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Structural characterization of hot wall deposited cadmium selenide thin films

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Abstract. Cadmium selenide (CdSe) films were prepared by the hot wall deposition technique with different quartz tube lengths under a vacuum of 6.6 mPa onto well cleaned glass substrates to optimize the tube length. The XRD analysis revealed that the films were polycrystalline in nature for smaller tube lengths and for smaller thicknesses, but with increasing thickness and increasing tube lengths a preferred orientation was observed on all substrates. The x-ray diffractograms of the films coated on 0.07 m length tube exhibit preferential orientation along the (103) direction for smaller thicknesses, which changes to the (002) direction as the thickness increases, but the films coated with other tube lengths show preferential orientation along the (002) plane parallel to the substrate at larger thicknesses. The lattice constants and the structural parameters *viz*., crystallite size (D), dislocation density (δ) and strain (ϵ), were calculated for all the films and it was found that as the tube length increases the grain size and strain decreases up to 0.13 m tube length and increases for 0.15 m tube length. The effects of the substrate were studied by coating the film on steatite, mica and ITO substrates using an optimized tube length (0.15 m). Films coated on glass, mica and ITO have a very smooth surface finish, reflecting like a mirror, and those on steatite have a matt finish. Depending on the adhesiveness and grain size the substrates were chosen for further applications. Studies on the effect of substrate temperature were carried out on the films obtained using 0.15 m quartz tube length. From Rutherford backscattering spectrometry analysis, it was found that as the tube length increases the Cd/Se ratio approaches unity.

1. Introduction

Cadmium chalcogenide semiconductors play a vital role in the development of science and technology, particularly cadmium selenide (CdSe) films with an absorption edge at approximately 730 nm and a direct forbidden energy gap of 1.7 eV. Usually CdSe films are n type and they are of interest for their applications as photoconductors (Shimizu *et al* 1971), solar cells (Guruszecki and Holmstrom 1993, Pal *et al* 1990), thin film transistors (Moersch *et al* 1989, Van Calster *et al* 1988), gas sensors (Smyntyna *et al* 1994, Patel *et al* 1994), acousto-optic devices (Bonnello and Fernandez 1993), vidicones (Schottmiller *et al* 1975), photographic photoreceptors (Curtis *et al* 1980) etc. A detailed review of the influence of substrate and source temperatures, type of substrate, chemical composition etc on the growth, structure and physical properties of the cadmium chalcogenide films has been published by Kalinkin *et al* (1978).

CdSe films obtained using vacuum evaporation (Jawalekar and Rao 1980, Russak and Reichman 1982), molecular beam epitaxy (MBE) (Luo *et al* 1991, Hyugaji and Miura 1985) and metal oxide molecular beam epitaxy (MOMBE) (Shizuo *et al* 1992) have already been studied extensively. Normally, the crystallinity and impurities incorporated during the deposition process strongly affect the basic characteristics. After the report of p-type conduction by Takeo *et al* (1994) in CdSe films prepared by MBE using a nitrogen plasma source the interest in this material has been renewed.

Among the various techniques available for the deposition of thin films, recently hot wall epitaxy has become a popular technique, since it is a relatively simple and economically viable technique and it has contributed significantly to the growth of high quality epitaxial thin films with smooth surfaces. Schikora *et al* (1986) have

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also claimed that hot wall deposited CdTe films on GaAs substrates show superior luminescence properties when compared to MBE and MOCVD (metal oxide chemical vapour deposition) films. Chaudhuri *et al* (1987) have used the hot wall technique to deposit CdSe thin films onto glass, NaCl and KCl substrates at different substrate temperatures. Pal *et al* (1990) have fabricated ZnTe/CdSe solar cells by the hot wall technique and studied I–V, C–V and spectral response measurements.

A systematic study on CdSe films deposited by the hot wall technique using different tube lengths on glass substrates will be of proven use to optimize the tube length to obtain stoichiometric films. Hence an effort is made in this paper to prepare CdSe films using four different tube lengths and to compare their structural and compositional parameters to identify the better films. Using the optimized tube length, films are prepared on glass substrates kept at different temperatures and their structural parameters are analysed. Also the effect of the nature of the substrate on the structural parameters is studied.

2. Experimental technique

2.1. Hot wall setup

The hot wall experimental setup (figure 1) was similar to the one used by Ramachandran and Vaya (1983). The main feature of the system used was the heated linear quartz tube (0.01 m diameter), which served to enclose and direct the vapour from the source to the substrate. The source, wall and substrate were heated independently using heater coil windings made of tungsten, kanthal and nichrome respectively. The heater wire was wound on the quartz wall with uniform spacing to provide the same temperature along its length. Care was also taken to avoid any cold areas within the quartz tube which would adversely affect not only the deposition rate but also the stoichiometry of the deposited films owing to the different partial pressures of the Cd and Se vapours. This was done by enclosing the quartz tube in an outer glass tube as shown in figure 1.

