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The Potentiostatic Electrodeposition of Indium Doped Aluminium Selenide Thin Films

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ABSTRACT

The In containing AlSe thin films were electrosynthesized by electrochemical co-deposition technique. The morphological properties of thin films were studied through the Scanning Electron Micrograph (SEM) while the structural features through X-Ray Diffraction technique (XRD). The deposition current along with the film thickness values, the charge carrier density, flat band potential, corrosion characteristics i.e., corrosion current, corrosion potential and corrosion rate were calculated.

Key words: Potentiostatic Electrodeposition; Saturated Calomel Electrode (SCE); Scanning Electron Micrograph (SEM); X-Ray Diffraction (XRD); Charge Carrier Density (N_D); Flat Band Potential (E_{fb}); Corrosion Current (Icorr); Corrosion Potential (Vcorr) and Corrosion Rate (CR).

INTRODUCTION

The In containing AlSe thin films have significance in various applications particularly in electronic devices. The electrodeposition of these films was carried out potentiostatically. Certain methods are performed for this purpose but here thin films were electrosynthesized by electrodeposition method[1-14] which is considered as the simple, effective and cheap to obtain Aluminium chalcogenide thin films. The kinetics of electrode reactions was studied by I-t characteristics. The charge carrier density and flat band potential were determined by the capacitance measurement. With the help of I-t characteristics deposition current and film thickness were also determined. These films were characterized by Scanning Electron Micrograph (SEM) and structural features of these films were studied by X-Ray Diffraction technique (XRD). In order to study the corrosion characteristics of the films polarization techniques were used.

RESULTS AND DISCUSSION

The electrodeposition of In doped AlSe thin films had been carried out at -0.700 mV vs SCE at room temperature. The current-potential behavior of AlSe was helpful to decide the deposition potential value. Fig. 1 shows the current-voltage behavior of AlSe solution. During the deposition of AlSe, the current decreases very fast upto a steady state values. Fig. 2 shows the variation of current with time for AlSe. These current-time

characteristics were also studied for different compositions of In containing AlSe and AlIn thin films. The decrease in the current indicates the total coverage of the deposits on the surface of the substrate as well as the quality of the deposited material. The deposition current and the film thickness were also estimated by the amount of charge passed through the electrolyte. The deposition current and film thickness (f_t) values are given in table 1. These values were also estimated for the different compositions of In containing AlSe thin films and AlIn. The film thickness value increases on the inclusion of In concentration.

The charge carrier density (N_D) and the flat band potential (E_{fb}) of these deposited films had also been studied with the help of Mott-Schottky plots[15-20]. Fig. 3 shows the Mott-Schottky plot for In containing AlSe film. The slope of these plots describes the N_D while the extrapolation and intersection of these curves at potential axis determines the E_{fb}. The values of N_D and E_{fb} are also shown in table 1 for all the compositions of AlSe, In containing AlSe and AlIn solutions. The charge carrier density values increases with increasing the concentration of Indium in the electroplating solution. The capacitance data indicates that the prepared films shows p-type semiconductor.

The polarization studies of the electrodeposited films had been carried out. The potential of the working electrode was varied with respect to SCE and corresponding current flowing between the working and counter electrode was measured to obtain Tafel Plots. In order to improve the resistance of the electrodeposited thin films against corrosion the inhibitor Benzotriazole was added to the electrolyte. The corrosion characteristics[21-24] had also been studied in the presence of inhibitor. Fig. 4 shows the tafel plot for 0.006M In containing AlSe thin film in the absence and in the presence of inhibitor. With the help of these tafel plots Icorr and Vcorr were estimated while the CR was calculated with the help of an expression:

