Effect of Triton X-100 surfactant on thiol-amine cosolvents assisted facile synthesized CdS thin films on glass substrate by spin coating method

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Abstract

In this article, the effect of Triton X-100 surfactant on the spin coated and vacuum annealed CdS thin films on glass substrates using thiol-amine cosolvents have been explored in details. The SEM study reveals that the surfactant assisted CdS films are uniform, homogeneous and smooth having less cracks, voids, pinholes compared to CdS films synthesized without surfactant. The X-ray diffraction spectra indicate the hexagonal structure of the surfactant-added films with (002) and (100) planes having relatively high intensity whereas a weak hexagonal (002) peak appears for the films deposited without surfactant. Two prominent Raman peaks of CdS thin films are observed at the wavenumber 207 and 212 cm^{-1} due to the multi-phonon vibration of hexagonal CdS. A relatively strong and broad FTIR peaks at 1647 and 670 cm^{-1} were observed due to the Cd-S bending and stretching vibration in the surfactant mediated films. Surfactant induced CdS films are nearly stoichiometric as manifested by the energy dispersive X-ray report. Finally, higher optical transmission and band gap have been achieved for the CdS thin films deposited with Triton X-100 surfactant. This study reveals that surfactant mediated CdS thin films with thiol-amine cosolvents can be exploited effectively in higher efficiency thin film solar cell technologies with low cost.

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Graphic abstract

Keywords CdS thin films · Thiol-amine cosolvents · Triton X-100 surfactant · XRD · SEM · FTIR

1 Introduction

Cadmium Sulfide (CdS) thin films have been commonly used as optoelectronic materials owing to the applications in photo-electrochemical, opto-electronic and large area electronic devices, semiconductor sensitized solar cells, transparent conductors [1–5], photo catalysts and field effect transistors (FETs) [6] as well. It is most widely used as the window layer in major second generation (2G) thin film solar cell (TFSC) technologies like Cadmium Telluride (CdTe), and Copper Indium Gallium di-Selenide (CIGS) based solar cells [1, 7–9]. However, surface morphology is a very important part of nanomaterials. Usually the crystalline structures, morphologies, and compositions influence the material properties of CdS thin films.

Surfactant is one of the most versatile compounds in the physio-chemical colloidal field of research [10, 11]. Surfactants can be utilized to modify material attributes like surface morphology, grain size, crystalline structure, brightness, porosity, pitting, internal stress, corrosion characteristics, even chemical compositions; thus, to enhance the power conversion efficiency of the TFSCs [12]. With no added water, Triton X-100 is a high-purity, water-soluble, colorless viscous fluid with a viscosity of ~ 270 centipoise at 25°C , which goes down to ~ 80 centipoise at 50°C . At

25°C it is soluble in water. It is supplied as 100% active ingredient [13, 14]. Triton X-100 surfactant has its application in hard surface cleaning [14], as wetting agent in coatings and paints [15, 16].

For the synthesis of CdS film, chemical bath deposition (CBD) is the most extensively used method [17–23]. Other than CBD, extensive diverse methods have been reported to deposit CdS thin-films including electrodeposition [24], spray pyrolysis [25], pulsed DC magnetron sputtering [26], molecular beam epitaxy (MBE) [27], chemical vapor deposition (CVD) [28], successive ionic layer absorption and reaction (SILAR) [29, 30], electron beam evaporation [31], pH-controlled growth solutions [32], spin coating [33] etc. Most recently, we have reported the direct solution-processed thiol-amine cosolvents assisted synthesis of air annealed CdS thin films using glass protector where Triton X-100 surfactant has been used for the uniform deposition of the films [34]. There are also few reports of the use of nonionic surfactants aided synthesis of CdS films [18, 35, 36]. To take away or resurgence of heavy, unusual, and costly metal ions, and organic contaminants from aqueous solutions, the use of various surfactants in metal ion flotation process has also been reported [37]. Throughout the last Duo decennial, effects of natural organic and synthetic inorganic surfactants on metal oxides including SnO_2 [38],

Ni(OH)₂ [39], TiO₂ [40], ZnO [41], WO₃ [42], metal chalcogenides such as CdS [18, 35, 36], PbS [43], CuS [44] have been inspected intensely. Capacity of the solubility of carbon nanotubes (CNTs) in the aqueous solutions of Triton X-100 surfactant has been reported [45]. The spin coating method is an easy cost-effective method to directly obtain sulfide films from sulfurizing oxyhydrate precursors [12]. Consequences of Triton X-100 surfactant aided development of Cu₂SnS₃ (CTS) thin-film by means of sol-gel spin coating technique has been reported [12] where Triton X-100 surfactant aided growth was believed as a promising means of getting superior conversion efficiency of CTS based TFSCs.

