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***Research Article***

# THICKNESS DEPENDENT PHYSICAL PROPERTIES OF SPRAY DEPOSITED NANOCRYSTALLINE $Cd_{0.5}Fe_{0.5}S$ THIN FILMS

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## ABSTRACT

Ternary nanostructured $Cd_{0.5}Fe_{0.5}S$ thin films were deposited onto glass substrate by spray pyrolysis method at 573 K temperature using ferric chloride and cadmium chloride as cationic and thiourea as anionic precursor. The films of thickness 132 to 312nm were prepared by changing the quantity of spray solution from 10 to 25mL. The structural and morphological properties of the nanostructured $Cd_{0.5}Fe_{0.5}S$ thin films were investigated by XRD, EDS, SEM and AFM analysis respectively. The spray deposited $Cd_{0.5}Fe_{0.5}S$ thin films are nanocrystalline in nature with hexagonal crystal structure. The electrical resistivity of semiconducting $Cd_{0.5}Fe_{0.5}S$ thin films is of the order of $10^6$ Ωcm and it decreases as the film thickness increases. The thermo-emf measurement confirms n-type conductivity of $Cd_{0.5}Fe_{0.5}S$ thin films. The optical band gap energy of the as deposited $Cd_{0.5}Fe_{0.5}S$ film increases from 2.20eV to 2.40eV as the film thickness decreases from 312 to 132nm.

***Keywords:*** *Thin Films; Nanostructures; Chemical Properties; Electrical Properties****;*** *Optical Properties*

## INTRODUCTION

Thin film science has occupied a prominent place in the basic research and the applications of semiconductor thin films in various electronic and optoelectronic devices have attracted much attention of modern technology. There has been increasing interest in the growth of nanostructured ternary semiconducting materials which offers the great opportunity to mingle the magnetic, electrical and optical properties into a single semiconductor material. The diluted magnetic semiconductors (DMSs) (Furdyna, 1988) have attracted much attention due to the wealth of scientific information and potential technological applications. DMSs are semiconductor alloys formed by randomly replacing a fraction of the cations with magnetic ions, e.g., $Fe^{2+}$ in CdS to form $Cd_xFe_{1-x}S$. In these materials, the large sp–d exchange interaction between magnetic ions and electrons in valence band can lead to a number of unusual electronic, optical and magneto-optical properties including the ability to magnetically tune the band gap (Twardowski *et al.*, 1983; Twardowski *et al.*, 1984; Furdyna and Samarth, 1987). These properties make DMSs promising candidates for fabricating magneto-optical devices such as magnetic field sensors, isolators and magneto-optical switches (Hwang *et al.*, 2004). Few reports were available on chemically deposited Fe based ternary composite thin films such as $Pb_{1-x}Fe_xSe$ (Bhardwaj *et al.*, 2008), $Cd_{1-x}Fe_xS$ (Gao *et al.*, 2013; Wu *et al.*, 2006; Liua *et al.*, 2007), $FeCdS_3$ (Ubale and Ibrahim, 2012; Ubale and Ibrahim, 2011), Fe: CdSe (Pawar *et al.*, 2008) Cd-Fe-Se (Rajpure *et al.*, 1999) etc.

The fast growing Semiconductor Industry based on nanomaterials needs cost effective simple deposition process. Development of such economic, low temperature high-quality film growth technique is the prime need of the Nanotechnology. The various physical deposition techniques require a vacuum system along with sophisticated instrumentation. These techniques deliver quality films, but the cost of deposition systems has limited their potential applications. However the chemical spray pyrolysis deposition method is non-vacuum, economic, simple and low energy consumption method. In the present work, chemical spray pyrolysis method was used to prepare $Cd_{0.5}Fe_{0.5}S$ thin films of thickness 132 to 312 nm by varying the quantity of spray solution. The effect of film thickness on structural, electrical and optical properties of nanostructured $Cd_{0.5}Fe_{0.5}S$ thin films is discussed.

***Experimental Details***

The spray system used for present work has vertical assembly that consists of spray nozzle, substrate heater, temperature controller and air compressor. To measure flow of precursor solution and air, liquid and gas flow meters are used. The horizontal motion of spray nozzle was controlled by stepping motor along with programmed microcontroller. The distance between spray nozzle and substrate heater was kept 28 cm. The film formation depends on the process of droplet landing, reaction and solvent evaporation rate, which are related to droplet size and momentum (Lampkin, 1979; Patil, 1999). The spraying solution was of 10 mL of 0.1M ferric chloride, 10 mL of 0.1M cadmium chloride and 20 mL of 0.1M thiourea in a measuring cylinder. This solution was then sprayed onto hot glass substrates using compressed air as a carrier gas. Several trials were conducted to optimize the different deposition parameters such as substrate temperature, substrate to nozzle distance, spray rate etc. (Table 1). The optimized deposition temperature was found to be ≈ 573 K. The films deposited below this temperature were discontinuous and less adhesive. The optimized spray rate was found to be 6 mL min$^{-1}$. Four different sets of $Cd_{0.5}Fe_{0.5}S$ thin films were prepared by spraying 10, 15, 20 and 25mL of spray solution. The films deposited below 10mL of spray solution are discontinuous and inhomogeneous in nature. However the films deposited above 25mL of spray solution are powdery and less adhesive in nature. The average thickness of the film was measured by the gravimetric method. The two-point dc probe method of dark electrical resistivity was used to study the variation of resistivity with temperature. A copper block was used as a sample holder cum heater with chromel–alumel thermocouple to measure the temperature. For the measurement of resistivity, a constant voltage was applied across the sample and the variation of current with temperature was noted using a digital nanoameter. The structural studies were carried out using Philips PW 1710 diffractometer, with Cu-Ka radiation of 1.5405 A$^0$. The optical characteristics were studied using Lambda 25 UV-VIS spectrophotometer (PerkinElmer) to find band gap energy. The surface morphological studies were carried out using JEOL 6380A scanning electron microscope and the atomic force microscope (AFM) was used to analyze microstructures by molecular imaging in two- or three-dimensions.

