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A short review of CdTe and CdSe films: growth and characterization

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Abstract: Cadmium based thin films including CdTe and CdSe show great potential for the use in the field of solar cells, optoelectronic and biomedical applications. This work gives a brief review of the preparation of CdSe, and CdTe films by using various deposition techniques. The chemical bath deposition method was used to deposit CdSe films using three precursors such as cadmium nitrate, cadmium sulfate and cadmium acetate are reported. Also, this article reviews on the experimental advances in electrodeposited of CdSe and screen printed cadmium telluride thin films. Various characterization techniques namely, X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDAX), scanning electron microscopy (SEM), UV-Visible spectrophotometer (UV-Vis) were used in the present research work have been discussed.

Keywords: chemical bath deposition, screen printing technique, electrodeposition, thin films, solar cells

Introduction

The production of selenides and tellurides thin films is inexpensive and simple. Some properties of these materials (CdSe and CdTe) are quite interesting and can be widely used in the optoelectronics [1], photoelectrochemical cell [2], solar energy cell [3], light-emitting diodes [4], photovoltaic devices [5], biomedical applications, thin film transistor, electroluminescent device, sensor, lasers, and gamma-ray detector. For example, solar cell application of CdTe in thin film form has achieved a commercial stage due to direct band gap [6,7] (1.2 to 1.7 eV) and its high absorption co-efficient for solar radiation [8]. The *n* and *p*-type semiconductors in bulk and thin film form [9] are strongly dependent on the CdTe growth condition.

There exists a wide variety of deposition methods (physical and chemical technique) available for the deposition of cadmium selenide and cadmium telluride thin films. This work presents the synthesis of CdSe and CdTe by using chemical bath deposition, screen printing technique, and the electrodeposition method. The properties of these materials will be briefing reviewed as well.

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Literature survey

Screen printed cadmium telluride thin films:

Recently, huge research has been demonstrated on the fabrication of high-performance photo-sensors (having high photo-responsivity and fast switching action) [10-12]. However, performances of devices are strongly influenced by properties of thin films. In order to improve the quality of thin films, the deposition technique such as closed-spaced sublimation [13], vacuum thermal evaporation [14,15], dip-coating [16], RF sputtering [17], chemical bath deposition [18], and screen printing [19] have been demonstrated. Among these, screen printing technique has drawn considerable attention due to its simplicity, low deposition cost [20], convenient method for large area preparation [21] of the thick films. This method was employed for coating surfaces of any geometry and morphology [22]. Apart from this, the polycrystalline nature of screen printed thin film makes this technique most appropriate for the fabrication of optoelectronic devices [23]. Moreover, the properties of obtained films can be modified by a sintering process.

The sintering temperature is one of the key parameters of this technique as it can change crystallinity as well as the cation/anion ratio. In

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crystalline form, CdTe has a cubic structure with a lattice constant of 6.487 Å and *Pmm2* space group [24].

For the deposition of thin films using screen printing technique, material preparation and subsequent sintering process are important steps. Material slurry was prepared using a mixture of 99.9 % pure cadmium telluride, cadmium chloride powders and ethylene glycol in order to synthesis cadmium telluride films. Small amounts of cadmium chloride (as 10 % by weight of CdTe) and ethylene glycol were added dropwise to make material slurry viscous enough to pass through the screen openings. Mixing and dispensing of mixture paste were achieved by a mortar and a pastel [25]. The obtained paste was printed over pre-cleaned glass substrates through the nonreactive screen (having uniform grids). The thickness of the prepared films was maintained by filling the substrate space kept unmasked between two layers of invisible tapes fixed along the sides of a glass slide (using squeegee in one glide). Printed films (4 µm thick) were then subjected to low temperature drying at 120 ℃, for 4 hours (by avoiding crack formation). Cadmium chloride ($CdCl_2$), is used to synthesize slurry and it works as a sintering flux. $CdCl_2$ has a melting point of about 568 ℃, but as it is hygroscopic in nature, the compound starts to evaporate at a temperature above 400 ℃. Thus, it satisfies the condition that sintering temperature must be greater than evaporation temperature [26] of sintering flux in order to make the deposited thin films free from it. Consequently, dried thin films were sintered at a temperature higher than 490 ℃.

