



Electrosynthesis of Y-S Films by Electrodeposition Method

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ABSTRACT

Electrosynthesis of Y-S films on the variety of substrates using non-aqueous bath has been studied. The range of deposition potential and substrate type was decided by plotting polarization curves. The effect of concentration of sulphur ions and temperature on deposition potential on stainless steel substrates has been studied. Ohmic contact between yttrium film and stainless steel was confirmed by I-V characteristics. The variation of terminal thickness with time has been studied. The films were characterized by optical absorption and XRD studies.

Keywords: Electrosynthesis, polarization curves, substrates, complexed bath, terminal thickness, ohmic nature.

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INTRODUCTION:

High-tech applications of rare earth metals with their unique properties are reported by some researchers [1, 2]. Electrodepositions of some of the rare earth metal chalcogenides from aqueous as well as non-aqueous media have been reported by some workers. *Le Tacon and et al* has been used the multisource evaporator system for the preparation of thin films of rare earth chalcogenides [3]. Some field compounds have found to be high field technology applications [4-6]. Superconducting and ferromagnetic states are observed in rare earth compounds, such work was published by *Umbach* in 1982 [7]. Many researchers reported the rare earth films and their characterization by various methods, especially by electrodeposition method [8,9]. In this paper we report on the electrosynthesis of yttrium sulphide from non-aqueous bath. Sodium acetate was used as a complexing agent and thioacetamide used as a sulphur ion source. Using polarization curves the deposition potentials were estimated. The electrodeposition was tested and studied on variety of substrates, while stainless steel substrate was used for final deposition. The concentration of the complexing agent was varied from 0.1M to 1.0M. Films are characterized by optical absorption and X-ray diffraction pattern.

MATERIAL AND METHODS

The electrodepositing cell consists of bakelite holder and solution container. Bakelite holder is used to hold counter electrode and substrate at appropriate distance. There is additional provision to bakelite holder to insert calomel electrode inside the bath. The molar solution of yttrium nitrate was prepared in formaldehyde. Substrates were sequentially cleaned by using zero grade polish paper, detergent powder, ultrasonic cleaner etc. While conducting glass substrates are specially cleaned by using dilute Hydrochloric acid and doubly distilled water. It was found that bath 0.05M Y (NO₃)₃ containing 0.05M CH₃CSNH₂ and 0.05M CH₃COONa composition was suitable for electrodeposition of yttrium sulphide from formaldehyde bath. The suitable deposition temperature range was 25°C to 50°C. The film thickness was found in between 4 to 5 μm. The characterization of film is carried out by XRD and Optical absorption. The X-ray diffraction studies showed that the films are polycrystalline and composition corresponding to YS_{1.71-1.76}. The band gap was calculated to be 1.85eV by optical absorption method.

RESULTS AND DISCUSSION

Fig.1 shows the polarization curves on variety of substrates for the reduction of Y-S. The nature of polarization curves were found to be sharp, hence it is concluded that the electrodeposition is possible at single outset potential. The deposition potentials are estimated from polarization curves for different substrates. These potentials are near about the individual potentials of yttrium and sulphur on to a stainless steel substrate. Thus deposition was taken on stainless steel substrate. The exact concentration

of thioacetamide i.e. sulphur ion was confirmed by plotting polarization curves for different concentrations on to a stainless steel substrates for desired deposition potential. As per *William Blum et al and Satpathy K. C. et al* work, free ion concentration of chalcogenides affects cathodic polarization [10, 11]. Fig. 2 shows the polarization curves for various sulphur ion concentrations on to a stainless-steel substrate. Table 1 is the estimated deposition potentials for various sulphur ion concentrations of sulphur ion source on to a stainless steel substrate with and without sodium acetate bath, shows the effect of concentration of sulphur ions on cathodic polarization. Fig.3 shows the polarization curves at various temperatures onto stainless steel substrates. Estimated deposition potentials for optimized bath at various temperatures in complexed bath are shown in Table 2. It was seen that dissociation of complex decreases the cathodic polarization due to increase in temperature.