In the present work, we unravel the effect of Triton X-100 surfactant on the surface morphology, crystallographic structure, optical and electrical properties of spin coated and vacuum annealed CdS thin films with thiol-amine cosolvents.

2 Experimental details

2.1 Materials

CdS powder (99.9999%), Triton X-100 surfactant, Ethylenediamine and 1,2 Ethanedithiol were used to prepare

CdS solution. All chemicals were collected from Sigma Aldrich and were used without further modification. The chemical structure of Triton X-100, Ethylenediamine and 1,2 Ethanedithiol are shown in Fig. 1a Triton X-100 is an eminent example for nonionic surfactant types in industrial applications. Frequently used names for Triton X-100 are TX-100, Polyethylene glycol p-(1,1,3,3-tetramethylbutyl)-phenyl ether, Polyoxyethylene octyl phenyl ether etc. Triton X-100 is a trade name of Polyoxyethylene glycol octyl phenol ethers: C₁₄H₂₂O(C₂H₄O)_x having a polyethylene oxide chain containing x = 9–10 ethylene oxide (EO) units [13, 46] as hydrophilic tail and an aromatic hydrocarbon (HC) lipophilic as hydrophobic head. The HC group is a 4-(1,1,1,3-tetramethylbutyl)-phenyl group.

2.2 Preparation of the CdS precursor solution

Two types of CdS solutions were prepared with and without Triton X-100 surfactant in this work as shown in Fig. 1b. Ethylenediamine and 1,2 Ethanedithiol were mixed at a volume ratio of 10:1 and then 1 wt% CdS powder was added in the prepared cosolvents. In the first recipe, the CdS powder added thiol-amine solution was stirred for 15 h at 50 °C temperature and 460 rpm. After stirring, the color of the solution looks yellow which was partially dissolved as demonstrated in Fig. 1b. In the second recipe, totally

Fig. 1 **a** Chemical structure of Triton X-100 (TX-100), 1,2 Ethanedithiol and Ethylenediamine **b** CdS solution prepared using thiol-amine cosolvents at 50 °C and 450 rpm and **c** The optical images of CdS thin films

uniform dissolved CdS solution was achieved by the same temperature and magnetic stirring with adding 2–3 drops of Triton X-100 surfactant in the solution. The final color of the solution was faint yellow as also illustrated in Fig. 1b.

2.3 Deposition of CdS thin films

Single layer vacuum annealed CdS thin film was deposited onto glass substrate (1×1 inch) prepared using CdS solution by the spin coating method with and without surfactant. In the beginning to remove gross contamination, the glass substrates were cleaned with piranha solution. Then the glass substrate was cleaned using acetone for 10 min, Isopropyl alcohol (IPA) cleaned for 12 min and finally and Deionized (DI) water cleaned for 12 min and dried by air blower.

The CdS solution was spin coated on cleaned substrates at a speed of 2000 rpm for 35 s which is a preferential speed for the uniform and smooth film deposition [34]. Then the deposited CdS thin films were dried at 90 °C for 15 min on a hot-plate to take away the residual solvents. Finally, the CdS thin films were annealed in a low vacuum chamber (0.07 Torr) at 300 °C for 5 min for the crystallization of the films and to avoid oxidation. This annealing temperature was chosen as it offered comparatively better surface morphology, crystallinity as well as the optical properties than others films annealed below and above 300 °C using simple open-air glass protector annealing system [34]. The entire process of CdS thin films deposition is shown in Fig. 2.

