

## Structural studies of synthesis of CdSe from Cd/Se bilayer

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Cadmium and selenium bilayer have successfully been made by a vacuum evaporation technique on cleaned glass substrates under the vacuum of the order of  $10^{-5}$  mbar. The samples of different thicknesses were prepared by varying Cd:Se ratio as 1:1, 1:1.5, 1:2. Rapid thermal annealing (RTA) of some bilayers have been carried out at different temperatures in nitrogen ambient. XRD measurements were made for structural analysis on these as-deposited and annealed samples. RTA studies could show the formation of cadmium selenide at the interface after annealing. XRF measurements were carried out for elemental analysis of these bilayers. XRF studies reveal that the films are found to be cadmium rich at room temperature. The results of CdSe formed from Cd/Se bilayer by RTA are compared with annealed sample.

**Keywords:** CdSe films, Wurtzite structure, Rapid thermal annealing

**IPC Code:** C30B

### 1 Introduction

In recent years more attention has been focused on II-VI semiconductor compounds due to their optoelectronic properties<sup>1,2</sup>. Cadmium selenide is an important member of group of binary compounds and it can be synthesized using various techniques<sup>3,4</sup>. Synthesis of CdSe using hot wall deposition technique, molecular beam epitaxy and laser ablation technique has earlier been reported<sup>5,6</sup>. CdSe is grown by evaporation technique<sup>7</sup>. In literature there are reports on CdSe compound evaporation on glass substrates at different conditions<sup>8</sup>. Earlier we have reported the structural analysis of Cd/Se bilayer by annealing in nitrogen ambient<sup>10</sup>.

The present paper reports on structural and elemental analysis of as-deposited Cd/Se bilayer prepared onto glass substrate using vacuum evaporation technique and their Rapid Thermal Annealing (RTA) in nitrogen ambient at different temperatures.

### 2 Experimental Details

The Cadmium and selenium (Cd/Se) bilayers were prepared by evaporating first cadmium (99.99%) and then selenium (99.99%) powder onto cleaned glass substrates under the vacuum ( $10^{-5}$  mbar). Samples with cadmium to selenium ratio of 1:1, 1:1.5, 1:2 were prepared. The substrate cooling arrangement was employed for cadmium deposition. Selenium was

deposited at the room temperature. The Rapid Thermal Annealing (RTA) of as-deposited samples was carried out in dry nitrogen ambient at 373, 423 and 473K for 60s/120s at each temperature. The structure of the bilayer samples was investigated by X-ray diffraction (XRD), on a Seimens D-500 spectrometer. The samples were scanned for the  $2\theta$  (20 to 80 degree) and were rotated at a speed of  $2^\circ/\text{min}$ . The elemental analysis was carried out on a Jeol JSX-322 EDXRF with a rhodium as the target.

### 3 Results and Discussion

#### 3.1 X-ray diffraction analysis

The X-Ray diffraction patterns of Cd/Se bilayer with Cd:Se ratio of 1:2 using  $\text{Cu-K}\alpha$  radiation ( $1.5405\text{\AA}$ ) are shown in Fig. 1(a). In as-deposited films the characteristics peaks (002), (100), (101), (103) and (004) for cadmium in the range of  $31^\circ$ - $75^\circ$  are clearly appeared and selenium is found to be amorphous. The reflection in (002) orientation for cadmium is observed to be prominent at  $2\theta$  value of  $31.8^\circ$ . The presence of sharp peaks indicates that the film is polycrystalline in nature. Fig. 1(b) shows XRD pattern for Rapid Thermal Annealed (RTA) Cd/Se bilayer at 373K for 60s in nitrogen ambient. The reflections from selenium are observed at  $23.5^\circ$  and  $29.8^\circ$  and all indexed as (100) and (101). The other reflections observed are presented in Table 1.