The substrate was kept at less than 1 mm distance above the quartz tube acting almost as a lid to close the tube. The substrate with its heater (holder) was attached to a substrate manipulator which can make a semicircle in the vacuum chamber. The front surface of the substrate was facing down towards the source. The substrate could be brought to the desired position (figure 2) above the hot wall after the required growth conditions were attained and stabilized. The substrate manipulator was designed in such a way that three to five similar films could be grown on different substrates without breaking the vacuum. The films were coated using different tube lengths to find the optimum length for evaporation. Films of different thicknesses (120–1800 nm) were prepared to study the effect of the tube length. Also films were coated onto different substrates such as mica, ITO (indium tin oxide) and steatite ($MgAl_2SiO_3$) in order to study various properties such as optical, photoelectrical and photodielectric. The whole setup was kept inside the chamber, which was evacuated to a pressure of 6.6 mPa prior to the growth of the film.

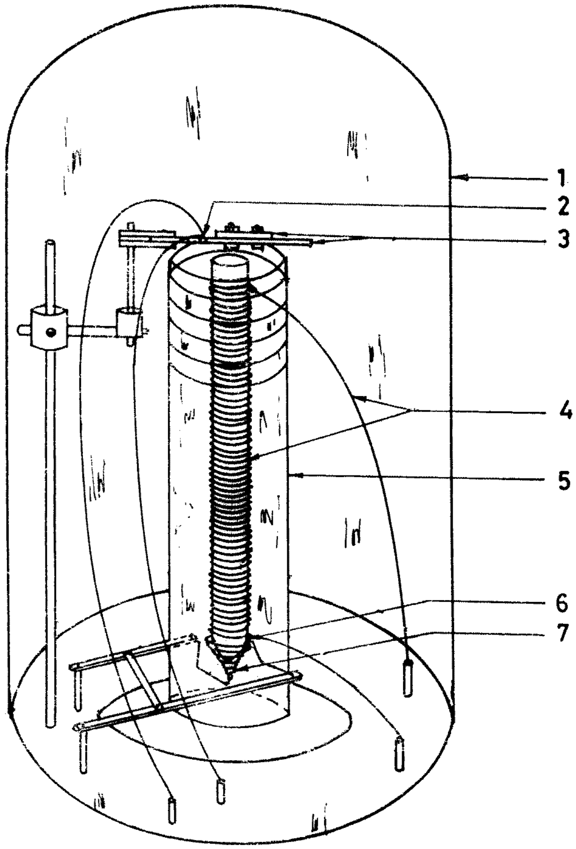

Figure 1. The hot wall experimental setup. (1) Glass bell jar; (2) thermocouple; (3) substrate manipulator cum heater; (4) kanthal wire heater; (5) outer glass tube; (6) quartz tube; (7) tungsten filament.

The quartz tube was charged with CdSe powder (CdSe 99.999%, Alrich, USA) and the films were deposited by keeping the source and wall temperatures around 1000 and 900 K respectively. Substrate temperature was varied from 400 to 700 K. Chromel–alumel thermocouples were used to measure the temperatures of source and wall and a copper–constantan thermocouple was used to measure the substrate temperature.

2.2. Structural analysis

The structure of the films were analysed using an x-ray diffractometer (JEOL 8030, using Cu Kα radiation with $\lambda = 0.15418$ nm). The line profiles were subjected to variance analysis (Mitra 1964) and, since the method is sensitive to variation near the tails of the peaks, a careful adjustment of the background was carried out following the methods of Mitra and Misra (1966). Also the profiles were corrected for instrumental broadening by subtracting the variance of the corresponding profile for standard well annealed samples. The crystallite sizes (D) were calculated using the Scherrer formula (Cullity 1956) from the full width at half maximum (β).

$$D = \frac{0.94\lambda}{\beta\cos\theta}. \tag{1}$$

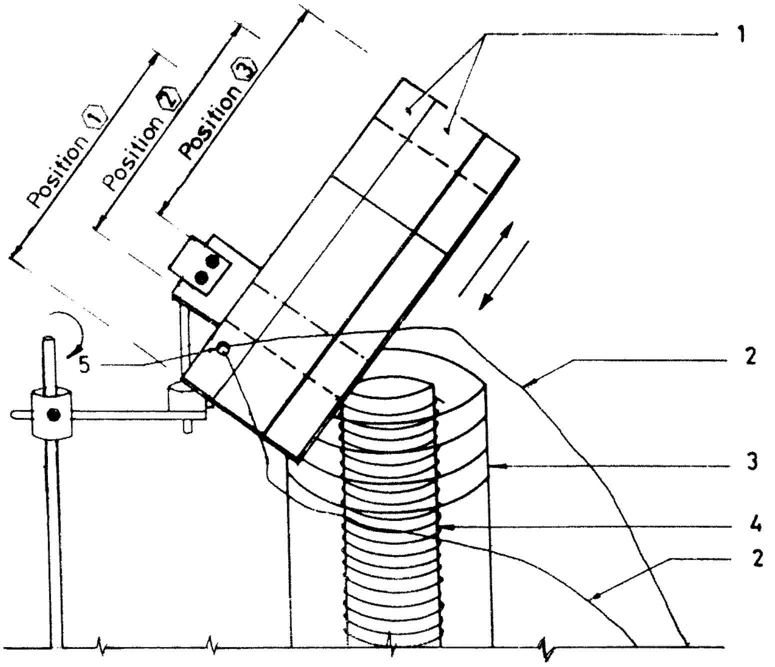

Figure 2. The substrate manipulator. (1) Aluminium plate; (2) nichrome wire; (3) outer glass tube; (4) inner quartz tube; (5) thermocouple.

