

# Influence of molar concentration on triethanolamine (TEA) added tin sulfide (SnS) thin films by SILAR method

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Abstract In the present work, thin films of Tin Sulphide (SnS) with TEA are deposited on glass substrate at room temperature by successive ionic layer adsorption and reaction method for solar cell application. The structural, topographical, morphalogical and optical properties of triethanolamine doped tin sulphide thin films are analyzed by X-ray diffraction, SEM, AFM, FTIR and UV-Vis analysis. The X-ray diffraction studies confirm that the deposited SnS films are amorphous and polycrystalline structure. From the SEM micrographs, it is clear that the film possesses almost smooth surface although some particles have distributed on the film because of the increasing concentration. The AFM image indicates that these coatings cover the surface of the substrate completely. A well ordered stair case structure of the SnS thin films are spread over the surface. Various functional groups present in the developed thin film are investigated by FT-IR analysis. The band gap energy decreases with increase in molar concentration as observed by UV-Vis studies and also it shows that the band gap decreases with the increasing particle size.

# 1 Introduction

Thin film coatings are widely used in photovoltaic devices [1] for its reduced size, simple preparation methodology and cost effectiveness. Among the various binary and

P. Mani maniprahaspathy@gmail.com ternary materials. Tin sulfide is having appreciable electrical and optical properties [2], such as large band gap and transparency in the visible region [3]. It can be used as an absorber layer for solar cells [4, 5] etc. Tin sulfide (SnS) is the most interesting material due to its high potential in device fabrication [6]. SnS is having high conversion efficiency ( $\sim 25$  %) which is obtainable in photovoltaic devices and its low cost, availability, toxicity and stability ascribe to SnS a unique position among the metal sulfides, probably to be shared only by  $FeS_2$  [1]. The narrow band gap and the interesting structural properties of SnS satisfy most of the criteria to make them prospective material in photoelectrochemical solar cells, as solar absorbers in thin film solar cells, near infrared detectors, as photovoltaic materials and in other optoelectronic systems. The SnS films show both direct and indirect absorptions at room temperature. It exhibits p-type conduction and high absorption coefficient [13]. Thin films of tin sulfides have been deposited already by different techniques such as: spray pyrolysis [7], chemical bath deposition [5, 8, 9], and metal organic chemical vapour deposition [6], electro chemical deposition [10], co evaporation [11] and spin coating [12], chemical deposition [13], electro deposition technique [14], thermal evaporation [4], electron beam evaporation [17] and successive ionic layer adsorption and reaction (SILAR) [18-22, 25-29].

The present work is focused to develop the thin films of tin sulfide (SnS) on a glass substrate at room temperature by SILAR method. In this technique, several coatings of tin nitrate (Sn (NO<sub>3</sub>)<sub>2</sub>) are made as cationic precursor and sodium sulfide (Na<sub>2</sub>S) as anionic precursor. The changes in the properties of SnS with complexing agent triethanolamine (TEA) [22] are studied for increasing molar concentration. SILAR technique is simple, cost effective, suitable for large area deposition, less time consuming and

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producing uniform, well adherent thin film. The deposition parameters are optimized to obtain good quality films. The structural, morphological, topographical and optical properties of developed triethanolamine added tin sulfide thin films are analyzed by XRD, FT-IR, SEM, AFM and optical properties are studied.

## 2 Experimental details

## 2.1 Preparation of SnS thin films with TEA

The SILAR method is an another version of the chemical bath deposition (CBD) and improves the difficulties of CBD. This method is having successive reactions at the surface of the substrate. Glass plate is cleaned at each reaction to enable



Step 1 : Immersion in cationic precursor TEA added tin nitrate Step 2 : Rinsing in distilled water Step 3 : Immersion in anionic precursor sodium sulfide

