Vol 3 Issue 9 Oct 2013

Impact Factor : 0.2105(GISI)

ISSN No : 2230-7850

Monthly Multidisciplinary Research Journal

Indian Streams Research Journal

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RNI MAHMUL/2011/38595 **ISSN No.2230-7850**

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Indian Streams Research Journal
Volume-3, Issue-9, Oct-2013
ISSN 2230-7850

Available online at www.isrj.net

Potentiostatic Electrochemical Preparation And Characterisation Of ZnCdSe

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Abstract: The electrodeposition of ZnCdSe polycrystalline semiconducting thin films from aqueous acidic bath without any additives onto mild steel substrate is described. The growth of ternary Zn/Cd selenides opens up the possibility of their applications for novel opto-electronic devices in the visible region of electromagnetic radiation. Electrodeposition technique is an important technique for effective application of such a valuable system. In this work ZnCdSe are cathodically electrosynthesized from aqueous solution containing Zinc Sulphate, Selenium dioxide and Cadmium Sulphate. Morphological and the structural studies of the electrodeposited material were determined by SEM, XRD while the film composition was determined by energy dispersive x-ray (EDAX) spectroscopy.

Keywords: Electrodeposition, corrosion rate, Film thickness, SEM, XRD and EDAX analysis.

INTRODUCTION

ZnCdSe is an important class of semiconducting materials which find extensive application in novel optoelectronic devices like blue-green lasers, light emitting diodes, and photovoltaic solar cells (1,2).Thin films of this alloy would prove extremely useful in the preparation of image intensifiers, cathode ray screens,thin-film transistors, photovoltaic and photoelectrochemical solar cells (3). it's one dimensional semiconducting nanostructures have attracted interest in device applications(4).In particular nanomaterials based on II-VI compounds are suitable candidates for nanoptics and nanosensoring(5,6). Due to their wide band gap, ZnCdSe films have been extensively studied as materials for lasers and light emitting diodes in the blue-green region of visible spectrum (7, 8).There are several reports on the electrodeposition of ZnSe from aqueous (9, 10) and non aqueous solutions (11), as well as from molten salt solutions (12).Zinc and selenium are codeposited in order to synthesize ternary ZnCdSe alloy (13-14).The elemental analysis of the sample is done by EDAX (energy dispersive analysis).

Materials and Methods: Electrodeposition process has been used in preparing thin film of ZnCdSe ternary alloy. The electrochemical assembly consists of a conventional three-electrode system.In this assembly titanium electrode (1cm × 1cm) served as a counter electrode, saturated calomel electrode (SCE) was used as a reference electrode and the mild steel substrate (1cm × 1cm) was used as a working electrode. The reagents used consist of cadmium sulphate heptahydrate, zinc sulphate heptahydrate and selenium dioxide. All the reagents were of analytical grade. The electroplating solutions were prepared in deionizied water.
Experiment The ZnCdSe thin film was potentiostatically electrodeposited at -0.700V Vs SCE. The deposition of ZnCdSe ternary alloy was carried out for 50 minutes. For data acquisition transistor based power supply unit Model 613 (systronics electronics limited) and multimeters were used as per the circuit requirement for the measurement of current and potential during eletrodeposition. Three different solutions were prepared by taking ZnSO4.7H2O, CdSO4.7H2O and SeO2 and one solution containing ZnSO4.7H2O, SeO2 was prepared. The pH of solution was adjusted between 3.5-4. The temperature of bath was maintained at 40±2oC. All the electrosynthesized samples were tested in 0.1 N HCl, 0.01 N HCl, 0.001 N HCl, 1 % NaCl, 0.01 % NaCl, 0.001 % NaCl, 0.1 N H2SO4, 0.01 N H2SO4 and 0.001 N H2SO4 to acquire the Current-Time data, Current-Voltage data and to construct Tafel graph. The SEM images and EDAX of the electrodeposited samples were obtained by SEM instrument model JEOL JSM 5600 while X ray diffraction pattern of the samples were obtained by X-ray diffractometer D8 Advance Bruker AXS.

RESULTS AND DISCUSSIONS

The present investigation deals with the electrodeposition of ZnCdSe alloy samples and their study on the aspects of corrosion parameters, SEM, XRD and EDAX analysis. The deposition potential of zinc, cadmium and selenium ions are different. Therefore to estimate the deposition potential for the codeposition of these ions, Current-Voltage data is acquired. Fig.1 shows a typical sigmoidal graph which mainly consists of three regions namely-diffusion current region, residual current region and limiting current region. The thin films were deposited for 50 minutes from all the different electroplating solutions having

different concentration of cadmium. The potential was maintained at a constant value of -7.00mv Vs SCE and the value of corresponding current is taken with respect to time. A typical current-time curve is shown is Fig.2. Initially the current value is high but as the deposition progresses on the substrate surface, its surface morphology changes due to which the current value becomes time invariant. The current-time data is used to determine the thickness of the deposited thin film. The thickness of the sample was calculated by using expression

