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Size Dependent Physical Properties of Spray Deposited Nanocrystalline $Cd_{0.5}Fe_{0.5}Se$ Thin Films

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Abstract Nanostructured $Cd_{0.5}Fe_{0.5}Se$ thin films were successfully deposited onto the glass substrates at 573 K temperature using spray pyrolysis technique. The films of thickness 156 to 290 nm were prepared by changing the quantity of spray solution from 10 to 25 mL. The structural and morphological properties of nanostructured $Cd_{0.5}Fe_{0.5}Se$ thin films were investigated by XRD, EDAX, SEM and AFM analysis respectively. The XRD analysis shows that the spray deposited $Cd_{0.5}Fe_{0.5}Se$ thin films are nanocrystalline in nature with hexagonal crystal structure. The as deposited $Cd_{0.5}Fe_{0.5}Se$ thin films are porous in nature with growth of nanotubes and nanodiscs depending upon quantity of spray solution. The AFM analysis shows uniform growth of $Cd_{0.5}Se_{0.5}Fe$ material that covers the whole substrate. The electrical resistivity of semiconducting $Cd_{0.5}Fe_{0.5}Se$ thin films is of the order of 10^6 Ωcm and it decreases as the film thickness increases. The thermo-emf measurement confirms the n-type conductivity of $Cd_{0.5}Fe_{0.5}Se$ thin films. The optical band gap energy of the deposited $Cd_{0.5}Fe_{0.5}Se$ thin film increases from 1.74eV to 2.08eV as the film thickness decreases from 290 to 156 nm.

Keywords Thin films, Nanostructures, Chemical synthesis, Electrical properties, Optical properties

1. Introduction

In the last few years, the science and technology of nanomaterials has created great excitement and expectations in the scientific word. The properties of materials with nanometric dimensions are significantly different from those of atoms or bulk materials which can be significantly utilized to develop new science as well as new products. In this regards the II–VI group semiconducting chalcogenide nanoparticles, especially sulphides and selenides have been investigated extensively owing to their interesting optoelectronic properties[1,2]. Cadmium selenide is important compound semiconductor material because of its major contribution in solar cells, photo detectors, light amplifiers, electrophotography, light emitting diodes, lasers, photoelectrochemical cells, gas sensors and biomedical imaging devices[3–6]. Electrical and optical properties of semiconducting films are very important from application point of view in various optoelectronic devices and these properties are extremely sensitive to ambient conditions and deposition technique used. The study of effect of various deposition parameters such as nature of precursors, type of substrate, deposition temperature, film thickness etc. on physical and chemical properties of the deposited material is necessary in order to explore its application[7,8] Therefore, study of such properties of the films with respect to their different growing as well as ambient conditions is a matter of profound importance. The nanocrystalline film exhibits several interesting phenomena originating from the quantum confinement effect, such as increase in the optical band gap with decrease in crystallite size[9–11]. As per literature few reports were available on chemically deposited Fe based ternary composite thin films such as $Pb_{1-x}Fe_xSe$[12], $Cd_{1-x}Fe_xS$[13,14], $FeCdS_3$[15,16], Fe:CdSe [17] Cd-Fe-Se[18] etc. But no report is available on size dependent structural, electrical and optical properties of $Cd_{0.5}Fe_{0.5}Se$ thin films. In the present work, chemical spray pyrolysis method was used to prepare $Cd_{0.5}Fe_{0.5}Se$ thin films of thickness 156 to 290 nm by varying the quantity of spray solution. The effect of film thickness on structural, electrical and optical properties of nanostructured $Cd_{0.5}Fe_{0.5}Se$ thin films is discussed.