Table1: Electrochemical Parameters of In containing AlSe thin films

S. No.	Composition			Deposition Current ($mAcm^{-2}$)		Film Thickness (10^{-5} cm)	Charge Carrier Density (N_D) (10^{-25} cm^{-3})	Flat Band Potential (E_{fb})
	Al	Se	In	Initial	Final			
1.	0.01	0.01	0.000	1.41	0.29	2.87	4.44	10.5
2.	0.01	0.01	0.002	2.23	0.72	3.03	7.14	10.9
3.	0.01	0.01	0.004	3.10	1.15	4.46	7.40	11.3
4.	0.01	0.01	0.006	4.30	1.71	6.85	9.52	12.5
5.	0.01	0.00	0.010	3.23	0.24	4.79	8.00	10.8

Table 2: Corrosion characteristics of deposited thin films in the absence and in the Presence of inhibitor

S. No.	Icorr (mA)	Absence of Inhibitor		Presence of Inhibitor		
		Vcorr (mV)	CR (MPY)	Icorr (mA)	Vcorr (mV)	CR (MPY)
1.	0.8	300	2.57	1.00	300	5.15
2.	1.2	200	5.18	2.21	250	11.34
3.	1.5	100	7.73	2.5	160	12.89
4.	2.6	150	12.89	4.2	80	21.66
5.	4.2	40	21.66	6.0	40	30.94

$$CR = \frac{Icorr \times 0.13 \times Eq.\ Wt.}{F \times d \times A}$$

Where,

Icorr = Corrosion Current of the deposited film.
Eq.Wt. = Equivalent Weight of AlSe.
F = Faraday's Constant.
D = Density of AlSe.
A = Area of the substrate.

These tafel plots were constructed for AlSe, In containing AlSe from 0.002 M to 0.006 M concentration and AlIn thin films, in the absence and in the presence of inhibitor. Table 2 shows the values of Icorr, Vcorr and CR in the absence and in the presence of inhibitor. These data shows that the values of Icorr and CR increases while Vcorr decreases with the increment of In in AlSe solution in absence and in presence of inhibitor.

Scanning Electron Micrograph (SEM) is the source of studying the surface morphology of