The strain (ϵ) was calculated from the slope of a $\beta\cos\theta$ versus $\sin\theta$ plot using the relation

$$\beta = \frac{\lambda}{D\cos\theta} - \epsilon\tan\theta. \qquad (2)$$

The dislocation density (δ), defined as the length of dislocation lines per unit volume of the crystal, was evaluated from the formula (Williamson and Smallman 1956)

$$\delta = \frac{1}{D^2}. \qquad (3)$$

The lattice parameters a and c were calculated from the equation

$$\frac{1}{d^2} = \frac{4}{3}\frac{h^2+hk+k^2}{a^2} + \frac{l^2}{c^2}. \qquad (4)$$

Film thicknesses were measured by the gravimetric method using a Metler micro-balance (model No 200). Also the thicknesses of some samples were evaluated from the interference pattern obtained from optical transmittance data recorded using a UV–visible–near IR spectrophotometer (Hitachi, U-3400) in the 350 nm to 2500 nm wavelength range and cross verified by the Taylor–Hobson Tally step method. The smoothness and thickness distribution throughout the film were tested using a surfometer (Planar Product, UK).

2.3. Composition analysis

The composition of the films was estimated by Rutherford backscattering spectrometry (RBS). A 3 MV tandem pelletron accelerator (Institute of Physics, Bhubaneswar, India) with a beam energy of 2 MeV along with a detector positioned at a scattering angle of 140°, were used to carry out the RBS studies.

3. Results and discussion

3.1. Structural analysis

3.1.1. Effect of tube length and thickness. Four quartz tubes of lengths 0.07, 0.10, 0.13 and 0.15 m were used separately to prepare different sets of cadmium selenide films on glass substrates. The substrate temperature was automatically raised to around 380 K under the normal coating conditions, due to the effect of radiations from the wall of the quartz tube. Visual observation showed that films were shining like a mirror. Also the surface analysis revealed that the films were smooth and the thickness distribution was uniform. The x-ray diffractograms of the films on glass substrates of various thicknesses obtained using different tube lengths are shown in figures 3–6. Films coated using 0.07 m tube show preferred orientation along the (103) direction for smaller thicknesses and the (002) orientation for larger thicknesses, but the films coated using 0.10, 0.13 and 0.15 m tubes show highly preferred orientation along the (002) direction irrespective of the thickness. Table 1 shows the calculated values of lattice parameters (a, c), crystallite size (D), dislocation density (δ) and strain (ϵ) for different tube lengths. Physical observation revealed that the films obtained using 0.07 m length tube are highly unstable. The films obtained using 0.13 and 0.15 m are stable compared to films coated using other tube lengths. The instability is due to higher concentration of selenium in the films as observed by Chan and Hill (1976) for vacuum evaporated CdSe films. Here, non-equilibrium in films for smaller tube lengths may be attributed to direct transmission of molecules from the source with out making sufficient wall collisions to attain thermal equilibrium. This was further confirmed from the composition analysis.

Table 1. Structural parameters for CdSe thin films of different thicknesses deposited using different quartz tube lengths.

Tube length	Thickness (nm)	Lattice parameter a (Å)	c (Å)	D (nm)	δ (10^{14} lin m^{-2})	ϵ (10^{-4} lin^{-2} m^{-4})
0.07 m	130	4.235	6.918	16.50	36.73	19.42
	455	4.292	7.024	31.22	10.26	13.85
	610	4.371	6.929	38.53	6.73	9.46
	1200	4.318	7.022	51.86	3.71	9.15
0.10 m	410	4.332	7.010	29.62	11.39	11.91
	620	4.351	6.981	32.22	9.63	9.46
	1200	4.311	7.022	48.38	4.27	8.63
0.13 m	140	4.276	6.937	21.70	21.24	13.00
	420	4.323	6.940	26.81	13.92	9.00
	615	4.321	6.946	28.43	12.37	8.16
	1400	4.330	7.090	43.46	5.29	7.85
0.15 m	160	4.317	7.062	27.03	13.68	12.32
	490	4.317	7.062	29.21	11.72	8.61
	720	4.275	7.09	39.10	6.54	7.86
	1200	4.275	7.09	42.22	5.61	7.86
	1800	4.29	7.09	41.51	5.81	7.91

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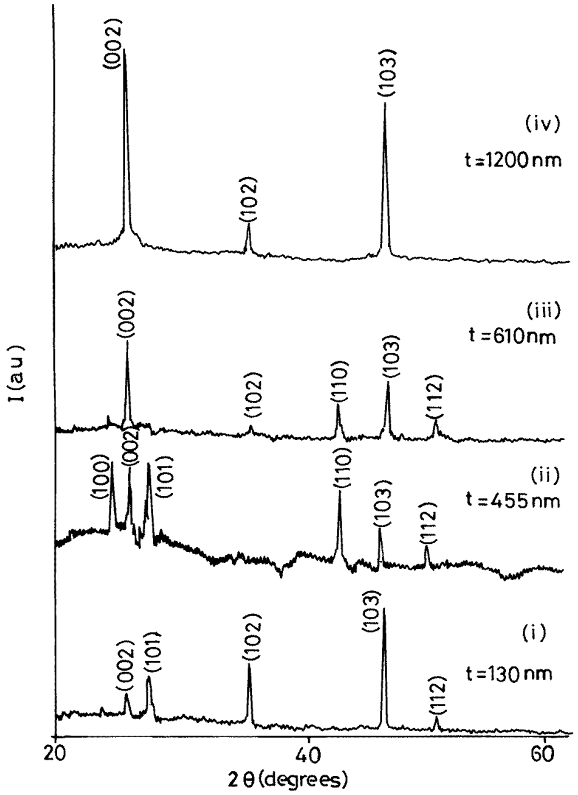