**Table 1: Optimized preparative parameters**

| Name of Parameter | Optimized value |
|---|---|
| Composition of spray solution | 10 mL ,0.1 M Ferric chloride + 10 mL,0.1M Cadmium chloride + 20 mL, 0.1 M Thiourea |
| Nature of substrate | Amorphous glass |
| Substrate temperature | 573 ±5K |
| Spray rate | 6 ml /min |
| Spray nozzle diameter | 0.5 mm |
| Nozzle to substrate distance | 28 cm |

## RESULTS AND DISCUSSION

***Structural Analysis***

The structural properties of $Cd_{0.5}Fe_{0.5}S$ thin films were investigated by X-ray diffraction technique. Figure1 shows the X-ray diffraction spectra of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution from 10 to 25mL. The observed 'd' values were compared with the standard data (JCPDS file CdS: 80-06, 6-314 and FeS:80-1026,1027) to confirm the structure of the deposited thin films (Table 2). The spray deposited $Cd_{0.5}Fe_{0.5}S$ films are nanocrystalline in nature with mixture of hexagonal CdS and FeS phases. The film deposited at 10mL spray solution shows amorphous nature as no prominent peak is observed. It may be because for 10mL of spray solution the film thickness is very small with tiny grains formed on the substrate. These grains are further grown with quantity of spray solution showing peaks in the XRD pattern above 10mL. The (211) orientation due to hexagonal CdS and (217), (002) orientation due to hexagonal FeS are repeated after 15mL of spray solution. However, the (112) orientation due to hexagonal CdS and (103) orientation due to hexagonal FeS are repeated after 15mL of spray solution, also (114) and (212) orientation due to hexagonal FeS are observed above 15mL of spray solution. This variation may be due to alloy formation in the film. The hexagonal nature of $Cd_{0.5}Fe_{0.5}S$ observed is in good agreement with the results reported by Badera *et al.,* (1984). Also, several workers have reported hexagonal structures of chemically deposited CdS and FeS thin films (Patil and Singh; Mahmoud, 2001; Rami *et al.,* 1999; Thanikaikarasan *et al.,* 2010). The crystallites size was calculated by using FWHM data and Debye Scherrer's formula (Mahmoud, 2001).

$$d = \frac{K\lambda}{\beta Cos\theta} \tag{1}$$

Where the constant 'k' is the shape factor = 0.94, λ is the wavelength used (0.154nm), β is the angular line width at half maximum intensity, θ is the Bragg's angle. The variation of grain size from 16 to 41 nm was observed with film thickness, which may be due to improved crystalline quantity of film. The strain (ε) and presence of dislocations strongly influences the physical and chemical properties of the films (Senthilarasu *et al.,* 2007). The strain (ε) was calculated from the formula,

$$\varepsilon = \frac{\beta Cos\theta}{4} \tag{2}$$

**Table 2: Comparison of observed and standard XRD data of $Cd_{0.5}Fe_{0.5}S$ thin films (JCPDS card CdS: 80-06, 6-314 and FeS:80-1026,1027)**

| Film | Observed data | | Standard data | | hkl | phase |
|---|---|---|---|---|---|---|
| | 2θ(degree) | d ($A^0$) | 2θ(degree) | d ($A^0$) | | |
| A | --- | --- | --- | --- | --- | --- |
| B | 18.807 | 4.714 | 18.701 | 5.871 | 0 0 2 | FeS |
| | 71.331 | 1.321 | 71.263 | 1.322 | 2 1 1 | CdS |
| | 74.307 | 1.275 | 74.400 | 1.274 | 2 1 7 | FeS |
| C | 18.807 | 4.714 | 18.701 | 5.871 | 0 0 2 | FeS |
| | 28.440 | 3.135 | 28.568 | 3.122 | 1 0 3 | FeS |
| | 43.942 | 2.058 | 43.180 | 2.093 | 1 1 4 | FeS |
| | 51.850 | 1.761 | 51.875 | 1.761 | 1 1 2 | CdS |
| | 71.331 | 1.321 | 71.263 | 1.322 | 2 1 1 | CdS |
| | 74.307 | 1.275 | 74.400 | 1.274 | 2 1 7 | FeS |
| D | 18.807 | 4.714 | 18.701 | 5.871 | 0 0 2 | FeS |
| | 28.540 | 3.112 | 28.568 | 3.122 | 1 0 3 | FeS |
| | 48.040 | 1.892 | 48.985 | 1.858 | 2 1 2 | FeS |
| | 51.850 | 1.761 | 51.875 | 1.761 | 1 1 2 | CdS |
| | 71.331 | 1.321 | 71.263 | 1.322 | 2 1 1 | CdS |
| | 74.307 | 1.275 | 74.400 | 1.274 | 2 1 7 | FeS |

The strain of the film on the substrate deposited using 15mL of spray solution is of the order of $19\times10^3$ $Lin^{-2}nm$ and it decreases to $14.21\times10^3$ $Lin^{-2}nm$ at 25mL. The decrease in the strain is because of increased film thickness with improved grain structure. The dislocation density (δ), defined as the length of dislocation lines per unit volume of the crystal, was evaluated from the formula (Chowdhury *et al.,* 2012),

$$\delta = \frac{1}{d^2} \tag{3}$$

The calculated values of the strain (ε) and dislocation density (δ) are given in Table 3. The dislocation density of $Cd_{0.5}Fe_{0.5}S$ is $10.86 \times 10^{-4}$ and decreases to $5.850 \times 10^{-4}$ $nm^{-2}$ with increase in thickness respectively and shows slight variation depending on crystallite size.