Table 1. EDAX data of screen printed CdTe thin films

Sample	**495°C**		**Ratio**	**500°C**		**Ratio**	**505°C**		**Ratio**	**Total%**
Element	**Cd**	**Te**		**Cd**	**Te**		**Cd**	**Te**		**CdTe**
Weight%	70.92	29.08	2.43879	54.88	45.12	1.21631	53.05	46.95	1.12993	100
Atomic%	73.46	26.54	2.7679	58	42	1.38095	56.2	43.8	1.28311	100

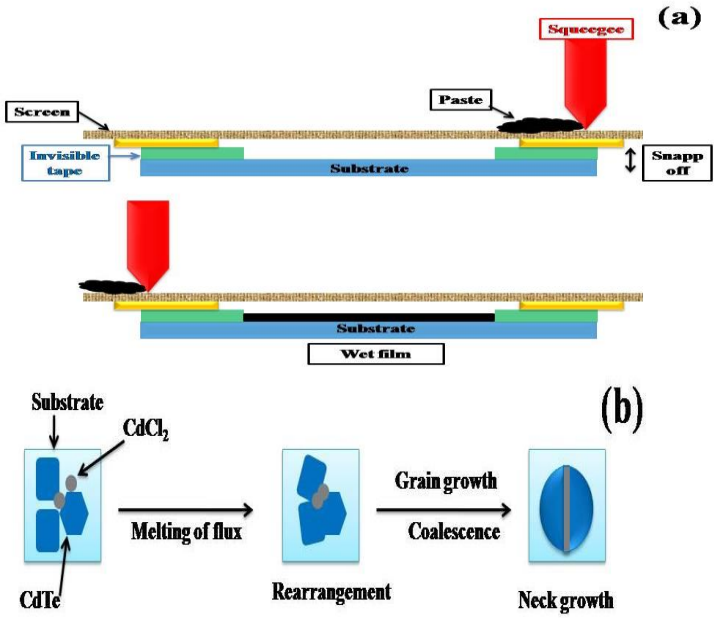

Figure 1. (a) Experimental set-up for screen printing and **(b)** growth stages of the deposited CdTe thin film.

Here, the slurry is composed of a main compound (CdTe), sintering flux ($CdCl_2$) and binder (ethylene glycol) which is further used to the printing of wet thin layer. It was followed by a low temperature drying to maintain continuity of the printed thin film. In next step, this dried film undergoes a sintering process, where sintering flux melts and creates shell surrounding the CdTe grains. Under a specific temperature-time cycle the compound first go through a process of rearrangement of grains in the presence of liquid phase of flux followed by dissolving small grains in to the flux to initiate the formation of large grains. This further promote coalescence of re-growth of grains and at the last stage of sintering neck growth between grains takes place that yields in to the formation of uniform and crack-free CdTe thin films on condensation [27]. At the end of the sintering process, the sintered flux as well as binder should be completely evaporated. The experimental set-up and growth stages of screen printed CdTe thin films are shown in Figure 1 (a) and (b).

