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PREPARATION AND CHARACTERIZATION OF NANOCOMPOSITE THIN FILMS OF CADMIUM SULPHIDE & ZINC SULPHIDE

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ABSTRACT

In this study, nanocomposite thin film of cadmium sulphide and zinc sulphide has been prepared by chemical bath deposition method. The ratio of cadmium sulphide and zinc sulphide was different for each deposited layer. The thin film was kept at room temperature overnight after deposition of each layer. The next layer was deposited on the thin film prepared one day earlier. Finally three layers of film are obtained. Cadmium acetate and zinc acetate are mixed in a proper proportion for the preparation of composite thin film. The synthesized films will be characterized by different techniques and properties will be studied. For the Investigation of surface morphology and micro structural features of film, SEM has been used. The elemental compositional analysis of materials has been identified by EDAX (energy dispersive X-ray analysis) also referred as EDX and EDS. Absorbance spectral study provided by UV-visible spectroscopy, which gives a measure of absorbance as a nature of band gap of semiconductor nano material and the value of band gap and particle size. X- ray diffraction will reveal the structural properties of the prepared films. SEM micrograph of the single, double and multilayer films at 50K magnification matches with the standard images. The particles are seen to be spherical and asymmetrical. The particles have seen to be spherical and asymmetrical. XRD have confirmed the polycrystalline growth for various arrangements of single layer, double layer and as-deposited multilayer thin film of CdS and CdZnS. All films found cubic and hexagonal structures. The EDS studies showed the presence of cadmium zinc and sulphur along with some impurities. It can be clearly seen that during the deposition of films, the impurities such as carbon, oxygen, and nitrogen found due to the presence of air. The thicknesses of the films were measured by optical interference method (multiple beam interferometers). The Band gap of thin films has investigated by Tauc's Plot. In this investigation, band gap for CdS thin film was 2.52 eV, for CdS-($Cd_{0.8}$ $Zn_{0.2}$) S thin film was 2.65 eV and for CdS- ($Cd_{0.8}$ $Zn_{0.2}$)S- ($Cd_{0.5}$ $Zn_{0.5}$)S thin film was 2.75 eV.

Keywords: Bandgap, Thin film, Nanotechnology, Nanomaterials, Zinc Sulphide.

1.INTRODUCTION

The most common and recently developed technique in the field of nanotechnology is called thin film technology. A material that meets these requirements is needed for the vast array of innovative procedures. The everyday routine has been significantly impacted by nanotechnology and nanomaterial research during the last few decades. The creation, analysis, and use of semiconducting nanoparticles have recently attracted a lot of attention. Due to the quantum size effect and the huge surface area, when the sizes of semiconducting nanoparticles are reduced to the nanosize range, their electrical and optical properties radically alter. Semiconducting particles can have their conductivity and optical characteristics changed.

The characteristics of thin films of (Cd-Zn) S are fascinating because they fall between those of ZnS and CdS [1–5]. It is regarded as a somewhat practical transparent metal sulphide for use in solar cells as a window. Its UV absorbance is lower and its band gaps are often more than 2.4 eV [1, 5, 6–8]. Cadmium Zinc Sulphide, a ternary compound that belongs to the II-VI group of compounds, with the chemical formula (Cd-Zn)S. For broad band-gap windows in heterojunction photovoltaic cells and photoconductive devices, (Cd-Zn)S thin films have been widely employed. CdS films have been shown to be effective in solar cell systems; however, the replacement of CdS with the higher band gap ternary compound (Cd-Zn) S has resulted in a decrease in window absorption losses and an increase in the (Cd-Zn)S has an excellent optical, electronic, and morphological property that made it an important key component in the area of nanotechnology and thin film based technology. The direct band gap of (Cd-Zn)S, which results in the visible region and is at 2.27 eV, attracted a lot of interest. Due to their high absorption coefficient, it finds extensive use in a variety of industries, including lasers, buffer layers in thin-film heterojunction solar cells, biological applications, non-linear optical devices, and display devices, photovoltaic cell and piezoelectric transducer [9–12].