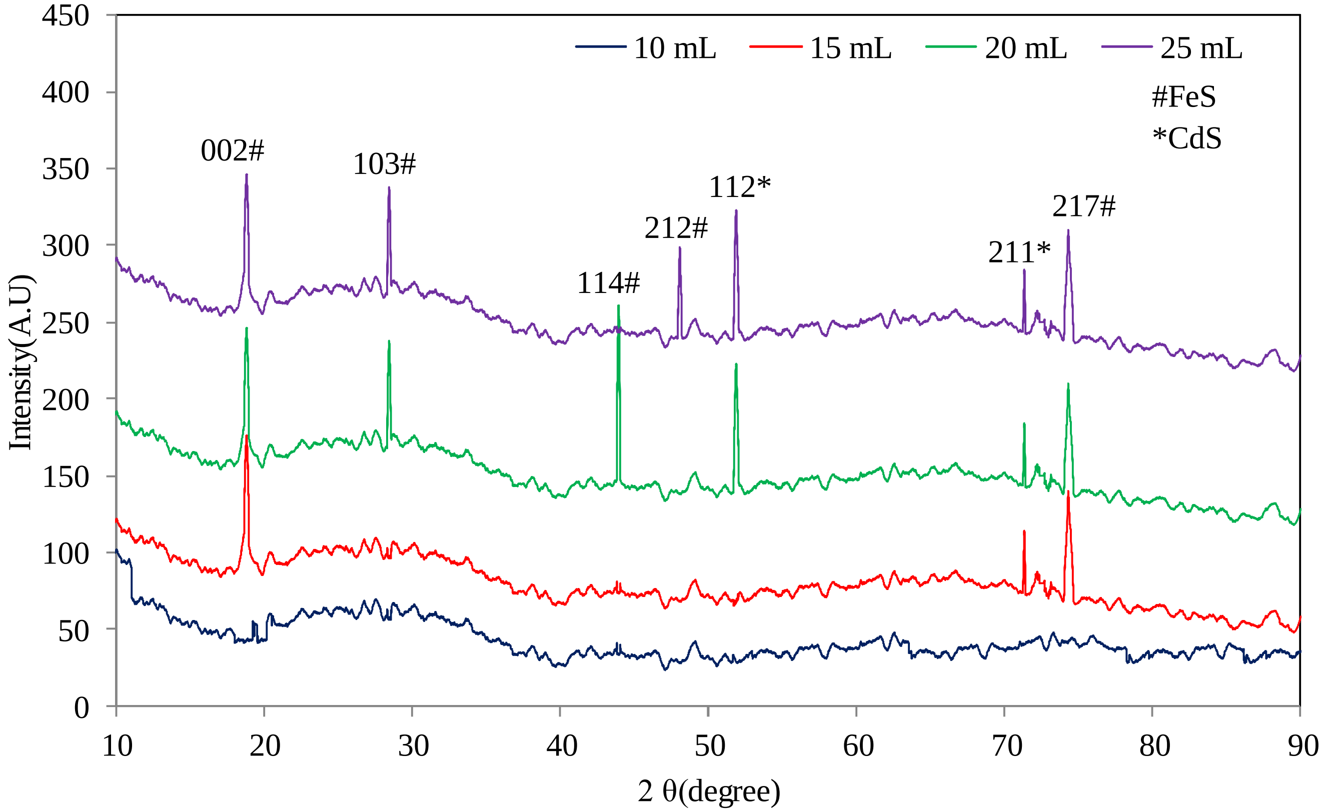


**Figure1: XRD patterns of composite $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution.**

**Table 3: Variation of crystallite size, dislocation density, strain, band gap energy and activation energy of $Cd_{0.5}Fe_{0.5}S$ thin films with spray volume**

| Film | Spray Volume | Film thickness (nm) | Crystallite Size D (nm) | Dislocation Density $\delta \times 10^4 (nm^{-2})$ | Strain $\varepsilon \times 10^3$ ($lin^{-2}nm$) | Band gap Energy Eg (eV) | Activation energy Ea (eV) |
|---|---|---|---|---|---|---|---|
| A | 10mL | 132 | ----- | ----- | --- | 2.40 | 0.052 |
| B | 15mL | 249 | 16.04 | 10.37 | 19.77 | 2.34 | 0.045 |
| C | 20mL | 266 | 38.47 | 6.756 | 19.06 | 2.28 | 0.041 |
| D | 25mL | 312 | 41.34 | 5.850 | 14.21 | 2.20 | 0.048 |

***Morphology***

The SEM micrographs of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying quantity of spray solution are shown in Figure 2. It is observed that the as deposited $Cd_{0.5}Fe_{0.5}S$ thin films are continuous with agglomeration of grains at some places at lower thickness. However the grain growth is observed with thickness. At 20mL of spray solution the film surface shows well defined porous morphology with overgrowth at some places. The porous network contains number of voids however, at 25mL spray solution the void size get decreased with irregular overgrowth. The overgrowth observed at 25mL of spray solution may be due to over deposition of excess quantity of spray solution.