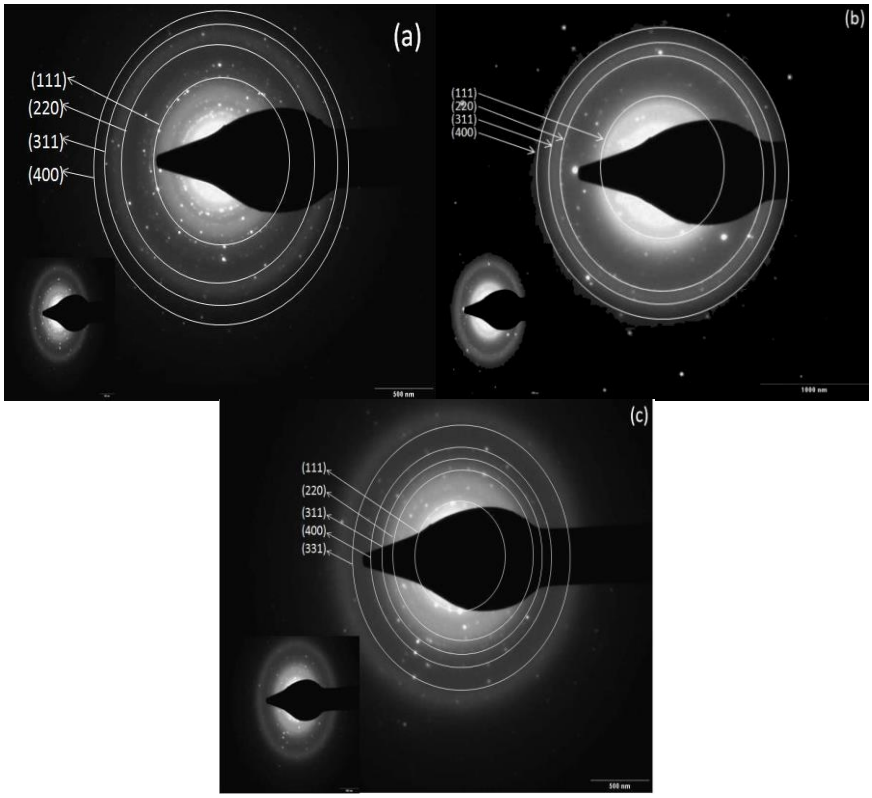

Figure 2. Selected area electron diffraction pattern (SAED) images with indexing for CdTe thin films sintered at (a) 495 ℃ (b) 500 ℃ (c) 505℃

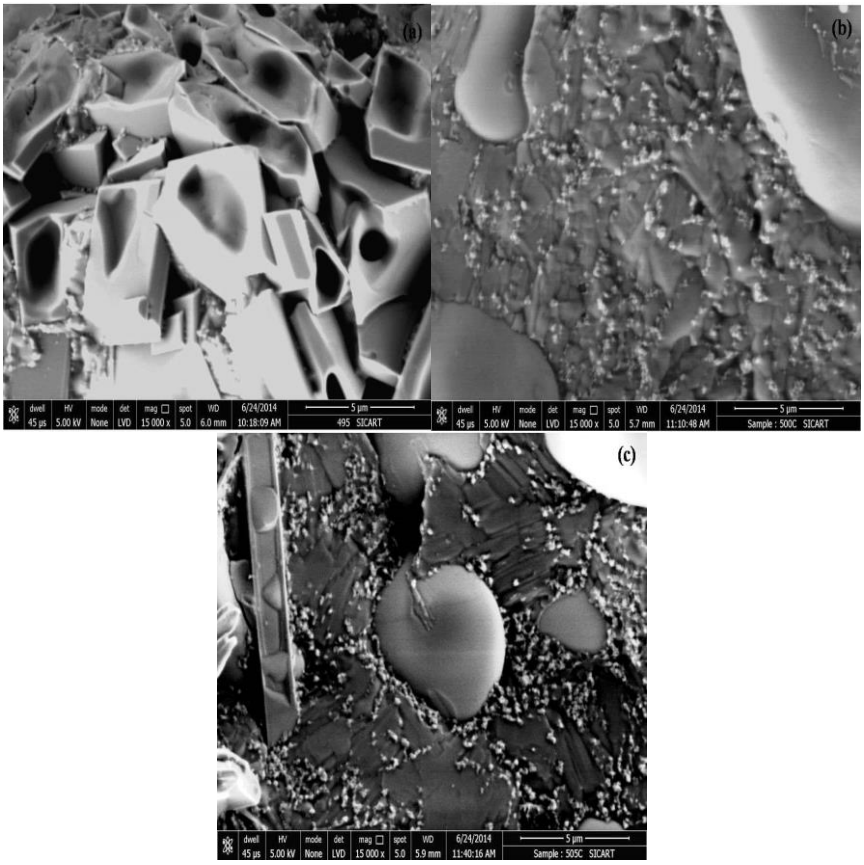

Figure 3. SEM micrographs of CdTe thin films sintered at (a) 495 ℃ (b) 500℃ and (c) 505℃.

The effect of sintering temperature (495, 500 and 505 ℃) on the stoichiometry of screen printed CdTe thin films is studied using energy dispersive X-ray spectroscopy (EDAX), as shown in Table 1. The ratio of atomic (%) improved through variation of sintering temperature which in turn can suppress the chalcogen vacancies. The diffraction spots along with rings, as observed in SAED pattern (Figure 2), depict the crystalline phase mixed with amorphous structure of deposited thin films. The sintering process initiates the crystallization, and hence improves the surface morphology as shown in SEM micrographs (Figure 3).