Fig. 2 Complete deposition process of CdS thin films with and without surfactant using thiol-amine co-solvents with spin coated at 2000 rpm and annealed in low vacuum

2.4 Characterization of the deposited CdS thin films

The BRUKER DekTak XTL thickness profiler was used to measure the thickness of the CdS thin films. The surface morphological analysis was performed using scanning electron microscopy (SEM) (Carl Zeiss, Model: EVO-18 Research). The structure of the synthesized spin coated films was studied by X-ray diffraction (XRD) with CuK α radiation ($\lambda = 1.5418 \text{ \AA}$). To investigate the local structure and molecular arrangements of deposited films, the Micro-Raman measurement was used with an excitation wavelength 532 nm of Nd:YAG laser (inVia, Renishaw) having 800 lines/mm grating, CCD detector of $1.4 \text{ cm}^{-1}/\text{pixel}$ and Eclipse notch filter. The Fourier transform infra-red (FTIR) spectra were also monitored in the wavenumber range of $400\text{--}4000 \text{ cm}^{-1}$ to understand the bonding structure between Cadmium (Cd) and Sulfur (S) in the CdS thin films. The energy dispersive X-ray (EDX) study was used for the elemental chemical analysis. To study the optical transmission of the synthesized films in the wavelength range of 360–1100 nm, T60 UV–visible Spectrophotometer (PG Instruments) was used. The temperature dependent resistivity and Hall measurement were also performed on CdS thin films.

3 Results and discussion

3.1 Morphological studies of CdS thin films

Figure 3 depicts the scanning electron microscopy (SEM) pictures of CdS thin films spin coated at 2000 rpm for 35 s from CdS solutions with and without surfactant. In coating procedure where droplets are hastily generated and essential to be stabilized, the dynamics of surfactant adsorption has remarkable significances. From Fig. 3, it is perceived that the surface of the films prepared from solution with surfactant possesses higher uniformity and homogeneity with less cracks and pinholes compared to that deposited from the solution without surfactant. Reasons behind these are because the TX-100 surfactant decreases the CdS film surface roughness and interfacial tension between the glass and CdS film to a great extent. Obtained results are well matched with reported articles [12, 47, 48]. For that reason, facile synthesized CdS thin film using thiol-amine cosolvents system along with Triton X-100 surfactant suggests higher surface uniformity than the film surface deposited using the complicated and costly methods and other solvents.

Fig. 3 The SEM micrographs of CdS thin films of different magnifications deposited from CdS solution (a) with- and (b) without-surfactant and annealed at 300 °C for 5 min, respectively

3.2 Structural studies of CdS thin films

3.2.1 XRD study of CdS thin films

The X-ray diffraction (XRD) patterns of CdS thin film synthesized at 2000 rpm for 35 s using CdS solution with thiolamine co-solvents and annealed at 300 °C for 5 min are exposed in Fig. 4. Surfactant mediated thin film showing 2θ equal to 13.20°, 15° and 25° corresponds to (002), (002) and (100) planes, respectively, revealing the hexagonal structure with high intensity compared to the film without surfactant [17, 49–54]. Although only (002) low intensity hexagonal plane were observed at $2\theta = 13.20^\circ$ and 15° without surfactant [49–51]. On the other hand, CdS thin films prepared with surfactant and annealed in air with glass protector has the prominent XRD peak at $2\theta = 20.5^\circ$ along (110) plane with cubic structure and other two weak peaks appeared at 15.5° and 23.1° with (002) and (100) planes, respectively with hexagonal structure [34]. It is evident from Fig. 4 that the intensity of all peaks increases for the CdS films with surfactant compared to that without surfactant. The (100) plane was disappeared for the CdS

thin films without surfactant. Therefore, TX-100 surfactant and vacuum annealing promote the growth of the aforementioned hexagonal planes. The results are consistent with earlier studies [12, 55, 56].

The crystallite sizes of CdS thin films were calculated from XRD data using Scherrer's equation [34]. The crystallite sizes for planes (002), (002) and (100) were found to be about 17.12, 15.80 and 11.50 nm, respectively for TX-100 assisted CdS film where the determined crystallite sizes surfactant free film at planes (002) and (002) were about 12.20 and 14.34 nm, respectively. Therefore, the XRD study indicates the synthesis of high quality CdS thin films by this facile spin coating technique from CdS solution with Triton X-100 surfactant.