The temperature is an important parameter in the deposition process. Anomalous effect of temperature on atomic layer deposition of titanium dioxide was reported by *Jaan Aarik and et al* [12]. *Jundale and Lokhande* have been reported the effect of temperature on deposition potentials of some rare earth chalcogenides in aqueous bath [13]. Similar effect was observed for the reduction of yttrium-sulphide in non-aqueous bath. Thickness- time variation on to a FTO coated glass was studied and shown in fig. 4. It increases with time initially up to $4.0 \mu\text{m}$, in first twenty minutes and further it decreases up to $2.5 \mu\text{m}$, in last 20 minutes.

Fig.5 is the I-V characteristics between Y-S and stainless steel in reverse and forward biased current, shows the ohmic contact between -0.4 to $+0.4$ volt.

Optical property:

Optical absorption was studied in the range of 400 to 1,000 nm for the yttrium sulphide films deposited on to a FTO coated glass at room temperature. Fig. 6 shows the variation of optical density with wavelength. The optical density starts decreasing with increase in wavelength. Fig.7 shows the plot of $(\alpha t h \nu^2)$ versus $(h\nu)$, from this band gap 'Eg' of the Y-S film is estimated. The estimated value of the band gap energy is 1.85eV.

Structural Property:

Films deposited on to a stainless steel of different concentration of sulphur ions are used to study structural properties of yttrium-sulphide substrates. 0.05M concentration of thioacetamide was found to be suitable for the deposition of Y-S films. Fig. 8 shows X-ray diffraction pattern of the same, which confirms the deposited films have the yttrium-sulphide composition. Table 3 shows comparative data for 'd' values of Y-S compound. It was confirmed that the material deposited is to be $\text{YS}_{1.72-1.78}$ [15].

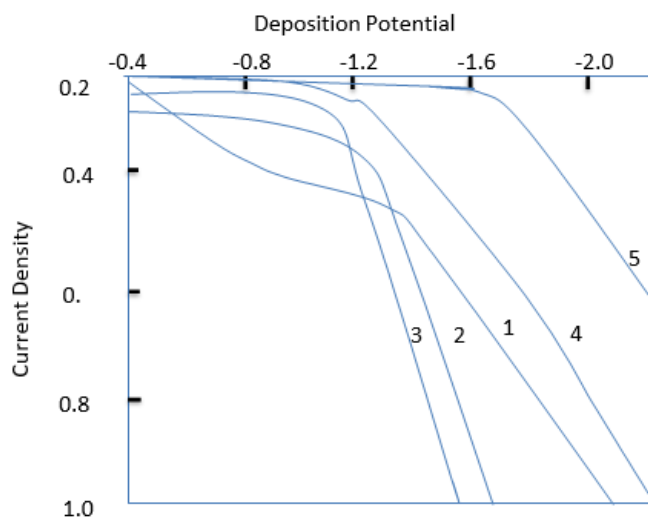


Fig.1: Polarization curves for the reduction of the sulphur onto different substrates.

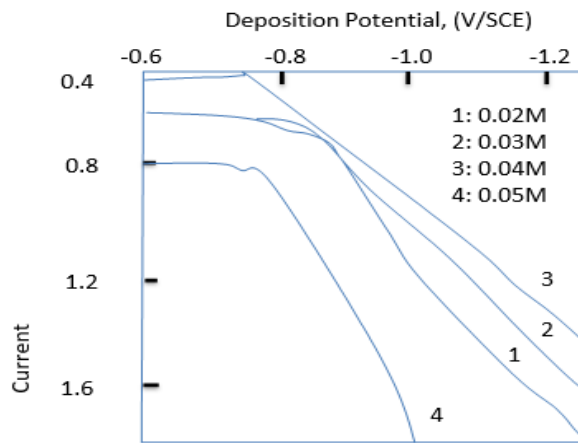


Fig. 2: The polarization curves for various sulphur ion concentrations on to a stainless-steel substrate.

Table 1: Estimated deposition potentials for various concentrations of sulphur ion source.