Figure 3. X-ray diffraction patterns of CdSe films deposited on glass substrates using 0.07 m quartz tube length of various thicknesses: (i) 130 nm; (ii) 455 nm; (iii) 610 nm; (iv) 1200 nm.

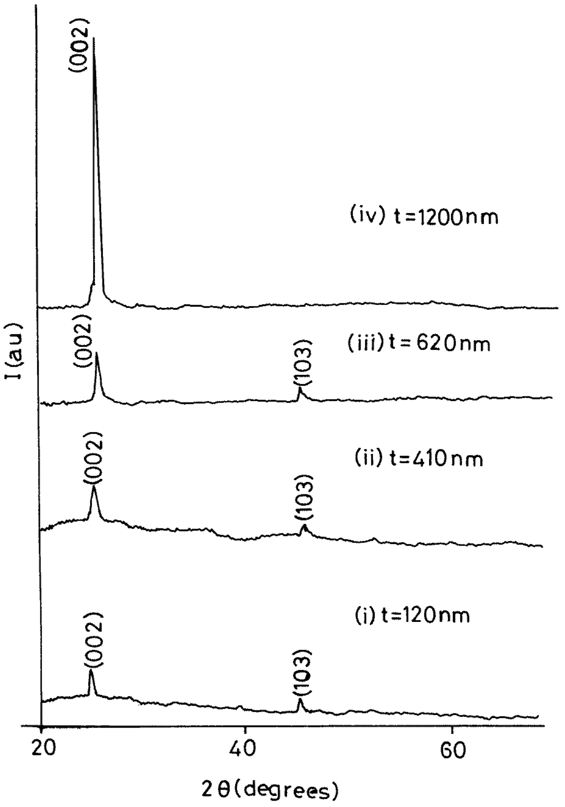

Figure 4. X-ray diffraction patterns of CdSe films deposited on glass substrates using 0.10 m quartz tube length of various thicknesses: (i) 120 nm; (ii) 410 nm; (iii) 620 nm; (iv) 1200 nm.

The calculated lattice parameters are in good agreement with the ASTM data and also with the results obtained by Seetha Rama Rao and Jayarama Reddy (1984) for vacuum evaporated films. It almost coincides with the lattice parameters calculated theoretically for CdSe nanocrystals (a = 4.3 Å and c = 7.020 Å for the 100 cleavage face) from the sp^3 tight binding model reported by Wang and Duke (1988). From table 1 it has been observed that the crystallite size increases but strain and dislocation density in the film decrease with the increase of film thickness