The CdxZn1-xS belongs to II-VI group. It has a variable band gap which is the function of composition between 2.42 eV for CdS and 3.70 eV for ZnS [13, 14]. Chemical deposition techniques are chosen over physical deposition techniques for the deposition of CdZnS thin films due to their cost effectiveness. The deposition of CdZnS thin films has been accomplished using a number of low-cost chemical methodologies and techniques, particularly Chemical Bath Deposition (CBD) [14,16], Spray Pyrolysis [17,18], Successive Ion Layer Adsorption and Reaction (SILAR) [19], Dip Coating [20], and Ink Screen-Printing [21]. Because controlled precipitation from a solution over a broad surface is feasible under ambient circumstances, CBD is a little more beneficial than the other chemical techniques of deposition [22]. The thin films that are produced are of an equivalent quality to those created using physical deposition techniques.

To understand the structural, morphological, and optical features for the feasibility of (Cd-Zn)S thin films in more advanced optoelectronic and photovoltaic devices, inclusion of Zn element in the CdS has been examined in the current study.

2. MATERIAL AND METHOD

Cadmium acetate and zinc acetate are the mixed in proper proportion. Triethanolamine, 30% aqueous ammonia solution, thiourea and PVP polymer are added to the cadmium acetate and zinc acetate according to the requirement for the thin film preparation. The thin film was deposited on the glass substrate**.**

2.1: Multilayer bulk thin film

The multilayer thin film was deposited on glass slides using the Chemical Bath Deposition *(*CBD) technique. All the apparatus required to prepare the solution were initially degreased in HCl, followed by acetone and lastly cleaned with triple distilled water. They were allowed to dry in open air. For the first layer deposition of CdS bulk thin film, the solution of cadmium acetate, triethanolamine, aqueous ammonia and thiourea was prepared. Instantly the solution is stirred on magnetic stirrer for 60 minutes. The bath temperature was set to 60°C and glass slides were placed vertically in the solution. The deposition of film in chemical bath was carried for 2 hours. The film was taken out from the bath and left to dry at room temperature overnight. The second layer of cadmium sulphide and zinc sulphide was deposited on the first layer. The solution prepared has cadmium acetate and zinc acetate in a certain ratio followed by triethanolamine, aqueous ammonia and thiourea. The stirring time of the solution was one hour. The bath was set to 60°C and the glass slide with earlier deposited thin film of CdS was placed vertically in the solution for 2 hours. Similarly the third layer of cadmium sulphide and zinc sulphide (different in concentration ratio from the second layer) was deposited on the second layer. This procedure resulted in the formation of cadmium sulphide and zinc sulphide multilayer bulk thin film.

2.2: Multilayer nano thin film

The similar procedure detailed above for the bulk film deposition was carried for the cadmium sulphide and zinc sulphide multilayer nano thin film deposition. The only difference was pvp which acted as the capping agent for the nano thin film formation. The solution is prepared in the similar manner for all the three films just that before stirring, pvp is added to the solution. After deposition of each layer of film, it is left to dry at room temperature.

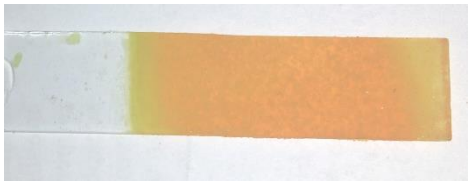


Fig.1: Single layer CdS nano film deposited on glass substrate.

Fig.2: Double layer CdS-($Cd_{0.8}Zn_{0.2}$)S nano film.

Fig.3: Multilayer CdS-($Cd_{0.8}Zn_{0.2}$)S-($Cd_{0.5}Zn_{0.5}$)S thin film.

3. RESULTS AND DISCUSSIONS

3.1: Absorption Spectral Studies

Optical spectroscopy is one of the most powerful and extensively employed analytical techniques in use and offer a very consistent methodology for characterization of materials. The optical absorption spectrum is a measure of absorbance as a function of wavelength of incident light. If the proportion of light scattered is small then it is important only to consider the light absorbed or transmitted. Absorption spectral studies also give information about the nature of band gap of semiconducting material and the value of band gap.

The optical absorption coefficient α describe the exponential decay of light intensity with the path within a solid. It is described using the relation

$$I=I_o\exp(-\alpha d) \quad \ldots (1)$$

Where I is the intensity transmitted through the sample at a particular wavelength.

I_o is the incident light intensity at the same wavelength and d is the film thickness.