2. Experimental Details

The $Cd_{0.5}Fe_{0.5}Se$ thin films were deposited onto glass substrates using spray pyrolysis technique at 573K temperature. The various deposition parameters were optimized by taking several trails and listed in Table 1. It was observed that in the deposition mechanism the nature of the substrate surface is very important in order to grow uniform film over the entire substrate surface. To get adhesive film, extreme cleaning of the substrate is required, since the

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contaminated oily substrate surface gives non-uniform and non-adhesive film growth. Hence cleaning of the substrate prior to the actual deposition is important. Commercially available glass micro slides of dimensions 26 mm×76 mm×2 mm were boiled in chromic acid for 30 min, then washed with liquid detergent and rinsed in acetone. Finally slides were ultrasonically cleaned with double distilled water for 15 min prior to the actual deposition. The SeO_2 solution was prepared by dissolving 1g of selenium metal powder (99% purity) with 10 mL nitric acid (HNO_3). It was then boiled for few minutes to get white residual powder inside the beaker. To it 100 mL of distilled water was added to prepare 0.1 M SeO_2 solution. For the deposition of $Cd_{0.5}Fe_{0.5}Se$ thin films the aqueous solutions of 10 mL of 0.1M ferric chloride, 0.1M $CdCl_2$ and 20 mL of 0.1M SeO_2 solutions were mixed together to prepare spray solution. It was observed that the films deposited at 573K are well adherent and uniform; however films deposited below were discontinuous and less adhesive, which may be due to incomplete thermal decomposition.

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 SeO_2
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

In order to study the size dependent physical properties of $Cd_{0.5}Fe_{0.5}Se$ thin films, volume of spray solution was changed from 10 to 25 mL to prepare films of thickness 156 to 290 nm. The average thickness of the as deposited $Cd_{0.5}Fe_{0.5}Se$ thin film was measured by the gravimetric method[19]. The structural characterization of $Cd_{0.5}Fe_{0.5}Se$ thin films were carried out by analyzing the X-ray diffraction patterns obtained by Philips PW 1710 diffractometer with Cu Kα radiation of wavelength 1.5405 A^0. Scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDAX) data were obtained from a JOEL’S 6380A Scanning electron microscope having resolution of 1 nm. Atomic force micrographs (AFM) were collected using Park Scientific Instrument. The optical absorption studies were carried out using Lambda 25 UV-VIS spectrophotometer (PerkinElmer) at normal incidence, in the wavelength range 350-1150 nm. To study the electrical characterization of the films, the dark electrical resistivity measurements were carried out using two point d.c. probe method in the temperature range 303-513K. The thermo-emf voltage developed across the film was measured to find the type of conductivity of $Cd_{0.5}Fe_{0.5}Se$ thin film.

3. Results and Discussion

3.1. Structural Analysis

The structural properties of $Cd_{0.5}Fe_{0.5}Se$ thin films were investigated by X-ray diffraction technique. Figure1 shows the X-ray diffraction spectra of $Cd_{0.5}Fe_{0.5}Se$ 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 CdSe: 80-459,2-230,77-2307 and FeSe: 75-0608) to confirm the structure of the deposited thin films (Table 2). The spray deposited $Cd_{0.5}Fe_{0.5}Se$ thin films are nanocrystalline in nature with mixture of hexagonal CdSe and FeSe 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 observed diffraction peaks at 2Θ angles 35.64, 45.98, 72.58, 82.49, 53.11, 50.35, 72.63, 79.38, 41.60, 63.77, 72.63 and 76.10 degree (marked by *) corresponds to the lattice planes (102), (103), (212), (006), (102), (103), (201), (212), (213), (110), (203), (212) and (300) due to hexagonal CdSe, however the diffraction peaks observed at 2Θ angles 50.38, 61.41, 67.82, 30.68, 32.60, 60.45, 61.34, 30.68, 50.90, 55.28 and 81.27 degree (marked by #) corresponds to lattice planes (110), (201), (202), (002), (101), (112), (201), (002), (110), (103) and (210) due to hexagonal FeSe respectively. The (103) orientation due to hexagonal CdSe is repeated for 15 and 20 mL of spray solution and (212) is repeated for 20 and 25 mL of spray solution while (002) orientation due to hexagonal FeSe is repeated after 20 mL of spray solution. This variation may be due to alloy formation in the film. Pawar et al[20] have reported hexagonal polycrystalline nature for Fe doped CdSe thin films deposited by electrodeposition method. The crystallite size was calculated by using FWHM data and Debye Scherer’s formula.

