

## Effect of Bath Temperature on Preparation of PVA and TEA Capped CdSe Nano Crystalline Thin Films

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### Abstract:

The CdSe thin films within PVA and TEA matrices have been successfully deposited on glass substrates by chemical bath deposition technique. The structural characterization of the deposited films capped within the PVA and TEA polymeric matrices showed cubic structure with the highest intense reflection peak along (111) plane. The surface micrograph and chemical composition of deposited films were confirmed by SEM and EDAX. The optical properties of the films within the two polymers were revealing by UV-visible and photoluminescence spectrometers. The particle sizes and their dependence of band gap with the varied bath temperature were studied.

**Keywords:** CdSe thin films, micro structural properties, optical properties, PVA, TEA etc.

### 1 INTRODUCTION

The size tunable group II-VI compound semiconductor nanocrystalline thin film has been the subject of considerable research in recent years due to their specific optoelectrical properties in the application of photo conductive devices, solar cells and LEDs<sup>1-5</sup>. Among group II-VI compound semiconductor, CdSe is one of the important compounds for its applications in modern technologies of solid state devices<sup>6</sup>. In material science, several techniques like Electron beam evaporation,<sup>7</sup> Sputtering<sup>8</sup>, Chemical bath deposition<sup>9-11</sup> Photoelectrochemical<sup>12</sup> Spray pyrolysis<sup>13</sup>, Successive ionic layer absorption and reaction method<sup>14</sup> Molecular beam epitaxy<sup>15</sup> have been employed for the deposition of CdSe thin films. Among these techniques chemical

bath deposition is one of the most suitable, cost effective and green route for deposition of CdSe thin films. The deposition of thin films by chemical bath deposition is strongly influenced by bath parameters such as time of deposition, bath temperature, solution pH and relative concentration of solution.<sup>16-17</sup> Deposition temperature is considered one of the most important parameters which influences the film morphology and dependence of crystal size within the CdSe nanocrystalline thin films. The grain size of CdSe nanocrystallite has been reported to increase with the increase of bath temperature, and thereby resulting in decrease of the band gap in the alkaline medium.<sup>18</sup> This nanocrystal also showed blue shifting by an energy gap of 0.5eV from the bulk value. The grain size of the prepared CdSe thin films decreases with increase of bath temperature at 323K, 333K and 353K as has been reported by Girija et. al<sup>19</sup>. However, it is known that the reports of the effect of bath temperature on grain sizes of CdSe nanoparticles did not use any kind of capping agents like polyvinyl alcohol (PVA) or triethanolamine (TEA). Use of capping agents normally arrest and keep the nanoparticles from agglomeration and ultimately modify size and quality of the nanoparticles. Therefore the present paper encompasses the reports of the effect of bath temperature on the optical behaviour of nanocrystalline films of CdSe in PVA and TEA matrices prepared in the alkaline medium *viz.* NH<sub>3</sub> in order to verify the ambiguous results reported by different researchers.

## 2 EXPERIMENTAL

### 2.1 Materials

The reagent grade chemicals such as Cadmium acetate (Cd(CH<sub>3</sub>COO)<sub>2</sub>), Sodium Sulfite (Na<sub>2</sub>SO<sub>3</sub>) and 25% Ammonia (E-Merck), Selenium powder (Lobba-Chem), PVA (C<sub>2</sub>H<sub>4</sub>O)<sub>n</sub> (sd- Fine Chem Ltd) and Triethylamine N(CH<sub>2</sub>CH<sub>3</sub>)<sub>3</sub> (s-d fine) were used in the preparation of samples without further purification.

### 2.2 Sample Preparation

Preparation for the deposition of PVA and TEA-capped CdSe nanocrystalline thin film on glass substrates were done at different bath temperatures, using chemical bath deposition method. Cadmium acetate and sodium selenosulfate were used as Cd and Se-sources respectively. Since selenium is not soluble in water, the stock solution of 0.25M sodium selenosulfate (Na<sub>2</sub>SeSO<sub>3</sub>) was prepared by refluxing 2g of Se powder with 20g of sodium sulfite (Na<sub>2</sub>SO<sub>3</sub>) in a three round bottom flask containing 100 ml of double distilled water at 70<sup>0</sup> C in N<sub>2</sub> atmosphere. The resulting freshly prepared Na<sub>2</sub> SeSO<sub>3</sub> was taken as the source of Se<sup>-2</sup> ions. The pH of the solution was adjusted at 9 with 25% of ammonia. The polymer (20ml of 2% (w/v) PVA or 15% (v/v) TEA) was added to the above metal ion solution with constant stirring (for 5seconds) to form the polymer metal ion solution at constant bath temperature. Previously cleaned glass substrates were vertically immersed into the solution for deposition on the surface. Deposition was performed at different bath temperature 298K, 308K and 323K. Deposited films were collected after 5 hrs of deposition and washed several times with double distilled water which then dried it at room temperature and reserved for further use. The so deposited nanocrystalline thin films were found to be uniform,

well adherent to substrates. The colour of the film ranges from orange to red orange in colour.