Sulphur ion concentrations	MCH ₃ CSNH ₂	Deposition potentials without with	
		sodium acetate	sodium acetate
0.02	MCH ₃ CSNH ₂	- 0.80	- 0.80
0.03	MCH ₃ CSNH ₂	- 0.80	- 0.85
0.04	MCH ₃ CSNH ₂	- 0.79	- 0.82
0.05	MCH ₃ CSNH ₂	- 0.78	- 0.75

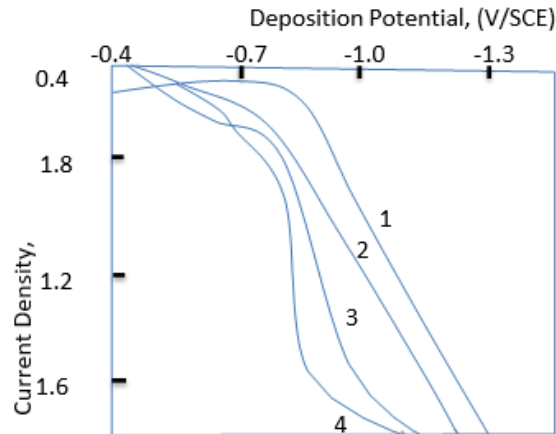


Fig. 3: Polarization curves at 1) 27°C, 2) 35°C, 3) 45°C and 55°C

Table 2: Estimated deposition potentials for optimized bath at various temperatures in a complexed bath.

Bath composition	Temperatures in			0 _C
	27	35	45	55
0.05M Y(NO ₃) ₃ - 0.05M CH ₃ CSNH ₂ - 0.05MCH ₃ COONa	- 0.75	- 0.73	- 0.72	- 0.70

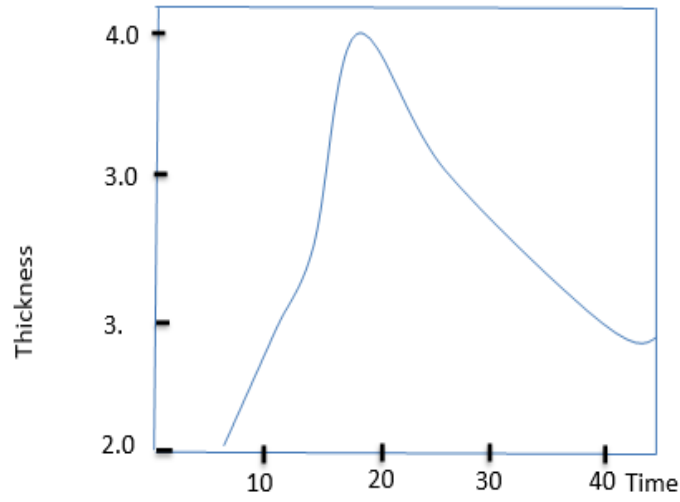


Fig. 4: Thickness variation of Y-S film deposited on FTO coated glass.

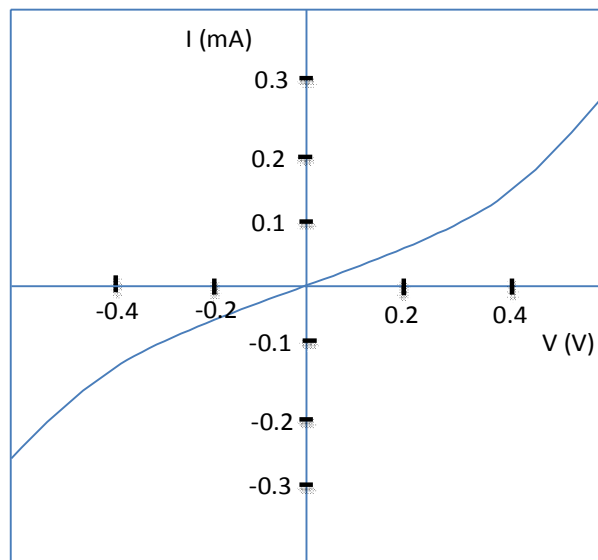


Fig. 5: I-V characteristics between Y-S and stainless steel.