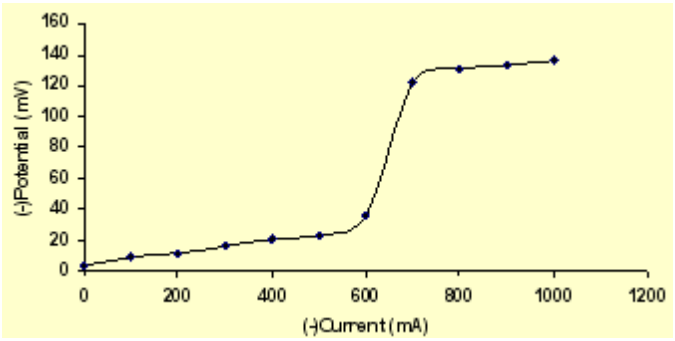

Fig. 1: Current Voltage behaviour of AlSe thin film

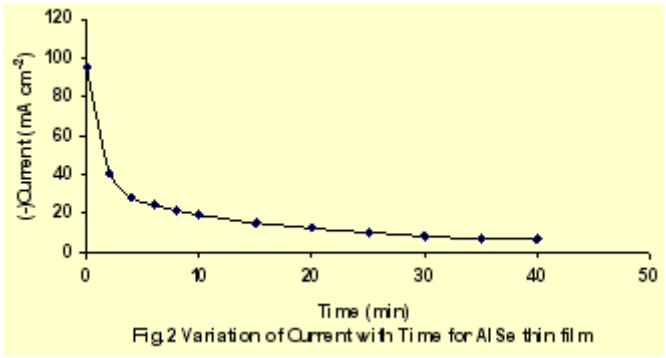

Fig. 2: Variation of Current with Time for AlSe thin film

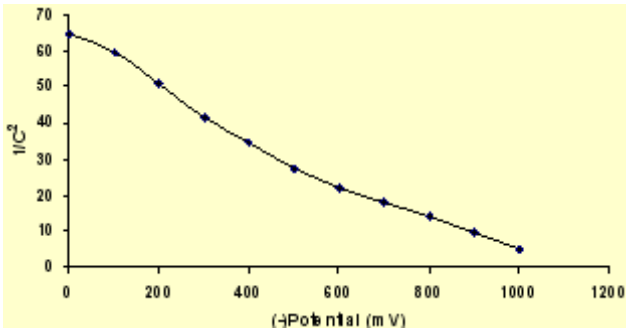

Fig. 3: Mott-Schottky plot for in containing AlSe thin film

the deposited films[25-30]. Fig. 5 shows the SEM of the electrodeposited (a) AlSe (b) 0.004 M In containing AlSe and (c) AlIn thin films respectively. The increment of Indium in different compositions in the electroplating solution affects the morphology of the films. The particle size of the deposited films range from 100 nm to 400 nm. The change in the particle size also supports the fact that there is an addition of Indium in different concentrations in the electroplating solution. The surface of the film shows the continuity and homogeneity.

The crystallite size of the deposited films was studied by X-Ray diffraction method[31-36] (XRD) using Scherrer's formula,

$$d = \frac{\lambda}{\beta \cos \theta}$$

Where,

λ = Wavelength used 1.5418 Aº

β = Angular line width at half maximum intensity in radians

θ = Bragg's angle grain size was found to be of the order of ~ 26nm in AlSe, ~ 22nm in In containing AlSe and ~42 nm in AlIn.

Fig. 6(a), 6(b) and 6(c) shows XRD pattern of AlSe, 0.004M In containing AlSe and AlIn thin films by SILAR method. Comparison of d-values with ASTM data for AlSe shows that the material is AlSe having hexagonal structure.

EXPERIMENTAL

The In doped AlSe thin films have been electrosynthesized at -0.700 mV vs SCE at room temperature. The starting materials were 0.01 M $AlK(SO_4)_2$ (Sd fine), 0.01 M SeO_2 (Sd fine) and 0.001 M $InCl_3$ (Sd fine). Among these materials, the concentrations of $AlK(SO_4)_2$ and SeO_2 were kept constant through out the experiment and the concentration of $InCl_3$ was varied from 0.002 M to 0.006 M. Different compositions of In were added in the total concentration of AlSe solution. Thus, five electrodes were prepared for each concentration of In in AlSe.

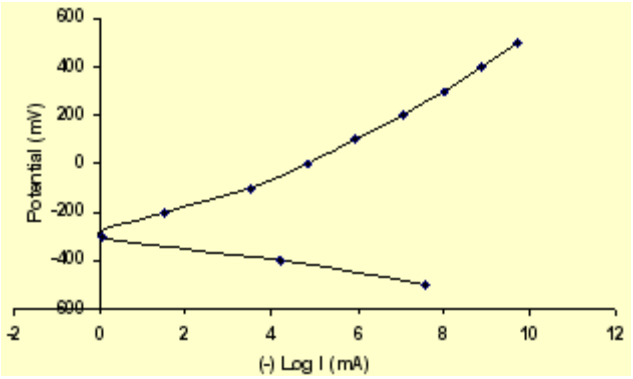

Fig. 4(a): Tafel Plot for 0.006 M in containing AlSe thin film in absence of inhibitor

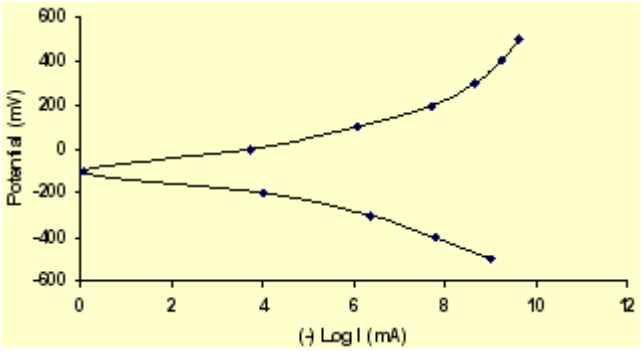

Fig. 4(b): Tafel Plot for 0.006 M in containing AlSe thin film in presence of inhibitor