3.2.2 Raman study of CdS thin films

Figure 5a shows the Raman spectra of CdS thin films deposited on a 500 μm thick silicon substrate (p-type: 1–5 $\Omega\text{-cm}$) with a coating speed of 2000 rpm and annealing at 300 °C for 5 min from a CdS solution with and without surfactant. The silicon substrates were cleaned using acetone, IPA and DI water, respectively prior to film deposition. It

Fig. 4 The XRD spectra of CdS thin films spin coated at 2000 rpm using CdS solution of with and without surfactant with thiol-amine co-solvents and annealed at 300 °C for 5 min

is revealed from the figure that the two Raman peaks are detected. The first prominent peak of CdS thin films is observed at the wavenumber 207 cm^{-1} due to multi phonon vibration of hexagonal CdS, which agrees well with the reported value [57–59]. Another prominent peak of CdS thin films appeared at the wavenumber 212 cm^{-1}

owing to E_2 multi phonon process of hexagonal CdS. This also matches well with the former reports [57, 58, 60].

Figure 5b shows the change in Raman peak intensity of CdS thin films with peak position prepared with and without surfactant, respectively. It is seen that the intensity of the both Raman peaks increases with surfactant, as shown in Fig. 5b which is also consistent with the XRD data. Thus, it can be inferred that from the Raman study, the surfactant mediated CdS thin films are superior to the films without surfactant.

3.2.3 FTIR study of CdS thin films

Figure 6 shows the FTIR spectra of CdS thin films synthesized with and without surfactant by spin coating at 2000 rpm and annealing temperatures 300 °C for 5 min using CdS solution in thiol-amine cosolvents. TX-100 surfactant has covalently bonded O-added hydrophilic groups that are attached to the hydrophobic parent structure. It is seen from the figure that there is a broad absorption band around a wavenumber of 3450 cm^{-1} which can be credited to the O–H stretching vibrations [61]. The absorption peak at 1647 cm^{-1} assigned owing to the Cd–S bending vibration and well agreed with reported works [62].

Fig. 5 The Raman spectra of **a** CdS thin films synthesized from CdS solution with and without surfactant annealed at 300 °C for 5 min and **b** The change of Raman peak intensity with peak position

Fig. 6 The FTIR spectra of CdS thin films synthesized with and without surfactant by spin coating at 2000 rpm using thiol-amine cosolvents system

Due to C-S vibrational mode, another peak assigned at 1383 cm^{-1} and C-N symmetry stretching indicated by the bands at 1120 cm^{-1} and C-O stretching vibration at 1001 cm^{-1} were observed, respectively and the result are in good agreement with earlier-reported works [63–65]. Another peak detected around 670 cm^{-1} is due to the Cd-S vibrational mode which agrees well with reported value of [66]. Moreover, the peak intensity increases with surfactant mediated CdS thin films which was also observed in XRD and Raman studies due to higher growth of CdS, supported by the surfactant.

3.2.4 The EDX study of CdS thin films

The chemical composition of CdS thin films was inspected via the energy dispersive X-ray (EDX) analysis.

To find an accurate chemical composition and check its uniformity, the analysis was performed at distinct spots. Figure 7a, b shows the EDX spectrum of spin coated CdS thin films on glass substrates with a speed of 2000 rpm and annealing temperature of $300\text{ }^{\circ}\text{C}$ for 5 min with and without surfactant, respectively. It shows that the films contain the elements Cd and S and the atomic % ratio is nearly 1(0.98) when surfactant was used as shown in Table 1. Therefore, it can be inferred that the deposited CdS thin films from CdS solution using thiol-amine cosolvents with surfactant are nearly stoichiometric with slightly enriched Sulfur (S). The results are in good agreement with recognizing the formation of the high-quality films with surfactant [67]. In contrast, a non-stoichiometric film of CdS was observed for CdS films without surfactant assisted deposition. As realized by these two spectra, a strong peak of Si was detected due to the glass substrate. At the high operating voltages, the electron beam enters the film and reaches the glass surface which outcomes the Si peak [68].

