Corrosion and Structural studies of Galvanostatically Electrodeposited ZnMgO Thin Films

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Abstract: ZnMgO thin films have been grown on Stainless steel substrate by galvanostatic electrodeposition from nitrate baths. The effect of bath composition was observed. A comparative study of corrosion parameters were done in three different electrolytes; NaCl, HCl, H_2SO_4 by tafel plot. 1-Phenyl-1H-tetrazole-5-thiol was used as inhibitor in deposition bath and its effect on corrosion rate was determined and found effective in corrosion inhibition of ZnMgO thin films. The morphology and structural characterization of thin films were done by Scanning Electron Microscopy (SEM) and X-Ray Diffraction pattern (XRD). Elemental composition has been determined by Energy Dispersive X-Ray spectroscopy (EDAX). XRD confirmed that the films possessed Hexagonal wurtzite structure. Structural changes were also observed by XRD studies which indicate a possibility of partial replacement of Zn atoms by Mg atoms in the lattice structure.

Keywords: Galvanostatic, Thin films, corrosion rate, inhibitor, SEM, XRD

1. Introduction

ZnO thin films have attracted great research interest in recent years due to its excellent electrical, electronics and optical properties. ZnO is a multifunctional material. It has a wide direct band gap (3.36 eV) and a large excitonic binding energy(60 meV) due to which it is used in many areas like light emitting diode [1], laser diode [2], gas sensor [3], [4] and solar cells[5]-[7]. Additional advantage of ZnO is, it is a bio safe and bio compatible material [8], [9]. ZnO thin films are also used in thin film transistors, uv- photodetector [10], wear resistance films [11] etc. Corrosion behaviour of ZnO and their composite films deposited on different substrates like steel, aluminium and brass were studied in different electrolytes [12]-[14]. Depending on the application the properties of ZnO thin films can be altered by alloying with another materials such as Cadmium (Cd), Iron (Fe), Nickel (Ni), Manganese (Mn), Magnesium (Mg) etc

Manganese doped ZnO were monitored as dilute magnetic semiconductor (DMS) which exhibit room temperature ferromagnetism [15]. Shanmugans et al [16] prepared Mg doped ZnO nanoparticles to analyse their structural properties. ZnO and MgO has lattice mismatch. ZnO is wurtzite while MgO is cubic but the ionic radii of Magnesium and Zinc are relatively similar so Zn^{2+} can be replaced by Mg²⁺ [17]. Fe doped ZnO thin films have been fabricated from different fabrication methods and most of the researches mainly focused on their ferromagnetic behaviour [18]-[20]. Structural and optical properties of ZnO thin films and their relative materials were also analysed by researchers [21]-[23].

Various physical and chemical methods have been used for thin film deposition such as Sol-Gel method [24], thermal evaporation [25], Radio frequency(RF) magnetron sputtering [26], Pulse laser deposition [27], Chemical bath deposition [28], electrodeposition [29], [30] etc. Despite the other techniques Electrodeposition is a promising technique for the preparation of alloy thin films from aqueous solution due to its simplicity and cost effectiveness. This technique doesn't need vacuum system or toxic gases. Electrodeposition process can be carried out on any conductive substrate like glass, polymers, metals, templates etc.

A corrosion inhibitor is a substance when added in a small concentration to an environment reduces the corrosion rate of a metal or alloy exposed to that environment. Organic compounds containing Nitrogen, Oxygen and Sulphur reduces the corrosion rate. Inhibitors act by selectively precipitating on cathodic areas to limit the diffusion of reducing species to the surface.

In present work ZnMgO films were electrodeposited from nitrate baths and their corrosion behaviour was tested in presence and absence of 1-Phenyl-1H-tetrazole-5-thiol used as inhibitor.



1-Phenyl-1H-tetrazole-5-thiol

2. Experimental Procedure

The composition of electroplating baths used for synthesis of ZnMgO thin films were 0.05 M Zn(NO₃)₂, 0.05-0.20 M Mg(NO₃)₂, 0.01 M NaNO₃, 0.01 M KCl. All chemicals were of A.R. grade and deposition solutions have been prepared in distilled water. All electrochemical experiments were carried out in a conventional three electrode cell without stirring. All potentials were referred to the saturated calomel electrode (SCE). Stainless steel 202 (SS 202) plates with surface area 1 cm^2 were used as working as well as counter electrode. SS 202 plates were cleaned with emery paper and washed successively with acetone and distilled water. For data

acquisition transistor based power supply and for current density and potential measurements digital multimeters were used during electrochemical co-deposition of alloy thin films. The pH of deposition bath was maintained 4 using dilute HNO₃ and thin film depositions were carried out at $55^{\circ}C \pm 1^{\circ}C$.