Step 4 : Rinsing in distilled water

Fig. 1 a Schematic representations of SILAR deposition for SnS thin films with TEA. b Experimental set up of SILAR deposition for SnS thin films with TEA

the heterogeneous reaction between the solid phase and the insoluble ions in the solution. Schematic representations and experimental set up of SILAR deposition method are shown in Fig. 1a, b. The glass plates are degreased with acetone and rinsed by concentrated nitric acid. The glass plate is rinsed with a detergent solution, washed with distilled water and dried in air. TEA added tin sulfide thin films are prepared using the beakers containing aqueous solutions of TEA added tin nitrate  $(Sn(NO_3)_2)$  and sodium sulfide  $[Na_2S]$ . Tin nitrate and sodium sulfide are kept at 1:1 molar ratio for all the concentrations. 4 ml of triethanolamine (TEA) added to tin nitrate  $(Sn(NO_3)_2)$  is taken in beaker (a). This few drops of triethanolamine (TEA) help in achieving uniformity of the thin films over the entire substrate surface [22]. This solution is taken as a cationic precursor solution. Thin film deposition process carried out by the parameters mentioned in Table 1.

Pre-washed glass plates are immersed in cationic precursor TEA added tin nitrate in beaker 1(a) for 35 s, so that Sn<sup>2+</sup> ions present in the solution can be adsorbed on the glass plates. Then, the substrate is rinsed in distilled water in beaker 1(b) for 35 s, so that, the excessively adsorbed Sn<sup>2+</sup> ions are rinsed away from the deposited layer which may result in saturated adsorbed layer of  $(Sn^{2+})$  cations on the glass plate. The cation coated films are immersed in anionic precursor in beaker 1(c) for 35 s for adsorption, the anions  $S^{2-}$  from the anionic sodium sulfide solution are introduced to the system and a solid substrate is formed on the interface. Hence the cations  $(Sn^{2+})$  can react with the newly introduced anions  $S^{2-}$ . The sulfide ions which do not react are removed by rinsing them in distilled water in beaker 1(d) for 35 s. Thus a SILAR cycle is comprised of these four parts. Molar concentrations of tin nitrate and sodium sulfide are varied as 0.05, 0.10, 0.15, 0.20 and 0.25 M. These operations are repeated for 30 cycles for all the concentrations in order to get an adherent film.

#### 2.2 Material characterization

Crystalline phases of the films are verified using a conventional Ultima3 theta–theta gonio X-ray diffractometer with Cu K $\alpha$  radiation ( $\lambda = 1.5406$  Å). Presences of molecular vibrations in the films are confirmed using FTIR spectrum for the wave number range of 400–4000 cm<sup>-1</sup>. The surface morphology studies of the film deposited on glass substrate are studied by scanning electron microscopy (SEM) technique using VEGA3 TESCAN unit. The surface topography is analyzed using atomic force microscopy (Park XE100 model, Hallmark).The dependence of optical transmittance and absorbance on the wavelength was carried out using Jasco Ultraviolet Spectrum in the range 200–800 nm.

Table 1Deposition parametersin SILAR controller settings forSnS thin films with TEA

Source of material	Tin nitrate	Distilled water	Sodium sulfide	Distilled water
Volume	100 ml	100 ml	100 ml	100 ml
Concentration	0.05–0.25 M	_	0.05-0.25 M	_
Temperature	31 °C	31 °C	31 °C	31 °C
Complexing agent	TEA (4 ml)	NIL	NIL	NIL
Start position	0	0	0	0
Dip length	75 mm	75 mm	75 mm	75 mm
Dip speed	$5 \text{ mm sec}^{-1}$			
Retrieval speed	$4 \text{ mm sec}^{-1}$			
Dip duration	35 s	35 s	35 s	35 s
Ex. dip duration	4 s	4 s	4 s	4 s
No. of cycles	30	30	30	30
Soluble in	Dist. water	-	Dist. water	_
Source of ions	Sn <sup>2+</sup>	_	$S^{2-}$	_