The absorption of an optical medium also referred to as absorbance is defined as

$$\text{Absorbance } A=\log(1/T) \quad \ldots(2)$$

Where $T=I/I_o$ is called transmittance.

Therefore α is directly proportional to absorbance and is given by

$$\alpha = 2.030 * A/d \quad \ldots (3)$$

Where α is the absorption coefficient and d is the thickness of the film.

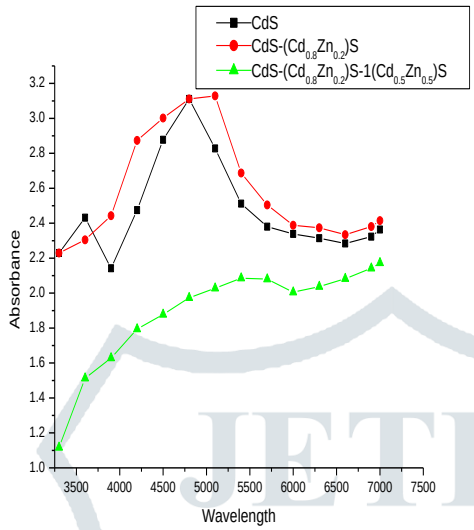


Fig.4: Absorption spectrum of multilayer CdS-($Cd_{0.8}Zn_{0.2}$)S-($Cd_{0.5}Zn_{0.5}$)S thin film .

The film shows good optical absorbance and sharp increase absorbance in the visible region. From the above Fig.4, it is clear that the character of the curves is similar, with absorption edge ranging between 4000 $\overset{0}{A}$ -5500 $\overset{0}{A}$. The strong absorbance of nanocrystalline CdS and (Cd-Zn)S films can be utilized in the design of optical and optoelectronic devices. The optical absorbance is a powerful method to find out the energy gap as well as optical properties of the sample.

Tauc's Plot and Band gap Determination

To test the nature of band gap and also to determine its values, plots between $(\alpha h\nu)^2$ and hν were drawn (Tauc's plot) where α is the absorption coefficient. The absorption coefficient α and band gap Eg for direct band gap materials are represented by the equation.

$$\alpha = c\,(h\nu - Eg)^{1/2}/h\nu \quad \ldots (4)$$

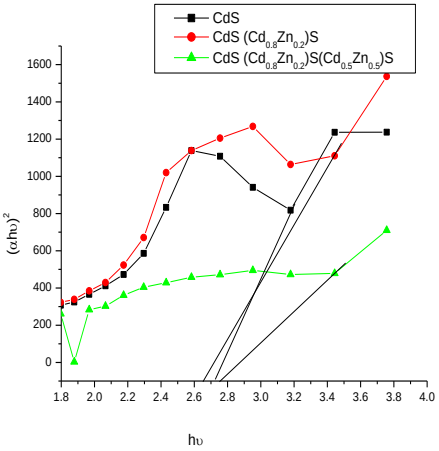


Fig.5: Tauc's plot multilayer CdS-($Cd_{0.8}$ $Zn_{0.2}$)S-($Cd_{0.5}$ $Zn_{0.5}$)S thin film (a) as deposited film (b) annealed film.

Table 1: Energy Band gap value of different arrangement of CdS, ($Cd_{0.8}$ $Zn_{0.2}$)S, and ($Cd_{0.5}$ $Zn_{0.5}$)S single layer, double layer and multilayer thin film

S no	Sample	As-deposited films Band gap(eV)
1	CdS thin film	2.52
2	CdS- ($Cd_{0.8}$ $Zn_{0.2}$)S thin film	2.65
3	CdS- ($Cd_{0.8}$ $Zn_{0.2}$)S- ($Cd_{0.5}$ $Zn_{0.5}$)S thin film	2.75

3.2: XRD Studies

X-Ray Diffraction is an analytical technique largely used for phase identification of a crystalline material and can provide information on unit cell dimension.