$$d = K\lambda/\beta \cos\Theta \quad (1)$$

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 crystallite size from 42 to 71 nm was observed as film thickness rises from 210 to 290 nm 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[21]. The strain (ε) was calculated from the formula,

$$\varepsilon = \beta \cos\theta/4 \quad (2)$$

The strain of the film on the substrate deposited using 15mL of spray solution is of the order of 5.9×10^4 $Lin^{-2}nm$ and it decreases to 2.5×10^4 $Lin^{-2}nm$ at 25mL. The decrease in the strain is because of increased film thickness with improved crystallite structure. The dislocation density (δ), defined as the length of dislocation lines per unit volume of the crystal, was evaluated from the formula[22],

$$\delta = 1/d^2 \quad (3)$$

The calculated values of the strain (ε) and dislocation density (δ) are given in Table 3. The dislocation density of $Cd_{0.5}Fe_{0.5}Se$ is of the order 5.62 × 10^{-4} and it decreases to 1.97 × 10^{-4} nm^{-2} with rise in thickness. The variation in dislocation density and strain with thickness may be due to film morphology.

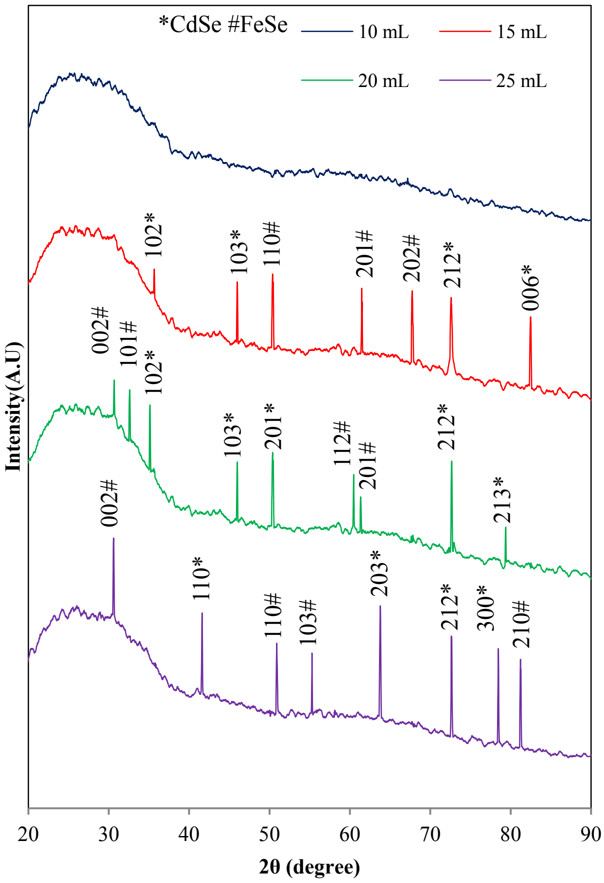

Figure 1. XRD images of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

3.2. Morphology

The SEM micrographs of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying quantity of spray solution are shown in Figure 2. It is observed that the deposited $Cd_{0.5}Fe_{0.5}Se$ thin films are homogeneous and porous in nature. At 10 mL of spray solution growth of nano grains on homogeneous background are observed. This grain growth is improved with quantity of spray solution showing well developed nano needle like network at 15mL. However, as the quantity of spray solution is increased above 15 mL growth of nano discs are observed. At 20 mL of spray solution the film surface shows clear overgrowth of well-developed nanodiscs on homogeneous background. The porous network observed at 15 mL spray solution contains number of voids however, above 15 mL spray solution the voids disappear with development of disc structure. The growth of disc structure above 15 mL of spray solution may be due to over deposition of excess quantity of spray solution. To study the stoichiometry of the film quantitative analysis was carried out using the EDAX technique. Figure 3 shows typical EDAX patterns of the $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying quantity of spray solution. The elemental analysis was carried out only for Fe, Cd and Se. However, there are some additional peaks corresponding to i, 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:Se are listed in Table 4. It is observed that the elemental composition in the film is almost in good agreement with experimental expected composition of $Cd_{0.5}Fe_{0.5}Se$.Two-dimensional surface morphology of the spray deposited $Cd_{0.5}Fe_{0.5}Se$ thin films was investigated from atomic force micrographs. Figure 4 shows the 2D AFM images of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution from 10 to 25mL. It can be seen that spray deposited $Cd_{0.5}Fe_{0.5}Se$ thin film have granular structure. In addition to granular nature, the film substrate is dense, uniform with porous nature are observed. Also it is observed that film deposited using 15 mL of spray solution are porous as that of other films, which supports the SEM analysis.