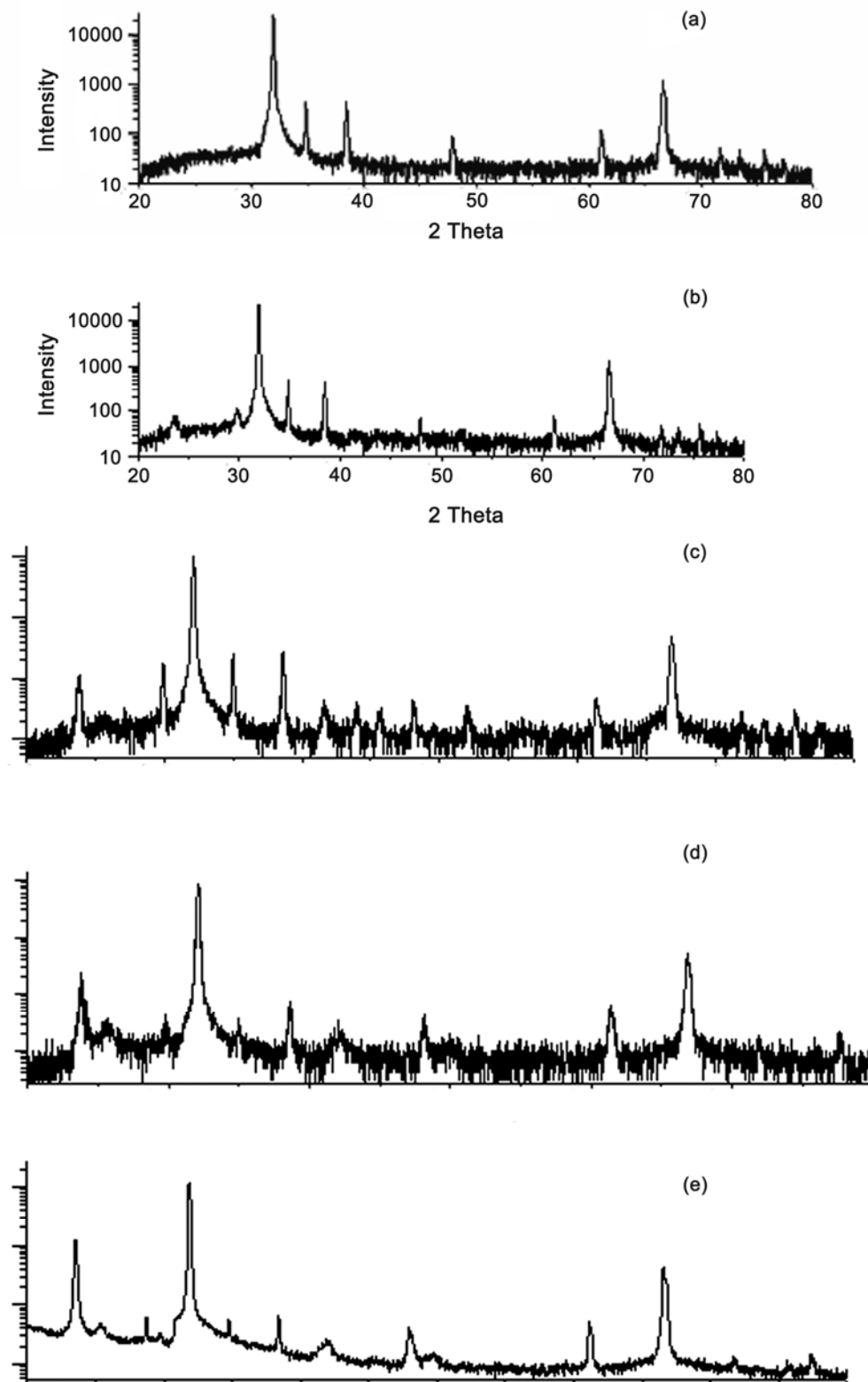


Fig. 1 — XRD pattern of Cd/Se bilayer films (a) as-deposited, (b) annealed at 373K for 60sec, (c) annealed at 423K for 60sec, (d) annealed at 473K for 60sec, (e) annealed at 473K for 120sec.