### 2.3 CHARACTERIZATION

The structure analysis of the as prepared films were done by using PAN analytical powder diffract meter (X' PertPRO) CuK $\alpha$  (1.540  $\text{\AA}$ ) radiation Ni filter. The surface morphology and elemental analysis of the as prepared films were studied by using FEI-QUANTA 250 fabricated with EDAX. The Absorption spectra of the as prepared films were studied by Shimadzu spectrometer 8400S.

### 3 RESULTS AND DISCUSSION

The visible spectra of the CdSe films capped with PVA and TEA at various concentration of the Cd $^{2+}$  ion at bath temperature 298K were shown in Figure 1. Similar patterns were also observed at bath temperatures 308K and 323K (Figure not included). Band gap ( $E_g$ ) of the CdSe nanocrystalline thin films capped within PVA and TEA matrices at 298K for different polymer concentration are reported along with the spectra in the Figure2. Band Gap ( $E_g$ ) has been evaluated from the intersection point of lines extrapolating to the slope at right side of the spectrum with the X-axis ( $h\nu$ ) in the plot of  $(\alpha h\nu)^2$  vs  $h\nu$  as shown in Figure 2 where  $\alpha$  is absorption co-efficient given by Tauc relation<sup>20</sup>

$$\alpha = A(h\nu - E_g)^n / h\nu$$

where,  $h\nu$  = photon energy, 'A' = a constant and 'n' depend on the nature of the transition (for direct transition,  $n = 1/2$  or  $2/3$  and for indirect transition  $n = 2$  or  $3$ ).

The variation of band gap of the CdSe thin film capped within PVA or TEA at different Cd $^{2+}$  ion concentration and at different bath temperature is reported in Table 1. The observed band gap value of the CdSe nanoparticle capped within PVA or TEA matrices were used while estimating particle size (R) in simplified Brus equation<sup>21-22</sup>

$$E_g = E_g(0) + \frac{h^2}{8R^2} \left[ \frac{1}{m_e^*} + \frac{1}{m_h^*} \right] \text{ and are also included in Table 1.}$$

where,  $E_g$  = band gap of nanocrystalline thin films,  $E_g(0)$  = bulk band gap,  $m_e^* = 0.13m_0$  and  $m_h^* = 0.45m_0$  ( $m_0 = 9.11 \times 10^{-31} \text{ Kg}$ ) are the effective masses of electron and hole, R = the radius of the nano particle, h = Planck constant and  $E_g(0) = 1.74 \text{ eV}$ .

The crystallographic phases of the CdSe films within the matrices of two capping groups TEA and PVA with varied Cd $^{2+}$  ion concentration at 298K were shown in Figure 3. The prominent sharp peaks were assigned to (111), (220) and (311) planes with the highest peak along (111). The variation of the average crystallite size of CdSe thin films capped with PVA and TEA matrices at different bath temperature and Cd $^{2+}$  concentration which are presented in Table 2 and 3 were determined using

Debye Scherer's formula,

$$D = \frac{0.9\lambda}{W \cos \theta}$$

Where  $\lambda = 1.54 \text{ \AA}$  is the wavelength of incident X-ray,  $W$  is the full width at half maxima,  $\theta$  is the angle of diffraction.

It is observed from the figure that CdSe thin films have better absorption spectra with sharp band edges within wavelength ranges 300-650nm. The absorption peaks around 713nm from bulk were observed to be shifting to the lower wavelength with increase in the  $\text{Cd}^{2+}$  ion concentration i.e., from 0.1M to 0.5M. Similar blue shifting was also observed with increasing temperature. The blue shifting of the absorption edge is the indication of the decreasing particle size of the CdSe with increasing in temperature.<sup>20</sup> The band gaps increases with increasing  $\text{Cd}^{2+}$  ion concentration. For example in case of 0.1M  $\text{Cd}^{2+}$  increase from 2.09eV(at 298K) to 4.09eV (at 323K) for PVA capped and from 2.00eV (at 298K) to 3.76eV(323K) for TEA capped CdSe thin films. The increase in the band gap with increasing  $\text{Cd}^{2+}$  ion concentration is the consequence of decreasing crystallite sizes in the film and is in agreement with the available literature.<sup>23</sup>