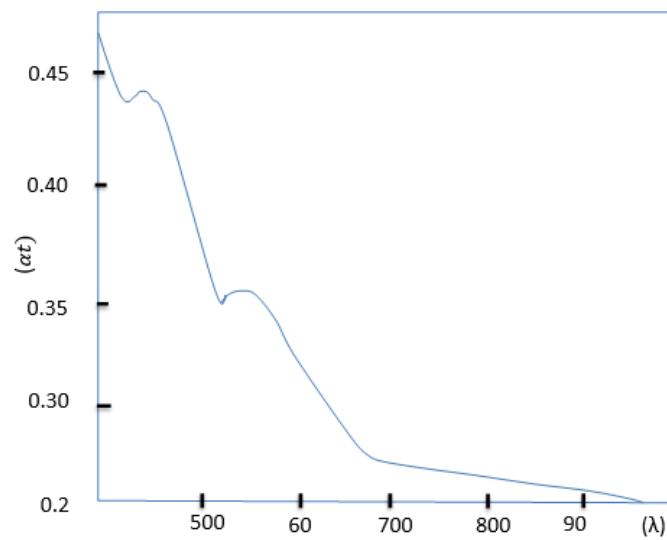


Fig. 6: Optical density (αt) versus wavelength (λ) spectra of Y-S film.

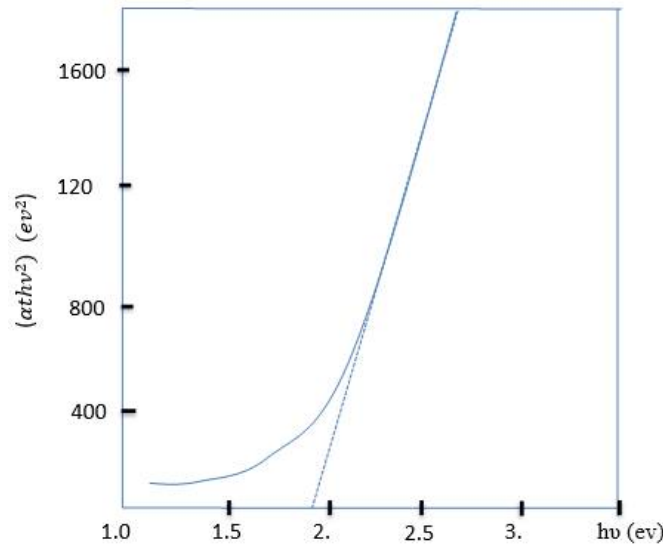


Fig. 7: Graph of (αhv^2) versus (hv) for Y-S Films

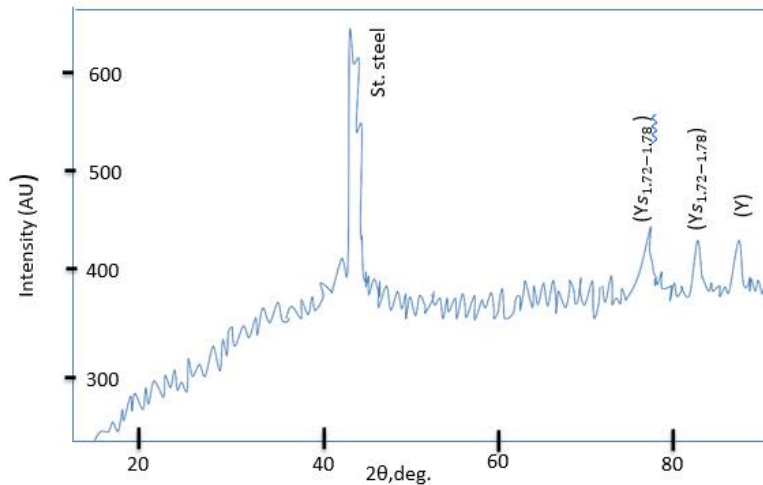


Fig. 8: XRD pattern of Y-S film deposited onto stainless substrate

Table 3: Observed and standard 'd' values of Y-S films.

Bath Composition	Observed 'd' values	Standard 'd' values
0.05M $Y(NO_3)_3$ - 0.05M CH_3CSNH_2 - 0.05M	1.27	1.26
CH_3COONa .	1.09	1.07

CONCLUSIONS

Our study shows that yttrium sulphide films can be deposited on stainlesssteel substrate and ITO coated glass. The optimized bath composition is 0.05M $Y(NO_3)_3$ – 0.05M CH_3CSNH_2 – 0.05M CH_3COONa in temperature range 27°C – 55°C. The band gap energy 'Eg' of the yttrium-sulphide is estimated to be 1.85eV and composition of the film is $YS_{1.72-1.78}$.

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