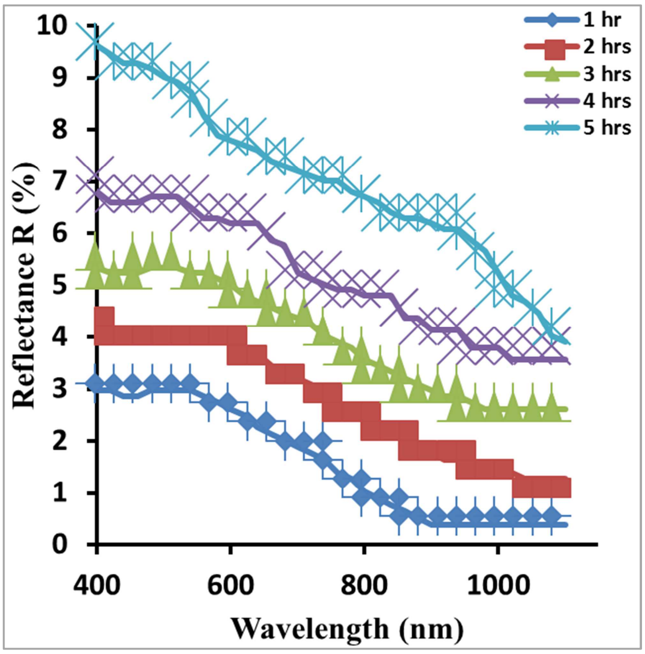

Figure 4. Plot of Reflectance against Wavelength of the As-grown Thin Films of CdAgS.

3.1.4. Refractive Index

Refractive index of the films is shown in Fig. 5 below. The films have refractive index that decreases as wavelength increases and increases as time of deposition increases. Least value of 1.42 at 400 nm to 1.13 at 1100 nm is attained by film deposited at 1 hour within VIS and NIR regions while highest value of 1.90 at 400 nm to 1.49 at 1100 nm is attainted by film deposited at 5 hours within VIS region.

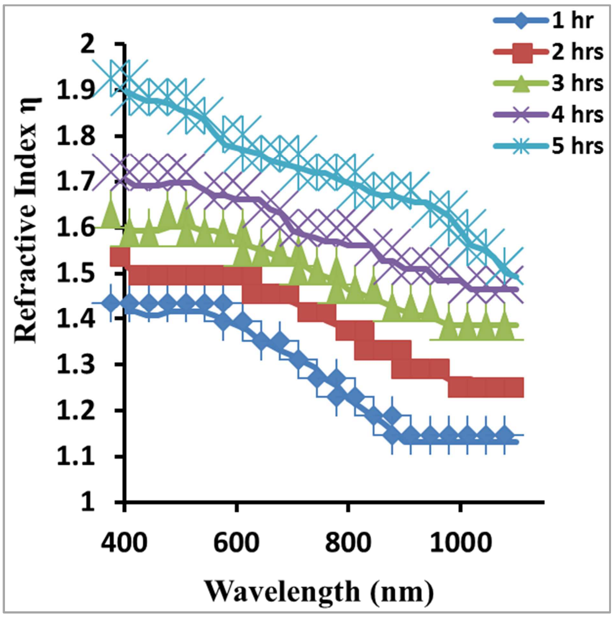

Figure 5. Plot of Refractive Index against Wavelength of the As-grown Thin Films of CdAgS.

3.1.5. Extinction Coefficient

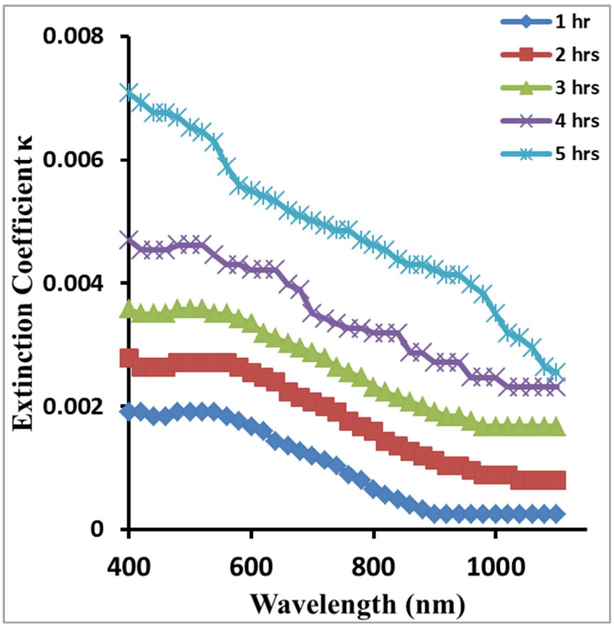

Figure 6. Plot of Extinction Coefficient against Wavelength of the As-grown Thin Films of CdAgS.