It is observed that results from XRD patterns were well matched and confirmed with the JCPDS and concluded that deposited CdSe films were of nanocrystalline having cubic structure.<sup>24</sup> The crystallographic analysis also revealed that there is no phase transition with varied bath temperature. The average crystallite sizes decrease with the increase of bath temperature and  $\text{Cd}^{2+}$  concentrations. This could be attributed to the increase of grain boundaries and the probability of impurity materials deposited or may be due to defects associated while increasing bath temperature.<sup>20</sup> Since rate of reaction increases with increase in the bath temperature more and more ions are produced however not all the ions get the chance to be deposited on the substrate surface and therefore settle down at the bottom of the container. The crystal defect plays a critical role when the size of the nanoparticle decreases from 10 to 1nm. In such case of CdSe, the percentage of surface atoms increases from 20 to 100% resulting unsaturated bonds or dangling bonds with the increase of surface energy thereby contributing to the varying magnitude of strain.<sup>25</sup> The lattice constant,  $a$  of the cubic phase nanocrystalline thin films capped with PVA and TEA determined using the relation  $a = d\sqrt{h^2 + k^2 + l^2}$  are included in Table 2 and 3. It is observed that lattice constant,  $a$ 's are different for different orientation of the same film. This can be attributed to the divergence of the X-rays beams diffraction and absorption of X-rays by the specimens, involving a number of systematic error in the measurement of  $\theta$  and  $d$  values.<sup>20, 26</sup> The corrected value of  $a$  is obtained from the Nelson- Riley graph by plotting lattice parameter,  $a$  for different planes with the error function<sup>27</sup>  $f(\theta)$ , which is given by  $f(\theta) = \frac{1}{2} \left[ \frac{\cos^2 \theta}{\cos \theta} + \frac{\cos^2 \theta}{\theta} \right]$ . The corrected value of "a" is determined from the graph by extrapolating the graph to  $\theta = 90^\circ$  as shown in fig 3. The collected value of lattice parameter 'a' for PVA and TEA-caped CdSe with varied bath temperature are slightly deviated from the standard JCPDS bulk value 6.077Å as shown in the

Table 2 and Table 3. From table it is inferred that the deviation of the lattice constant, 'a' of the as prepared CdSe thin films from the standard value (6.077Å) are under strain. When the nanoparticles reduced its size with varied temperature an extra free energy is associated with the surface which tried to increase the fraction of the particle's total free energy. This excess free energy is used up in changing its lattice constant, resulting the contraction or expansion in 'a' axes for cubic structure.

The dislocation density, 'δ' of the PVA and TEA-capped CdSe nano-thin films are determined from the Williamson and Smallman's formula<sup>28</sup>  $\delta = n / D^2$

For minimum dislocation, the factor n = 1 and D = average crystallite size The dislocation density measured the quantity of the dislocation present in the material and the dislocation density in prepared films are found to be in the order of 10<sup>16</sup> lines/m<sup>2</sup>.

The micro strain, ε of the as prepared CdSe thin films are calculated from the relation<sup>28</sup>

$$\varepsilon = \beta \cot\theta / 4$$

Where β = full width at half at half maxima and θ = Bragg's angle in radians. The micro structural parameters of the CdSe films for both the capping groups are shown in Table 4. The variation of dislocation density δ and micro strain, ε with particle size (Cd ions concentration) are shown in Fig 4. It is observed that the dislocation density and micro strain increases with decrease in particle size. The micro strain produced in this case creates deviation of lattice constant 'a' from its bulk value 6.077Å<sup>29</sup>, leading to either elongation or compression of the lattice. The decrease in crystallite size with increase of bath temperature of the as prepared CdSe thin films indicated the increase in defects like dislocation density the compressive strain (micro strain). The estimated values of particle sizes are in good agreement with those of values from XRD.

A comparison of the particle sizes in the Table 1 shows that particle sizes of CdSe nanocrystalline thin films within the two matrices of the capping groups PVA and TEA are decreases with the increase of concentration and bath temperature. It is observed that particle sizes in case of CdSe filmed capped with PVA is comparatively greater than from the filmed capped with TEA thereby decreasing size of the cavity available in the matrices. But in the case of PVA polymer the matrix may becomes more intriguing at higher bath temperature leading to smaller cavity of the PVA matrix and thus the size get arrested and stabilize from further agglomeration.

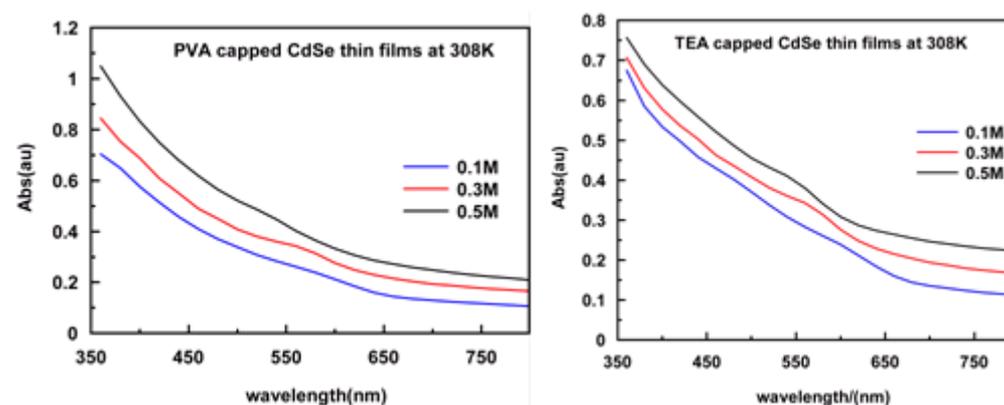
The surface morphology of the capped nanocrystalline films were showed in Fig. (9 ). The SEM –micrograph of the PVA and TEA caped CdSe nanocrystalline thin films composed of spherically shaped and well defined grains. The good adherent thick CdSe films are obtained in both the matrices but some cracks are observed in the films at lower temperature. This macroscopic defect is not observed in the CdSe films at higher temperature.