Table 2. Comparison of observed and standard XRD data of $Cd_{0.5}Fe_{0.5}Se$ thin films. (JCPDS card CdSe: 80-459,2-230,77-2307 and FeSe: 75-0608)

Film	Observed data		Standard data		hkl	phase
	2θ (degree)	d (A^0)	2θ (degree)	d (A^0)		
10 mL	---	---	---	---	---	---
15 mL	35.641	2.516	35.136	2.552	1 0 2	CdSe
	45.982	1.972	45.81	1.979	1 0 3	CdSe
	50.387	1.809	50.524	1.805	1 1 0	FeSe
	61.411	1.502	61.321	1.510	2 0 1	FeSe
	67.823	1.380	67.879	1.379	2 0 2	FeSe
	72.585	1.301	72.296	1.305	2 1 2	CdSe
	82.491	1.168	82.496	1.168	0 0 6	CdSe
20 mL	30.686	2.911	30.431	2.935	0 0 2	FeSe
	32.606	2.744	32.42	2.759	1 0 1	FeSe
	35.118	2.553	35.107	2.554	1 0 2	CdSe
	45.982	1.972	45.81	1.979	1 0 3	CdSe
	50.353	1.810	50.463	1.807	2 0 1	CdSe
	60.453	1.530	60.133	1.537	1 1 2	FeSe
	61.343	1.510	61.321	1.510	2 0 1	FeSe
	72.633	1.300	72.296	1.305	2 1 2	CdSe
	79.386	1.206	79.435	1.205	2 1 3	CdSe
25 mL	30.686	2.911	30.431	2.935	0 0 2	FeSe
	41.606	2.168	41.784	2.16	1 1 0	CdSe
	50.909	1.792	50.524	1.805	1 1 0	FeSe
	55.284	1.660	55.346	1.658	1 0 3	FeSe
	63.771	1.458	63.734	1.459	2 0 3	CdSe
	72.633	1.300	72.296	1.305	2 1 2	CdSe
	76.104	1.249	76.735	1.241	3 0 0	CdSe
	81.275	1.182	81.368	1.181	2 1 0	FeSe