# 3 Results and discussion

#### 3.1 XRD analysis

The X-ray diffraction patterns of the SnS thin films deposited in different molar concentrations such as 0.05, 0.10, 0.15, 0.20, 0.25 M by SILAR technique on glass substrates at room temperature are shown in Fig. 2a-e. It is confirmed from the Fig. 2a-d 0.05, 0.10, 0.15 and 0.20 M, the deposited films are found to have hexagonal crystal structure and agree well with the standard data from JCPDS card No. 89-3198. The observed d spacing and hkl planes values of Hexagonal phase SnS films for corresponding  $2\theta$  values are tabulated in Table 1. The reflection peaks are indexed and the corresponding values of interplanar spacing d are calculated and compared with the standard values. From Fig. 2c 0.15 M, it is observed that additional peaks present at  $2\theta$  values of  $28.102^{\circ}$ ,  $47.675^{\circ}$ and 55.008° corresponding to the hkl planes (111), (220) and (311) respectively. Figure 2d 0.20 M, shows an additional peaks at 2 $\theta$  values of 28.367° and 47.173° corresponding to the hkl planes (111) and (220) respectively for the increase of concentration. Fig. 2c, d reveals that the films are in mixed hexagonal and cubic structure for increasing concentration from 0.15 to 0.20 M. Figure 2e 0.25 M confirms that the deposited films are found to have cubic crystal structure and agree well with the standard data from JCPDS card No. 89-2755. The observed d spacing and hkl planes values of cubic phase SnS films for corresponding 2 $\theta$  values are tabulated in Tables 2 and 3.

The grain size (D) of the cubic SnS film is calculated from the line broadening of the (111) and (200) diffraction peak. According to the Scherrer equation:

$$D = \frac{0.9 * \lambda}{\beta \cos \theta} \tag{1}$$

where  $\lambda$  is the wavelength of Cu-K $\alpha$  radiation (1.5406 Å), 0.9 is  $\beta$  is the broadening of a diffraction peak measured at full width and half of the maximum (FWHM) intensity and  $\theta$  is the diffraction angle. The calculated average grain sizes of SnS thin films are around 12 and 8 nm for 0.25 M concentrations of (111) and (200) diffraction peaks.



Fig. 2 XRD patterns of SnS thin films with TEA for different molar concentrations

 Table 2
 hkl plane and d spacing for corresponding 2θ values

 hexagonal phase in XRD
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20	hkl planes	d spacing
29.012	(101)	3.0538
30.300	(004)	2.9569
33.235	(102)	2.6978
37.200	(103)	2.4188
41.47	(104)	2.1752
45.955	(006)	1.9714
48.272	(105)	1.8835
49.508	(110)	1.8413
53.224	(106)	1.7164

**Table 3** hkl plane and d spacing for corresponding 2θ values cubic phase in XRD

20	hkl planes	d spacing
28.336	(111)	3.1249
33.449	(200)	2.6771
58.188	(222)	1.5844

The dislocation density  $(\rho)$  of the deposited film is also calculated from the diffractogram by using the formula,

$$\rho = \frac{1}{D^2} \tag{2}$$

The value of micro strain  $(\epsilon)$  is obtained using the relation:

$$\varepsilon = \frac{\beta \cos \theta}{4} \tag{3}$$

The calculated value of dislocation density is found to be  $6.70 \times 10^{15}$  and  $15.475 \times 10^{15}$  line m<sup>-2</sup>. The calculated values of micro strains are  $2.838 \times 10^{-3}$  and  $4.3132 \times 10^{-3}$  line<sup>-2</sup> m<sup>-4</sup> for 0.25 M concentrations of (111) and (200) diffraction peaks. These observations indicate that, the deposited SnS thin films are in nano scale. The XRD pattern of SnS film indexed to the cubic structure estimates the maximum crystalline size as 12 nm from the Debye–Scherrer formula which compared with the reported value (99 nm) [5, 14, 15, 17].

### 3.2 SEM analysis

SEM is used to investigate the effect on the surface of TEA added tin sulfide thin films with different molar concentration. Figure 3 shows the SEM micrograph of the surface of different molar concentration films at different magnification. The images recorded at 5, 2 and 1  $\mu$ m resolution shows the aggregates composed of tin sulfide thin films. The image of 0.05 M samples has a uniform surface morphology revealing a rather smooth surface. One interesting observation from (0.05 M) is the appearance of stair case like surface. In the case of 0.10 M, a drastic change in the surface morphology is observed as compared to 0.05 M.