The quantitative analysis of the as prepared nanocrystalline films were done by using EDAX technique fabricated to the SEM as shown in Figure (10) .The strong peaks of Si and O observed in the elemental analysis revealed that the high operating voltage of the electron beam penetrates the thin films and reaches the glass surface resulting successful deposition of as prepared PVA and TEA caped CdSe nano

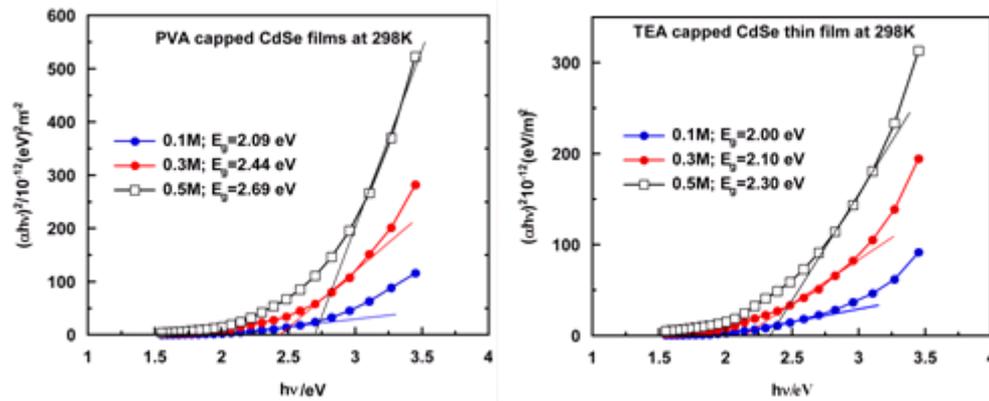
crystalline thin films. The EDAX elemental analysis confirmed the presence Cd and Se in the prepared films. The observed values of the weight percentage of Cd and Se are approximately agreeable with the calculated values as shown in the Table 5.

## 5 CONCLUSION

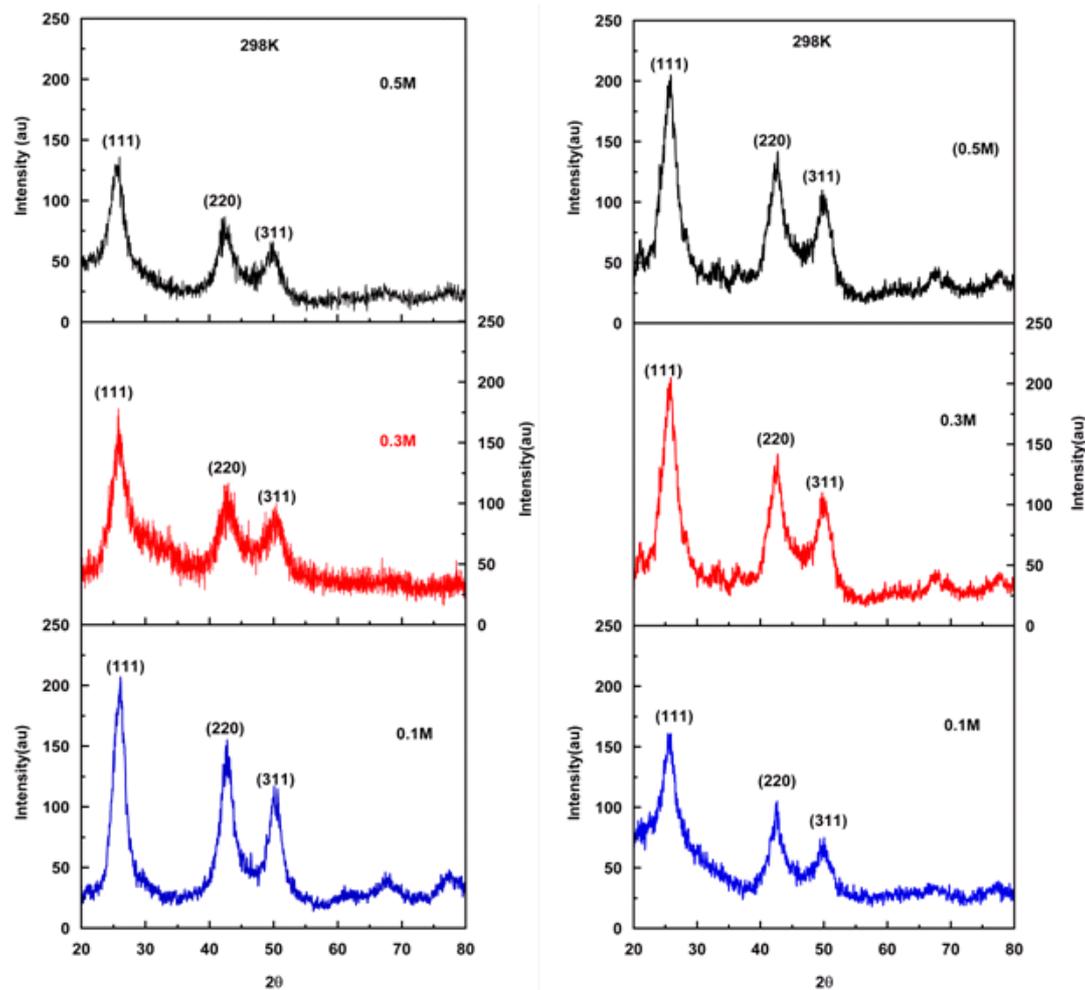
The TEA and PVA-capped CdSe nanocrystalline thin films were successfully prepared through easy, economical and environmentally friendly chemical route at different bath temperatures 298 K, 308 K and 323 K. The as prepared nanocrystalline thin films in both the polymers matrices are cubic in structure with lattice parameters fairly in agreement with the standard JCPDS values. The particle sizes are increased with the increase of bath temperature and  $\text{Cd}^{+2}$  concentrations. The optical analysis of the as prepared CdSe thin films in the two capping groups revealed blue shifting in relation to the bulk value which confirmed the quantum confinement effect. The SEM micrograph CdSe films revealed no macroscopic defect at higher bath temperature. The agreeable wt % of calculated and observed values of Cd and Se in the prepared films was confirmed by EDAX analysis. Similar patterns were observed at the temperatures the 308K and 323K. It is also found that the particle size of the CdSe nanocrystalline thin film capped with PVA and TEA polymers are tunable by varying the bath temperature as well as the precursor concentration.



