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Effect of bath concentration, temperature on the growth and properties of chemical bath deposited ZnS films

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ABSTRACT

Uniform, specular and adherent ZnS films were grown by chemical bath deposition process with ammonia as the only complexing agent and thioacetamide as the source of sulphur. The effects of bath temperature, concentration of the zinc salt, thioacetamide and ammonia on the growth of ZnS films were investigated with an objective to optimize the bath concentration and temperature. This optimization was done to reduce the deposition time and achieve the desired thickness without the need for repeated depositions in fresh baths. Structural, morphological and optical properties of the films deposited at 60 °C (optimized bath temperature) with different concentrations of thioacetamide in the bath are reported. Film formation mechanism and reason for the film peel-off from the substrate at higher zinc salt as well as thioacetamide concentrations are explained. Crystallinity of the film and the surface roughness were found to increase with increase in thioacetamide concentration in the bath. Films deposited under optimized conditions were found to be nanocrystalline, cubic in structure. FTIR studies revealed the presence of trace amount of hydroxide. The optical band gap was found to increase from 3.70 eV to 3.95 eV with decrease in thioacetamide concentration. The average transmittance of films deposited at different bath temperatures was found to be >80% in the wavelength range 400–1500 nm.

1. Introduction

Zinc sulphide is an important wide band gap ($E_g = 3.68$ eV [1]) semiconductor with potential applications in electroluminescent and photoluminescent devices, thin film solar cells, light emitting diodes etc. Zinc sulphide thin films are being investigated as an alternate buffer layer to CdS, which is toxic, in copper indium gallium selenide (CIGS) and cadmium telluride (CdTe) thin film solar cells. Because of higher band gap compared to CdS, solar cells with ZnS as the window layer are expected to show higher photocurrent in the blue wavelength region of the solar radiation spectrum. CIGS solar cells with ZnS as window/buffer layer have exhibited the efficiency close to 18.6% [2] against the record efficiency of 20.3% with CdS [3]. Inspite of expected benefits in the case of ZnS, CIGS device efficiency is less than by about 1% compared to CdS layer. Thus there is a need to have a thorough understanding of ZnS film growth to obtain films with better quality to improve the device efficiency. Several techniques are used for the growth of ZnS films such as electron beam evaporation, vacuum evaporation, R.F sputtering, atomic layer deposition, pulsed laser deposition, spray pyrolysis, MOCVD and chemical bath deposition (CBD). CBD is a simple and low-cost technique amenable for scaling up and is currently used to deposit CdS buffer layer in CIGS solar cells. In recent years, several investigators have therefore attempted to deposit ZnS thin films by CBD. Various complexing agents like ammonia [4–6], triethanolamine [7,8], tartaric acid [9] have been attempted. Additional complexing agents like hydrazine hydrate [9–12], trisodium citrate [13–15] have also been used along with ammonia in some cases. Mostly thiourea was used as the source of sulphur. The thickness obtained was in the range 120–160 nm and the deposition times were long (1–2 h). Repeated dips in fresh baths have been carried out by some others [9,12,13] to achieve thicker films. However, from the technological point of view, long process times and repeated depositions reduce throughput and enhance the cost. Thus there is a need to reduce the process time and complexity. To achieve this objective, a systematic study of the effect of bath temperature and concentrations of bath constituents on the growth and properties of CBD grown ZnS films has been carried out to optimize the growth conditions. We have used thioacetamide as the source of sulphur and ammonia as the only complexing agent. The stability constant of zinc–ammonia complex is higher compared to the stability constants of zinc complexes with other agents like hydrazine hydrate and triethanolamine [16]. Thioacetamide readily dissolves in water giving

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stable solution which hydrolyzes very slowly, uniformly and homogeneously. However, in acidic or alkaline solutions or at elevated temperatures, the rate of hydrolysis of thioacetamide increases, and the rate being greater in alkaline solution than in acidic solution [17]. The present paper reports the results of the investigations on the effects of bath temperature and concentrations of bath constituents on the growth and properties of CBD ZnS films.

2. Experimental

The recipe for the deposition of ZnS film consists of aqueous solution of zinc sulphate heptahydrate (0.03 M–0.10 M), ammonia (7 M–11 M) and thioacetamide (0.02 M–0.10 M). The starting solution pH was in the range 11–12 based on the concentrations chosen. Chemically and ultrasonically cleaned soda-lime glass slides were used as substrates. For optical absorption studies, films were deposited on quartz substrates. In order to study the effect of bath temperature, depositions were carried out at different bath temperatures in the range 30 °C–80 °C. The bath temperature could be maintained to an accuracy of ±2 °C using a thermostatic control. To study the film growth as a function of time, at each bath temperature substrates were removed from the bath at intervals of 10, 15, 30, 60 and 120 min, cleaned with deionized water and dried. From these studies, the optimized bath temperature for the growth of ZnS films was found to be 60 °C. In order to investigate the effect of bath constituents on the growth of the films, initially zinc salt concentration in the solution was varied from 0.03 M to 0.10 M, keeping the bath temperature, concentrations of thioacetamide (TA) and ammonia fixed. At each concentration, the growth rate was determined from the study of thickness dependence with time. Similarly, keeping the bath temperature as well as zinc salt and thioacetamide concentrations in the bath fixed, ammonia concentration in the bath was varied to understand the effect of ammonia on the growth process. Finally, with optimized bath temperature, zinc salt and ammonia concentrations fixed, the thioacetamide concentration in the bath was varied to understand its effect on the film formation.