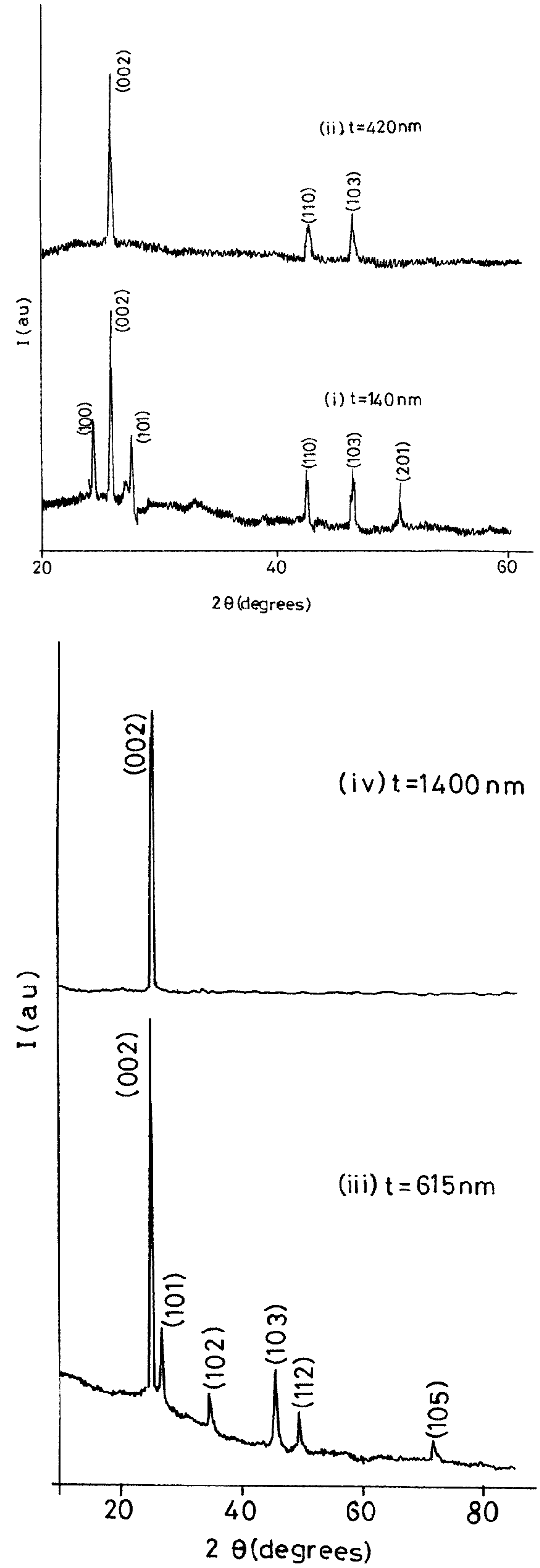

Figure 5. X-ray diffraction patterns of CdSe films deposited on glass substrates using 0.13 m quartz tube length of various thicknesses: (i) 140 nm; (ii) 420 nm; (iii) 615 nm; (iv) 1400 nm.

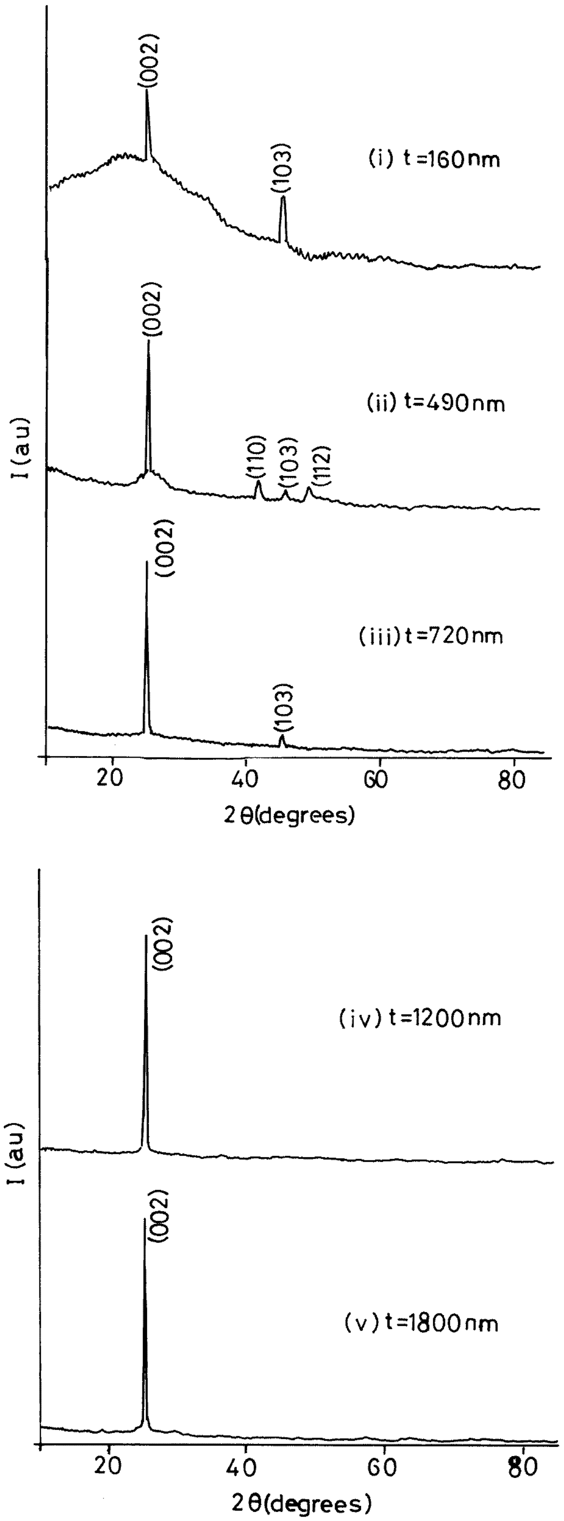

Figure 6. X-ray diffraction patterns of CdSe films deposited on glass substrates using 0.15 m quartz tube length of various thicknesses: (i) 160 nm; (ii) 490 nm; (iii) 720 nm; (iv) 1200 nm; (v) 1800 nm.

for all tube lengths. A similar trend of the dependence of grain size, dislocation density and strain on the thickness was reported by Pal *et al* (1993) for vacuum deposited

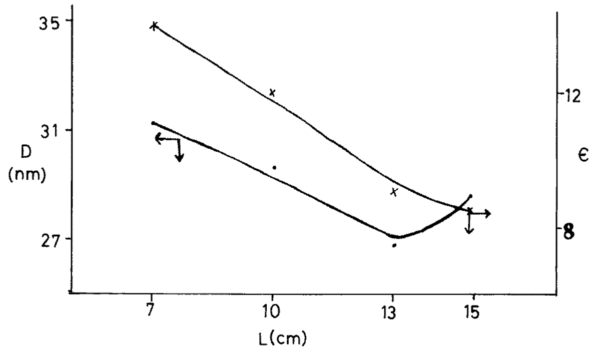

Figure 7. Tube length versus grain size (D) and strain (ϵ) ($\times 10^{-4}$ lin^{-2} m^{-4}) (for 0.07 m, $t = 455$ nm; for 0.10 m, $t = 410$ nm; for 0.13 m, $t = 420$ nm; for 0.15 m, $t = 490$ nm).