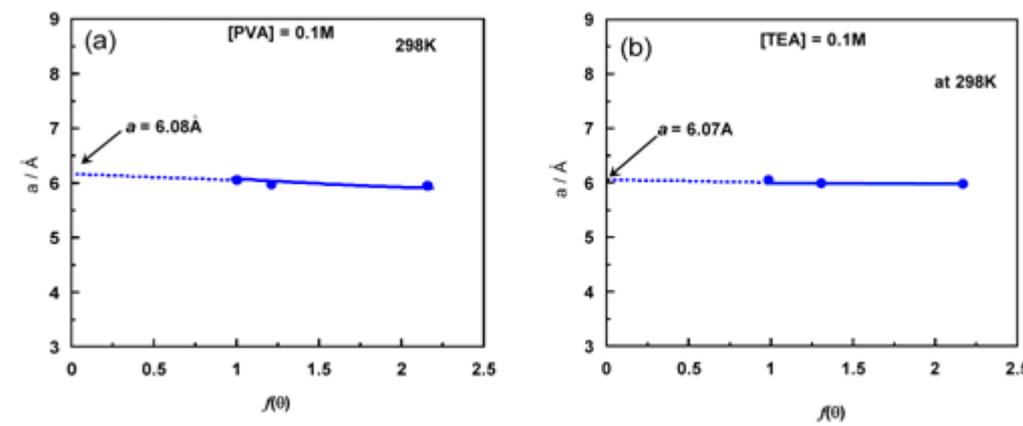
**Figure 1:** Absorption spectra of CdSe thin films deposited in the (a) PVA and (b) TEA matrices.



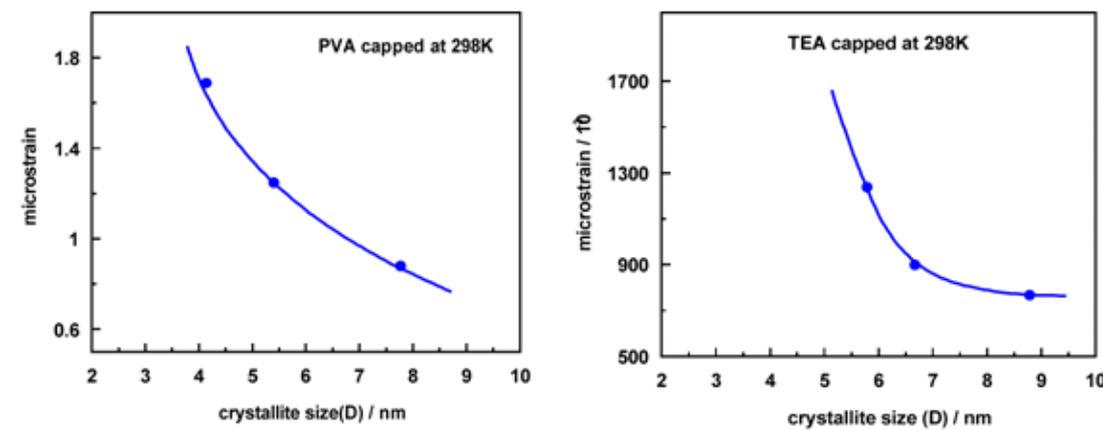
**Figure 2:** Plots of  $(\alpha h\nu)^2$  vs  $h\nu$  for CdSe thin films deposited in the (a) PVA and TEA(b) matrices.