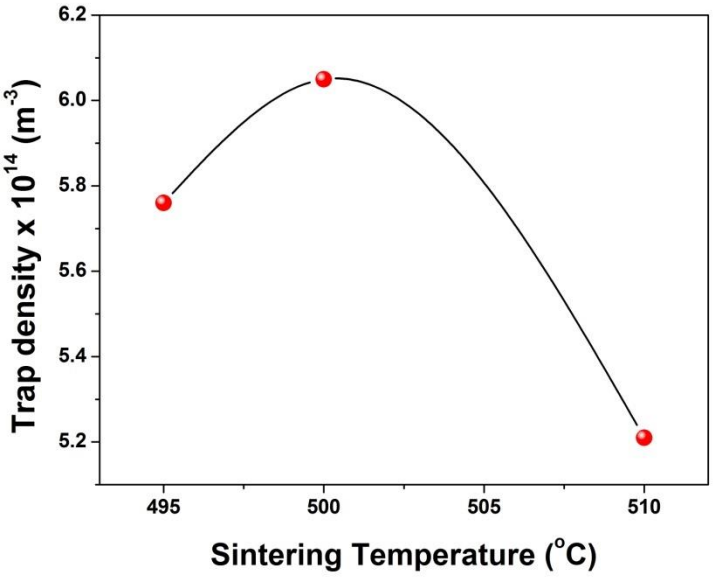

Figure 4. Variation of trap density with sintering temperature.

Conductive adhesive silver paste (copper wires are used for making contacts) was used in order to study the charge carrier conduction mechanism. The *V-I* characteristics were measured and the obtained data were analysed using space charge limited current (SCLC) theory to evaluate trap density. Figure 4 shows the variation in trap density with sintering temperature, suggesting that the performance of the device can be tuned by controlling trap density using proper sintering temperature.

Electrodeposited cadmium selenide thin films:

Cadmium selenide films have been prepared using various methods including thermal evaporation [28], chemical bath deposition [29], spray-pyrolysis [30], and electrodeposition [31] methods. Among these techniques, electrodeposition method is an attractive method for depositing semiconductor films, owing to its cheap [32], low temperature [33], single step process, large area deposition capability and high through-put technique for fabricating nanostructured thin films. It is the conventional process of coating a thin layer [34] of one metal on top of a different metal to modify its surface properties [35] by improve heat tolerance, reduce wear and friction. Electric current [36] was employed to reduce the cations [37] from an electrolyte and coat those materials (as a thin film) onto the surface of the substrate. Nowadays, this technique is already a major technology for mass production of large-area metallic protective coatings in industry, and photovoltaic as well.

For the deposition of CdSe thin films, indium tin oxide coated (ITO) glass slide (sheet resistance 10 ohm.cm^{-2}) was used as a cathode, and a graphite rod was used as an anode. An electrolytic bath containing 0.08 M of cadmium chloride and 0.005 M of selenous acid. The solution was then stirred for 15 min (using a Teflon coated magnetic paddle and stirrer) to ensure that all precursors were completely dissolved in distilled water. The pH was adjusted to 1.9 by adding nitric acid. The deposition was carried out in two electrode electrodeposition system at depositional potential 1.85 V under various deposition times (1, 2, 3 min). After the deposition, the films were washed with distilled water and dried in air for few minutes. Then, the as-deposited films were annealed at 450 ºC in the air for 60 minutes (in a muffle furnace) with a ramp rate of 2 º Cmin^{-1} to raise the temperature and followed by normal cooling to room temperature.

Figures 5 (a)-(c) show the XRD patterns of CdSe thin films prepared under various different deposition times. CdSe films are polycrystalline with the hexagonal (wurtzite) crystal structure. The films deposited at a shorter time (1 and 2 minutes) show two diffraction peaks corresponding to crystalline planes (002) and (201).