CdSe films. When thickness increases the average grain size increases because larger grains begin to form on the surface of smaller grains up to 1.2 μm for all tube lengths, but for films coated on 0.15 m tube length above 1.2 μm a decrease in grain size and an increase of dislocation density were observed, which may be due to the formation of new grains on the surface of larger grains. The size of the grains does not increase indefinitely with thickness but reaches a level, depending on growth conditions, where average grain size begins to oscillate with increasing thickness and the results become more difficult to analyse experimentally. Similar observations were made by Lopez-Otero (1977) for hot wall coated PbTe films.

Figure 7 relates the grain size and strain to the tube length. It was observed that when the tube length was increased the grain size and strain decreased up to 0.13 m and a slight increase in both the parameters was observed thereafter as shown in table 1.

Figure 8 shows a plot of normalized diffraction peak intensities I/t (ratio of peak intensity to the film thickness) versus thickness of the films for 0.07 and 0.15 m tube lengths. The graph indicates clearly the change in orientation of the film for 0.07 m tube length from (103) to (002) as thickness increases. For the films deposited using 0.15 m length tube as the thickness increases the (103) peak is suppressed and a gradual increase of the (002) peak was observed. A similar trend was observed for the films deposited using 0.10 and 0.13 m tube lengths. Hence it is suggested that the film thickness has the predominant effect on the (002) peak intensity regardless of the tube length. Similar observations, *viz.*, increase in peak intensity with thickness, were made on hot wall coated Zn_3P_2 films under different growth conditions by Tetsuji *et al* (1988) but there were no reports of change in orientation with tube length as observed in the present study.

3.1.2. Optimization of tube length. In the hot wall technique, in order to optimize the quartz tube length for the CdSe film a compromise was made to obtain average grains considering the fact of interdiffusion effects, because (i) for optical applications and in the fields of ion implantation we require very smooth surfaces but (ii) for higher mobility the grains must be larger because carrier mean paths larger than the dimensions of the grains cannot be obtained (Lopez-Otero 1977). With tube length of 0.15 m, films with average grain size and more preferred orientation along the (002) plane were obtained. Hence 0.15 m tube length is ideal for preparing CdSe films for further applications.

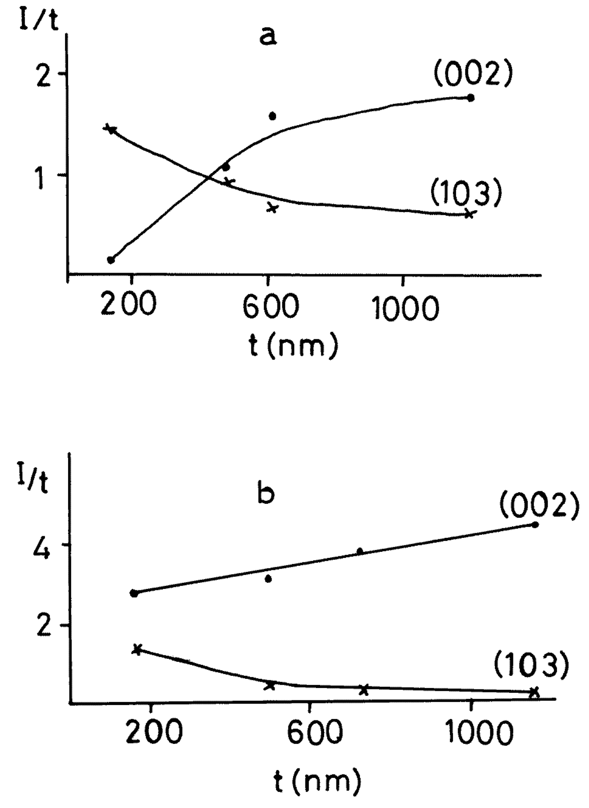

Figure 8. Normalized diffraction peak intensity (I/t) versus thickness (t) of the cadmium selenide thin films deposited using (a) 0.07 m and (b) 0.15 m tube lengths.

Apart from the above parameters, from the technical considerations (Ramachandran and Vaya 1983) of film deposition the following facts are also considered to optimize the tube length. For lower values of tube length, the number of wall collisions is smaller and the probability of direct transmission is greater. This leads to non-equilibrium during growth and in the films, which can be explicitly seen from the composition analysis. For thermal equilibrium, the direct transmission of molecules should be a minimum and molecules must attain thermal equilibrium with the heated wall before the exit. For larger tube lengths the number of wall collisions is sufficient for the molecules to attain thermal equilibrium before they flow out of the system and the direct transmission is a minimum. Also the molecular flux density distribution at the exit plane from the centre to the wall is more uniform for larger tube lengths (Ramachandran and Vaya 1983). Therefore for CdSe films a hot wall system with a tube length of 0.15 m is chosen for optimum growth conditions.

