SYNTHESIS AND CHARACTERIZATION OF PVP-CAPED CADMIUM SULFIDE (CDS) THIN FILMS BY DIP COATING METHOD

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Abstract

Cadmium sulfide (CdS) thin film was prepared on glass substrate by a chemical bath deposition dip coating technique. Precursors are aqueous cadmium chloride,Distilled water, thiourea are the basic medium, Polyvinylpyrrolidone (PVP) as a dispersant, Ethylene glycol (EG) as a solvent and monoethanolamine as stabilizing agent respectively. The abovesolutions was prepared and maintained under 80°C for 2 hours stirring time. After prepared (CdS) thin films was annealed with different annealed temperatures at 350°C, 400°C and 500°C for 2 hours. Synthesized thin films have been characterized with structural and morphological and optical properties of films obtained by CBD using X-ray diffraction (XRD), Scanning and Electron Microscope (SEM) and Ultraviolet Visible spectrophotometer (UV-Vis). X-ray diffraction studies shows the orientation analysis that the prepared samples have the hexagonal crystal structure that the CdS thin film deposited with capping agent was grown on an amorphous phase. SEM image reveals grains tied up in a fibrous-like porous structure uniformly distributed over the surface of the substrate for the CdS films. CdS thin film deposited homogeneous, uniform grain size and good adhesion to the substrate.

Keywords: CdS thin films, XRD, SEM, and UV-Vis, Dip coating

1. Introduction

Cadmium sulfide (CdS) is an important II–VI groupsemiconductor due to its direct band gap transition with band-gap energy of 2.42 eV at room temperature and unique electrical, optical and photoconductive properties, supplying for wide technologyapplications, such as thin film solar cells[1], photo-electro catalysis devices [2] and other semiconducting devices [3].There are various methods reported in the literature to fabricate nanoparticles by physical [4, 5], chemical methods [6] and ion implantation [7].Different techniques have been used to deposit CdS thin films such as RF sputtering, pulsed laser deposition, molecular beam epitaxial, thermal evaporation, sol-gel spin coating, sol-gel dip coating, electrochemical deposition, and spray-pyrolysis [8–10].sol gel is commonly used because it is a simple, inexpensive and reliable process that produces uniform and easily adhering films and this work focuses on thin CdS films deposited with sol-gel dip coating techniques on glass substrates [11]. The effect of annealing temperature on the structural and optical properties of CdS thin films using waste water was investigated. In the present study we have synthesized CdS thin films through sol-gel dip coating technique. CdS thin films are extensively characterized using X-Ray diffractometer XRD, SEM, and UV–Vis spectrophotometer.

2. Experimental

2.1. Preparation procedure

Chemicals: cadmium chloride, thiourea, ethylene glycol, monoethanolamine, Polyvinylpyrrolidone. The synthesis of CdS Nano crystals was performed as follows: 1.141 g cadmium chloride ($CdCl_2$) was dissolved into 50 ml ethylene glycol with 1.3 ml monoethanolamine and 0.1 g Polyvinylpyrrolidone (PVP). The **'S'** precursor solution was prepared by dissolving 0.0761 g thiourea (CH_4N_2S) into 10 ml ethylene glycol.

Then the **'Cd'** precursor solution was added with above solution under 80^{o}C for 2 hours stirring time.The pre-cleaned substrates immersed vertically in to prepared CdS solution for 5 min dip durations and dried 5 min at 90^{o}C, the above dip and dry process were repeated 10 times to improvethe film thickness by using automatic dip coating unit with infrareddryer and as-deposited thin films were annealed at 350°C, 400°C and 500°C for 2 hours in muffle furnace, in order to improve the crystalinity.

2.1. Characterization techniques

The structural properties are analyzed using X-ray diffraction (XRD, JEOL diffract meter) with Cu Ka radiation (k = 1.54056 Å).The morphology is observed by SEM (JEOL Model JSM–6390LV).The optical study is measured by UV–Vis spectrophotometer (JASCO V-500) in the wavelength range of 200–800 nm.

3. Results and discussion

3.1. XRD analysis

The XRD patterns of the samples synthesized at as deposited, 350°C, 400°C, and 500°C samples. The XRD diffraction (h k l) peaks are observed at 2θ values of 24.80^{0}, 26.40^{0} and 28.20^{0} corresponding to the lattice planes (100), (002) and (101) respectively at 350°C . Peaks are observed at 2θ values of 24.60^{0}, 26.35^{0} and 28.17^{0} corresponding to the lattice planes (100), (002) and (101) respectively at 400°C. Peaks annealed at 500^{0}C shows 2θ values of 24.74^{0}, 26.43^{0}, 28.18^{0} and corresponding to the lattice planes (100), (002), (101) and (110) respectively. They are in agreement with the standard JCPDS 065-3414 card for hexagonal wurzite CdS. The intensity of the peak is observed at thin film annealed at 500°C is higher than the film annealed at 400°C. The observed sharp peak compared to bulk CdS sample indicates the presence of good crystalline nature, which is good for photo catalytic reaction. The

Crystalline size is calculated by Scherrer's equation [12].