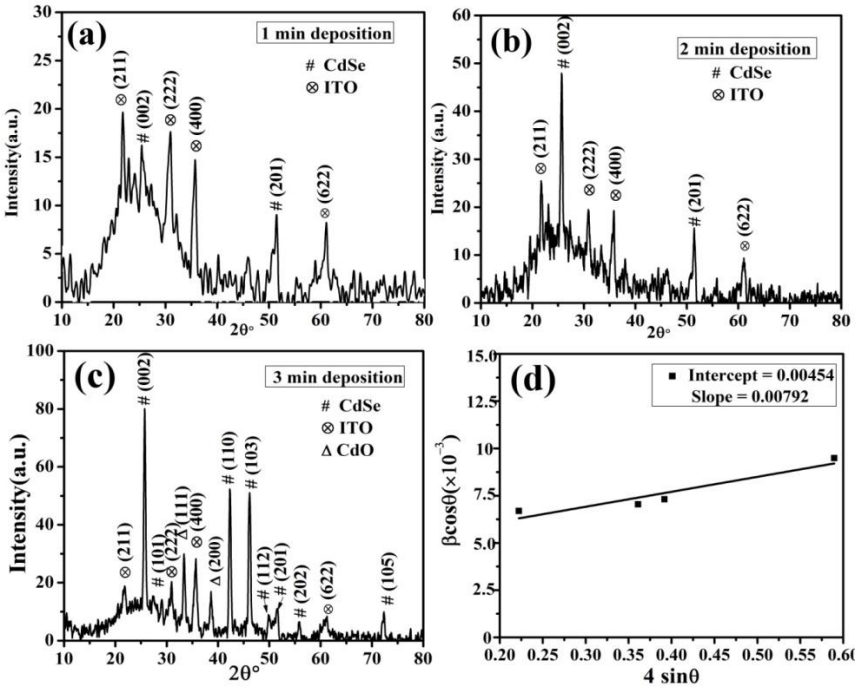

Figure 5. [(a) (b) and (c)]: The X-ray diffraction patterns of CdSe thin films prepared at different deposition times (1, 2 and 3 minutes) [(d)] The W-H analysis of CdSe thin films prepared for 3 min.

The number of CdSe peaks increased to eight for the films prepared for 3 minutes. The intensity of the peak (002) increased as the deposition time was increased also. However, the presence of two CdO peaks indicating less favourable conditions for the formation of CdSe films. In the background, a small hump was found on as-deposited CdSe thin film and it was due to the partial amorphous nature of ITO coated glass substrates [38]. The particle sizes are calculated by Williamson-Hall [39].

$$\beta_{hkl}cos\theta = \frac{K\lambda}{D} + 4\varepsilon sin\theta \qquad (1)$$

Where D is the particle size in nanometers, λ is the wavelength of the radiation (λ=1.5406 Å), K is a constant and θ is the peak position. From this formula, the average crystalline size is found to be about 30 nm for the films prepared for 3 min (Figure 4d).

Figure 6 shows the optical absorbance data for the CdSe films prepared at different deposition times. It is clear that the films prepared at the longer time (3 minutes) possess higher absorption if compared to other deposition times. This could be due to more material successfully deposited onto the substrate surface; the thicker film could be formed. The absorption data have been analysed using the following relation for near edge optical absorption of semiconductors.

$$\alpha = \left(\frac{K}{h\nu}\right)[h\nu - E_g]^n \qquad (2)$$

Where α is absorption co-efficient, $h\nu$ is the photon energy, K is a constant value, E_g is the band gap and n is a constant (n equals to 2 for allowed indirect semiconductor) [40].

Figures 6(b, c, d) show the plot of $(\alpha h\nu)^2$ versus $h\nu$ for CdSe films prepared at different deposition times. The band gap value could be determined by extrapolating the linear portion of these plots to the energy axis. It was observed that the band gap values reduce (from 2.43, 2.28 to 1.93 eV) as the thickness of the film increased (from 0.6, 2.5 to 6 µm).