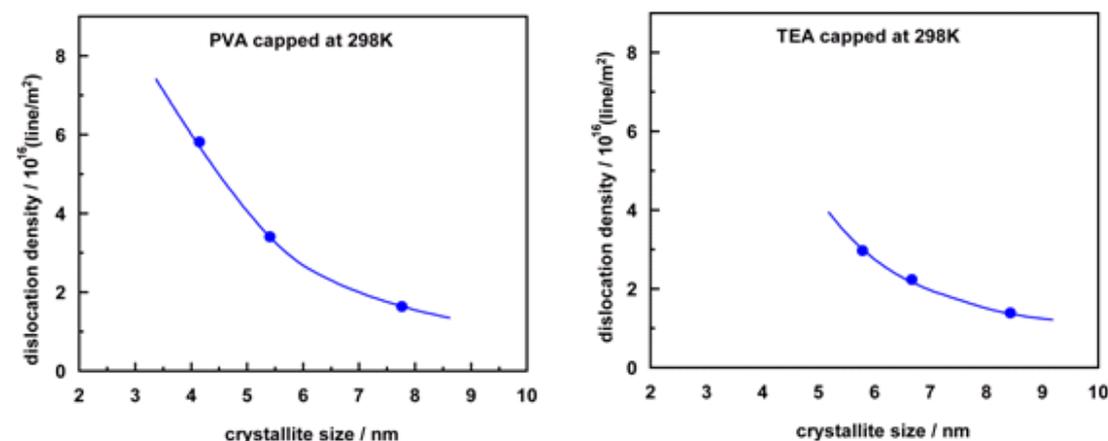
**Figure 3:** XRD Pattern of (a) PVA and (b) TEA capped CdSe thin films.



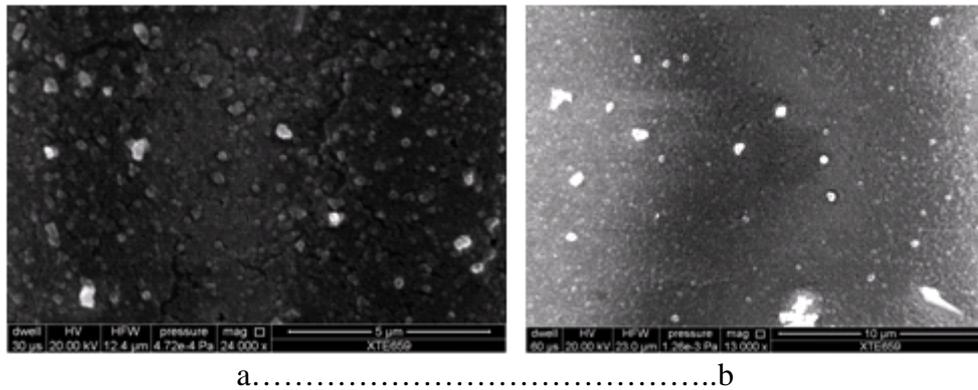
**Figure 4:** Nelson-Riley Plot of CdSe capped with PVA (a) and TEA (b) thin films.



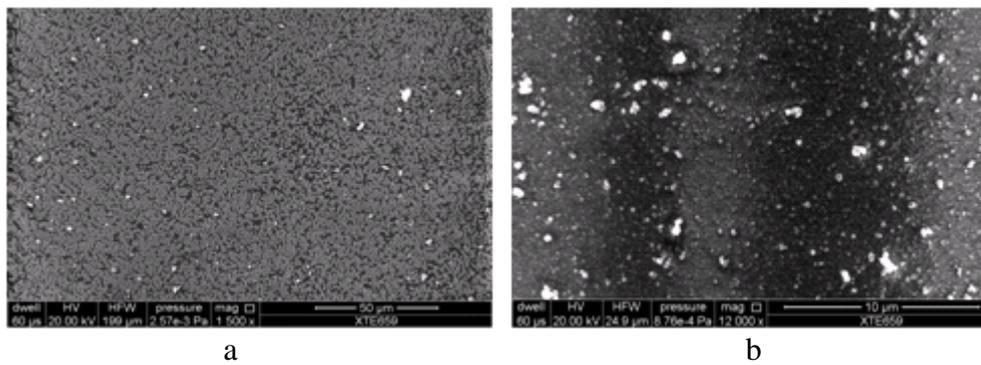
**Figure 5:** Variation of dislocation density with crystallite size of (a) PVA and (b) TEA capped CdSe film at 298K.