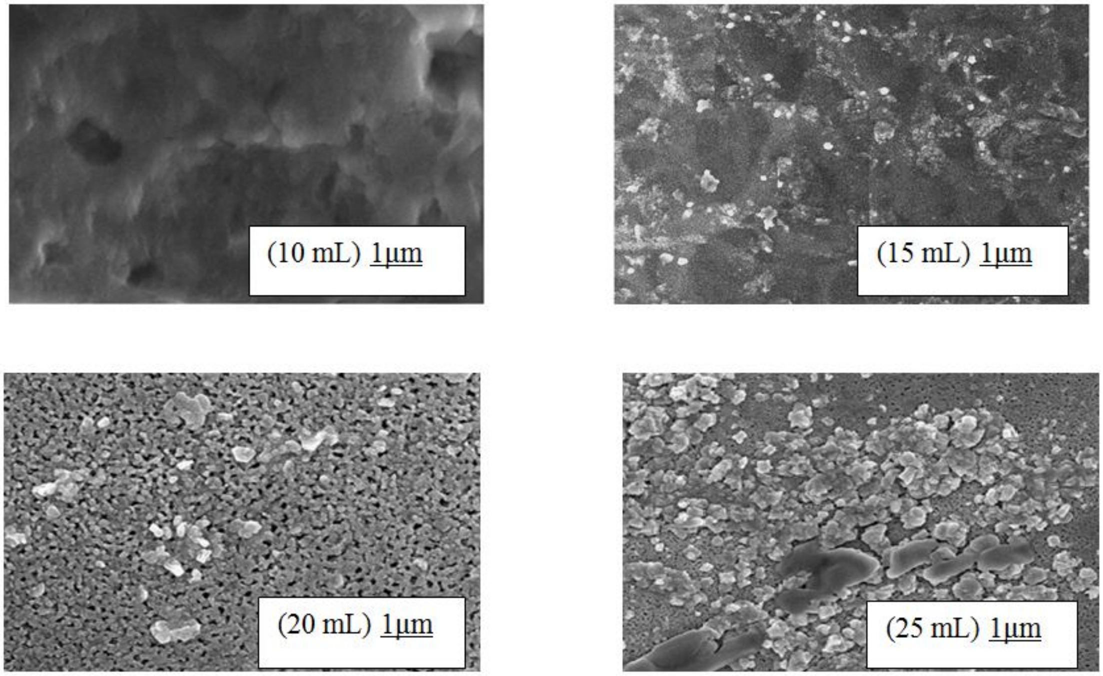


**Figure 2: SEM images of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

To study the stoichiometry of the film quantitative analysis was carried out using the EDAX technique. Figure 4 shows the 2D AFM images of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution from 10 to 25mL. It can be seen that spray deposited $Cd_{0.5}Fe_{0.5}S$ thin film have granular structure. In addition to granular nature, the film substrate is dense, uniform with porous nature are observed. Figure 3 shows typical EDAX patterns of the $Cd_{0.5}Fe_{0.5}S$ thin film of different thickness.

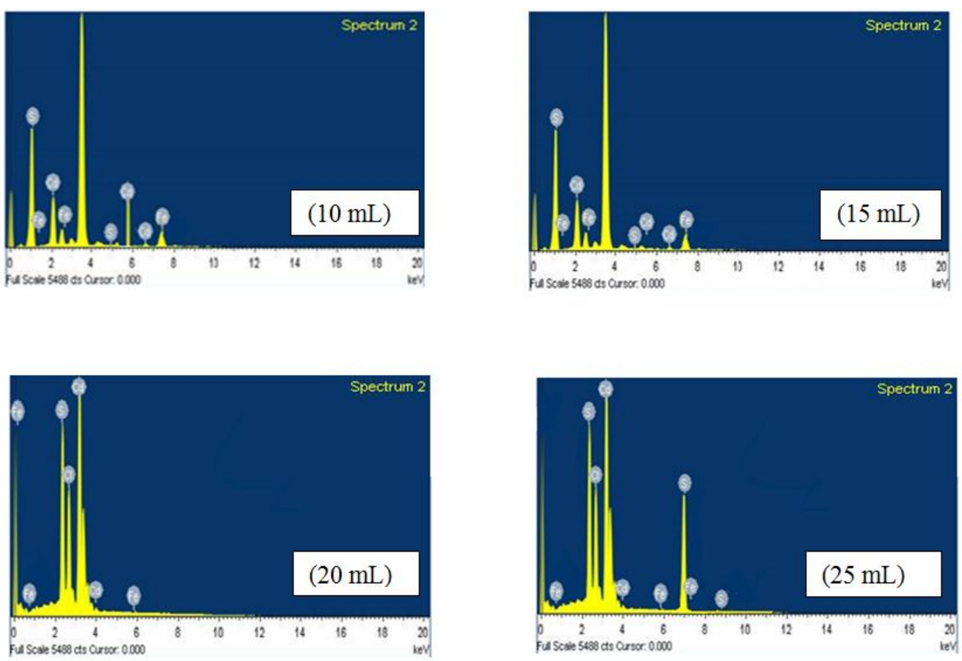


**Figure 3: EDAX spectrum of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

The elemental analysis was carried out only for Fe, Cd and S. However, there are some additional peaks corresponding to Si, O, Ca, Mg, etc., in the EDAX spectra, which could be due to the presence of these elements in the glass substrate. The average atomic percentage ratio of Cd: Fe: S is listed in Table 4, and it shows that the elemental composition in the film is almost in good agreement with experimental expected composition of $Cd_{0.5}Fe_{0.5}S$. Figure 4 shows two-dimensional surface morphology of the spray deposited $Cd_{0.5}Fe_{0.5}S$ thin films investigated by atomic force micrographs.

