Chapter V

Photoelectrochemical Properties of Zn$_2$SnO$_4$ /CdS Thin Films
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Photoelectrochemical Properties of Zn$_2$SnO$_4$ /CdS Thin Films

5.1 Introduction

5.1.1 Construction of PEC solar cell

A PEC solar cell is an electrochemical cell in which one or both electrodes are semiconductors such that irradiation of the semiconductor results in the non-spontaneous flow of current in the external circuit. This type of cell is different from a photogalvanic cell in the manner that light absorption occurs in the solution instead of electrode. Figure 5.1(a) represents PEC cell that has elements like semiconductor photoanode, an electrolyte and a counter electrode. The photoelectrode and counter electrode are 1 cm. apart.

![PEC Cell Diagram]

Figure 5.1 (a) - A typical electrochemical photovoltaic cell and (b) - Current flow and energy level diagram for n- semiconductor PEC cell.

5.1.2 Requirements of PEC cells

Good performance of the PEC cell is achieved by following requirements.

5.1.2.1 Semiconductor photoelectrode

The semiconductor photoelectrode is the important part of the PEC cell. The property of a semiconductor photoelectrode shows the changes in the electrical properties of a cell. The electrons send to the conduction band flow towards the interior, while the holes, the minority carriers, come to the surface of the semiconductor.

1. The maximum region of the solar spectrum should be covered by the band gap ($E_g$) of the photoelectrode materials.
2. It should have high stability in the dark as well as under illumination.
3. It should be of the direct band gap type along with high optical absorption coefficient.
4. There should be high mobility and life time of charge carriers in the material.
5. Thickness should be sufficiently large to absorb all the incident radiation.
6. Cost of manufacturing and efficiency should be agreed.
7. There should be large diffusion length of minority carriers.
8. Shunt resistance $R_{sh}$ should be large enough and Series resistance $R_s$ should as small as possible ideally $R_s = 0$ and $R_{sh} = \infty$.

5.1.2.2 Electrolyte

Electrolyte is an important parameter in the PEC cell. Electrolytes consist of the oxidized species and the reduced species. Mixtures of totally ionic molten salts and molecular inorganic solvents with supporting electrolytes have been introduced for aqueous electrolytes.

Requirements of electrolytes for the PEC cells:
1. There should be proper Reduction-oxidation reactions to match with the semiconductor band edges.
2. Charge transfer rates of oxidized and reduced species should be high at both semiconductor and counter electrode.
3. There should be high photo and thermal stability of oxidized, reduced species and solvent components over useful solar spectrum and operating temperature range.
4. Supporting electrolyte concentration and oxidized, reduced species in
5. Solvent should be enough to reach required current densities.

5.1.2.3 The Counter electrode

For better performance in the PEC cell, the requirements of the counter electrode are:
1. The counter electrode should not react with electrolyte.
2. Fast charge transfer between redox species in electrolyte and counter electrode.
3. To avoid concentration polarization the area should be large.
4. The low reduction potential reaction is desirable for counter electrode.
5. It should have low cost. The most commonly used counter electrodes are platinum and graphite.

5.1.3 Survey of Literature

5.1.3.1 Survey of literature on CdS
Since last 60 years, metal chalcogenides (sulphides, selenides and tellurides) have been studied for their important applications such as photoconductive cells, photovoltaic cells and other optical devices. CdS thin film is one of the important examples of film SC electrodes. CdS nanocrystalline thin films are used as window material for CdS/CdTe solar cells and it continues as a subject of intense research due to its possible application in solar cells [1-5].