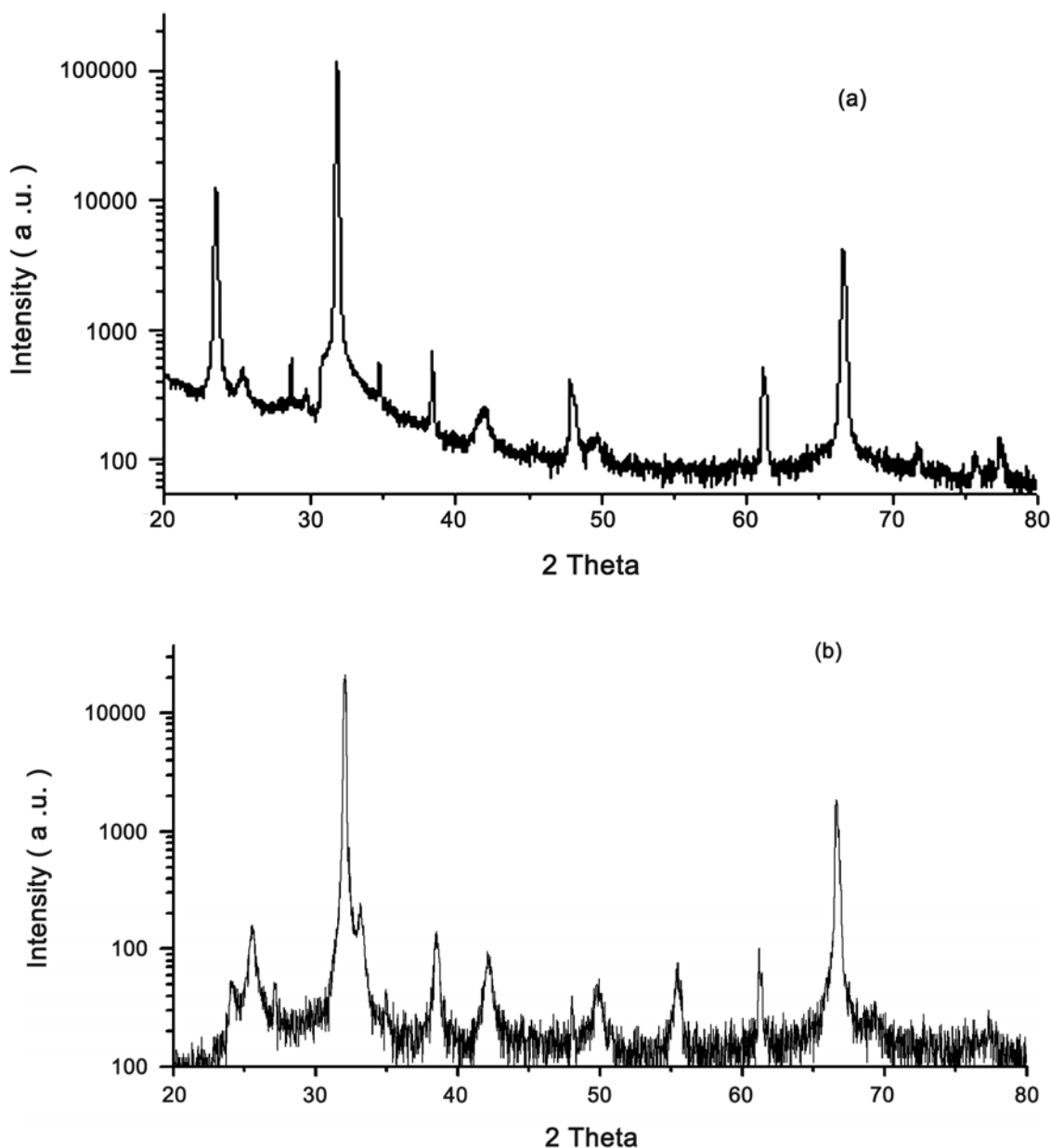


Fig. 2 — XRD pattern for Cd/Se bilayer films (a) RTA at 473K for 120 sec, (b) Furnace annealed at 473K for 2½ hour

Fig. 1(c) shows XRD pattern for Rapid Thermal Annealed Cd/Se bilayer at 423K for 60s in nitrogen ambient. It shows the formation of CdSe with orientation of the film to be prominent in (100) at  $2\theta$  value of  $23.8^\circ$ . The other reflections from CdSe are observed in (102), (110), (103), (210) and (105) orientations. The film is found to be polycrystalline with a hexagonal CdSe phase formation, which corresponds to wurtzite structure. At 423K the presence of cadmium and selenium is also observed and presented in Table 1. The XRD pattern for Rapid

Thermal Annealed Cd/Se bilayer at 473K for 60s in nitrogen ambient is shown in Fig. 1(d), it shows very few reflections from cadmium and selenium with a decrease in intensity of the peaks. The intensity of the CdSe peak in (100) orientation is found to be increase. Fig. 1(e) shows XRD pattern of further RTA of the Cd/Se bilayer at the same temperature of 473K for 120 seconds. The peak of CdSe at  $2\theta$  value of  $23.8^\circ$  in (100) orientation is found to be more intense with peak intensity of  $\sim 861$  whereas other reflections from cadmium and selenium are less intense.