The d values and lattice constants were determined using the formulae given below:

$$d^2_{hkl} = 1/ (h^2/a^2 + k^2/b^2 + l^2/c^2) \quad \dots (5)$$

$$d^2 = a^2/ (h^2 + k^2 + l^2) \text{ cubic (for 'a')} \quad \dots (6)$$

$$d^2_{hkl} = 3a^2/\{ 4* (h^2 + hk + k^2)\} \text{ hexagonal (for 'a')} \quad \dots (7)$$

$$d^2_{hkl} = 1/ [\{4*(h^2 + hk + k^2)/ 3a^2\} + l^2/c^2] \text{ hexagonal (for 'c')} \quad \dots (8)$$

where h, k and l are the miller indices and 'd' is the interplanar spacing.

The particle size 'D' can be determined by the Debye-Scherer's formula

$$D = (0.94\lambda)/ (\beta_{1/2} * \cos \Theta) \quad \dots (9)$$

Where $\beta_{1/2}$ represents the full width at half maximum for the peaks expressed in radians, λ is wavelength and 'Θ' is the Bragg's angle.

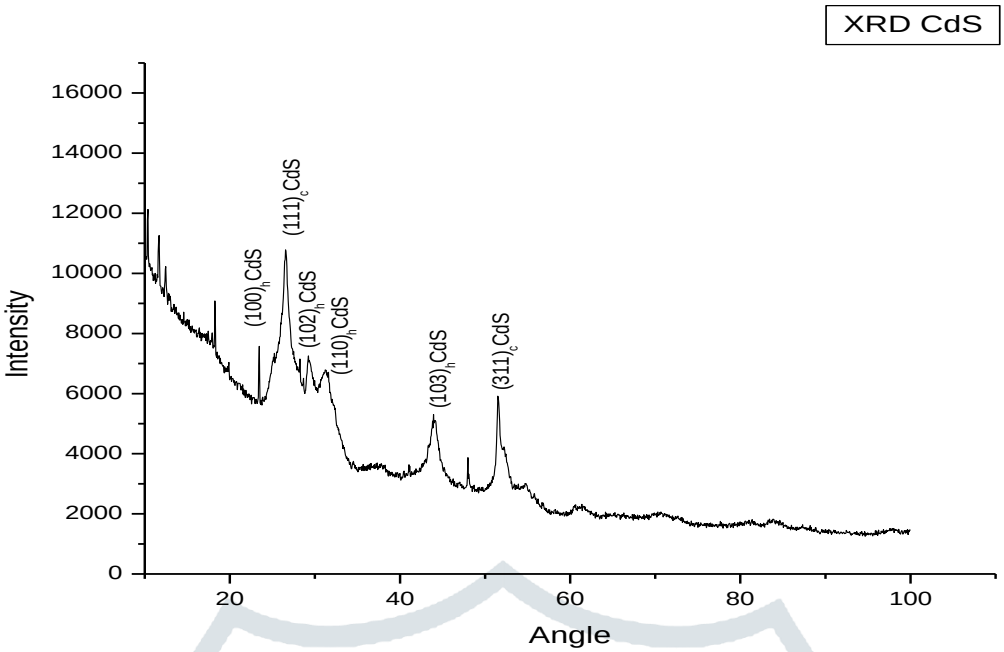


Fig.6 (a): XRD diffractogram of CdS nano film prepared on glass substrate.

Table 2: XRD data of CdS nano film prepared on glass substrate.

d value(A^0)		Relative Intensity		hkl	Lattice Constant	
(Obs)	(Rep)	(Rel)	(Rep)		(Obs)	(Rep)
3.55	3.58	100	75	100h CdS	a=4.09	a=4.13 c=6.71
3.28	3.36	6313	100	111c CdS	a=5.68	a=5.818
2.45	2.45	63.00	25	102h CdS	a=2.83 c=7.29	a=4.13 c=6.71
2.05	2.06	49.19	57	110h CdS	a=2.36	a=4.13 c=6.71
1.89	1.89	35.90	42	103h CdS	a=2.18 c=6.90	a=4.13 c=6.71
1.51	1.09	48.55	60	311c CdS	a=5.00	a=5.818