Fig. 6 shows the graph of extinction coefficient against wavelength. The extinction coefficient is an optical constant which is a factor that determines the extent to which radiation is absorbed in a material. It increases as time of deposition increases and decrease as wavelength increases. The extinction coefficients of the films are low which show that the films absorb little of electromagnetic radiation that pass through it. Film of 5 hours has the highest extinction coefficient of 7.08×10^{-3} at 400 nm which decrease to 2.55×10^{-3} at 1100 nm. Film of 1 hour has the least extinction coefficient of 1.91×10^{-3} at 400 nm to which decreases to 2.38×10^{-4} at 1100 nm.

3.1.6. Band Gap Energy

The plot of absorption coefficient squared (α^2) against the photon energy is shown in Fig. 7 and Fig. 8. The straight nature of the plot indicates the existence of direct transition. The band gap was determined by extrapolating the straight portion to the photon energy axis at $\alpha^2 = 0$.

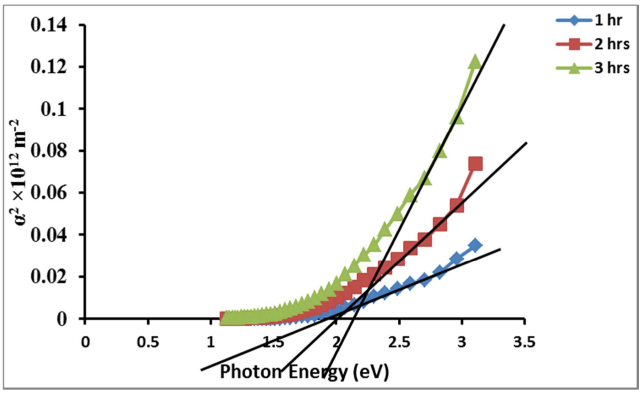

Figure 7. Plot of Absorption Coefficient Squared against Photon Energy of grown Thin Films of CdAgS at 1 hr, 2 hrs and 3 hrs.

The values of the band gap energy are shown in table 2 below for all the films. The graph of band gap energy against deposition time shows that band gap energy increases with increase in deposition time as shown in Fig. 9. This implies that CdAgS thin film has band gap energy that is between its parents binary semiconducting thin films of CdS (2.4 eV) as reported by [11] and AgS (1.78 eV – 2.09 eV) as reported by [16].

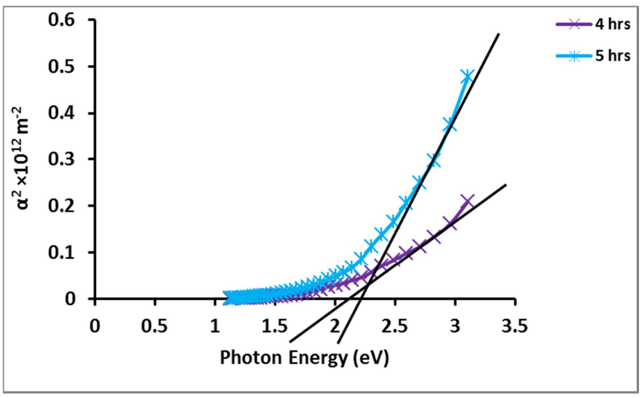

Figure 8. Plot of Absorption Coefficient Squared against Photon Energy of grown Thin Films of CdAgS at 4 hrs and 5 hrs.

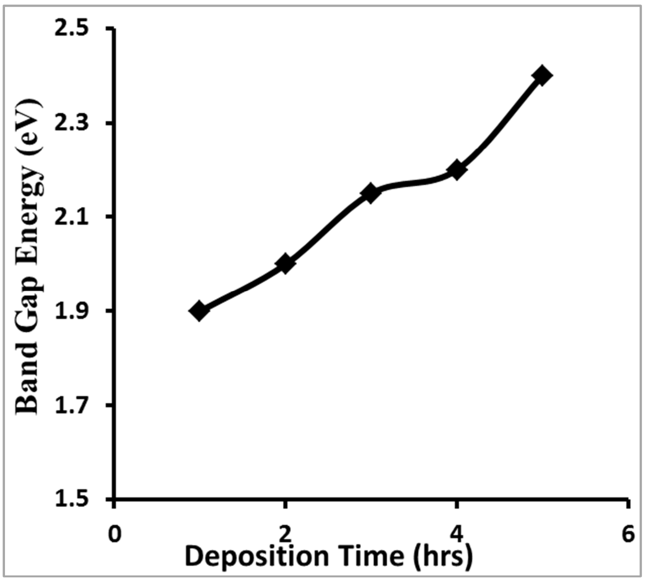

Figure 9. Graph of Band Gap Energy (eV) against Deposition Time (hrs).