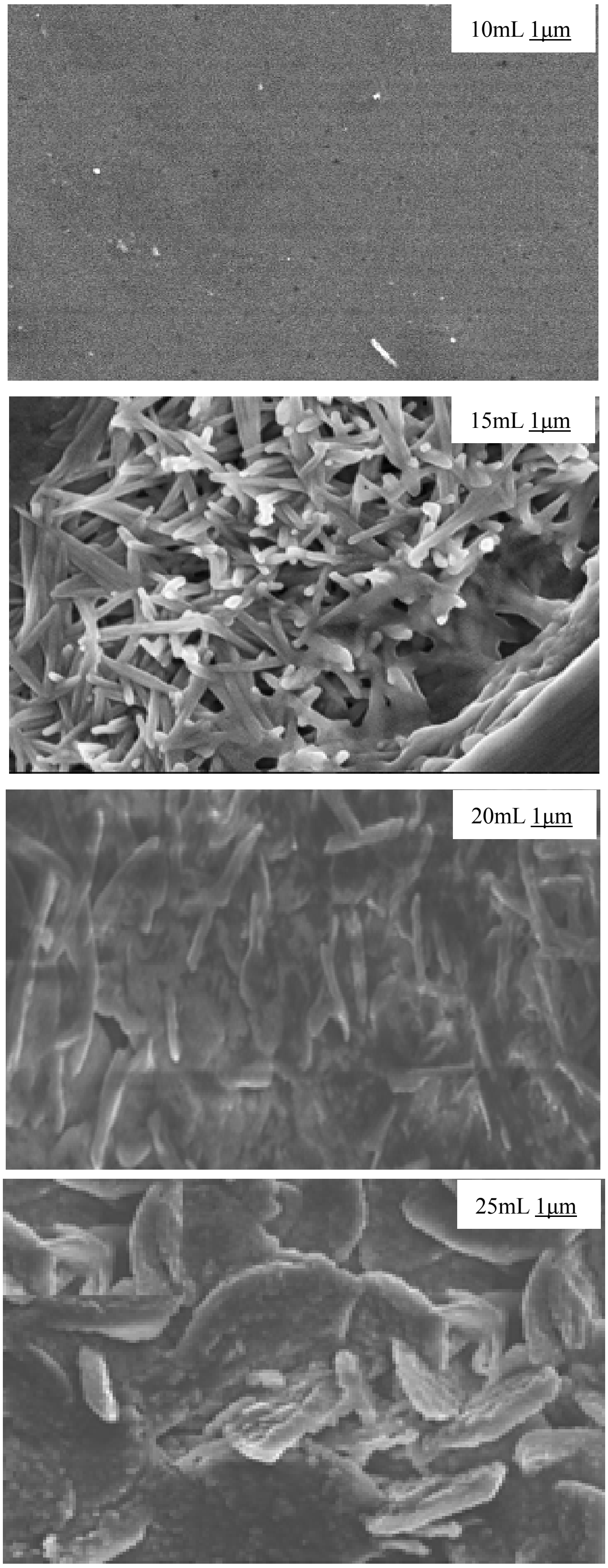

Figure 2. SEM images of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

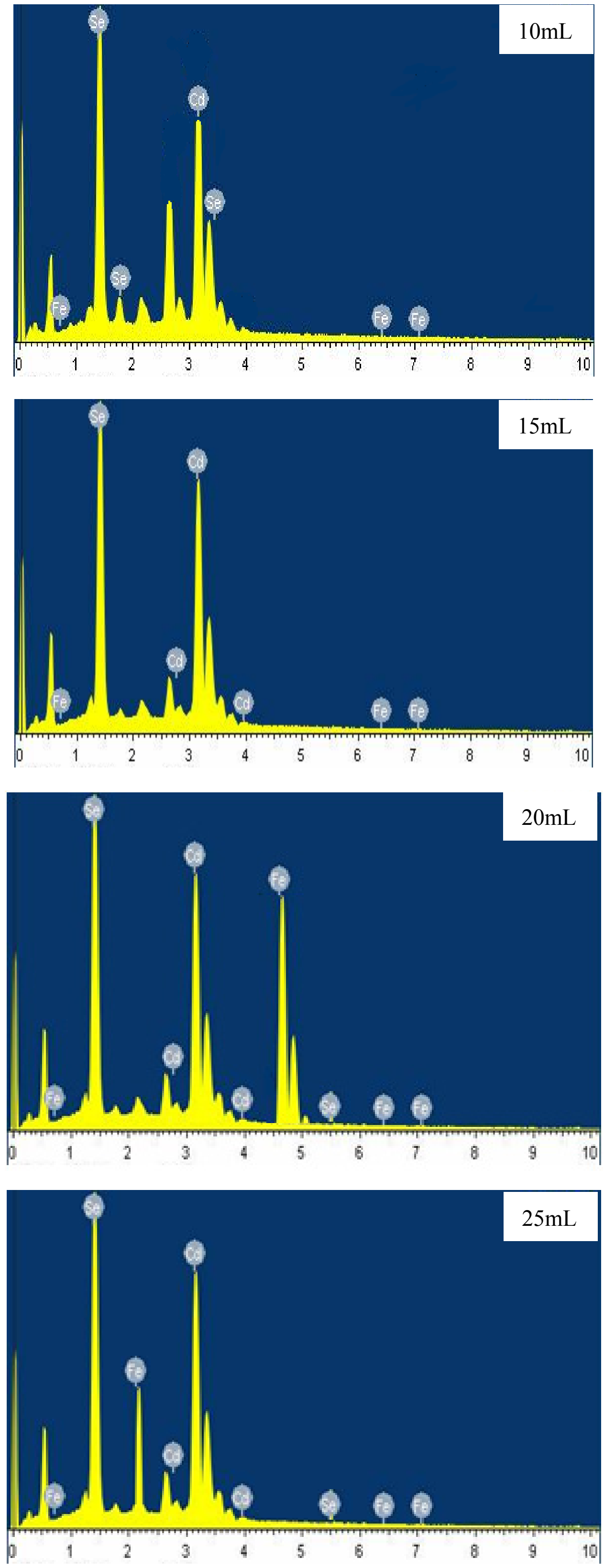

Figure 3. EDAX spectrum of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

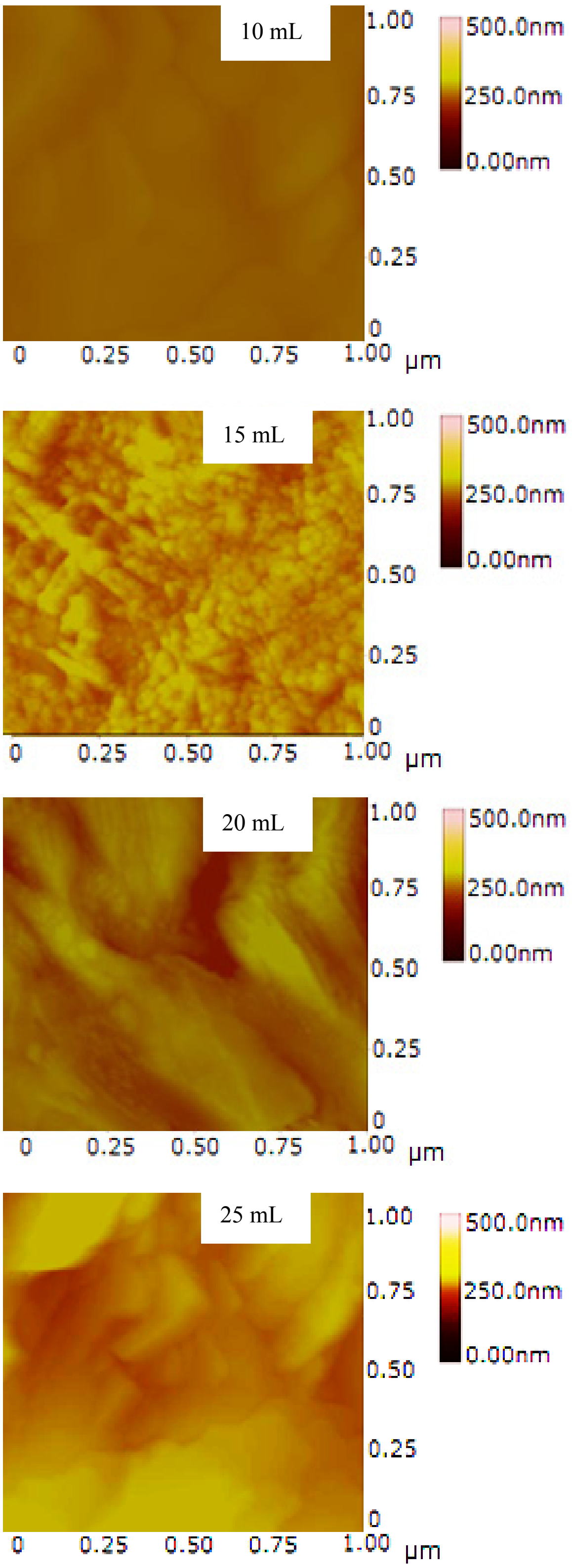

Figure 4. AFM of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

3.3. Electrical Analysis

Figure 5 shows the IV- characteristics of $Cd_{0.5}Fe_{0.5}Se$ thin films. The linear nature of graph confirms that silver used makes ohmic contact to the film. Also it is observed that, the current flowing through the film at constant applied voltage increases with film thickness, indicating that film conductivity increases with quantity of spray solution. The 'dc' electrical resistivity of $Cd_{0.5}Fe_{0.5}Se$ thin film was measured in the temperature range 303–513K by using two-probe method.

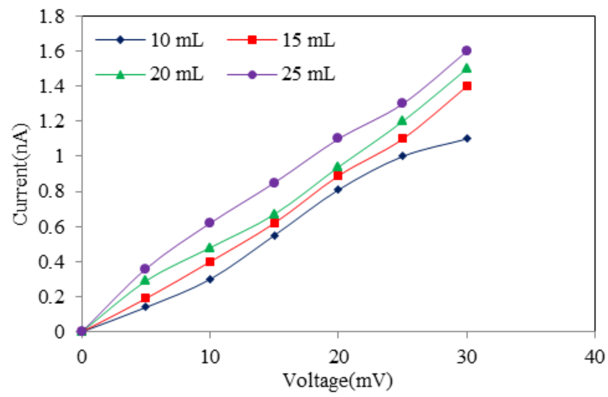

Figure 5. I-V characteristic of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