The film thickness was determined from the deposited mass measured using a METLER AE240 microbalance and the bulk density. The uncertainty in determination of the film thickness is found to be ±10%. The spectral transmittance of these films was recorded using a JASCO UV–VIS–NIR (Model V 570) spectrophotometer. The X-ray diffraction (XRD) patterns of these films were recorded in glancing angle mode (glancing angle = 0.3°) using a SEIFERT Model 3003 TT X-ray diffractometer with Cu K_α (0.15406 nm) radiation. The microstructure of the films was recorded using field emission scanning electron microscope (FEI). Infrared spectrum of the film deposited on silicon substrate was recorded using Fourier Transform infrared spectrophotometer (Bruker Tensor 27) in the range 400–4000 cm^{-1}. Atomic force microscopy images were taken in the contact mode using Nanoscope–E, Digital Instruments, USA.

3. Results and discussion

3.1. Effect of bath temperature (t_b)

Fig. 1 shows ZnS film thickness as a function of deposition time for different bath temperatures (t_b). It shows two distinct regions: (i) quasi-linear region and (ii) saturation region. As the bath temperature increases, it is observed that the onset of saturation commences earlier. At higher bath temperatures, the film grows relatively faster and reaches saturation earlier. The deposition times to reach the onset of saturation were found to vary mostly from 15 to 30 min based on the bath temperature. Films obtained at bath temperatures of 70 °C and 80 °C were found to be non-uniform. Uniform and specular ZnS films were obtained at $t_b = 60$ °C. It is possible to calculate the initial growth rate from the quasi-linear region. Fig. 2 shows the log (growth rate) against 1000/T_b for bath temperatures in the range 30 °C–60 °C ($T_b = 273 + t_b$). The activation energy (E_a) for the deposition process is found to be ~2.5 $kcalmol^{-1}$. The low activation energy suggests that the growth rate is predominantly a temperature controlled one rather than chemically controlled process [4].

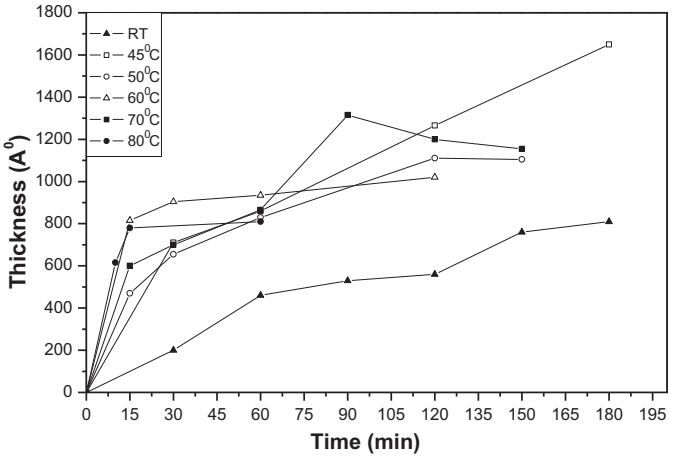

Fig. 1. Thickness variation as a function of time at different bath temperatures. Bath composition: [$ZnSO_4$] = 0.05 M, [TA] = 0.02 M, [NH_3] = 9.8 M.

3.2. Influence of bath constituents

3.2.1. Effect of ammonia concentration

Fig. 3(a) shows the effect of ammonia concentration on the growth rate of ZnS films when zinc salt concentration was 0.05 M, thioacetamide concentration was kept as 0.02 M and temperature of the bath was maintained at 60 °C. For low ammonia concentrations, the complexation is weak due to which the availability of

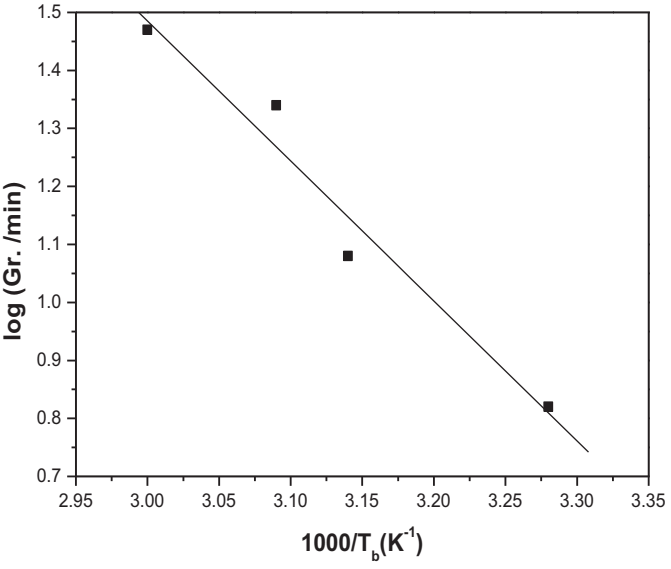

Fig. 2. Logarithm of growth rate as a function of (1000/T_b).