Four electrolytic solutions were prepared by taking fix concentration of other chemicals with different concentration of $Mg(NO_3)_2$. 1-Phenyl-1H-tetrazole-5-thiol was also added as inhibitor to these solutions and their effect on deposited films were studied.

The electrodeposition was done using galvanostatic method and the potential-time dependence for the deposition of ZnMgO alloy thin films on stainless steel substrate obtained at constant current density 5 mA/cm² for 10 minutes at 55°C \pm 1°C.

The film thickness was estimated using relationship (1)

F.T.=
$$(i *t * eq.wt.) / (F*D*A)$$
 (1)

Here,

F.T. = film thickness

- Eq.wt. = equivalent weight of deposited alloy
- I = current density (μ A/cm²)

t = time (second)

F = faraday 's constant

D = density of alloy (gm/cm3)

A = Area of substrate(cm²)

The corrosion parameters of deposited alloy thin films were determined by Tafel Polarization curve. The corrosion rates were calculated using equation (2)

$$C.R. = (0.13 * I_{corr} * eq.wt.) / (D_* A)$$
 (2)

Here,

C.R. = corrosion rate (MPY)

 $I_{corr} = corrosion current density (\mu A/cm²)$

Eq.wt.= equivalent weight of deposited alloy

D= density of alloy (gm/cm3)

A= Area of substrate (cm^2)

The morphology and composition of thin films were investigated by FModel: JEOL JSM 5600 Scanning electron microscope (SEM) and EDS model: INCA Oxford. Crystalline phase of the deposited films were characterised by Bruker D8 Advance X-Ray Diffractometer (XRD) with a monochromatized CuK α irradiation ($\lambda = 0.154$ nm).

3. Result and Discussion

ZnMgO thin films were obtained from different deposition baths at constant current density of 5 mA/cm² for 10 minutes. It was observed that at higher Mg concentration in the deposition bath, Mg was alloyed with ZnO to form ZnMgO. KCl was added in the bath to increase the specific conductance and secondary current distribution.

In order to obtain the suitable current for deposition first of all, the deposition was performed at different current densities i.e. 1mA, 2mA, 3mA, 4mA, 5mA and 6mA respectively. A smooth surface and corrosion rate was observed at 5 mA/cm², then all deposition experiments were carried out on this current density. The variation in potential

with time was noted. Figure 1 shows the variation in potential with time at fixed current density 5mA/cm2.





During the deposition potential first increases and then it decreases very fast up to a steady state value. The steady state indicates that the coverage of working electrode surface is nearly complete. The estimated film thickness of ZnMgO thin films is 2-2.5 μ m.

3.1 Corrosion studies

The corrosion behaviour of electrodeposited thin films on SS202 substrate from different deposition baths were studied using Tafel polarization method. The polarization studies were made by cathodic and anodic polarization data shown in figure 2(a-d).



Figure 2(a): Tafel plot for 0.05M Zn-0.05M Mg alloy thin Film



Figure 2(b): Tafel plot for 0.05M Zn-0.20M Mg alloy thin Film

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Figure 2(c): Tafel plot for 0.05M Zn-0.05M Mg alloy thin Film in presence of inhibitor

The alloy thin films were tested in three different corrosive mediums i.e. HCl, H_2SO_4 and NaCl at three different concentrations. Table 1,2 and 3 show the corrosion parameters of deposited films in different corrosive mediums.



Figure 2(d): Tafel plot for 0.05M Zn-0.20M Mg alloy thin Film in presence of inhibitor

It is observed that film having lower Mg concentration gives better corrosion protection. As the Mg concentration decreases in the deposition solution corrosion rate also decreases. Addition of 1-Phenyl-1H-tetrazole-5-thiol as inhibitor is also beneficial for corrosion protection. After addition of inhibitor, corrosion rate of ZnMgO alloy thin films decreases and it is much lower than ZnMgO alloy thin films synthesized in absence of inhibitor.