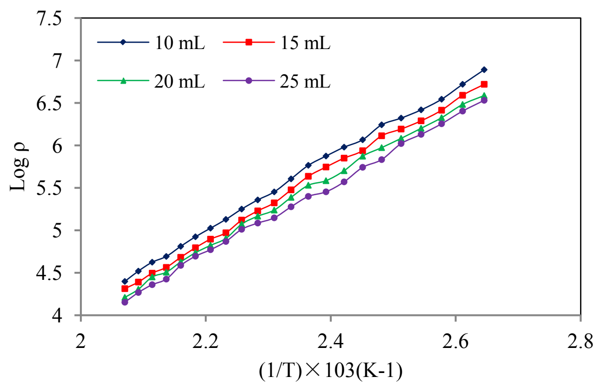

Figure 6. Variation of Log of resistivity with 1/T of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

Figure 6 shows the variation of logρ with reciprocal of temperature (1000/T). It is seen that resistivity decreases with temperature indicating semiconducting nature of $Cd_{0.5}Fe_{0.5}Se$ thin films. The resistivity follows the relation[23],

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

Where 'ρ' is resistivity at temperature 'T', ρ_0 is a constant, 'K' is the Boltzmann constant (1.38×10^{-23} J/k) and 'E_a' is the activation energy required for conduction.

The resistivity of the $Cd_{0.5}Fe_{0.5}Se$ film at 303 K is of the order of 7.8×10^6 Ωcm and it decreases to 3.6×10^6 Ωcm as the quantity of spray solution increased from 10 to 25mL. This decrease in resistivity may be due to improved crystalline nature of the film with decrease in dislocation density as compared in XRD and SEM studies.The activation energy of $Cd_{0.5}Fe_{0.5}S$ thin film deposited at 10 mL of spray solution is of the order of 0.13eV and it decreases to 0.10 eV as the film thickness increases (Table 3). These results are in good agreement with previous results on FeSe and CdSe[24,25].

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

Spray Volume	Film thickness (nm)	Crystallite Size D (nm)	Dislocation Density $\delta \times 10^4 (nm^{-2})$	Strain $\varepsilon \times 10^4 (lin^{-2} nm)$	Band gap Energy Eg (eV)	Activation energy Ea (eV)
10mL	156	-----	-----	-----	2.08	0.13
15mL	210	42.20	5.62	5.9	1.95	0.12
20mL	240	64.61	2.39	3.6	1.82	0.11
25mL	290	71.24	1.97	2.5	1.74	0.10

Table 4. Experimental and observed elemental composition in EDAX spectra for as-deposited $Cd_{0.5}Fe_{0.5}Se$ thin films

Spray Volume	Final atomic percentage in the film by EDAX analysis (%)		
	Cd	Fe	Se
10 mL	24.4	26.4	49.2
15 mL	30.2	19.5	53.2
20 mL	23.4	27.1	49.5
25 mL	23.9	27.7	48.4

The thermo-emf generated across $Cd_{0.5}Fe_{0.5}Se$ films was measured as a function of temperature difference in dark (Figure 7). The polarity of the generated thermo-emf was negative at the cold end with respect to the hot end, which confirms that $Cd_{0.5}Fe_{0.5}Se$ thin films are of n-type. The thermo-emf generated at 210 K applied temperature difference is 2.4mV for the film deposited using 10 mL of spray solution and it increases to 4.6mV as the quantity of spray solution was increased to 25 mL. This rise in thermo-emf may be due to improved crystalline quality of the film with quantity of spray solution.