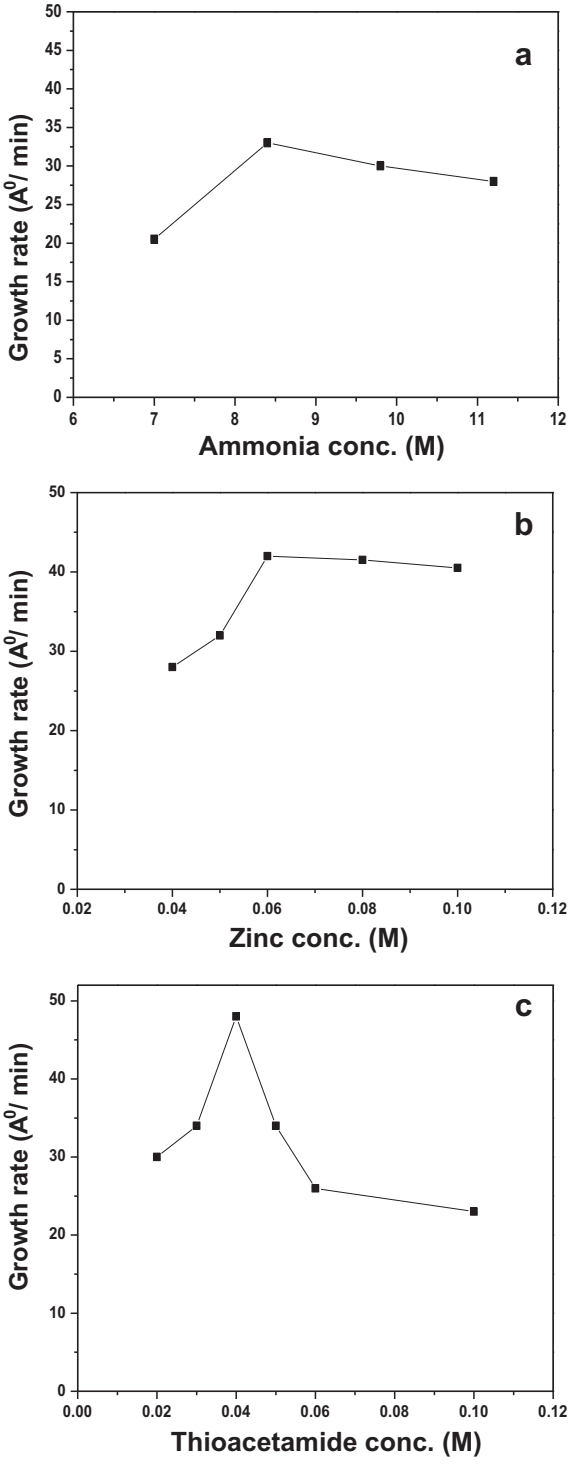

Fig. 3. Film growth rate as a function of (a) ammonia concentration (b) zinc concentration (c) thioacetamide concentration.

free Zn^{2+} in the bath is more. These free Zn^{2+} ions readily combine with S^{2-} ions leading to homogeneous precipitation of ZnS in the bath resulting in lower growth rate. With increase in the concentration of ammonia, the complexation is stronger and due to the controlled release of Zn^{2+} ions from zinc amine, the growth rate improves up to $[NH_3] = 8.4$ M. With further increase of ammonia concentration in the bath, there is a slight decrease in the growth rate due to decrease in the release of free Zn^{2+} ions from the complexing agent. It was observed that films deposited from a bath with $[NH_3] = 9.8$ M were relatively more specular and uniform compared to the film deposited from a bath with $[NH_3] = 8.4$ M. Hence the optimum $[NH_3]$ concentration for the growth of ZnS film was chosen as 9.8 M.

3.2.2. Effect of zinc concentration

In order to understand the effect of zinc salt concentration in the bath on the growth of ZnS films, its concentration in the solution was varied from 0.03 M to 0.10 M keeping the concentrations of ammonia and thioacetamide constant. The bath temperature was maintained at 60 °C. It was observed that practically there was no film formation for zinc salt concentration <0.04 M. Fig. 3(b) shows the effect of zinc salt concentration on growth rate. The growth rate is found to increase with increase in zinc concentration in the range 0.04 M–0.06 M and remains almost constant thereafter. However, for zinc concentration higher than 0.05 M, the films were non-uniform and appeared patchy, probably due to dissolution of a part of the film back into the solution.