Table 2. Band Gap Energy and Corresponding Deposition Time.

Time (hrs)	Band Gap	Thickness (μm)
1	1.90	0.086
2	2.00	0.130
3	2.15	0.147
4	2.20	0.174
5	2.40	0.199

3.2. Surface Structure Analysis

Surface morphology of deposited CdAgS films is presented in Fig. 10 and Fig. 11 respectively. The microstructure of the films reveals that the crystallites are of varying sizes and orientation. This property suggests that the grown films are polycrystalline. ImageJ for Microscopy Image Analysis [35] software was used to determine the average particle size of the films from the micrograph images. The average particle size of 94.54 nm and 732.45 nm were obtained for films deposited at 2 hours and 4 hours.

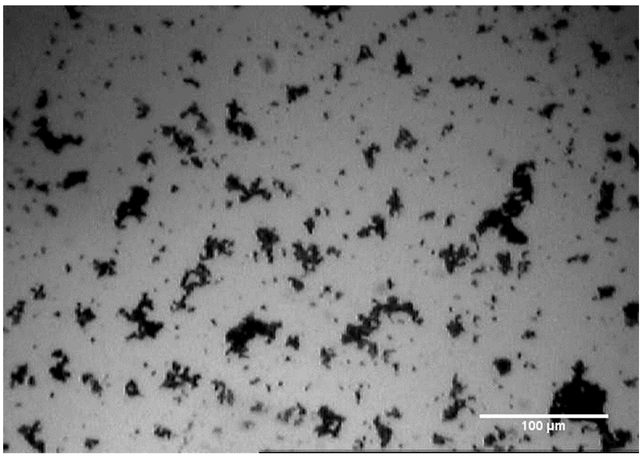

Figure 10. Micrograph of Cadmium Silver Ternary Thin Film Deposited at 2 hours.

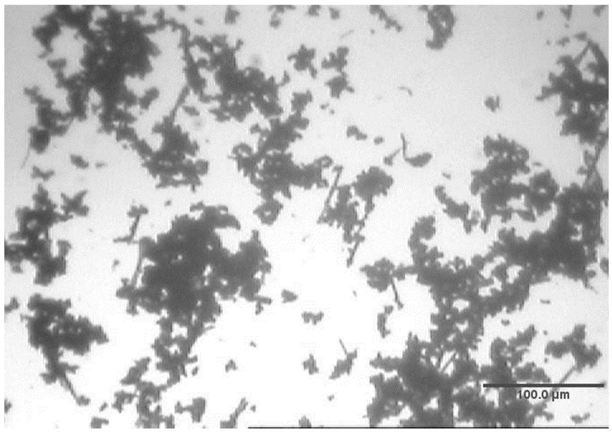

Figure 11. Micrograph of Cadmium Silver Ternary Thin Film Deposited at 4 hours.

4. Conclusion

Ternary thin films of Cadmium Silver Sulphide (CdAgS) have been grown on glass substrate using Chemical Bath Method at difference deposition time and characterized using a spectrophotometer to determine its optical properties. These optical properties studied vary with time, absorbance, reflectance, refractive index, extinction coefficient and band gap energy of these films increase as time of deposition increases while transmittance decreases as time of deposition increases. These films have low absorbance and high transmittance in VIS and NIR regions. The reflectance of the films is low < 15%. Refractive index of the films is between 1.12 – 1.90 and extinction coefficient of 1.83×10^{-3} - 2.39×10^{-4}. These results above suggest that the thin films can suitably be applied in the following: (i) solar cell fabrication, (ii) for the screening off UV radiation that is harmful to human beings and animals due to its low absorbance, high transmittance and low reflectance in VIS and NIR regions, (iii) optoelectronic devices, (iv) architectural design for cooling or heating buildings, (v) coating of windscreen and driving mirrors to reduce the effect of dazzling of light into driver's eyes etc. These deductions agree with the findings of other researchers on similar films [36], [37]. These values of the band gap energy 1.90 eV to 2.40 eV for CdAgS show that it is possible to tailor the band gap energy of Cadmium sulphide (CdS) semiconducting film by combining it with Silver Sulphide (AgS).

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