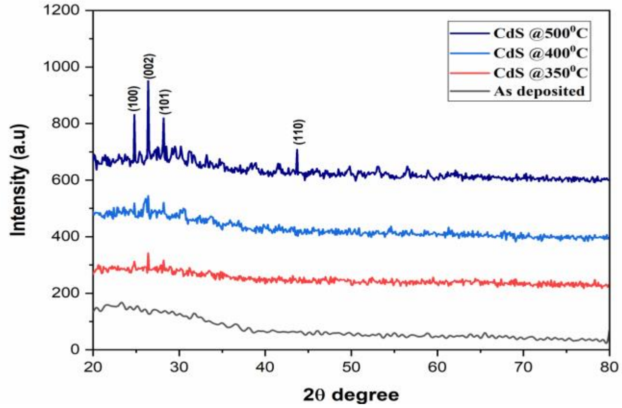


Fig. 1 Comparison of X-ray diffraction pattern of CdS thin films as deposited,

annealed at 350^{0}C, 400^{0}C and 500^{0}C

From the above XRD pattern it is clearly seen that as the annealing temperature increases from 350^{0}C, 400^{0}C and 500^{0}C the intensities of the (100), (002) and (101) diffraction peaks are gradually increased. Particularly at 500^{0}C annealing temperature, the intensity of (002) peak is higher when compared to other peaks. XRD shows that the annealing temperature of 500^{0}C has better wurzite structure with c-axis orientation.

The average crystallite size of the undoped CdS thin films for various annealed temperature 350^{0}C, 400^{0}C and 500^{0}C were calculated. The average crystallite size is found to be 7.83nm, 12.47nm and 31.72nm respectively. From the above result as annealing temperature increases from 350^{0}C, 400^{0}C and 500^{0}C the crystallite size has been increased. This implies that the annealing temperature influence the particle size.

3.2 SEM images

Fig.(2)shows the SEM image of prepared sample of CdS thin films at different annealing temperature. Morphology of CdS thin films consists of nanorod structure. Whenincrease annealing temperature morphology become spherical into small cubic structure on Morphology. The SEM image show uniform surface with well-defined grain boundaries with very smoothand the crystalline size are fine. No cluster formation is observed and the grains appear homogeneous and uniform nucleation throughout the surface.

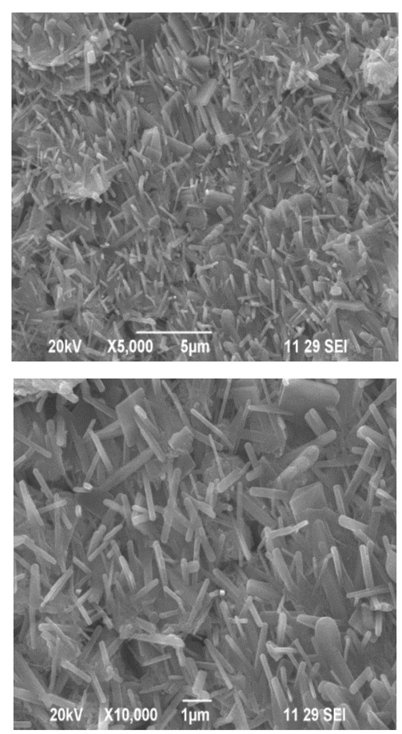


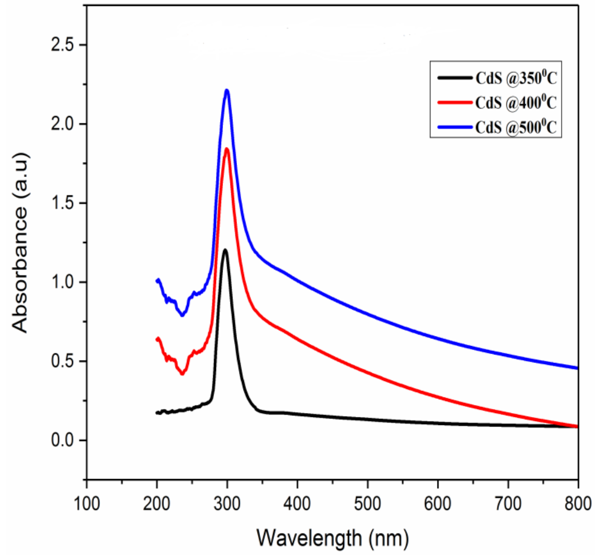
Fig. 2 SEM image of Undoped CdS thin films

While increasing the annealing temperature, the grains become denser and larger it can be considered as an amalgamation process by the thermal treatment. It is believed that annealing treatment causes film crystallization and removal of organic residue from as deposited thin films. The high magnification of SEM image reveals that the product consists of as deposited and annealed CdS thin films have relatively even grain size, but increase in density.