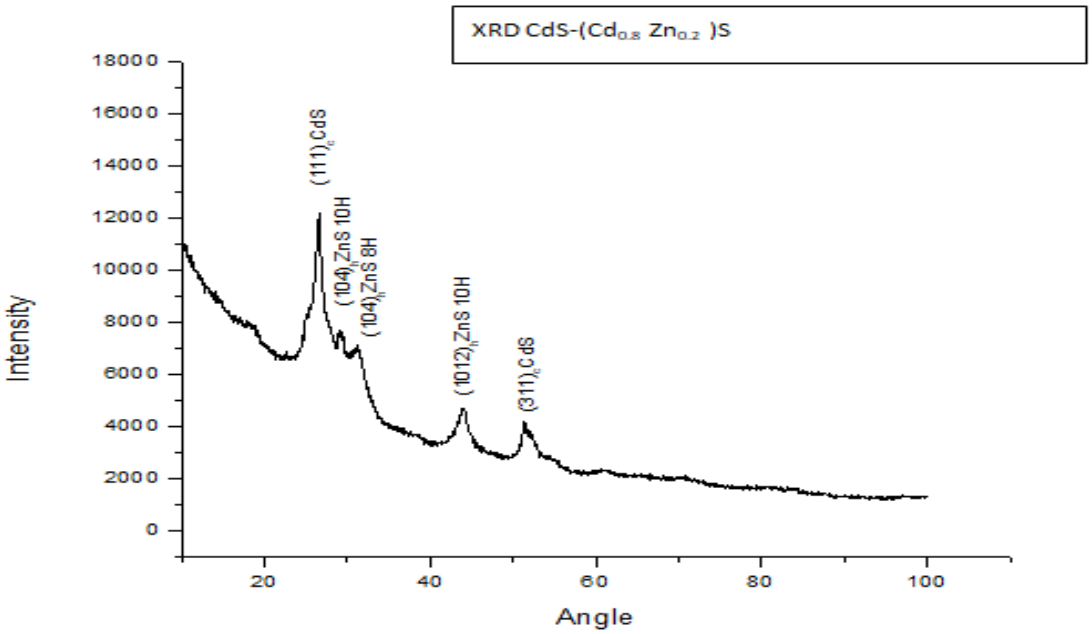


Fig.6(b): XRD diffractogram of double layer CdS-($Cd_{0.8}$ $Zn_{0.2}$)S thin film.

Table 3: XRD data of double layer CdS-($Cd_{0.8}$ $Zn_{0.2}$)S thin film prepared on glass substrate.

d Value(A^0)		Relative Intensity		hkl	Lattice Constant	
(Obs)	(Rep)	(Rel)	(Rep)		(Obs)	(Rep)
3.35	3.36	100	100	111c CdS	a=5.80	a=5.818
3.05	3.05	75.23	4	104h ZnS 10H	a=3.52 c=31.39	a=3.82 c=31.20
2.83	2.81	65.93	2	104h ZnS 8H	a=3.26 a=21.85	a=3.82 c=24.96
2.05	2.08	41.25	2	1012h ZnS 10H	a=2.36 c=31.34	a=3.52 c=31.20
1.51	1.09	48.55	60	311c CdS	a=5.00	a=5.818