3.2.3. Effect of thioacetamide concentration

'The thioacetamide concentration in the bath was varied from 0.01 M to 0.10 M keeping the bath temperature (60 °C), concentrations of NH_3 and zinc salt constant. The effect of thioacetamide concentration on the growth of ZnS film is shown in Fig. 3(c). When the thioacetamide concentration was <0.02 M, it was observed that there was no significant film growth. As the thioacetamide concentration in the bath increases, the growth rate increases up to a concentration of 0.04 M beyond which it is found to decrease. During the experimentation, it was observed that above 0.04 M thioacetamide concentration in the bath, the film partially peels-off from the substrate resulting in lower thickness. Thus the decrease in growth rate at higher thioacetamide concentration is due to partial dissolution of the film back into the solution. For thioacetamide concentration in excess of 0.10 M, initially film formation was observed but as the time progresses, the substrate was seen to be completely clean without any film due to complete dissolution of the same in the bath. The reason for the dissolution/partial peel-off at higher thioacetamide and zinc salt concentrations can be explained by considering the reaction kinetics.

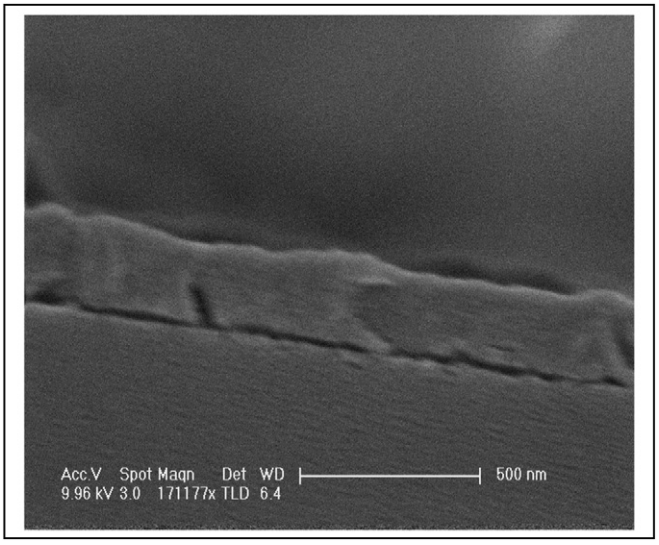

Fig. 4. SEM cross-sectional image of a typical ZnS film deposited under optimized conditions.

3.2.4. Growth kinetics

In order to understand how the concentrations of bath constituents (zinc salt, ammonia and thioacetamide) and temperature influence the availability of free Zn^{2+} ions and hence the growth of ZnS film, it is essential to consider the various competing chemical reactions occurring simultaneously in the bath and their equilibrium chemical kinetics.

Zinc sulphate dissociates giving rise to Zn^{2+} according to the equation

$$ZnSO_4 \rightarrow Zn^{2+} + SO_4{}^{2-} \quad (1)$$

On hydrolysis of ammonia, OH^- is obtained according to the equation

$$NH_3 + H_2O \rightarrow NH_4OH \rightleftharpoons NH_4{}^+ + OH^-$$

$$\text{with equilibrium constant}(k) = \frac{\left[NH_4{}^+\right][OH^-]}{[NH_4OH]} = 1.8 \times 10^{-5} \text{ at } 25\ ^\circ C \quad (2)$$