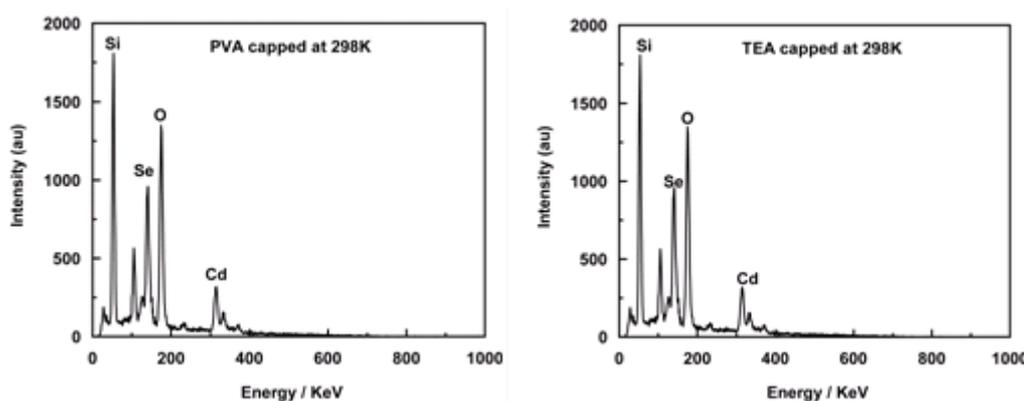
**Figure 6:** Variation of microstrain with crystallite size of (a) PVA and (b) TEA capped CdSe films.



**Figure 7:** SEM micrograph of PVA capped CdSe nanocrystalline films of at bath temp (a) 298 K and (b) 323 K



**Figure 8:** SEM micrograph of TEA capped CdSe nanocrystalline films of at bath temp (a) 298 K and (b) 323 K



**Figure 9:** EDAX analysis of (a) PVA and (b) TEA capped CdSe for 0.1M at 298K

**Table 1:** Band Gap values and particle size of CdSe thin films

Temp/K	[Cd <sup>2+</sup> ]/M	Particle size		Band gap	
		Diameter (EMA) (PVA capped)	Diameter (EMA) (TEA capped)	PVA capped	TEA capped
298	0.1	6.49	7.53	2.09	2.00
	0.3	4.59	6.40	2.44	2.10
	0.5	3.94	5.10	2.69	2.30
308	0.1	4.47	4.62	2.48	2.43
	0.3	3.18	4.09	3.23	2.60
	0.5	2.79	3.18	3.64	3.20
323	0.1	2.75	4.02	3.70	2.65
	0.3	2.78	2.81	3.64	3.60
	0.5	2.51	2.70	4.09	3.76

**Table 2:** Structural parameters of PVA capped nanocrystalline thin films at different bath temperature

Temp/K	[Cd(CH <sub>3</sub> COO) <sub>2</sub> ]/M	Plane (hkl)	2θ		Average crystallite size	'd' value		'a' value	
			Observed value	Std. Value		Observed value	Std. Value	Observed value	Std. Value
298	0.1	(111)	26.042	25.648	7.76	3.4421	3.510	5.961	6.077
		(220)	42.723	42.000		2.116	2.149	5.984	
		(311)	50.008	49.698		1.823	1.833	6.046	
	0.3	(111)	25.796	25.648	5.40	3.453	3.510	5.980	
		(220)	42.463	42.000		2.128	2,149	6.018	
		(311)	50.131	49.698		1.889	1.833	6.265	
	0.5	(111)	25.787	25.648	4,14	3.454	3.510	5.982	
		(220)	42.560	42.000		2.124	2,149	6.018	
		(311)	49.960	49.698		1.822	1.833	6.042	
308	0.1	(111)	25.796	25.648	5.43	3.453	3.510	5.980	
		(220)	42.464	42.000		2.128	2.149	6.018	
		(311)	50.130	49.698		1.819	1.833	6.032	
	0.3	(111)	25.344	25.648	3.08	3.525	3.510	6.105	
		(220)	42.478	42.000		2.121	2,149	5.999	
		(311)	49.871	49.698		1.827	1.833	6.059	
	0.5	(111)	25.517	25.648	2.90	3.487	3.510	6.039	
		(220)	42.578	42.000		2.120	2,149	5.796	
		(311)	49.871	49.698		1.848	1.833	6.053	
323	0.1	(111)	25.885	25.648	3.04	3.499	3.510	5.796	
		(220)	42.878	42.000		2.107	2.149	6.060	
		(311)	39.902	49.698		1.826	1.833	6.321	
	0.3	(111)	25.843	25.648	2.83	3.444	3.510	6.056	

		(220)	42.801	42.000		2.111	2,149	5.965	
		(311)	49.526	49.698		1.839	1.833	5.097	
	0.5	(111)	25.922	25.648	2.27	3.433	3.510	6.099	
		(220)	43.163	42.000		2.094	2,149	5.444	
		(311)	49.763	49.698		1.830	1.833	5.092	