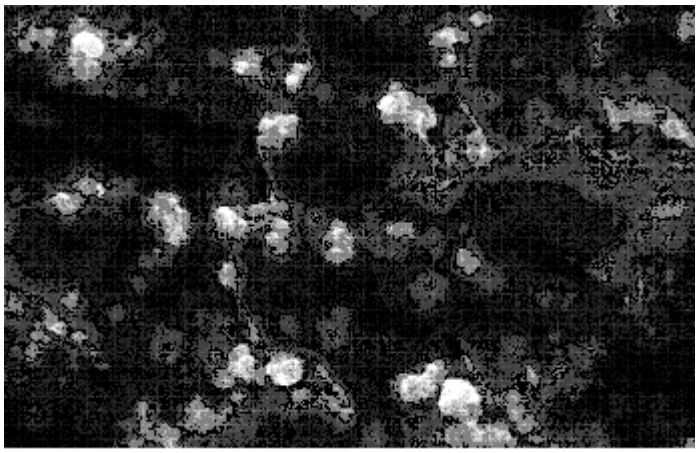

Fig. 5(a): SEM of AlSe thin film

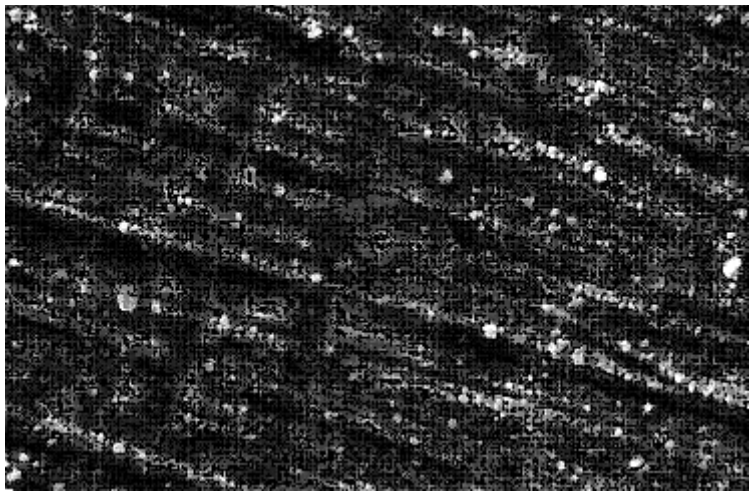

Fig. 5(b): SEM of 0.004 M In containing AlSe thin film

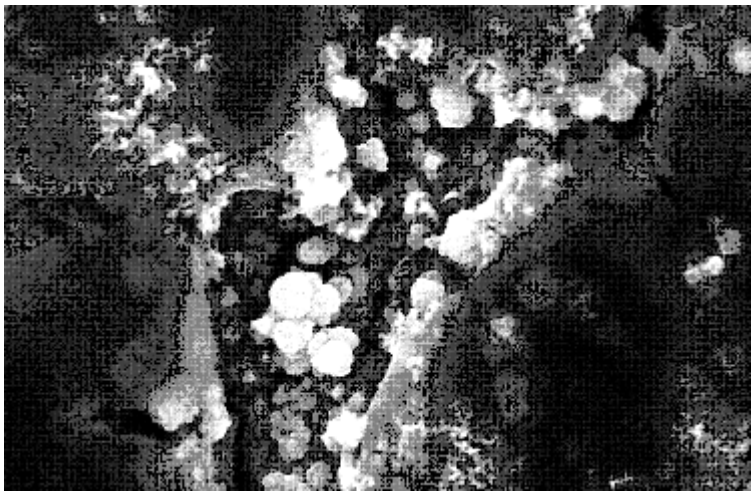

Fig. 5(c): SEM of AlIn thin film

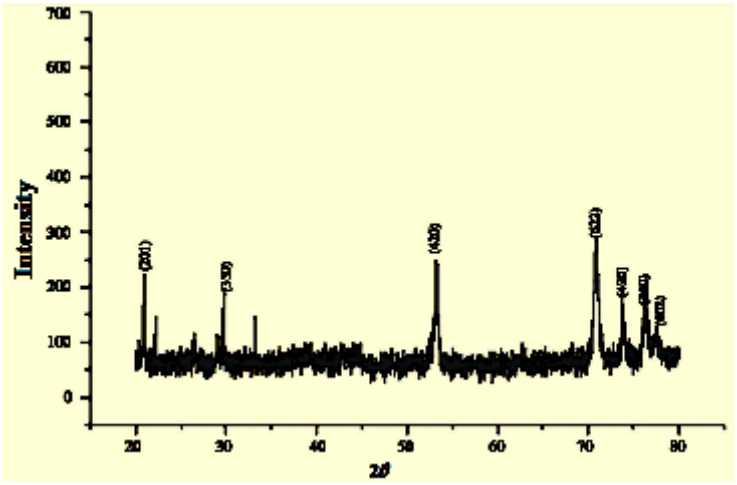

Fig. 6(a): XRD pattern of AlSe

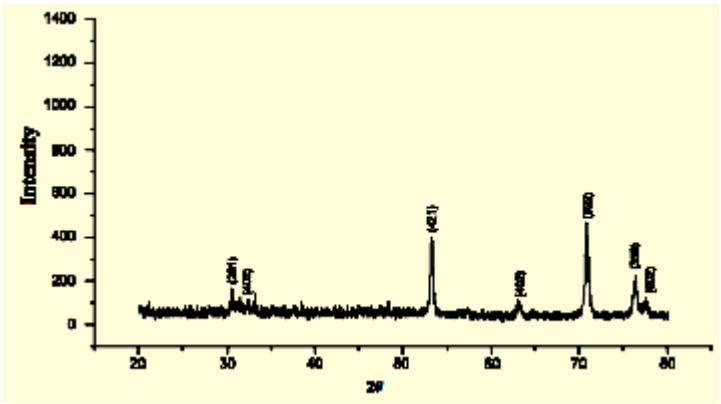

Fig. 6(b): XRD pattern for 0.004 M In containing AlSe thin film

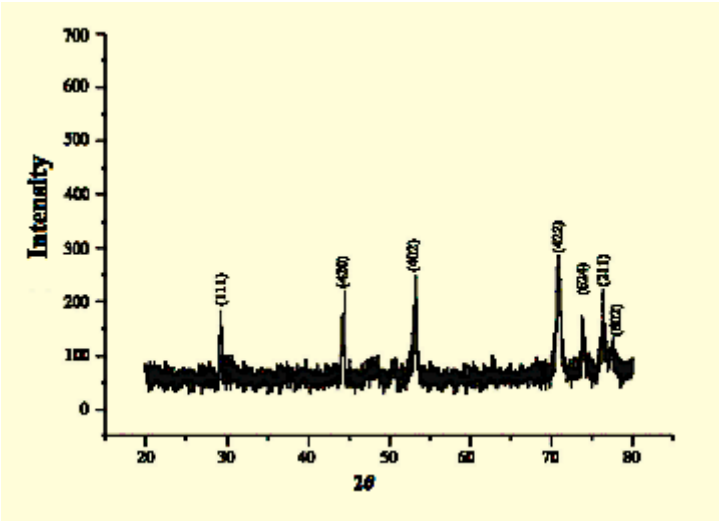

Fig. 6(c): XRD pattern for AlIn thin film

Two Ti plates were used among which one was used as working electrode, another as counter electrode and SCE as reference electrode. All the three electrodes were dipped in electrolyte for some time in order to attain the state of equilibrium. The deposition of electrode was carried out for one hour. The deposited films obtained were then investigated under different parameters. The corrosion characteristics of these deposited films were determined. For this determination, the redox solution $AlK(SO_4)_2$ and KI/I_2 was prepared. The deposited films were dipped in this redox solution to test the corrosion parameters in absence and in presence of inhibitor. Tafel plots were also constructed to determine the Icorr, Vcorr and CR. The capacitance of the films was measured by the LCR meter (systronic). The characterization of these deposited films was done with the help of SEM and the structural features were determined with the help of XRD.

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