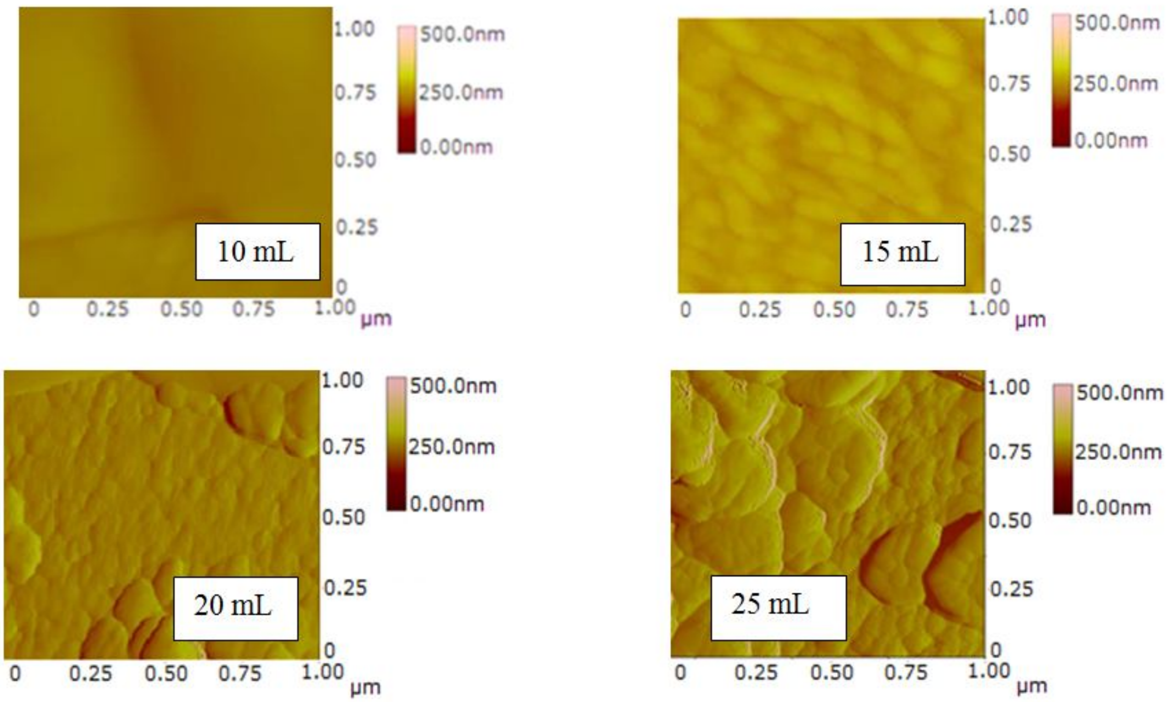


**Figure 4: AFM of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

**Table 4: Elemental composition of $Cd_{0.5}Fe_{0.5}S$ thin films**

| **Film** | **Spray Volume** | **Final atomic percentage in the film by EDAX analysis (%)** | | |
|---|---|---|---|---|
| | | Cd | Fe | S |
| A | 10mL | 25.2 | 25.1 | 49.7 |
| B | 15mL | 24.7 | 26.6 | 48.7 |
| C | 20mL | 25.3 | 25.5 | 49.2 |
| D | 25mL | 24.9 | 26.3 | 48.8 |

***Electrical Analysis***

Figure 5 shows the IV-characteristics of $Cd_{0.5}Fe_{0.5}S$ thin films. The linear nature of the curve confirms that silver forms ohmic contact with film. The current rises with thickness which may be due to increase in carrier concentration. The electrical resistivity of the $Cd_{0.}Fe_{0.5}S$ thin films deposited by varying quantity of spray solution was measured using the d.c. two probe method in air. Figure 6 shows the variation of log ρ with reciprocal of temperature (1000/T). It is seen that resistivity decreases with temperature indicating semiconducting nature of $Cd_{0.5}Fe_{0.5}S$ thin films. The resistivity follows the relation,

$$\rho=\rho_0 \exp (E_a/KT) \qquad (4)$$

Where 'ρ' is resistivity at temperature 'T', $\rho_0$ is a constant, 'K' is the Boltzmann constant (1.38 x $10^{-23}$J/k) and '$E_a$' is the activation energy required for conduction.