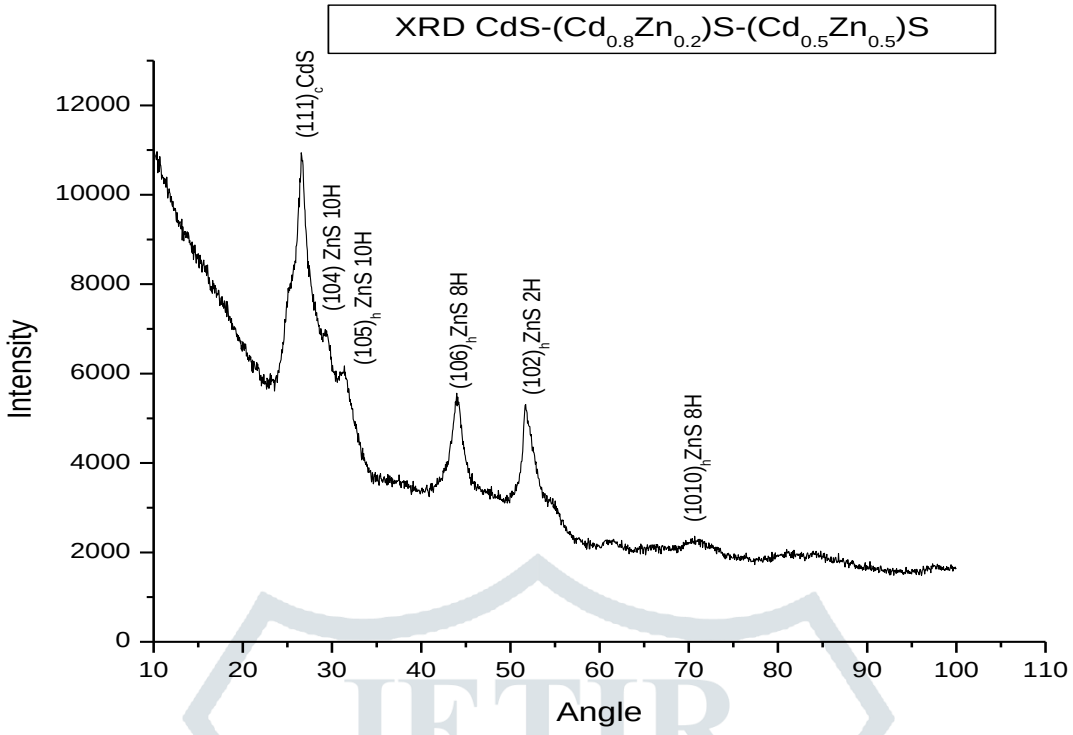


Fig. 6(c): XRD pattern of multilayer thin film CdS-($Cd_{0.8}$ $Zn_{0.2}$)S-($Cd_{0.5}Zn_{0.5}$)S.

Table 4: XRD data of multilayer CdS-($Cd_{0.8}$ $Zn_{0.2}$)S-($Cd_{0.5}Zn_{0.5}$)S thin film.

d Value(A^0)		Relative Intensity		hkl	Lattice Constant	
(Obs)	(Rep)	(Rel)	(Rep)		(Obs)	(Rep)
3.35	3.36	100	100	111c CdS	a=5.80	a=5.818
3.05	3.05	75.23	4	104 ZnS 10H	a=3.52 c=31.39	a=3.82 c=31.20
2.94	2.93	76.76	6	105h ZnS 10H	a=3.39 c=32.05	a=3.82 c=31.20
2.61	2.61	33.39	2	106h ZnS 8H	a=2.73 c=25.47	a=3.82 c=24.96
2.31	2.27	30.15	29	102h ZnS 2H	a=2.66 c=6.45	a=3.82 c=6.26
1.98	1.97	30.71	2	1010 ZnS 8H	a=2.28 c=24.71	a=3.82 c=24.96

Fig. 6 shows the XRD peaks appearing at 26.548^0 and 51.496^0 corresponds to plane (111) and (311) and shows the cubic phase of CdS thin film whereas the peaks at 23.8570^0, 31.223^0, 33.93^0 and 43.97^0 shows the hexagonal phase of planes (100), (102), (110), (103) of CdS film. From this it is confirmed that the films formed is matching to the standard film. Fig. 5.2.2, 5.2.3 shows the XRD patterns of double layer, multilayer and

multilayer arrangements of thin films deposited. The XRD peaks in the (Cd Zn)S film corresponding to the diffraction from (111) and (311) cubic phases of CdS. In addition (0010),(102), (104),(105),(106),(108),(1010) (1012) planes of the hexagonal phase of ZnS in the XRD peaks in the (Cd Zn)S film . Results are very much closer to the standard values. The mixed phases of CdS and ZnS are a result of the effect of the oxygen, carbon and a few other impurities present in the environment.

The particle sizes of the CdS single layer film, CdS-($Cd_{0.8}$ $Zn_{0.2}$)S double layer film and CdS-($Cd_{0.8}$ $Zn_{0.2}$)S-($Cd_{0.5}$ $Zn_{0.5}$)S multilayer films were calculated by the Debye Scherrer's formula mentioned earlier. Particle sizes were found to be in the nano range.

