MONITORING INTRINSIC STRESS INDUCED IN THE CdSe THIN FILMS DURING DEPOSITION BY DOUBLE EXPOSURE HOLOGRAPHIC INTERFEROMETRIC TECHNIQUE

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The double exposure holographic interferometry (DEHI) technique is used to study the intrinsic stress induced in the CdSe thin film. Cadmium selenide (CdSe) thin films have been deposited onto stainless steel and fluorine doped tin oxide (FTO) coated glass substrates by electrodeposition technique. For deposition Cadmium Sulphate (CdSO₄) and Selenium dioxide (SeO₂) are used as precursors. The structural and optical properties of the as deposited films have been studied using X-ray diffraction (XRD), Optical absorption respectively. The stress of the CdSe thin films was carried out by Double Exposure Holographic Interferometric Technique (DEHI).

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1. Introduction

Holography is an interference method of recording the light waves diffracted by a subject illuminated with coherent light. Holographic interferometry is a precise technique for measuring changes in the dimensions of an object. The double exposure holographic interferometry technique has been widely accepted as a viable tool for non-destructive testing of materials. The double exposure holographic interferometry (DEHI) involves the recording of images of an object before stressing and after stressing. It is highly accurate displacement measurement method employed for the determination of amplitude of vibration of metal disc [1-2]. It can also be applied to many engineering problems, especially continuous comparison of the surface displacement relative to an initial position [3]. Most of the industrial applications of thin films require mechanically stable coatings. The mechanical stability of the thin film depends mainly on the adhesive of the films to the substrates and on the intrinsic stress induce 4s in the films during deposition.

Various methods so far adopted for the preparation of CdSe thin films include cathodic electrodeposition, chemical bath deposition, evaporation, co-evaporation, spray pyrolysis, slurry painting and pellet sintering [4-14].

The literature survey reveals that no reports are available on the determination of CdSe film thickness and stress to the substrate using double exposure holographic interferometry technique. The present study concentrates on the synthesis and characterization of electrodeposited CdSe thin films prepared from aqueous acidic bath. The thickness and stress of the films have been determined by DEHI technique for various deposition times. The samples are then characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and optical absorption.

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2. Experimental

Thin films preparation:

CdSe thin films were electrodeposited on conducting stainless steel and FTO coated glass substrate from aqueous acidic bath containing 0.05 M CdSO₄ and 0.01 M SeO₂ as precursor sources of Cd and Se ions. The uniform and well adherent thin films have been deposited at room temperature. Before using stainless steel substrates in the electrolytic bath, they were polished by smooth polish paper (Zero fine grades) and cleaned with double distilled water. In order to remove the oily substance from the surfaces, cleaned substrates were etched in 10 % H_2SO_4 for 2 min. and finally ultrasonically cleaned with double distilled water and dried with hot air/ alcoholic vapors.

Electrodeposition of CdSe thin films was carried out using potentiostat in potentiostatic mode 230 ml of an electrolyte was used without stirring, pure graphite was used as an anode and cathode was either stainless steel or FTO coated glass substrate. The deposition potential measurement was carried out with respect to standard calomel electrode (SCE) as a reference electrode. Yellow coloured uniform and stoichiometric CdSe thin films were deposited on to stainless steel and FTO coated glass substrates. For optimized preparative parameters having bath compositions 0.05 M CdSO₄ and 0.01 M SeO₂ in volumetric proportion as 1:1 and deposition potential of -0.82V V/SCE.

2.1 Holographic experimental set up:

Double exposure holographic interferometry (DEHI) was used to record the holograms. In this technique comparison of the surface displacement of an object relative to its initial position causes in interference pattern observed on the object [15,16]. All the optical components used in the recording were arranged on a vibration-isolated table [17]. This system avoids all the vibrations reaching on to the top of the table from the ground. The substrate was illuminated by a 5 mW He-Ne laser ($\lambda = 6328$ A°). A beam splitter (70: 30) was used to split the laser beam into two paths: object beam and the reference beam as shown in fig. 1. The reference beam was impinged via mirrors directly on to the holographic plate where as the object beam scattered from the stainless steel substrate. The hologram was recorded on the 8E HD 75 AGFA holographic plate before and after the deposition of CdSe thin film. The reconstructed images of these interferograms were captured with a high resolution CCD camera.



2.2 Measurement of stress to the substrate:

The adhesion of thin films to their substrates is influenced by factors such as the proper matching of the substrate and thin film materials the cleaning techniques and the method of the deposition. In the present investigation, we utilized the DEHI technique for the study of thickness, stress to substrate. Also the structural, morphological and optical properties have been studied. However residual stresses are known to cause more damage to the film by crazing or in the worst case, by peeling of the film itself. The study of the residual stresses in optical thin film has assumed greater importance because of the development of high power laser systems which use coated optical compounds.