3.3 Optical properties of CdS thin films

Figure 8 delineates the optical transmittance spectra of CdS thin films prepared by spin coating technique at 2000 rpm and annealed at $300\text{ }^{\circ}\text{C}$ for 5 min from CdS solution with and without surfactant, respectively. The transmittance increases from 60 to 80% when surfactant is used. Therefore, CdS films with surfactant offer higher transmittance compared to that without surfactant. This may happen due to the uniform film surface as well as crystallization of the films for using surfactant [69]. However, the transmittance of CdS was found to be decreased swiftly for wavelengths below 550 nm. It should be worth noting here that high transmittance in the visible region is one of the key features for window materials.

Fig. 7 The EDX micrograph of CdS thin films (a) with and (b) without surfactant coated at 2000 rpm for 35 s and annealed at $300\text{ }^{\circ}\text{C}$ temperature for 5 min

Table 1 The EDX elemental composition of CdS thin film spin coated at 2000 rpm and annealed at 300 °C with and without surfactant, respectively

CdS thin films	Element	Weight (%)	Atomic (%)	Atomic error (%)	[Cd]/[S]
With Triton X-100 surfactant	C	9.01	9.29	±0.09	0.98
	N	5.63	6.08	±0.10	
	Si	20.12	21.01	±0.15	
	Cd	29.02	31.49	±0.23	
	S	36.22	32.13	±0.27	
	Total	100	100		
Without Triton X-100 surfactant	C	18.11	18.42	±0.13	0.85
	N	12.34	12.87	±0.16	
	Si	20.11	20.73	±0.19	
	Cd	21.19	22.10	±0.25	
	S	28.25	25.88	±0.22	
	Total	100	100

Fig. 8 The transmittance spectra of CdS thin films synthesized at 2000 rpm for 35 s and annealed at 300 °C for 5 min

The absorption coefficient (α) was considered from the transmittance spectra in the strong absorption region using Eq (1):

$$\alpha = \frac{\ln(100/T)}{d} \quad (1)$$

where T is the transmittance and d ($= 300$ nm) is the thickness of the film.

The high transmittance and the low absorption coefficient obtained for CdS thin films due to added surfactant in the wavelength area above 550 nm make them appropriate for window materials in solar cell. Since CdS is a direct band gap material, the band gap (E_g) of the CdS thin film was gained by extrapolating the linear portion of $(\alpha h\nu)^2$ versus $h\nu$ plot to $\alpha = 0$ as shown in Fig. 9. The obtained E_g of 1.95 eV increases to 2.4 eV when

Fig. 9 The plot of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) for CdS thin films coated at 2000 rpm for 35 s and annealed at 300 °C for 5 min

surfactant is used. Since the crystal quality improves with nanoparticle films deposited on the substrate surface, band gap energy is increased [69–71].

3.4 Effect of heat-treatment on the resistivity of CdS thin films

Figure 10 reveals the effect of heat treatment on the resistivity of 300 nm thick spin coated CdS thin film prepared from CdS solution with and without surfactant, respectively. The resistivity of the CdS thin films sharply fall at a temperature of 375 K for every cycle of heating–cooling processes whereas it does not implicitly change over a wide temperature range of 380–575 K during the heating–cooling cycles. It is also noticed from the figure that heat treatments are almost reversible after three cycles of operation in the investigated series of temperature from

Fig. 10 The effect of heat treatment on the resistivity of 300 nm thick spin coated CdS thin film prepared from CdS solution (a) with- and (b) without- TX-100 surfactant

300 to 575 K which confirms the stability of the CdS thin films.

4 Conclusions

CdS films were successfully deposited on glass substrates by spin coating method using CdS solution in Ethylenediamine and 1,2 Ethanedithiol cosolvents with and without Triton X-100 surfactant where annealing was performed in low vacuum. The synthesized films were characterized by XRD, EDX, Raman, FTIR, SEM, UV–vis etc. for investigating the morphology, molecular composition, crystalline structure and optical properties of the deposited films. The surfactant assists to form CdS films with better uniformity and homogeneity with nearly stoichiometric composition. Accordingly, structural, optical and heat-treatment properties of surfactant mediated CdS films are superior to the CdS films deposited without surfactant. Therefore, it can be concluded that surfactant mediated CdS thin films deposited by this simple and low-cost spin coating method may pave the way of fabricating very cost effective high efficiency CdS/CdTe and CdS/CIGS based thin film solar cells in near future.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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