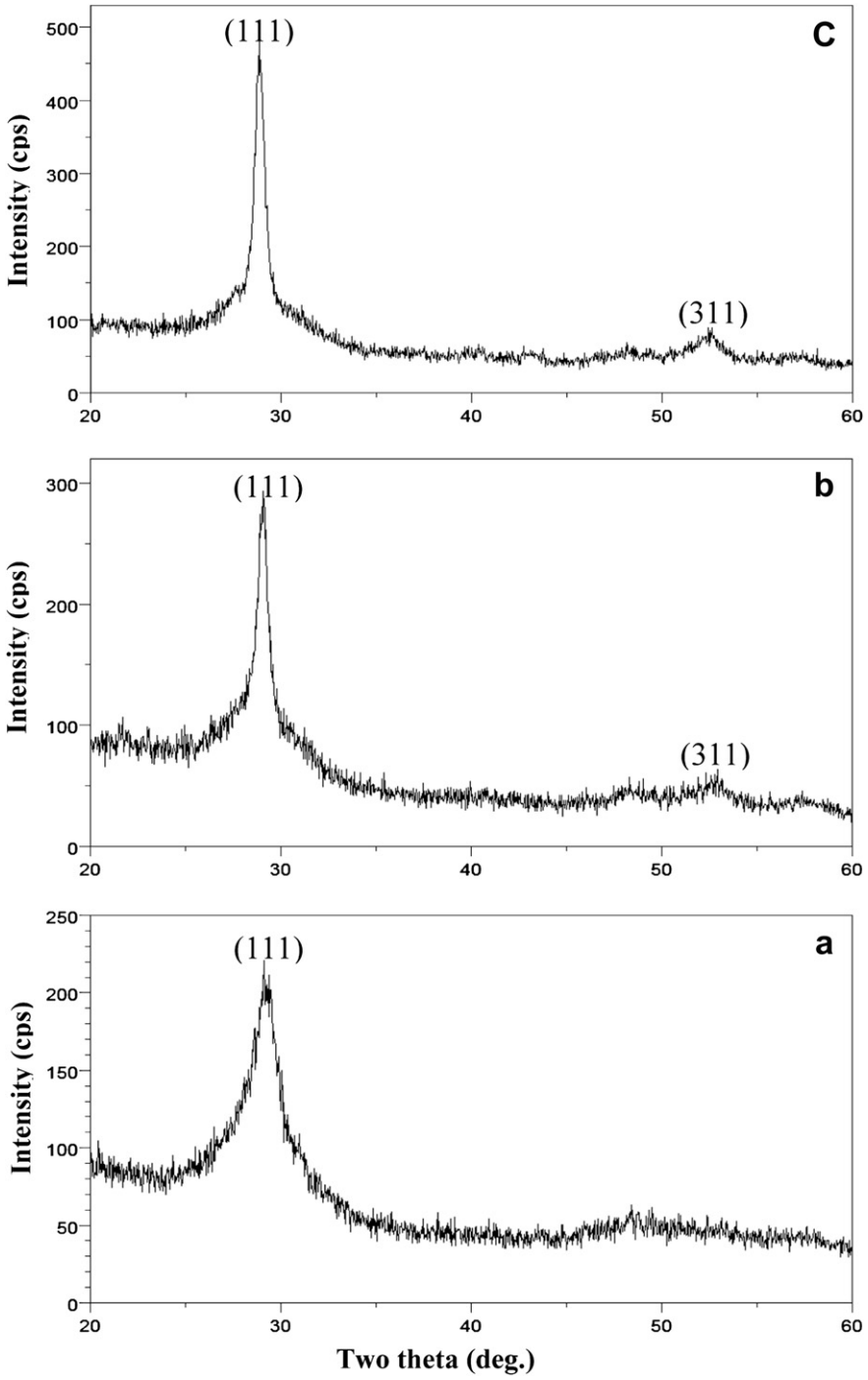

Fig. 5. GIXRD pattern of CBD ZnS films deposited from chemical baths with different thioacetamide concentrations keeping $[ZnSO_4] = 0.05$ M, $[NH_3] = 9.8$ M and $t_b = 60\ ^\circ C$. (a) [TA] = 0.02 M, (b) [TA] = 0.04 M, (c) [TA] = 0.06 M.

Zn^{2+} reacts with OH^- forming zinc hydroxide according to the reaction

$$Zn^{2+} + 2OH^- \rightleftharpoons Zn(OH)_2 \quad \text{with solubility product } (K_{SP}) = 2 \times 10^{-17} \tag{3}$$

Zn^{2+} ion reacts with NH_3 forming zinc tetramine

$$Zn^{2+} + 4NH_3 \rightleftharpoons Zn\left[(NH_3)_4\right]^{2+} \text{ with}$$

$$k = \frac{[Zn^{2+}][NH_3]^4}{\left[Zn\left[(NH_3)_4\right]^{2+}\right]} = 10^{8.9} \tag{4}$$

In an alkaline medium, the hydrolysis of thioacetamide is given by [18]

$$CH_3CSNH_2 + 2OH^- \rightleftharpoons CH_3COO^- + NH_3 + HS^-$$

Subsequently,

$$HS^- + OH^- \rightleftharpoons S^{2-} + H_2O$$

The free Zn^{2+} ions released from zinc amine react with S^{2-} released from the thioacetamide source giving rise to

$$Zn^{2+} + S^{2-} \rightleftharpoons ZnS\downarrow \tag{5}$$

with $k = [ZnS]/[Zn^{2+}][S^{2-}] = 10^{24.7}$. Zn^{2+} ions are in simultaneous equilibrium with OH^-, NH_3 and S^{2-} as seen from Eqs. (3)–(5) and their concentration in the bath depends on equilibrium kinetics of these competing chemical reactions.

The rate of forward reaction in Eq. (5) can be deliberately increased by increasing concentration of Zn^{2+} or S^{2-} in the solution. Thus, as the thioacetamide or zinc salt concentration in the solution increases, ZnS film growth as well as precipitation in the bath increases. However, as the forward reaction proceeds with time, the concentration of reactants in the solution decreases. When the concentration of the reactants (Zn^{2+} or S^{2-}) in the vicinity of the substrate falls below the concentration levels permitted by the chemical equilibrium given by Eq. (5), the reverse reaction takes place due to which the film dissociates to compensate for the deficiency. This accounts for the film peel-off and the decrease of the growth rate at higher concentrations of zinc salt or thioacetamide as observed in Fig. 3(b) and Fig. 3(c). Such high concentrations of zinc salt or thioacetamide are therefore not useful, although the initial growth rate is higher.

The optimum bath conditions for ZnS film growth from our observations were found to be $[ZnSO_4] = 0.05$ M, $[TA] = 0.04$ M and

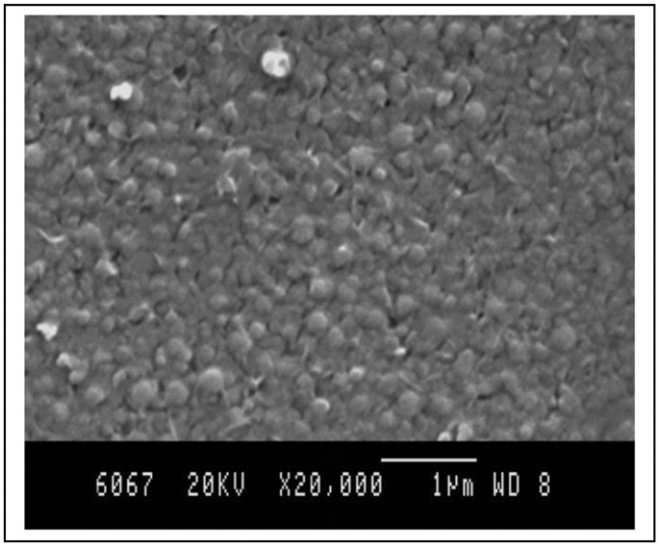

Fig. 6. SEM image of a typical ZnS film deposited under optimized conditions.