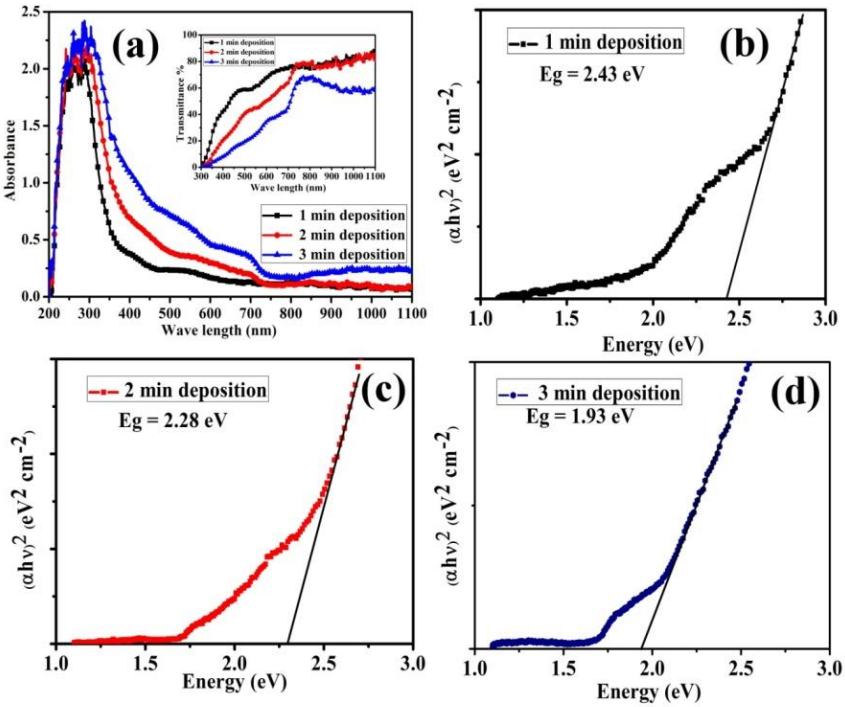

Figure 6. (a) UV-Visible absorption and transmittance spectra of CdSe films prepared at different deposition times. (b), (c) and (d) Tauc plots for CdSe films.

The surface morphology of obtained films was investigated using field emission scanning electron microscopy (FESEM) as shown in Figures 7 (a, b, c). FESEM studies indicate that the film thickness increased (the average grain size also increases [41]) as the deposition time was increased [42]. The grains somehow produced compact morphology structure over the substrate, dense packed, homogeneous and crack free. CdSe films prepared at 1 minute were non-uniform, irregular shape, and less dense hexagonal form (Figure 7a, 7a'). However, films prepared at 2 minutes were more homogeneous and extremely dense (Figure 7b, 7b'). The closely packed grains provide a pinhole-free morphology could be seen for the films prepared at 3 minutes (Figure 7c, 7c').

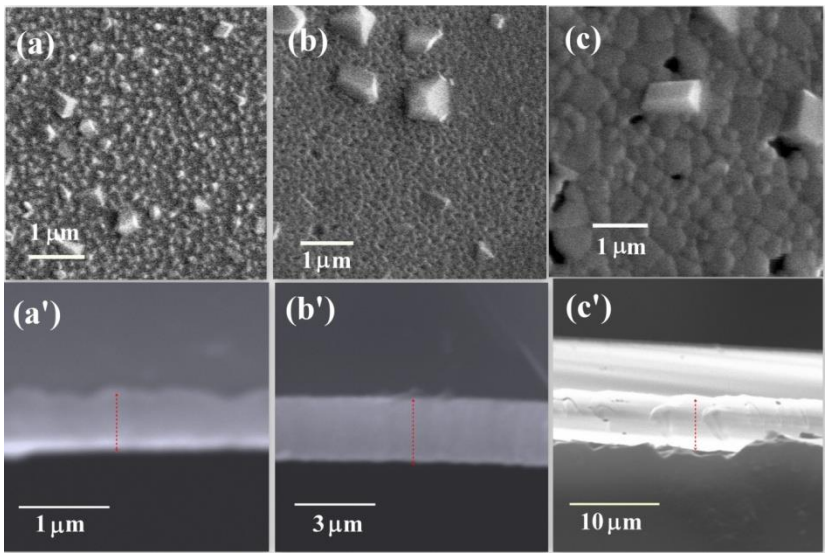

Figure 7. (a), (b) and (c) are the surface morphology and 2(a'), 2(b') and 2(c') are the cross-section images of FESEM micrograph of CdSe thin films

Chemical bath deposited cadmium selenide thin films:

Chemical bath deposition method is a slow process. Nowadays, there are many researchers [43,44] select chemical bath deposition method [45,46] to produce thin films [47-49] onto various substrates such as indium tin oxide [50-53], fluorine-doped tin oxide [54-56], a microscope glass slide [57-60] and soda

lime glass [61-63]. It has many advantages over the other ones such as low cost, large area production in lower temperature, and simple process. The process of precipitation of a substance from the solution onto substrate depends mainly on the formation of nucleus centre and subsequent growth of a film [64].