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Sr	Time of	Number of	Thickness of thin	Stress in film in ×
No.	deposition	fringes	film in (µm)	$10^9 (dyne/cm^2)$
	(min)			
1	1	13	0.21	8.33
2	2	9	0.3432	3.5266
3	3	7	0.71715	1.3126
4	5	5	0.8349	0.8053

Table. 2

Sr. No.	Standard	Observed 'd' (A ^o)	Plan (h k l)
	'd' (A')		
1	1.98	2.03	(103)
2	1.80	1.80	(201)
3	1.24	1.27	(300)
4	1.17	1.17	(302)
5	1.07	1.08	(220)

A simple non-destructive technique for the qualitative measurement of stress is given by the formula [18]

$$S = \frac{t_s^2 Y_s \Delta}{3l^2 t_e} \tag{1}$$

where S- is stress, which is to be calculated

 t_s - the substrate thickness can be measured by using micrometer gauge meter

 t_f - thin film thickness

 Y_{s} - Young's modulus of elasticity for the substrate

l- length of the substrate on which the film is deposited

 Δ - deflection of the substrate equal to $n\lambda/2$, n is the no. of fringes.

Structural characterization was carried out using a Philips PW-1710 X-ray diffractometer (Cu K_{α} radiation). Optical absorption studies of the films deposited onto FTO coated glass substrates in the wavelength range of 350-850 nm were done with the help of UV-vis- NIR

spectrophotometer (Hitachi Model 330). The surface morphology of the films was studied by scanning electron microscope.

3. Results and discussion

3.1 X-RD studies

A typical X-ray diffraction pattern recorded on stainless steel substrate for CdSe film deposited at optimized preparative parameters at room temperature is shown in Fig. 2. The XRD data are indexed with the help of JCPDS file [19]. The planes (1 1 0), (2 0 1), (2 1 2) and (2 2 0) are corresponding to CdSe hexagonal crystal structure. The shift in some of the peak position is attributed to internal strain existing in the crystallites due to disproportionate array of the constituents. Hence, the XRD data are therefore in agreement with polycrystalline nature of the material with hexagonal crystal structure. The similar results are obtained by Datta et al. [20].



Fig. 2 X-ray diffraction patterns of CdSe thin films for 20 and 30 min time.

3.2 Morphological studies

Fig. 3 (a-b) shows the SEM images of CdSe thin film at different (20-30 min.) on stainless steel substrate at room temperature. Increase in the thickness of the films shows a substantial granular growth and dense porous network structure. Increase in overgrowth of surface particles with thickness is well known for chemically deposited films [21]. From SEM, it is observed that as deposited CdSe films were homogeneous without cracks and covered by layered flacks to the surface on the substrate.





b

Fig. 3 SEM image of CdSe thin films for 20 and 30 min time.

3.3 Optical studies

CdSe thin film deposited at optimized preparative parameters on FTO coated glass substrate was characterized by optical absorption technique. The absorption data are further analyzed for near edge optical absorption of the semiconductor using a classical

relation (2),

$$a = \frac{A(h\nu - Eg)^n}{h\nu}$$
(2)

where the symbols have their usual meaning. For allowed direct transition, n = 1/2. The value of absorption coefficient is found to be of the order of 10^4 cm⁻¹ that supports the direct bandgap nature of the semiconductor. The plot of $(\alpha hv)^2$ versus hv is shown in Fig. 4. The straight portion is extrapolated to the energy axis at $\alpha = 0$, which gives the band gap energy (Eg) of CdSe to be 2.5 eV, which is slightly greater than the standard value reported for CdSe material [22]. This may be due to the smaller crystallite size and fine grains present in the semiconductor thin films [23, 24].



Fig. 4 Plot of $(ahm)^2$ vs. hm for CdSe thin films for 20 min.

3.4 Double Exposure Holographic Interferometry (DEHI)

Fig. 5 shows recorded holograms of CdSe thin films developed on the holographic plate from the hologram study. From the hologram study, it is observed that as deposition time increases, the number of fringes localized on the surface of stainless steel substrate increases and consequently decreases the fringe width. Hence the thickness of film increases and intrinsic stress in the film decreases. The variation of CdSe film thickness with deposition time is shown in Fig.6. Initially CdSe film thickness was increased linearly up to 5 min. and it becomes saturates. After certain limits, stress in the film decreased and leads to rupture and failure of thin film.

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Fig. 5 Holograms of CdSe thin film for deposition time: (a) 50 min; (b) 10 min; (c) 20 min; and (d) 30 min.



Fig. 6 The variation of CdSe film films for 20 and 30 min time.



Fig. 7 The variation of CdSe film stress to the substrate with deposition time 20 min and 30 min.

Fig. 7 shows the variation of CdSe thin film stress with deposition time. It is seen that as time of the film deposition increases, stress developed in the film decreases. [25]

4. Conclusion

In conclusion CdSe thin film can be successfully deposited by using electro deposition technique from aqueous medium. The determination of thickness and stress of the thin films can be measured in situ by using Double Exposure Holographic Interferometry Technique. The XRD study reveals the formation of CdSe thin films are polycrystalline with hexagonal crystal structure. The optical absorption studies shows that the as-deposited CdSe film has optical direct band gap of 2.4 ev.

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