From the above results of all SEM images is shown in Figure 2 clearly seen that, at annealing temperature of 500⁰C, best nanorods were grown. As annealing temperature increases the grains have been agglomerated with increase in width and height.

3.3 UV-Vis Absorbance and Transmittance study

Optical properties were characterized by UV–Vis absorbance and transmission spectra analysis shown in Fig. 3 (a & b). Prepared samplesare strongly absorbs in UV region and highly transparent in the visible region and a sharp fall in transmission is observed below 300 nm region. It is found that the absorption edge shifts toward longer wavelength with annealing (500°C) samples [12&13].



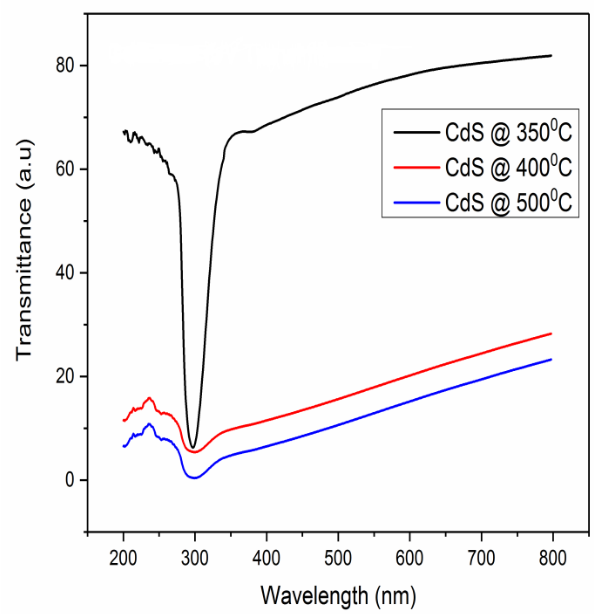


Fig. 3 (a & b) UV–Vis absorbance and transmission spectra of CdS undoped thin films

UV absorbance peaks were found at 294 nm, 288 nm and 300 nm for the samples annealed at 350⁰C, 400⁰C and 500⁰C respectively. The absorbance peaks is shifted to a higher wave length when annealing temperature is increased. As we know that the optical absorbance determines the optical band gap and CdS had a direct band gap.

Optical band gap of CdS thin films was found with increasing in annealing temperatures at 350⁰C, 400⁰C and 500⁰C, while the band gap decreases from 4.18 eV, 4.10 eV to 4.03 eV respectively. The optical transmittance percentages of prepared CdS thin films were found to decrease from 82% to 23% for the increasing annealing temperature of 350⁰C, 400⁰C and 500⁰C. The prepared CdS thin films at 500⁰C shows smaller band gap and maximum UV absorbance intensity and hence the annealed temperature were optimized as 500⁰C for further studies, towards photocatalytic application.

4. Conclusion

Cadmium sulfide (CdS) thin films deposited on glass substrates at various temperatures 350°C, 400°C and 500°C by Sol-Gel dip coating technique. The influence of annealing temperature on structural, optical and photo catalytic properties was investigated.The structural and optical studies were confirmed prepared CdS thin film at 500°C more suitable for dye degradation. The average crystallite size is found to be 7.83nm, 12.47nm and 31.72nm. Annealing temperature increases from 350⁰C, 400⁰C and 500⁰C the crystallite size has been increased. This implies that the annealing temperature influence the particle size.

SEM images shown as annealing temperature increases the grains have been agglomerated with increase in width and height. Optical band gap of CdS thin films was found with increasing in annealing temperatures at 350⁰C, 400⁰C and 500⁰C, while the band gap decreases from 4.18 eV, 4.10 eV to 4.03 eV respectively. Furthermore CdS thin film at 500°C shows excellent degradation performance for MB dye owing of large surface to volume ratio, crystalline size, and reduced band gap which promote electron hole pair under visible light. Hence based on this investigation we concluded CdS thin films annealed at 500°C acts as potential catalyst for wastewater treatment.

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