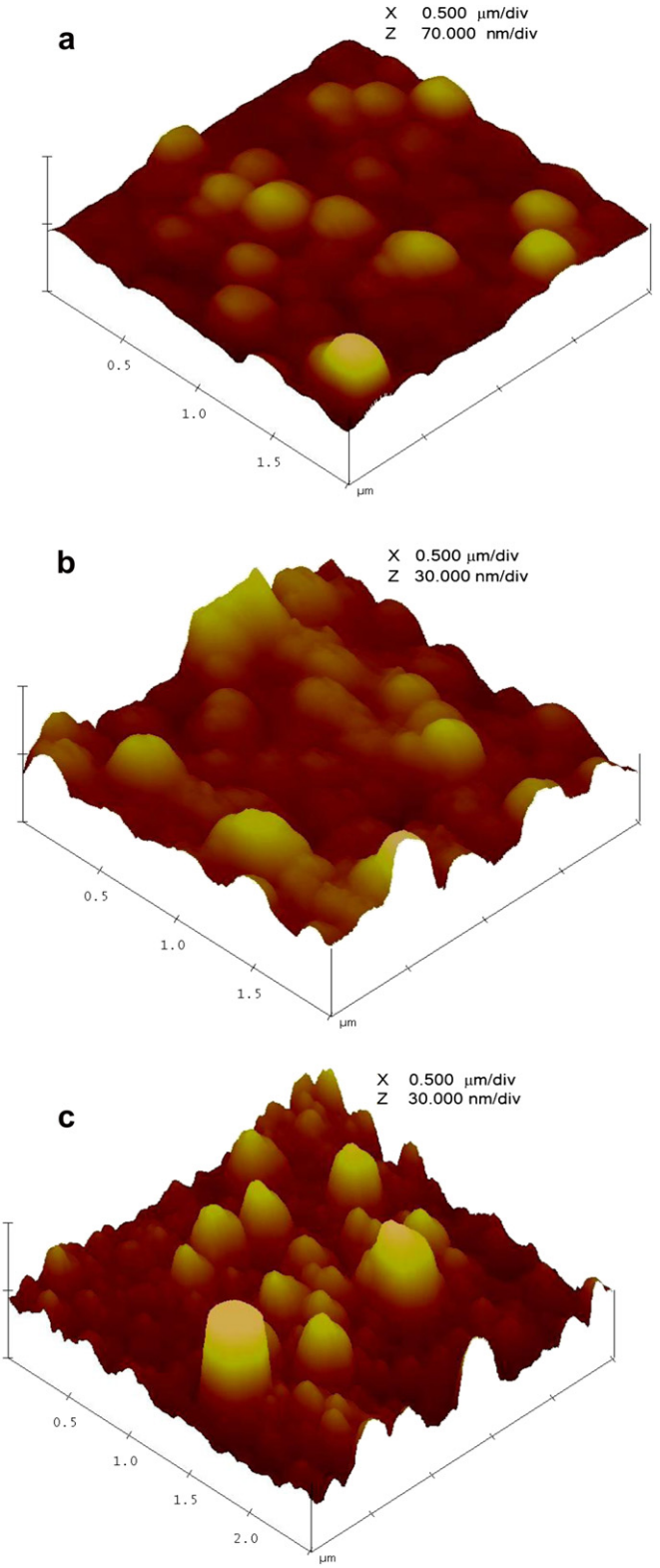

Fig. 7. AFM images of CBD ZnS films deposited from chemical baths with different thioacetamide concentrations keeping $[ZnSO_4] = 0.05$ M, $[NH_3] = 9.8$ M and $t_b = 60$ °C. (a) $[TA] = 0.02$ M, (b) $[TA] = 0.04$ M, (c) $[TA] = 0.06$ M.

$[NH_3]$ = 9.8 M at a bath temperature of 60 °C. Film thickness determined using mass of the film and bulk density was found to be 160 nm. The uncertainty in the measurement is ±10%. The thickness was also determined from SEM cross-sectional image (Fig. 4) and the average thickness is found to be 190 nm. Considering the limitations of the former method, the agreement is reasonable. The deposition time to obtain this thickness under these optimized conditions was found to be about 30 min. The reported deposition times to realize a thickness of 100–150 nm were in the range 40 min–4 h [19–22].

In addition to growth kinetic studies, the influence of concentration of bath constituents and temperature on the structural, morphological and optical properties was also investigated. It was seen that thioacetamide concentration profoundly affects the properties. The effects of thioacetamide concentration and bath temperature were investigated in detail and the results are presented below.