From the SEM micrographs, it is clear that the film possesses almost smooth surface although some particles have distraction on the film because of the increasing concentration. SEM micrographs of 0.15 and 0.20 M seem to be agglomerates of the particles in the film and also the degree of roughness of the surfaces increased with increasing concentrations. The SEM micrograph taken for 0.25 M shows that the film surface appear partially smooth but there are few isolated islands and minute particles over the surface. This indicates that the segregation of the increasing concentration and interaction of TEA in the host medium.

#### **3.3 AFM analysis**

SnS with TEA thin films are examined by AFM for its surface roughness, particle height and for its grain size. The 3D and 2D AFM images of SnS thin films (0.20 M) are recorded in 5 and 10  $\mu$ m as shown in Figs. 4, 5 and 6. The scanned area is focused for 10  $\mu$ m. Significantly, the change in topography of the thin film is observed for two different magnification areas. It can be observed that, there are well ordered stair case structure of the SnS thin films are spread over the surface. The AFM image indicates that these coatings cover the surface of the substrate completely. Surface roughness, grain size and surface height are tabulated for 10 and 5  $\mu$ m magnification in Table 4.

From 2D AFM image of SnS film for 5  $\mu$ m magnification, pits and peaks vary between -1.023 and 0.678 nm. Minimum and maximum roughness is around 0.16–0.34 nm, which confirms the uniform coating. The particle size is irregular and the particle height varies from 0.44 to 1.27 nm. For the decrease in maximum particle height, the smoothness increases. It is observed that, when the particles are having equal height, the roughness decreases and the smoothness increases for the deposited surface of the material.

For 10  $\mu$ m magnification, pits and peaks varies from -3.26 to 1.95 nm. The particle height varies from 1.24 to 3.72 nm. Surface roughness varies between 0.3 and 0.93 nm. For the closer area of magnification, surface roughness, grain size and surface height decreases comparitively. If focus reduced to very small area to get maximum magnification, grain size of tiny particles also found considerably. The different size of particles will not affect the smoothness of the surface. But change in surface height may affect the smoothness. To attain effective electron conversion, the smoothness is a significant factor.

#### 3.4 FT-IR analysis

The FTIR spectrum of SnS composite thin film is shown in Fig. 7. FTIR measurements have been made in the wave number range 400–4000 cm<sup>-1</sup>. The weak absorption peak at 3521 and 2911 cm<sup>-1</sup> is assigned to O–H stretching vibration of the adsorbed water in the sample. The presence of water is also confirmed by its bending vibration observed at 1800 cm<sup>-1</sup>. Broad peak is observed between 1800 and 1000 cm<sup>-1</sup> is possibly due to stretching vibration of sulfide group. A small absorption peak is observed at 569 and 686.66 cm<sup>-1</sup> are possibly due to the stretching vibration of Sn-S. The absorption peak appears around 2081.25 cm<sup>-1</sup> may be due to C=C vibration suggesting the organic contamination from the source materials [17, 22–24].



Fig. 3 SEM magnification series of 5, 2 and 1  $\mu$ m in 0.05, 0.10, 0.15, 0.20 and 0.25 M of SnS thin film with addition of TEA for different molar concentration



Fig. 4 AFM 3D images of SnS with TEA for 0.20 M in 5 and 10 µm magnification



Fig. 5 AFM 2D image of SnS with TEA for 0.20 M in 5  $\mu$ m magnification

### 3.5 UV-visible analysis

#### 3.5.1 Optical absorbance analysis

The optical absorption property of the SnS thin film with different molar concentrations such as 0.05, 0.10, 0.15, 0.20 and 0.25 M is analyzed from 300 to 800 nm and shown in Fig. 8. The films show better

absorption in the visible region and lower absorbance in the near-IR region. The increasing absorbance property is observed in the visible and IR regions with increasing molar concentrations. Figure 8 shows the absorbance peak at 380 nm wavelength invariantly for all the concentrations. For the increasing concentration the cutoff wavelength increases from 650 to 700 nm.