To provide further evidence for the claim of formation of high quality crystalline films deposited using 0.15 m tube length, an observation of FWHM values is made; these have been used as a quantitative measure of the film structural

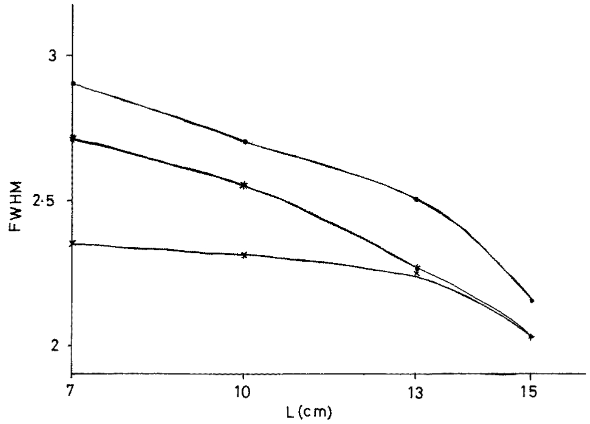

Figure 9. The variation of FWHM with tube length for various thicknesses (●, 400 nm; *, 650 nm; ×, 1200 nm).

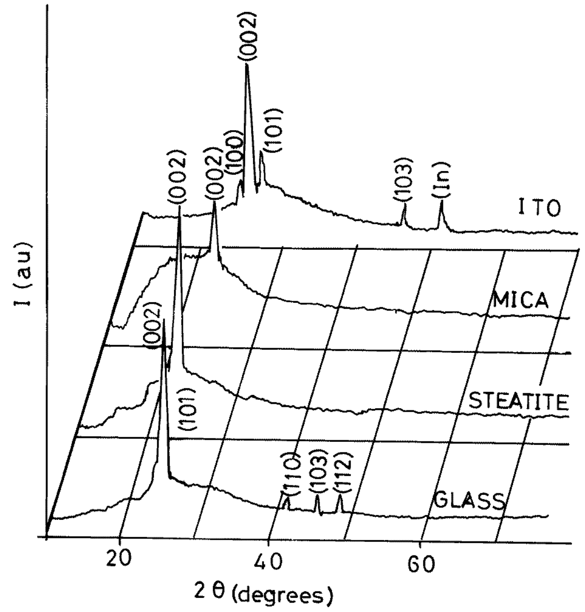

Figure 10. X-ray diffraction patterns of CdSe films deposited on glass, steatite mica and ITO substrates using a 0.15 m length tube.

quality: the smaller the line width the higher the quality of the film (Korenstein and MacLeod 1988). Figure 9 clearly shows the variation of FWHM of CdSe films (thicknesses in the neighbourhood of 450, 600 and 1200 nm) with tube length on glass substrates. The FWHM values are larger for smaller tube lengths and decrease as the tube length increases. Also as the thickness increases the FWHM value decreases, which shows very high quality crystalline films at higher thicknesses.

3.1.3. The effect of the substrate. In order to study the effect of the nature of the substrates on the structural properties of hot wall deposited CdSe films, films were prepared on glass (t = 490 nm), steatite (t = 510 nm), mica (t = 550 nm) and ITO (t = 615 nm) substrates using the optimized tube length of 0.15 m. From the surface analysis the films were found to possess uniform thickness distribution on all the substrates. Further, it has been observed that the films coated on glass, mica and ITO substrates have very smooth surface finish, but the films on

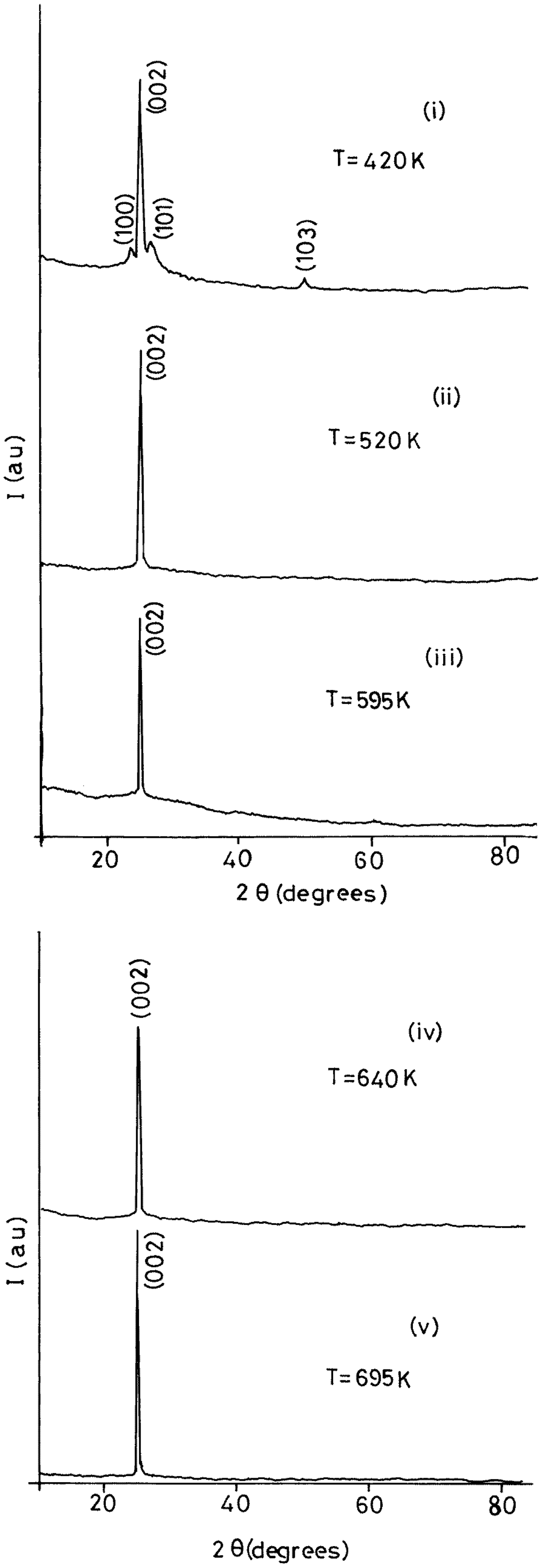

Figure 11. X-ray diffraction patterns of CdSe films of about 500 nm thickness at different glass substrate temperatures: (i) 420 K; (ii) 520 K; (iii) 595 K; (iv) 640 K; (v) 695 K.