Fable 1: Corrosion Rate	of ZnMgO	thin films in	n NaCl
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Concen	Concentration in						
electroplat	ing solution	on					
			0.1 MNaCl		0.01 M NaCl		0.001 M NaCl
Conc. Of	Conc. Of	Without	With inhibitor (0.0005	Without	With inhibitor (0.0005	Without	With inhibitor (0.0005 M
$Zn(NO_3)_2$	$Mg(NO_3)_2$	inhibitor	M 1-Phenyl-1H-	inhibitor M 1-Phenyl-1H-		inhibitor	1-Phenyl-1H-tetrazole-5-
			tetrazole-5-thiol)		tetrazole-5-thiol)		thiol)
0.05 M	0.05 M	0.1188	0.1009	0.1028	0.0801	0.0473	0.0301
0.05 M	0.10 M	0.1688	0.1148	0.1657	0.1023	0.0572	0.0342
0.05 M	0.15 M	0.1681	0.1586	0.1498	0.1414	0.0689	0.0422
0.05 M	0.20 M	0.3313	0.2760	0.1974	0.1552	0.0742	0.0413

Table 2: Corrosion Rate	of	ZnMgO	thin	films	in	H_2SC)4
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Concent	ration in	Corrosion Rate (mm/year)					
electroplati	electroplating solution						
		$0.1 M H_2 SO_4$		$0.01 M H_2 SO_4$		$0.001 M H_2 SO_4$	
Conc. Of	Conc. Of	Without	With inhibitor	Without	With inhibitor (0.0005 M	Without	With inhibitor (0.0005 M
$Zn(NO_3)_2$	$Mg(NO_3)_2$	inhibitor	(0.0005 M 1-Phenyl-	inhibitor	inhibitor 1-Phenyl-1H-tetrazole-		1-Phenyl-1H-tetrazole-5-
			1H-tetrazole-5-thiol)		5-thiol)		thiol)
0.05 M	0.05 M	0.4591	0.1139	0.3862	0.0899	0.1157	0.0636
0.05 M	0.10 M	0.4543	0.1621	0.4162	0.1288	0.1317	0.0861
0.05 M	0.15 M	0.5315	0.5016	0.3976	0.2820	0.1587	0.1061
0.05 M	0.20 M	0.6241	0.4637	0.4418	0.3145	0.1863	0.1547

Table 3: Corrosion Rate of ZnMgO thin films in HCl

Concentr	ation in	Corrosion Rate (mm/year)						
electroplating solution		0.1 M HCl			0.01 M HCl		0.001 M HCl	
$Zn(NO_3)_2$	$Mg(NO_3)_2$	Without	With inhibitor (0.0005 M	Without With inhibitor (0.0005		Without	With inhibitor (0.0005	
		inhibitor	1-Phenyl-1H-tetrazole-5-	inhibitor M 1-Phenyl-1H-		inhibitor	M 1-Phenyl-1H-	
			thiol)		tetrazole-5-thiol)		tetrazole-5-thiol)	
0.05 M	0.05 M	1.5374	1.0691	0.4334	0.3191	0.1222	0.0801	
0.05 M	0.10 M	1.8596	0.9660	0.5240	0.3055	0.1476	0.1023	
0.05 M	0.15 M	2.6635	1.6807	0.6894	0.5314	0.1681	0.1497	
0.05 M	0.20 M	3.1282	2.0699	0.9342	0.5835	0.2485	0.1845	

3.2 Morphology and compositional studies

Figure 3(a-d) show the Scanning electron microscopy (SEM) images. The images show that films are continuous but rough and nonhomogenous in nature. The particles are irregular in shape.



Figure3(a): SEM of 0.05MZn-0.05M Mg alloy thin film



Figure 3(b): SEM of 0.05M Zn-0.20M Mg alloy thin film



Figure 3(c): SEM of 0.05MZn-0.05 M Mg alloy thin film in Presence of inhibitor



Figure 3(d): SEM of 0.05MZn-0.20M Mg alloy thin film in Presence of inhibitor

In Figure 3(b) rod like particles are clearly visible. Addition of Mg increases the roughness of surface and leads to the particle aggregation. Addition of inhibitor leads more dense films.