$$\text{Film thickness} = \frac{i \times t \times E_w}{FdA}$$

The area under the current-time curve was used for the calculation of thickness of samples prepared from different electroplating solutions. The thickness of thin film prepared from solution containing Zn(0.1M)+Se(0.01M) and the solution containing Zn(0.1M) +Se(0.01M) +Cd(0.01M) is nearly same while, the thickness of thin film prepared from the solutions containing Zn(0.1M) +Se(0.01M) +Cd(0.05M) and Zn(0.1M) +Se(0.01M) +Cd(0.10M) are comparatively higher. After electrochemical synthesis of the thin film, the samples were tested for their stability in the corroding solutions of HCl, NaCl, and H2SO4 of different concentrations. First cathodic polarisation of the sample is done by sweeping the potential between reference electrode and the working electrode meanwhile the corresponding readings of the current was noted. Similarly, after changing the leads of the multimeters anodic polarisation was done by sweeping the potential in the reverse direction. The corresponding current and potential readings were again noted down. With the help of the data obtained from cathodic and anodic polarisation Tafel plot was constructed. A typical Tafel curves is shown in Fig .3

The corrosion rate (milli inches per year) of deposited the thin film is determined by the expression given below.

$$CR\ (mpy) = \frac{0.13 \times I_{corr} \times E_w}{F \times d \times A}$$

Where,

CR= corrosion rate in milli inches per year

I_{corr}= corrosion current

F= Faraday's constant

A= area of cross section of the working electrode

d= density of alloy

The calculated values of corrosion rate (Table 1.) indicates that there is a decrease in the corrosion rate as well as corrosion current on increasing the dilution of the corroding solutions viz., HCl, H2SO4, NaCl respectively. This also shows that corrosion decreases with the increasing dilution of the corrosive environment. The SEM images of the sample show cauliflower like structure of the deposited particle (Fig.5-6). There is an increment in the bulk of particle being deposited on the mild steel substrate surface along with increase in cadmium ion concentration in the electroplating solutions. The EDAX of the sample are shown in fig.8. The peaks obtained in the EDAX indicate the inclusion of zinc, cadmium and selenium in the deposited thin film. From EDAX analysis atomic percentage of deposited elements is obtained. The percentage of cadmium in the electroplating solution is plotted against the percentage of cadmium deposited in the thin film. This graph indicates increase in the percentage of deposited cadmium with the increase in its concentration in the electroplating solutions (Fig .9).

The XRD patterns of all the samples were indexed successfully to determine the crystal structure, grain size and surface stress in the sample. The peaks obtained in the XRD pattern were compared with the reference pattern in the JCPS card standard peaks. The crystal structure was found to be Hexagonal close Packing (HCP). XRD of the sample (Fig.4) indicates the polycrystalline nature of the thin film. The grain size is determined by scherer equation, which was found to lie between 10-100nm.

Williamson's Hall plot is obtained by plotting $4\sin\theta$ Vs $\beta\cos\theta$. The slope and intercept of the curve gives crystallite size and strain in the deposited samples (Fig.7). The negative slopes represent compressive strain while the positive slopes indicate extensive strain in the sample.

Table1. Testing of electrodeposited thin fims in experimental solutions of different Concentrations

Electroplating solution	Experimental solution H_2SO_4	Corrosion Rate (in 10^{-3} mpy)	Experimental solution HCl	Corrosion rate (in 10^{-3} mpy)	Experimental solution NaCl	Corrosion rate (in 10^{-3} mpy)
Zn(0.1M) + Se(0.01M)+ Cd(0.01M)	0.1N H_2SO_4	7.5	0.1 N HCl	9.0	1% NaCl	8.2
	0.01N H_2SO_4	3.2	0.01N HCl	5.8	0.01% NaCl	3.1
	0.001N H_2SO_4	1.6	0.001N HCl	0.27	0.001% NaCl	2.2
Zn(0.1M) + Se(0.01M)+ Cd(0.05M)	0.1N H_2SO_4	0.43	0.1 N HCl	0.29	1% NaCl	0.8
	0.01N H_2SO_4	0.04	0.01N HCl	0.45	0.01% NaCl	0.6
	0.001N H_2SO_4	0.08	0.001N HCl	0.13	0.001% NaCl	0.2
Zn(0.1M) + Se(0.01M)+ Cd(0.10M)	0.1N H_2SO_4	0.15	0.1 N HCl	0.3	1% NaCl	0.2
	0.01N H_2SO_4	0.113	0.01N HCl	0.14	0.01% NaCl	0.1
	0.001N H_2SO_4	0.119	0.001N HCl	0.12	0.001% NaCl	0.15
Zn(0.1M) + Se(0.01M)	0.1N H_2SO_4	1.47	0.1 N HCl	1.58	1% NaCl	1.34
	0.01N H_2SO_4	0.98	0.01N HCl	1.05	0.01% NaCl	0.54
	0.001N H_2SO_4	0.33	0.001N HCl	0.38	0.001% NaCl	0.18