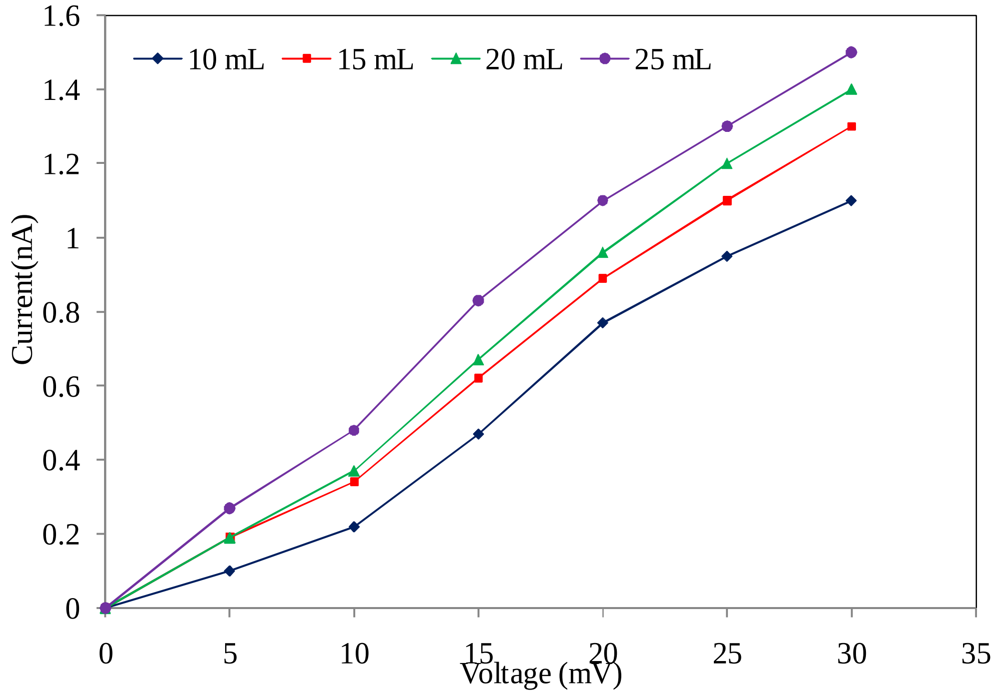


**Figure 5: I-V characteristic of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

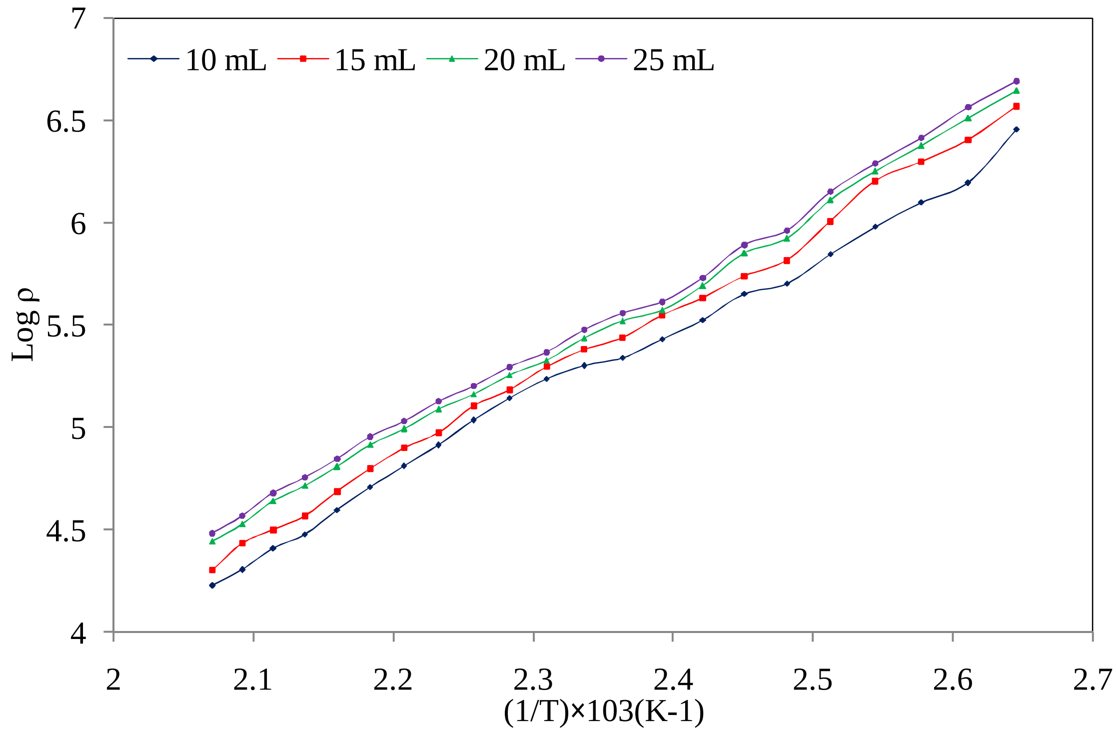


**Figure 6: Variation of Log of resistivity with 1/T of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

The resistivity of the $Cd_{0.5}Fe_{0.5}S$ film at 303 K is of the order of $6.4 \times 10^6 \Omega cm$ and it decreases to $3.9 \times 10^6 \Omega cm$ as the quantity of spray solution increased from 10 to 25mL. This decrease in resistivity may be due to improved crystalline nature of film. Table 3 shows activation energies of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying quantity of spray solution. The activation energy of $Cd_{0.5}Fe_{0.5}S$ thin films is of the order of 0.054eV and it decreases to 0.048 eV as the film thickness increases.

The thermo-emf developed across hot-cold junction of $Cd_{0.5}Fe_{0.5}S$ thin film in dark was measured as a function of temperature difference (Figure 7). The polarity of the generated thermo-emf was negative at the cold end with respect to the hot end, which confirms that $Cd_{0.5}Fe_{0.5}S$ films are of n-type.