**Table 3:** Structural parameters of TEA capped nanocrystalline thin films at different bath temperature.

Temp /K	[Cd(CH <sub>3</sub> COO) <sub>2</sub> ]/M	Plane (hkl)	2θ		Average crystallite size	'd' value		'a' value	
			Observed value	Std. Value		Observed value	Std. Value	Observed value	Std. Value
298	0.1	(111)	25.644	25.648	8.42	3.435	3.510	5.949	6.077
		(220)	42.552	42.000		2.124	2,149	6.007	
		(311)	49.827	49.698		1.830	1.833	6.069	
	0.3	(111)	25.851	25.648	6.66	3.344	3.510	5.958	
		(220)	42.701	42.000		2.112	2,149	5.973	
		(311)	50.195	49.698		1.817	1.833	6.026	
	0.5	(111)	26.021	25.648	5.78	3.424	3.510	5.930	
		(220)	42.822	42.000		2.110	2,149	5.967	
		(311)	49.835	49.698		1.828	1.833	6.079	
308	0.1	(111)	25.893	25.648	5.50	3.440	3.510	5.957	
		(220)	42.712	42.000		2.114	2,149	6.078	
		(311)	49.884	49.698		1.828	1.833	6.062	
	0.3	(111)	25.628	25.648	4.41	3.476	3.510	6.020	
		(220)	42.699	42.000		2.117	2,149	5.987	
		(311)	49.942	49.698		1.824	1.833	6.049	
	0.5	(111)	25.769	25.648	3.59	3.454	3.510	6.021	
		(220)	42.740	42.000		2.113	2,149	5.976	
		(311)	49.820	49.698		1.828	1.833	6.079	
323	0.1	(111)	25.471	25.648	3.64	3.494	3.510	6.051	
		(220)	42.539	42.000		2.123	2,149	6.004	
		(311)	49.703	49.698		1.832	1.833	6.076	
	0.3	(111)	25.586	25.648	2.92	3.478	3.510	6.024	
		(220)	42.539	42.000		2.123	2,149	6.004	
		(311)	49.629	49.698		1.835	1.833	6.086	
	0.5	(111)	25.755	25.648	2.80	3.456	3.510	5.985	
		(220)	42.717	42.00		2.115	2,149	5.982	
		(311)	49.937	49.698		1.824	1.833	6.046	

**Table 4a:** Microstructural parameters of PVA capped CdSe nanocrystalline thin films

Temp / (K)	[Cd <sup>2+</sup> ] / (M)	Observed lattice constant a (Å)	Collected lattice constant, a (Å)	Average crystallite size(D) ( nm)	Dislocation Density( $\delta$ ) $\times 10^{16}$ lines/m <sup>2</sup>	Micro strain( $\epsilon$ )
298	0.1	5.961	5.949	7.76	1.65	0.88
		5.984				
		6.046				
	0.3	5.982	6.012	5.40	3.42	1.25
		6.007				
		6.042				
	0.5	5.980	6.076	4.14	5.83	1.69
		6.018				
		6.265				
308	0.1	6.018	6.040	5.43	3.39	1.19
		6.007				
		6.050				
		5.999				
	0.3	5.980	6.031	3.08	10.54	2.13
		6.100				
		6.039				
	0.5	6.020	6.035	2.90	11.84	2.23
		6.000				
6.075						
323	0.1	6.051	6.043	3.04	10.82	2.10
		6.004				
		6.076				
	0.3	6.024	6.037	2.83	12.45	2.42
		6.004				
		6.086				
	0.5	5.985	6.004	2.27	19.32	2.78
		5.982				
		6.046				

**Table 4b:** Microstructural parameters of TEA-capped CdSe nanocrystalline thin films

Temp / (K)	[Cd <sup>2+</sup> ] / (M)	Observed lattice constant a (Å)	Collected lattice constant, a (Å)	Average crystallite Size( nm)	Dislocation Density( $\delta$ ) $\times 10^{16}$ lines/m <sup>2</sup>	Micro strain( $\epsilon$ )
298	0.1	5.949	6.007	8.42	1.4	0.77
		6.007				
		6.069				
	0.3	5.958	5.970	6.66	2.25	0.90
		5.973				
		6.026				
	0.5	5.930	5.900	5.78	2.98	1.24
		5.967				
		6.079				
308	0.1	5.957	6.034	5.50	3.30	1.21
		6.078				
		6.062				
	0.3	6.020	6.038	4.41	5.12	1.30
		6.087				
		6.049				
	0.5	6.021	6.098	3.559	8.13	1.77
		5.976				
		6.079				
323	0.1	6.051	6.013	3.64	7.54	1.98
		6.004				
		6.076				
	0.3	6.024	6.037	2.90	11.72	2.22
		6.004				
		6.086				
	0.5	5.985	6.003	2.80	12.74	2.65
		5.982				
		6.046				

**Table 5:** Elemental composition of PVA and TEA capped CdSe thin films.

Temp/ (K)	[Cd <sup>+2</sup> ]/M	capping	Calculated		observed	
			Cd (Wt%)	Se (Wt%)	Cd (Wt%)	Se (Wt%)
298	0.1	PVA	36.29	56.5	35.46	64.54
308	0.3		63.02	36.29	57.39	36.66
298	0.1	TEA	36.29	56.5	33.10	62.85
308	0.3		63.02	36.29	52.57	37.25

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