Table 1 — XRD measurement of Cd/Se bilayer film Rapid Thermal Annealed at different temperatures

Annealing temperature/ Time	2θ	Dexp. (Å)	Dstd. (Å)	(hkl)	Crystal structure	
373K (60 s)	23.55	3.776	3.78	100	Se (H)	
	29.83	2.992	3.005	101	Se (H)	
	32.07	2.789	2.809	002	Cd (H)	
	34.87	2.571	2.580	100	Cd (H)	
	38.39	2.343	2.345	101	Cd (H)	
	47.90	1.898	1.901	102	Cd (H)	
	61.10	1.516	1.516	103	Cd (H)	
	66.72	1.401	1.404	004	Cd (H)	
	71.81	1.313	1.316	112	Cd (H)	
	75.64	1.256	1.258	201	Cd (H)	
	23K (60 s)	23.82	3.732	3.72	100	CdSe (H)
		29.90	2.985	3.005	101	Se (H)
		32.07	2.788	2.809	002	Cd (H)
		35.05	2.558	2.554	102	CdSe (H)
38.57		2.332	2.345	101	Cd (H)	
41.75		2.162	2.151	110	CdSe (H)	
44.20		2.047	2.071	102	Se (H)	
48.06		1.891	1.901	102	Cd (H)	
51.95		1.759	1.766	201	Se (H)	
61.44		1.503	1.516	103	Cd (H)	
66.91		1.397	1.404	004	Cd (H)	
473K (60 s)	23.91	3.719	3.72	100	CdSe (H)	
	29.80	2.996	3.005	101	Se (H)	
	32.07	2.788	2.809	002	Cd (H)	
	35.05	2.558	2.554	102	CdSe (H)	
	38.57	2.332	2.345	101	Cd (H)	
	42.28	2.136	2.151	110	CdSe (H)	
	48.25	1.885	1.863	200	CdSe (H)	
			1.90	102	Cd (H)	
	61.44	1.508	1.516	103	Cd (H)	
	67.07	1.394	1.404	004	Cd (H)	
			1.407	210	CdSe (H)	
473K (120 s)	23.81	3.732	3.72	100	CdSe (H)	
	25.68	3.465	3.51	002	CdSe (H)	
	29.96	2.980	3.005	101	Se (H)	
	32.15	2.781	2.809	002	Cd (H)	
	34.99	2.562	2.554	102	CdSe (H)	
	38.63	2.328	2.345	101	Cd (H)	
	42.17	2.140	2.151	110	CdSe (H)	
	48.09	1.890	1.863	200	CdSe (H)	
			1.90	102	Cd (H)	
	61.31	1.510	1.516	103	Cd (H)	
	66.76	1.399	1.404	004	Cd (H)	
			1.407	210	CdSe (H)	

Table 2 — XRF measurement of as-deposited Cd/Se bilayer film for different ratio

Cd/Se Bilayer	Ratio	Molecular percentage of Cadmium	Molecular percentage of selenium
	1:1	92.6740	7.324
	1:1.5	88.9688	11.0312
	1:2	83.9932	16.0068

RTA studies have been made for different Cd:Se ratios of 1:1, 1:1.5, 1:2 but the stoichiometric CdSe formation could take place for the Cd/Se bilayer ratio of 1:2 at an annealing temperature of 473K. The comparison of XRD spectra for CdSe formed at 473K of Cd/Se bilayer for 2½ hr in nitrogen ambient with that of Rapid Thermal annealing at 473K for 120s is shown in Fig. 2. In furnace annealing the CdSe film is prominent in (002) orientation at 2θ value of 25.6° only after annealing, for a longer duration whereas in RTA the orientation is found to be prominent in (100) direction after annealing the film for 120s at 473K. In both the annealing films are polycrystalline in nature with a hexagonal phase corresponding to wurtzite structure. The difference in the orientation of the film is due to recrystallization of the film.

### 3.2 X-ray fluorescence

The quantitative/elemental analysis of Cd/Se bilayer samples of ratio 1:1, 1:1.5, 1:2 shows that the room temperature deposits of the films are cadmium rich i.e. the percentage of selenium incorporated in the film is less compared to that of cadmium. Results are given in Table 1.

### 4 Conclusion

The investigated CdSe from Cd/Se bilayer films have revealed polycrystalline nature. XRD measurements show an improved crystallinity of CdSe in Rapid Thermal Annealing (RTA) films with orientation in (100) plane whereas XRD of the annealed films shows the crystallites oriented in (002) plane due to recrystallization. The CdSe has a hexagonal phase corresponding to wurtzite structure. Quantitative analysis reveals that Cd/Se bilayer are cadmium rich at the room temperature.

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