Fig. 6 AFM 2D image of SnS with TEA for 0.20 M in 10  $\mu$ m magnification

Table 4 The surface properties of SnS film with TEA for 0.20 M in 5 and 10 µm magnification

	5 µm			10 μm		
	Grain size $(\Delta x)$ (nm)	Surfaces roughness(Ra) (nm)	Surface height (Rz)(nm)	Grain size $(\Delta x)$ (nm)	Surfaces roughness (Ra) (nm)	Surface height (Rz) (nm)
Min	60	0.16	0.44	83	0.30	1.24
Max	71	0.34	1.27	240	0.93	3.72

#### 3.5.2 Optical transmittance analysis

Optical transmittance spectrum of TEA added SnS thin film with different molar concentrations is shown in Fig. 9. The transmittance is found to agree well with absorbance at 381 nm wavelength. The optical transmittance 93.99, 93.48, 93.30, 92.38 and 92.23 % for the corresponding 0.05, 0.10, 0.15, 0.20 and 0.25 M concentration is observed from the spectrum. From the Fig. 9, it is observed that, transmission decreases for the increasing molar concentrations. No absorption found between 900 and 700 nm. For the increasing molar concentration, the coated material promotes electrons from the valence band to the conduction band for the absorption of minimum energy for the wavelength ranging from 650 to 700 nm.

#### 3.5.3 Band gap analysis

The band gap energy of the thin film samples is calculated using the equation below.

$$(Ahv)^n = k(hv - E_g) \tag{4}$$

where v is the frequency, h is Planck's constant; k equals a constant while n carries the value of either 2 or 1/2.

Thus, a plot of  $(Ahv)^2$  versus hv is a curved line. The band gap energy of the film can be determined by the extrapolation of the linear regions in the plot of  $(Ahv)^2$ versus hv. From the Fig. 10, the band gap energy of the TEA added SnS thin film with 0.05, 0.10, 0.15, 0.20 and 0.25 M are 2.44, 2.36, 2.23, 2.10 and 1.89 eV respectively



Fig. 7 FTIR spectra of the SnS films with TEA for different concentrations  $% \left( {{{\left[ {{{\rm{TE}}} \right]}_{{\rm{TE}}}}_{{\rm{TE}}}} \right)$ 



Fig. 8 Optical absorbance of SnS thin films with TEA



Fig. 9 Transmittance spectrums of the SnS thin films with TEA



Fig. 10 Band gap analysis of the SnS thin films with TEA from transmittance spectrum



Fig. 11 The effect of increasing molar concentration on optical properties of SnS thin fims with TEA

are observed. The values are having good agreement with the reported value [16, 17].

#### 3.5.4 Effect of molar concentration on optical properties

Effect of molar concentration on optical absorption, transmittance and band gap energy is shown in Fig. 11. Table 5 shows the comparative analysis on the effect of various molar concentration on optical properties of the

Table 5 The effect of
increasing molar concentration
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Molar concentration (M)	Optical absorbance	Optical transmittance (%)	Band gap energy (eV)
0.05	0.0707	93.99	2.44
0.10	0.0819	93.48	2.36
0.15	0.0837	93.30	2.23
0.20	0.0889	92.38	2.10
0.25	0.1296	92.23	1.89

deposited thin films. The optical absorption of the deposited films increases with the increasing concentration. The optical transmittance and band gap energy of the deposited films decreases for the increasing concentration.

## 4 Conclusion

TEA added SnS thin films are deposited by the SILAR technique using the equimolar and equivolume solutions of Tin nitrate and sodium sulfide. The films with higher concentrations are having dominant cubic phase. It is around 12 nm and shows good appearance in the deposition. FTIR spectrum shows the strong and weak bands associated with the deposited films. From the SEM micrographs, it is clear that the film possesses almost smooth surface although some particles have distraction on the film because of the increasing concentration. AFM examination explains its surface roughness, particle height and for its grain size. When the particles are having equal height, the roughness decreases and the smoothness increases for the deposited surface of the material. To attain effective electron conversion, the smoothness is a significant factor. The band gap energy decreases from 2.44 to 1.89 eV for increasing molar concentration from 0.05 to 0.25 M. Results shows the TEA added SnS thin film is one of the potential candidate for solar cell applications.

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