3.3. Structural properties

Fig. 5 shows the GIXRD pattern of the films deposited with different thioacetamide concentrations in the bath. The films were found to be nanocrystalline with (111) preferred orientation. The data agrees with reported JCPDS data [05–566, 77–2100] for cubic structure. The width of the diffraction peak is found to decrease with increase in thioacetamide concentration indicating improvement in crystallinity. The crystallite size, calculated using Debye–Scherrer's formula [23], is found to vary from 2.5 nm to 10 nm as the concentration of thioacetamide varied from 0.02 M to 0.06 M. It is worth mentioning that Scherrer's formula gives the crystallite size normal to X-ray beam direction [24] and does not give lateral grain dimension as in the case of SEM microstructure.

3.4. Microstructure

Fig. 6 shows the SEM image of ZnS film deposited under optimized conditions. The figure shows the grains are distributed uniformly on the surface of the film. The average grain size is found to be 200 nm.

Fig. 7 shows the AFM images of the ZnS film deposited from baths with different thioacetamide concentrations. The average grain size is found to increase from 220 nm to 290 nm and the rms roughness increases from 5.9 nm to 12.7 nm, as the thioacetamide concentration in bath increases from 0.02 M to 0.06 M.

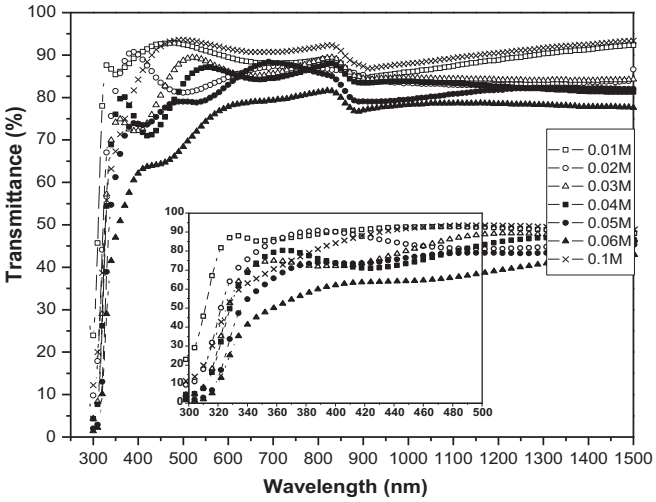

Fig. 9. Transmission spectra of CBD ZnS films deposited with different thioacetamide concentrations. Inset: Expanded view of the fundamental absorption region.

3.5. Fourier transform IR study

Fig. 8 shows the Fourier transform infrared (FTIR) spectrum of ZnS film deposited under optimized conditions on silicon substrate to know the presence of secondary phases like ZnO, $Zn(OH)_2$ etc. The spectrum shows a broad peak centred at 3360 cm^{-1} and a small peak at 1630 cm^{-1} which are due to stretching and bending modes

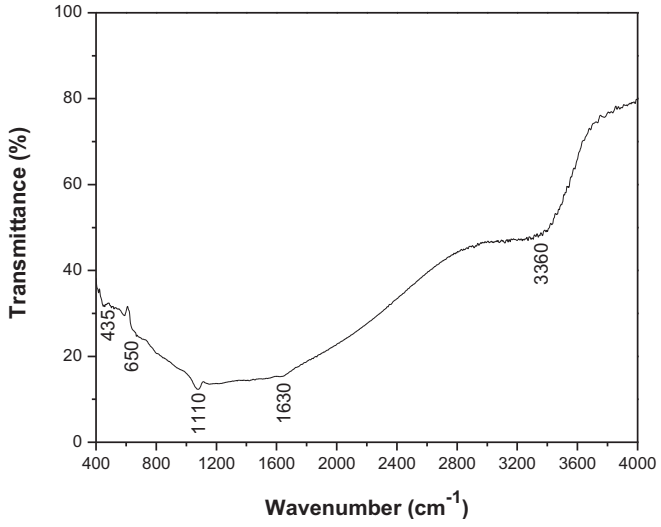

Fig. 8. FTIR spectrum of a typical ZnS film deposited under optimized conditions: $[ZnSO_4]$ = 0.05 M, [TA] = 0.04 M, $[NH_3]$ = 9.8 M and t_b = 60 °C.