The *n*-type chemical bath deposited CdSe films were deposited on glass substrate at 70 ℃ having a thickness of 1000 nm from a solution containing sodium selenosulfate (Se^{2-} ion source), triethanolamine (complexing agent) and cadmium nitrate (Cd^{2+} ion source). The surface morphology of the as-deposited CdSe films was observed to be rods and accumulation grains [65]. The Hall Effect measurements were conducted at room temperature indicate the band gap, carrier concentration, mobility and resistivity value of as-deposited films are 1.81 eV, 1.53 X 10^{12} cm^{-3}, 39 $cm^2V^{-1}s^{-1}$, and 1.07 X 10^5 Ω.cm, respectively. Scanning electron microscopy studies reveal that the corn-like nanowire with the diameter of 30-50 nm could be observed at higher annealing temperature (300-500 ℃), can improve the efficiency of the solar cell. Highly crystallized hexagonal CdSe films were synthesized at pH 10, 180 minutes and 50 ℃ [66]. Nanorod morphology (length range from 20-85.5 µm, width range from 1.8-10.9 µm) with variable film thickness (465 to 605 nm) was successfully prepared using a chemical bath deposition method. Optical properties such as refractive index (1.16 to 1.54), extinction coefficient (5.1 X 10^{-4} to 4.9 X 10^{-3}), real dielectric constant (2.36 to 1.36) and band gap (1.7 to 2.3 eV) were investigated under various pH values (pH 10-13). Kariper [67] conclude that increasing the pH from 7 to 10 could increase the film thickness (74.6 to 138.2 nm) because of the presence of hydroxide ions in the media. Further, the researcher explains that transmittance (16.7 to 52.5 %) and reflectance (16.7 to 39.7 %) data did not vary based on film thickness due to either cadmium or selenium was dominant in specific experimental conditions.

Influence of annealing process on the properties of CdSe films was studied by Bakiyaraj and Dhanasekaran [68]. The crystallinity (cubic to hexagonal), morphology (spherical shaped to nanorod with star shaped) and band gap (2 to 1.9 eV) have been changed by annealing process (450 ℃ and 60 minutes). CdSe films were deposited on fluorine-doped tin oxide using chemical bath deposition and electrodeposition method [69]. The obtained results reflect that thicker film (900 nm), larger particle size (6.9 nm), higher power conversion efficiency (1.72 %) and PL intensity than the chemical bath deposited films. They confirm that electrodeposited films have a lower bandgap due to bigger particle size and the emission wavelength (photoluminescent) becomes longer. Chemical bath deposited CdSe films were prepared using raw materials such as cadmium acetate, tartaric acid and sodium selenosulphate [70]. These films well adherent to the substrate, red in colour and pin-hole free. The percentage of atomic of Cd:Se was 49.4:50.6 based on the EDAX analysis. The obtained films indicate a preferential orientation along the (111) cubic phase of CdSe at the position of 25.5°.

The chemical bath contains sodium citrate, cadmium chloride and sodium selenosulphate was used to synthesize homogeneous stoichiometric CdSe films with various thicknesses (400-500 nm) on a glass substrate for 4 hours [71]. Structural analysis showed that the deposited films at 70 and 80 ℃ have hexagonal with a crystallite size of 20 nm while cubic structure (crystallite size of 4 nm) at room temperature. The high quality of CdSe films have been prepared on a glass substrate using cadmium sulfate, ammonia (complexing agent) and sodium selenosulphate, under magnetic stirring, at 60-70 ℃. The obtained films exhibit reddish in colour, homogeneous, adherent and optically transparent. SEM analysis showed the size and shape of nanoparticles mainly depended on experimental conditions. As shown in XRD patterns, all the diffraction peaks of cadmium selenide films such as (111), (220) and (311) plane can be indexed to the cubic structure [72]. The band gap is found to decrease from 3.52 to 1.84 eV with the increase of deposition temperature from room temperature to 50 ℃. Rutherford backscattering spectroscopy technique indicates the excess of cadmium ions rather than selenium ions by depth profile viewing the concentration of films.

Conclusion

In this article, we present a short review on the preparation of CdTe and CdSe films. The widely used preparation methods such as chemical bath deposition, electro deposition method and screen printing technique are surveyed. The obtained films have attracted much interest in different electronic and optoelectronic devices, due to very low production costs. Optical properties show that band gap values are strongly dependent on film thickness.

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