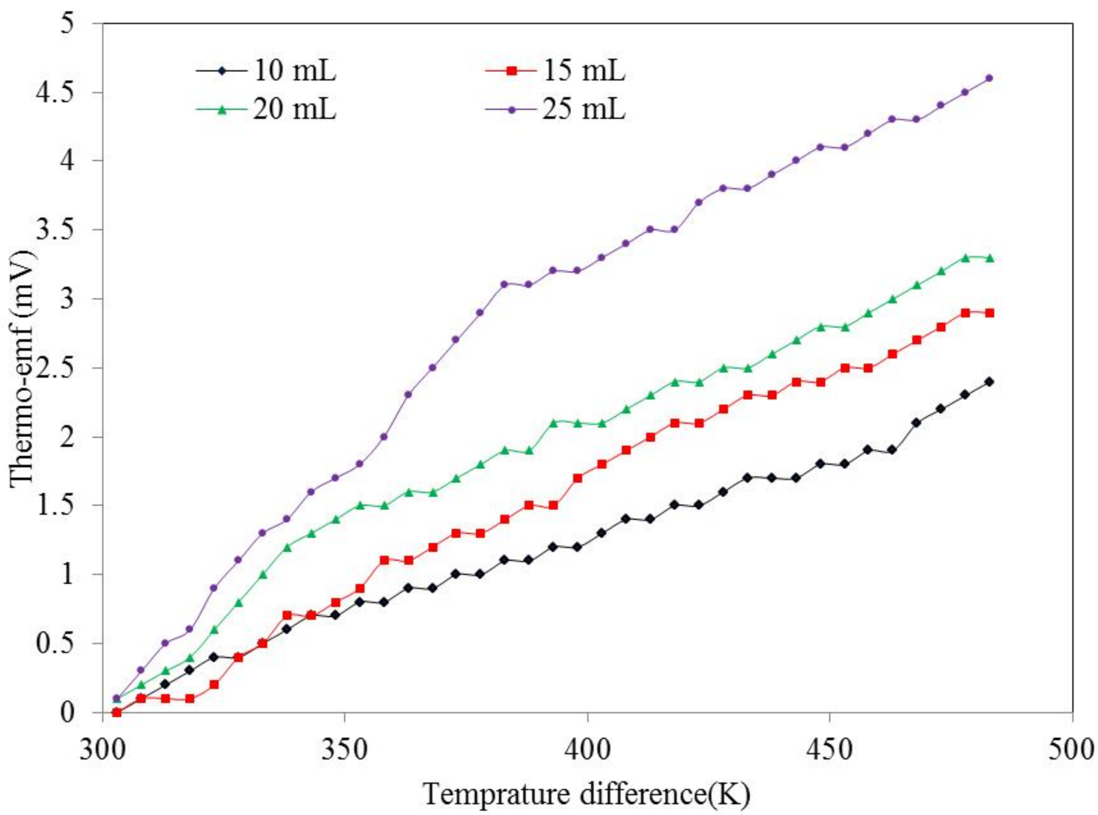


**Figure 7: Variation of thermo emf (mV) with temperature difference of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

***Optical Analysis***

The study of optical properties of thin films has special significance in the world of science, technology and industry for the development of new optoelectronics devices. The optical band gap energy of the semiconductor is an important parameter that plays a major role in the construction of photovoltaic cells. The optical absorption of the material is analyzed to find its optical band gap energy. In the present work optical absorption of $Cd_{0.5}Fe_{0.5}S$ thin film deposited onto glass substrates by by varying quantity of spray solution was studied in the wavelength range 350 to 1100 nm. The variation of optical absorption (αt) with wavelength (nm) for $Cd_{0.5}Fe_{0.5}S$ thin film is shown in figure 8. The shift in absorption edge observed in the spectra shows variation in the band gap energy depending on the thickness.

The nature of transition is determined by using the relation,

$$\alpha = \frac{A(h\upsilon - E_g)^n}{h\upsilon} \tag{5}$$

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Where 'α' is absorption coefficient, A is constant, 'hυ' is photon energy and 'Eg' is the optical band gap energy. The exponent 'n' depends on the nature of the transition, n=1/2, 2, 3/2 or 3 for allowed direct, allowed direct, forbidden direct or forbidden indirect transitions, respectively.