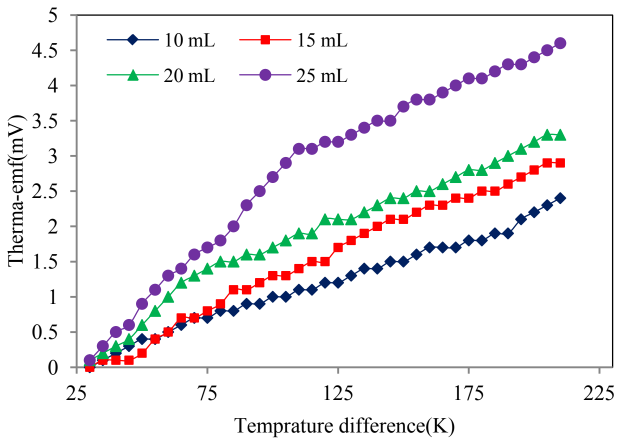

Figure 7. Variation of thermo emf (mV) with temperature difference of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

3.4. Optical Analysis

The variation of optical absorption with wavelength was analyzed to find the nature of transition involved and to estimate optical band gap. In the present work optical absorption of $Cd_{0.5}Fe_{0.5}Se$ thin film deposited onto glass substrates by varying quantity of spray solution was studied in the wavelength range 350 to 1150 nm. The variation of optical absorption (αt) with wavelength (nm) for $Cd_{0.5}Fe_{0.5}Se$ thin film is shown in figure 8. The nature of transition was determined by using the relation[26],

$$(\alpha h\upsilon)^{1/n} = A(h\upsilon - Eg) \quad (5)$$

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.

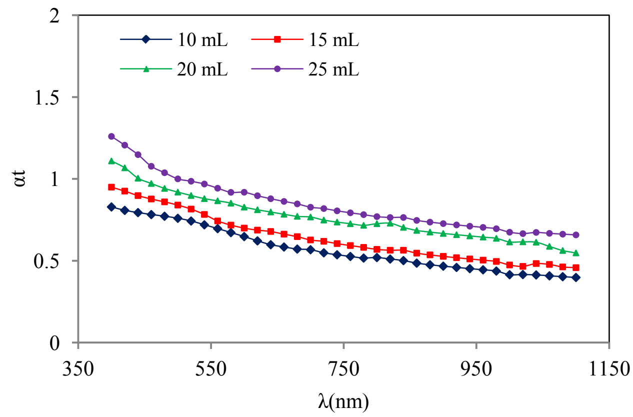

Figure 8. Variation of optical absorption vs. wavelength of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

The plots of $(\alpha h\upsilon)^2$ vs. hυ (Figure 9) shows that the spray deposited $Cd_{0.5}Fe_{0.5}Se$ thin film exhibits direct band transition. The spray deposited $Cd_{0.5}Fe_{0.5}Se$ film shows decrease in band gap energy (2.08eV to 1.74 eV) depending on thickness, which can be utilized in the development of various types of optoelectronic devices.

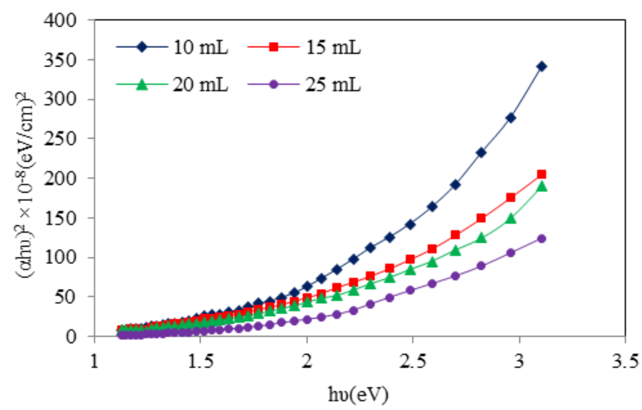

Figure 9. V The plots of $(\alpha h\upsilon)^2$ vs. hυ of $Cd_{0.5}Fe_{0.5}Se$ thin films deposited by varying the quantity of spray solution

4. Conclusions

Porous nanocrystalline $Cd_{0.5}Fe_{0.5}Se$ thin films were successfully spray deposited by varying quantity of spray solution. Structural characterization confirms that $Cd_{0.5}Fe_{0.5}Se$ thin films are nanocrystalline in nature with hexagonal lattice. The crystallite size, dislocation density and strain of the film on the substrate are found to depend on the film thickness. The film morphology is highly influenced by the quantity of spray solution. At 15mL quantity of spray solution, the growth of nano-needle like network is observed which is converted into nano-discs above 15mL. The electrical characterization shows that spray deposited $Cd_{0.5}Fe_{0.5}Se$ thin films are semiconducting in nature with n-type conductivity.

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