Table 5: Particle size of single layer CdS film, double layer CdS-($Cd_{0.8}Zn_{0.2}$)S film and (as deposited and annealed) multilayer CdS-($Cd_{0.8}Zn_{0.2}$)S-($Cd_{0.5}Zn_{0.5}$)S thin film

S no	Sample	D(nm)
1	CdS	75.33
2	CdS-($Cd_{0.8}$ $Zn_{0.2}$)S	71.16
3	CdS-($Cd_{0.8}$ $Zn_{0.2}$)S- ($Cd_{0.5}$ $Zn_{0.5}$)S	74.12

3.3: SEM and EDS Studies

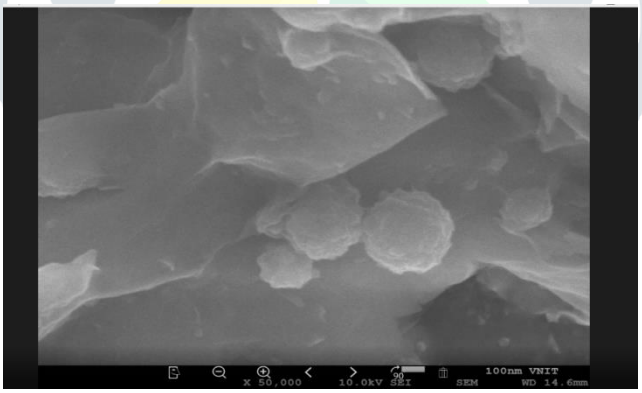


Fig. 7(a): SEM micrograph of CdS nano film at 50Kmagnification.

Fig. 7(a) shows the SEM micrograph of the CdS film at 50K magnification. The images match with the standard images of the CdS films. The surface of CdS is homogeneous with some large particles deposited on it. The particles are seen to be spherical and symmetrical. The layer is seen to be containing isolated grains of CdS formed above a compact polycrystalline CdS layer.

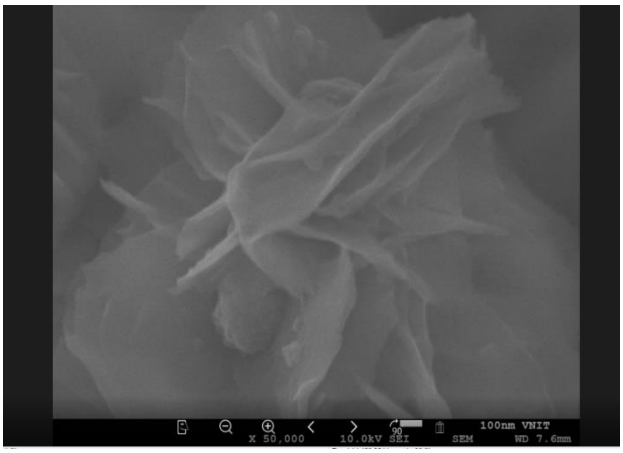


Fig.7(b): SEM micrograph of CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S at 50K magnification.

The Fig. 7(b) shows the SEM images of double layer thin film of arrangement CdS-(Cd $_{0.8}$ Zn$_{0.2}$)S. From the image layered structure can be observed.

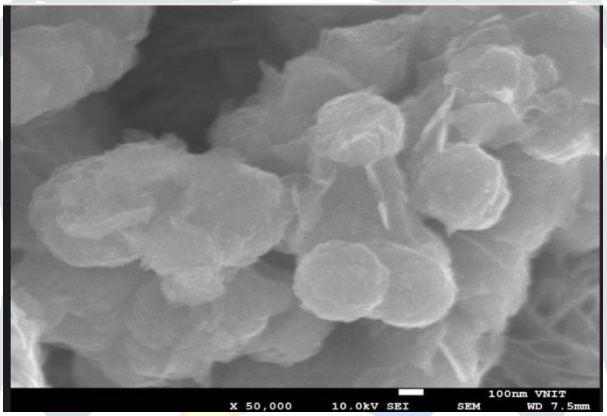
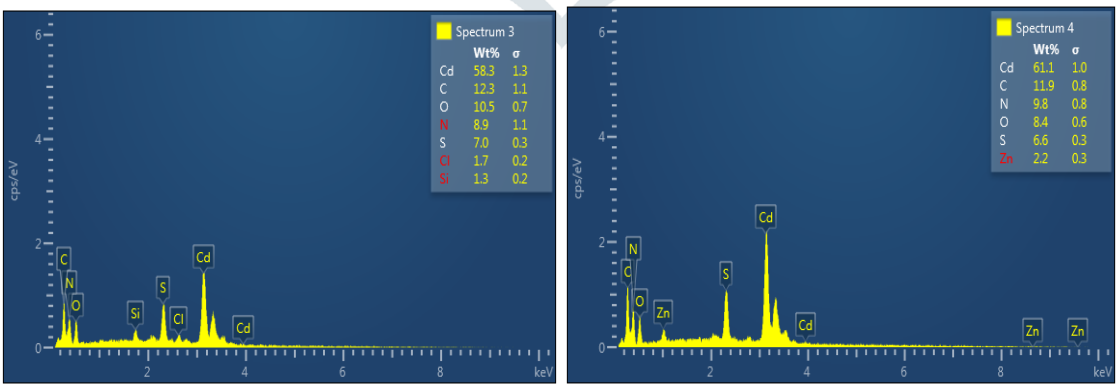
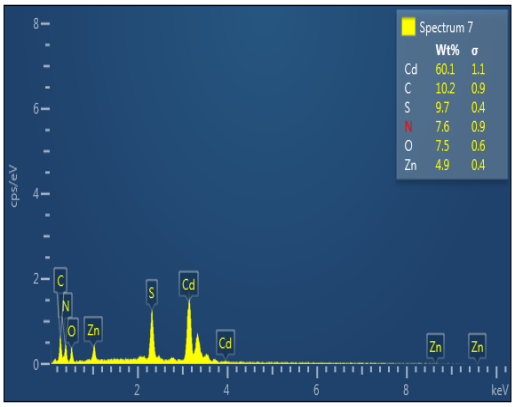