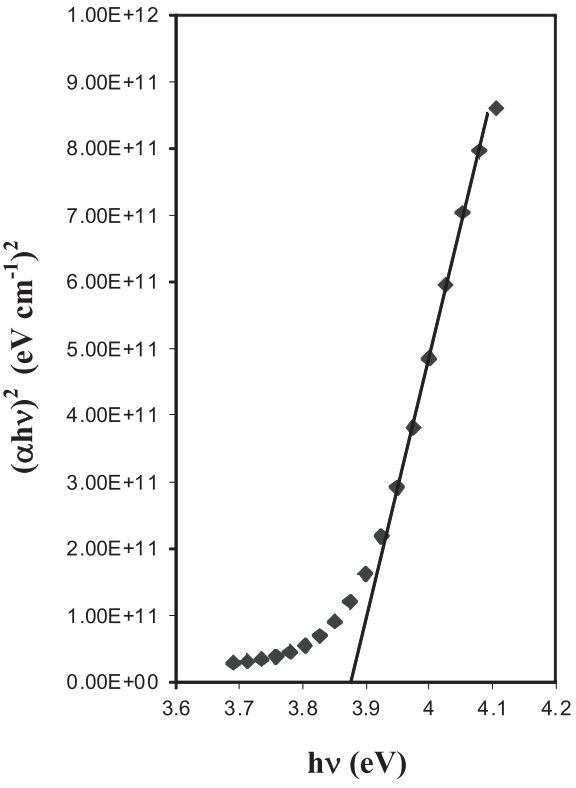

Fig. 10. $(\alpha h\nu)^2$ Versus $h\nu$ plot for a typical CBD ZnS film deposited under optimized conditions: $[ZnSO_4]$ = 0.05 M, [TA] = 0.04 M, $[NH_3]$ = 9.8 M and t_b = 60 °C.

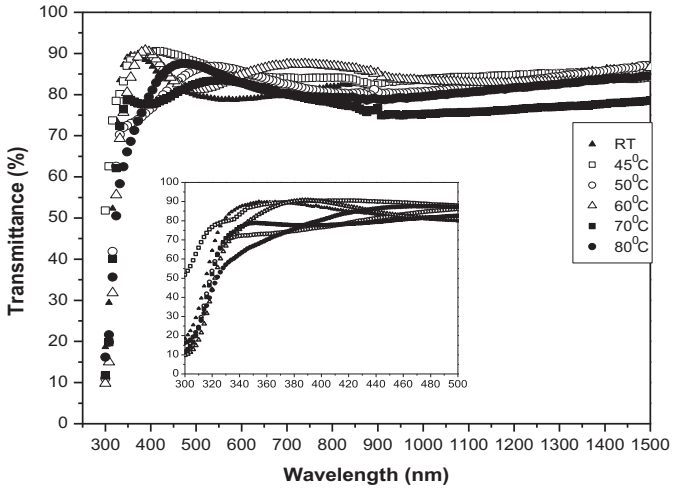

Fig. 11. Transmission spectra of CBD ZnS films deposited at different bath temperatures.

of H–OH. This might be due to trace amount of adsorbed water present on the film surface [7,25]. The spectrum shows two small peaks at 1110 cm^{-1} and 650 cm^{-1} which are assigned to stretching and bending modes of Zn–OH. A very small hump observed at 435 cm^{-1} is attributed to Zn–O bond [7,26]. Based on this study, it is felt that the film contains some amount of $Zn(OH)_2$ and ZnO.

3.6. Optical properties

Fig. 9 shows the spectral transmittance curves of ZnS films deposited at various thioacetamide concentrations. The optical absorption coefficient (α) of these films was calculated using the formula $\alpha = \ln(1/T)/t$, where 't' is thickness of the film [27]. The direct optical band gap (E_g) of the films was determined by extrapolating the linear portion of the $(\alpha h\nu)^2$ versus $h\nu$ plot and taking the intercept on $h\nu$-axis. It is observed that as thioacetamide concentration increases, the fundamental absorption edge shifts slightly towards higher wavelengths (shown in the inset) indicating that the optical band gap decreases with increase in thioacetamide concentration. The band gap was found to change from 3.70 eV to 3.95 eV as thioacetamide concentration decreases. The increase of the band gap with decrease in thioacetamide concentration might be due to decrease in crystallinity. A typical $(\alpha h\nu)^2$ versus $h\nu$ plot for films deposited under optimized thioacetamide concentration of 0.04 M is shown in Fig. 10 and the band gap is found to be 3.87 ± 0.02 eV.

The spectral transmittance curves of ZnS films deposited at different bath temperatures are shown in Fig. 11. All the films exhibit an average transmittance greater than 80% in the wavelength range 400 nm–1500 nm. As seen from the inset, a slight shift is observed in the fundamental absorption edge in films deposited at lower bath temperatures. The optical band gap is found to lie in the range 3.85 eV–3.95 eV.

4. Conclusions

Highly transparent, specular and adherent ZnS films were deposited successfully by chemical bath deposition technique with ammonia as the only complexing agent and thioacetamide as the source of sulphur. The effects of bath temperature as well as the concentrations of the bath constituents on the growth, structural and optical properties have been investigated to optimize the bath and deposition conditions. Considering the growth kinetics, the reason for the film peel-off from the substrate at higher zinc salt as well as thioacetamide concentrations is clearly explained. ZnS films of about 160 nm thickness could be obtained in a single-step deposition process in about 30 min at 60 °C with only one complexing agent. Structural characterization revealed that the films were nanocrystalline and the crystallite size was found to increase with increase in thioacetamide concentration. FTIR studies revealed the signatures corresponding to hydroxide and oxide groups indicating the presence of small amount of ZnO and $Zn(OH)_2$. The optical band gap of the films was found to decrease with increase in thioacetamide concentration owing to improvement in crystallinity. The band gap of the films under optimized deposition conditions is found to be 3.87 eV.

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