EDAX analysis shows the percentage composition and atomic percentage of ZnMgO alloy thin films. EDAX analysis (figure 4) confirmed the presence of Zn, Mg and O elements in the films. 3-4% of Mg incorporation in ZnMgO thin films was seen in EDAX analysis.



Figure 4(a): EDAX of 0.05M Zn-0.20M Mg alloy thin film



Figure 4(b): EDAX of 0.05MZn-0.20M Mg alloy thin film in presence of inhibitor

3.3 Structural analysis

Figure 5(a,b) shows the X-ray diffraction patterns obtained for ZnMgO thin films electro synthesized from different deposition baths.







Figure 5(b): XRD of 0.05M Zn-0.20M Mg alloy thin film in Presence of inhibitor

All the diffraction lines in diffraction pattern of ZnMgO were compared with the reference pattern JCPDS card standard peaks. The studies show ZnO like peaks of wurtzite structure. Few additional peaks were also seen in diffraction pattern which indicates partial structural changes this may be due to replacement of Zn atoms by Mg atoms in the lattice

Volume 6 Issue 4, April 2017 <u>www.ijsr.net</u> Licensed Under Creative Commons Attribution CC BY structure. The crystallite size is determined by Scherrer equation (eq.3) which was found to be less than 50 nm. Crystallite size

$$D = \frac{k \lambda}{\beta \cos \theta} \tag{3}$$

 λ = wavelength of incident beam (nm)

k= scherrer's constant

 β = peak broadening (FWHM)

 θ = scattering angle in radian

The dislocation density of ZnMgO thin films was calculated with the help of equation 4.

$$\delta = 1/D \tag{4}$$

here,

 δ = dislocation density D=crystallite size

The crystallite size and dislocation density of electrodeposited ZnMgO thin films is shown in table 4 for 002 planes. The micro strain in the deposited films was determined by Williamson Hall plot (figure 6)which indicates an decrease in strain with increasing Magnesium concentration in deposition bath in absence of inhibitor while reverse is observed in presence of inhibitor.



Figure 6(a): Williamson Hall plot for 0.05M Zn-0.20 M Mg alloy thin film



Figure 6(b): Williamson Hall plot for 0.05M Zn-0.20 M Mg alloy thin film in presence of inhibitor

S. No.	Concent	tration in el	ectroplating solution	Interplaner	FWHM	Crystallite	Dislocation	Strain
	$Zn(NO_3)_2$	$Mg(NO_3)_2$	inhibitor (1-Phenyl-	spacing (d)	(β)	size (nm)	density (nm)	<i>є х 10⁻³</i>
			1H-tetrazole-5-thiol)	<i>(nm)</i>				
1	0.05 M	0.05 M	-	0.2612	0.2626	31.6518	0.0316	3.57
2	0.05 M	0.10 M	-	0.2609	0.2459	33.8195	0.0296	3.53
3	0.05 M	0.15 M	-	0.2609	0.2492	33.3608	0.0300	0.74
4	0.05 M	0.20 M	-	0.2615	0.2937	28.3500	0.0353	0.77
5	0.05 M	0.05 M	0.0005 M	0.2608	0.2373	35.0486	0.0285	1.50
6	0.05 M	0.10 M	0.0005 M	0.2611	0.2347	35.4273	0.0282	1.60
7	0.05 M	0.15 M	0.0005 M	0.2609	0.2752	30.2106	0.0331	3.29
8	0.05 M	0.20 M	0.0005 M	0.2607	0.2380	34.9473	0.0286	3.51

Table 3:	Xrd	parameters	of Zn	MgO	thin	films
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4. Conclusion

The oxide of Zinc and Magnesium was obtained by keeping temperature higher than 50°C. The corrosion rate of samples in all three corroding solutions decreased while decreasing the concentration of Magnesium in deposition bath. SEM showed the non homogenous and rough development of alloy thin films on substrate. EDAX confirmed the percentage of Zinc , Magnesium and Oxygen in deposited films. It is found that Magnesium incorporated in films when added to the deposition bath in higher concentration. The XRD analysis shows that the thin films are polycrystalline in nature having hexagonal wurtzite structure. Structural changes were also seen in few samples. This may be due to replacement of some Zn atoms by Mg atoms in crystal lattice. The grain size in deposited films was less than 50 nm.

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