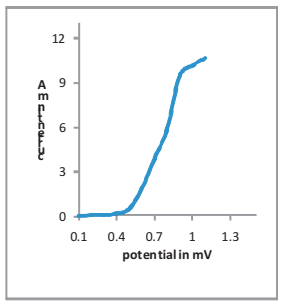

Fig.1 Current **Vs** Potential

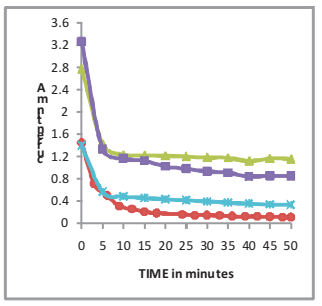

Fig.2 Current **Vs** Time

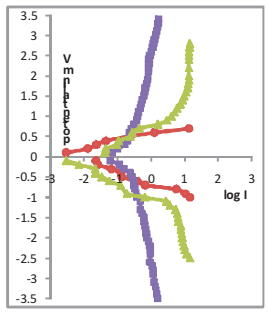

Fig.3 Superimposition of typical tafel graphs

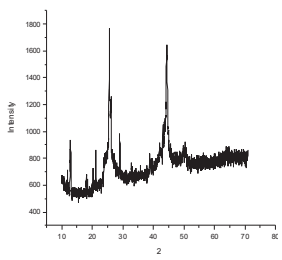

Fig.4 XRD of one of the sample

Fig.5 SEM image of ZnCdSe sample

Fig.6 SEM image of ZnSe sample

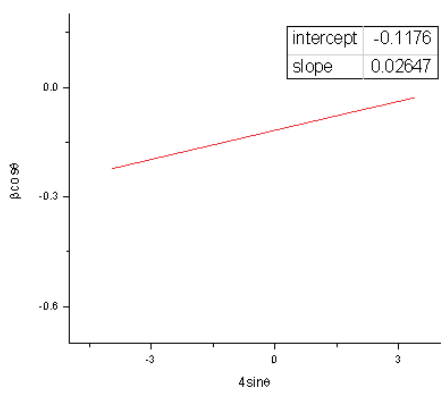

Fig.7 Williamson Hall plot

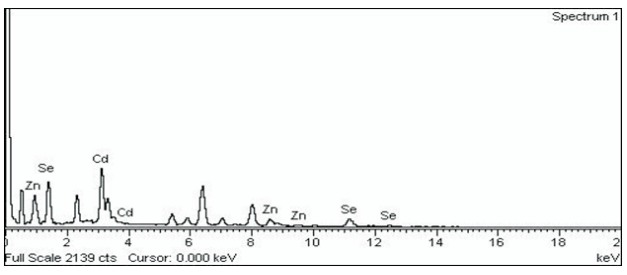

Fig 8. EDAX of one of the prepared sample

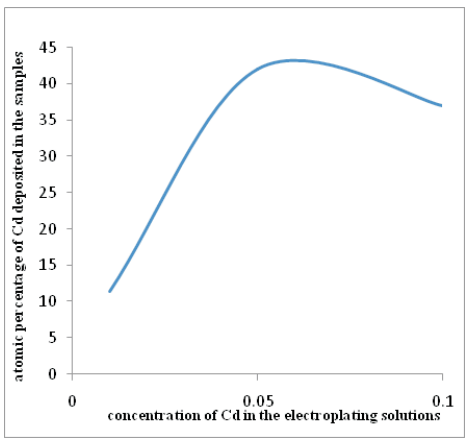

Fig.9 Atomic % of Cd in thin film Vs Cd ion concentration in electroplating solution

CONCLUSION

On the basis of current-voltage data derived from the cathodic and anodic polarisation tafel plot were constructed to determine the corrosion rate. The corrosion rate of the sample was found to decrease with the increase in the concentration of cadmium in the electroplating solution.The corrosion rate was also found to decrease with the increasing dilution of corroding solutions i.e., HCl, H2SO4 & NaCl solutions. Atomic percentage of eletrodeposited cadmium was found to increase with the increase in the concentration of cadmium in the electroplating solution. The deposition of ZnCdSe thin films is confirmed by XRD & EDAX of the samples. The XRD pattern recorded shows that the thin films are polycrystalline in nature with a (HCP) hexagonal close packing. Morphological and structural details of the sample were studied by scanning electron microscope (SEM). SEM images shows cauliflower like structure and the deposited thin film were found to be uniform. The grain was calculated which lies between 10-100nm.

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