Table 2. Structural parameters of CdSe thin films on various substrates using a 0.15 m quartz tube.

Substrate	Lattice parameter a (Å)	 c (Å)	D (nm)	δ (10^{14} lin m^{-2})	ϵ (10^{-4} lin^{-2} m^{-4})
Glass (490 nm)	4.317	7.062	29.2	11.72	8.61
Steatite (510 nm)	4.337	7.073	34.1	8.6	8.8
Mica (550 nm)	4.299	7.007	34.0	8.65	19.1
ITO (615 nm)	4.254	7.062	28.55	12.27	11.92

Table 3. Structural parameters of CdSe films of 500 nm thickness deposited using 0.15 m quartz tube at various substrate (glass) temperatures.

Temperature (K)	Lattice parameter a (Å)	 c (Å)	D (nm)	δ (10^{14} lin m^{-2})	ϵ (10^{-4} lin^{-2} m^{-4})
420	4.317	7.118	28.02	12.73	12.92
520	4.251	7.062	30.14	11.01	11.32
595	4.205	7.09	34.16	8.57	10.07
640	4.205	7.09	38.32	6.81	8.96
695	4.205	7.09	45.74	4.78	7.51

steatite substrate were found to have rough surface finish with greater adhesion.

Figure 10 shows the x-ray diffraction patterns for the CdSe films on glass, steatite, mica and ITO substrates. Table 2 gives a comparative look at lattice parameters, crystallite size, dislocation density and strain on glass, steatite, mica and ITO substrates for the films coated using 0.15 m tube length. It was observed that the strain is a minimum for the films deposited on glass and steatite substrates. The grain size is a minimum for films on glass substrates and a maximum for films on steatite substrates. From the above observations it can be concluded that steatite substrates are more suitable for the electrical and photoelectrical applications which require larger grains in the films (Lopez-Otero 1977).

3.1.4. The effect of substrate temperature. X-ray diffractograms of films of about 500 nm thickness coated using 0.15 m length tube on glass substrates at different substrate temperatures are shown in figure 11. These films show highly preferred orientation in the (002) direction like a single crystal with increased substrate temperature. A similar type of orientation has been reported for molecular beam deposited CdSe films by Masahiko and Tadao (1985). Table 3 shows the calculated values of crystallite size, dislocation density and strain of CdSe films. Similar trends of increase in grain size but decrease of dislocation density and strain with increase of substrate temperature was observed for vacuum evaporated CdSe films (Pal *et al* 1993). Since the particle size and strain are manifestations of dislocation network in the films, the decrease in both these parameters indicates the formation of high quality films.

3.2. Composition analysis

The composition analysis by RBS was made for one representative film each from 0.07, 0.10, 0.13 and 0.15 m length tubes deposited at 380 K. The thicknesses of the films analysed for composition lie in the range of 500 nm. The results are presented in table 4: the Cd/Se ratio for the films increases with increasing tube length. From the vapour pressure data (Gunther 1966) it can be interpreted that tubes with smaller lengths will give rise to CdSe films with a dominance of Se over Cd, but at the same time, if the tube length is increased Cd and Se approach equal proportions and reach almost a 1:1 ratio for 0.15 m tube length. This variation in composition may be due to direct transmission of vapour species from source to substrate without attaining thermal equilibrium. This result further confirms the optimum tube length for CdSe films. Similar composition analyses were made by Gurumurugan (1995) and Balasundaram (1994) to estimate the Cadmium in CdO films and selenium in GeSe films respectively.

Table 4. The variation of composition with tube length for films deposited at 380 K and t about 500 nm.

Tube length (m)	Composition Cd/Se
0.07	0.81
0.10	0.84
0.13	0.92
0.15	1.05

4. Conclusions

Hot wall deposition apparatus was fabricated to deposit three to five similar films in a single pumpdown. Cadmium selenide films were deposited using different tube lengths on glass substrates. From x-ray diffractograms, the structural parameters, *viz*., lattice constants, crystallite size, dislocation density and strain were calculated. The results are in good agreement with the reported values.

Comparison of the results indicates that the optimum tube length for the hot wall deposition of CdSe films is 0.15 m. Composition analysis using the RBS technique confirms the stoichiometric nature of the CdSe films prepared using 0.15 m tube length. Studies on the films coated on different substrates show that steatite substrates are more suitable for preparing CdSe films for photo-electrical applications. Substrate temperature studies reveal that single-crystalline-like films could be obtained at higher substrate temperatures. Finally, it can be emphatically pointed out from the present work that one does not have to sacrifice the simplicity and versatility of vacuum evaporation methods in order to obtain high quality thin film materials.

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