Fig.7(c): SEM micrograph of CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S-(Cd $_{0.5}$Zn $_{0.5}$)S thin film at 50K magnification.

From the Fig. 7(c) it is clear that the surface of the film is homogeneous with some large particles deposited on it. The particles are seen to be spherical and symmetrical. It is therefore concluded that the film is following a multilayer growth pattern.



(a) (b)



(c)

Fig 8: EDS images of (a) CdS nano film, (b) CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S nano film, (c) CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S-(Cd $_{0.5}$ Zn $_{0.5}$)S nano film

In material characterization, to verify how an element is distributed laterally and to find the inclusions on the surface is significant. This is most suitably done by using EDS technique. Fig.8 (a), (b) and (c) shows the EDX spectrum of CdS, CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S and CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S-(Cd $_{0.5}$ Zn $_{0.5}$)S films respectively. The spectrum confirms the Cd and S atoms are presented in the CdS film. From Fig.8 (b) and (c) spectrums the presence of Cd, Zn and S is confirmed. There are impurities atoms present on the film. The compositional analysis shows the presence of C, O and N in all the three spectrums however in CdS EDS spectrum Cl and Si are also oberserved along with C, O and N. The presence of carbon and oxygen may be attributed to air which were incorporated during synthesis. The stoichiometry ratio of CdS was found to 58.3%Cd and 7%S. The presence of chlorine may be due to residues of the byproduct from the synthesis. The stoichiometry ratio of Cd:Zn:S was found to 61.1%Cd, 2.2%Zn and 6.6%S in double layer thin film and that of multilayer thin film CdS-(Cd $_{0.8}$ Zn $_{0.2}$)S-(Cd $_{0.5}$ Zn $_{0.5}$)S is 60.1 % Cd, 4.9% Zn and 9.7% S.

4. CONCLUSIONS

The cadmium sulphide and cadmium zinc sulphide multilayer thin film was successfully deposited on glass substrate using chemical bath deposition (CBD) technique. The thickness measurement studies showed that the films prepared were getting deposited and were in the thin film range. The absorbance spectrum studies for the multilayer bulk thin film is better than mono layer and double layer thin film. The double layer nano thin film shows better absorbance spectrum than the mono layer nano thin film and multilayer nano thin film. The band gap energy of the CdS films was calculated and it was found to be as per the generalized results.

XRD characterization have confirmed the polycrystalline growth for various arrangement of CdS and CdZnS single layer, double layer, as-deposited multilayer thin film. All films have cubic and hexagonal structures.

SEM micrograph of the single, double and multilayer films at 50K magnification matches with the standard images. The surface of films is homogeneous with some large particle deposited on it. The particles are seen to be spherical and asymmetrical.

EDS studies show the presence of cadmium zinc and sulphur along with some impurities. The impurities such as carbon, oxygen, and nitrogen, are attributed to the presence of air during the deposition.

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