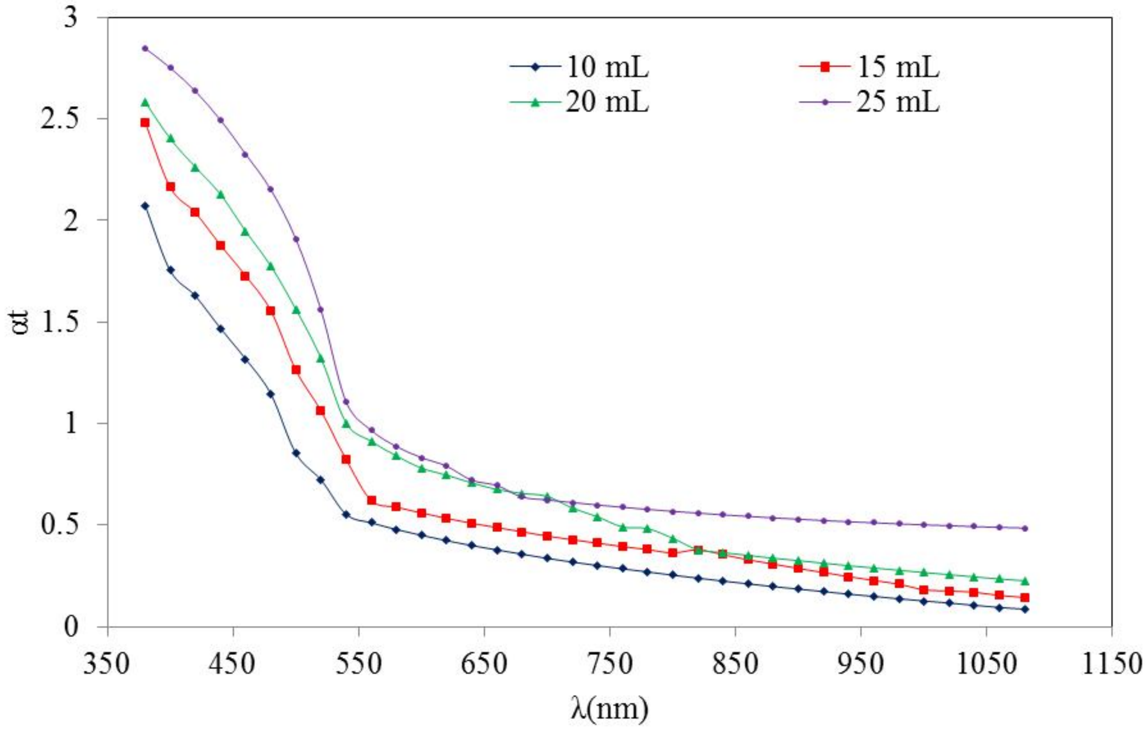


**Figure 8: Variation of optical absorption vs. wavelength of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

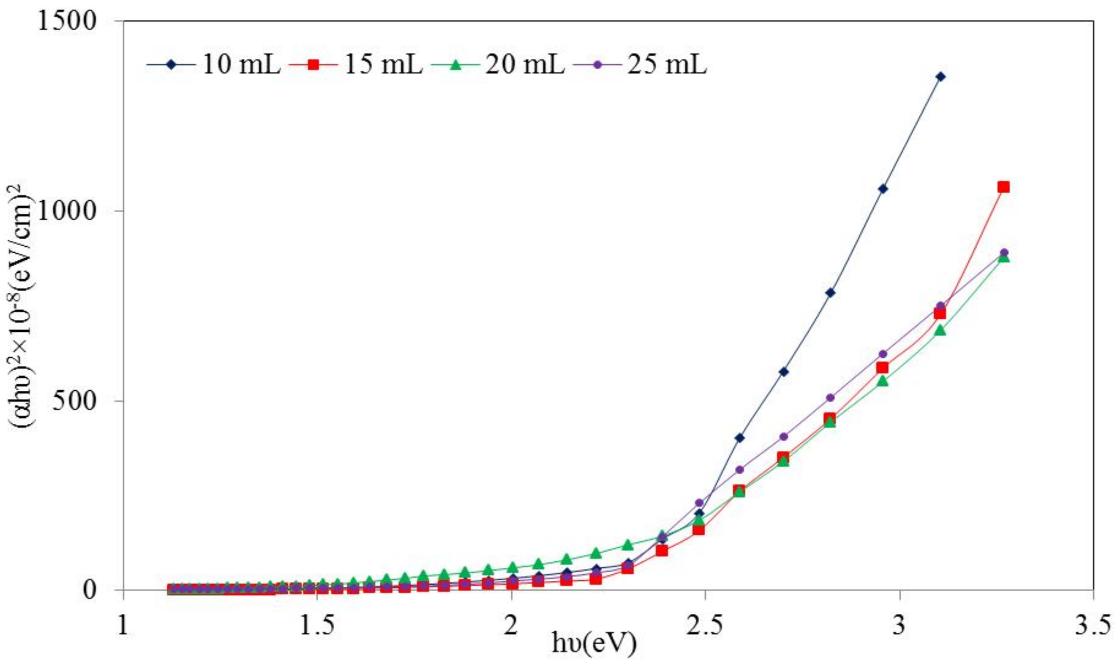


**Figure 9: Variation of Band gap of $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying the quantity of spray solution**

The plots of $(\alpha h\upsilon)^2$ vs. hυ (Figure 9) shows that the spray deposited $Cd_{0.5}Fe_{0.5}S$ thin film exhibits direct band transition. A finite energy shift in the absorption edge of the nanocrystalline material is generally expected towards higher energy side in the spectrum as that of bulk counterpart. The spray deposited $Cd_{0.5}Fe_{0.5}S$ film shows decrease in band gap energy (2.40eV to 2.20 eV) depending on thickness, which can be utilized in the development of various types of optoelectronic devices.

***Conclusion***

The structural, electrical and optical properties of nanostructured sprayed $Cd_{0.5}Fe_{0.5}S$ thin films deposited by varying quantity of spray solution have been reported. $Cd_{0.5}Fe_{0.5}S$ thin films are nanocrystalline in nature with hexagonal phase. The crystallite size, dislocation density and strain of the film on the substrate are found to depend on film thickness. The electrical characterization shows that $Cd_{0.5}Fe_{0.5}S$ thin films are semiconducting in nature with n-type conductivity. The nanocrystalline films deposited at 20 mL of spray solution are porous in nature and can be utilized for photovoltaic application.

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