5.1.3.2 Literature survey of chemical bath deposited CdS thin films
CdS thin films have been prepared by several techniques, such as electrodeposition, chemical bath deposition (CBD), physical vapour deposition and screen printing (SP). Today, there has been an urgent need for efficient, low temperature and low-cost deposition methods for thin films. Chemical bath deposition (CBD) is a soft solution process capable of producing high-quality thin film at relatively low temperature. The CBD growth mechanism consists of different stages like mass transport of reactants, adsorption, surface diffusion, reaction, nucleation and growth. Uniform, adherent and large-area thin films can be produced by simple and low cost CBD method for thin-film electronics applications such as solar cells [6]. The first application of CBD was reported by Reynolds [7] for the fabrication of lead sulphide photoconductive detectors in 1884. Chopra reported the first general review for CBD [8] in 1982. Later on Lokhande [9] published a review on the deposition of metal chalcogenides. Lincot [10] in 1998 reported review along with growth kinetics analysis. Nair [11] and Savadogo [12] reported reviews on solar energy-related issues. The characterization of the post-deposited films and optimization of the deposition processes are equally important together with deposition technique. Many researchers worked on preparation of CdS thin films to obtain smart optoelectronic properties.
Kaur et.al and Pandya et al. reported the CdS as a window layer in CIGS cells [13, 14]. The band gap of the window layer should be high and should possess low thickness to keep low series resistance so that it could be used in hetero-junction solar cells. The high band gap and low thickness of CdS thin film by wet chemical method was tried by number of investigators. Mokrushin et al. and Kitaev et.al studied the potential application of CdS [15, 16]. Moutinho H.R. and co-workers deposited CdS thin film by close-spaced sublimation (CSS) and chemical bath deposition (CBD), to study why CBD CdS is used to obtain the best PV devices. Unlike the CSS CdS, CBD CdS films grew conformal on the underlying SnO$_2$ film, which was used as a front contact. The conformal growth encourages a film with a more uniform thickness, minimizing shunting paths [17]. Ramanathan et al. reported about 19 % efficiency of CIGS with CdS buffer layer [18].

The quantum size effect is the increase in the band gap of semiconductor due to a decrease in the particle size. Size quantized CdS thin films are applied for the window layer in solar cells. Louis Brus and co-workers reported experimental and theoretical work in the size-dependent development of bulk electronic properties in semiconductor crystallites [19]. The optical effects with small colloidal particles of CdS (<50 Å) observed due to quantization in three dimensions was reported by Nozik and co-workers [20]. Electro chemical atomic layer epitaxy (ECALE) method for size quantized CdS thin film for photoelectrochemical application was reported by Torimoto et al. [21]. Vossmeier et al. [22] reported advanced synthetic routes for the preparation of monodispersed CdS clusters. Synthesis of CdS with different crystalline size by a precipitation technique with CdSO$_4$, thiourea and NH$_4$OH precursors was reported by K.K. Nanda et al.[23]. They used CdSO$_4$, thiourea and NH$_4$OH as precursors. By controlling the reaction time period/thickness, temperature and pH of the solution, different crystalline sizes were obtained. Nanocrystalline CdS presenting quantum size effect were prepared at 300 K with solution pH=11.7. They observed blue shift of 0.74 eV due to quantum size effect in CdS nanoparticles.

Yao and co-workers [24] reported preparation and photoelectrochemical characterization of CdS particles in nanospaces of expandable mica. Using XPS
spectra Jinesh and co-workers [25] find out the quantum confinement in chemically deposited CdS. They use XPS spectra for the investigation. They also reported that increased pH of the bath shows CdS clusters of finite size which are embedded in Cd(OH)₂ surroundings making the clusters electrically isolated and thus presenting carrier confinement.

Polymers are commonly used for the synthesis of semiconductor nanoparticles because the polymer matrices offer advantages like solubility, easy processability and control of the growth and morphology of the nanoparticles. Many workers [26-31] prepared CdS nanoparticles embedded in different matrixes like polystyrene, polyvinyl alcohol, polyethylene glycol. Z. Qian and co-workers [32] reported CdS-PAMAM composite nanocrystalline thin film useful for photoelectrochemical sensors. Hwang and co-workers [33] reported CdS nanoparticles on the surface of single walled carbon nanotubes (SWNTs).

It is seen that photoelectrochemical performance of CdS thin films can be improved by increasing surface area or by post deposition treatment. Pandey and coworkers and Tiwari [34, 35] reported that polycrystalline CdS thin films show good photoelectrochemical behavior in a polysulphide electrolyte with high conversion efficiency and stability due to surface treatment.

The effect of Cd:S ratio on the photoconducting properties of CdS films have been reported by Jadhav et al. [36]. They chemically deposited CdS films with different Cd:S ratios and studied their electrical and structural properties. The photoconducting studies showed that the photoconductivity has been improved with Cd:S ratio as 1:0.2. Patil and co-workers [37] reported the enhancement in PEC performance due to effect of complexing agents. The air annealed CdS thin film with triethanolamine (TEA) and ammonia as complexing agents shows the better short circuit current. Hillal and co-workers [38] reported that by pre-annealing the electrode at 250°C followed by its slow cooling enhances the PEC performance of CdS thin film prepared by CBD. The electronic quality of chemical bath deposited CdS layers for photovoltaic solar cells was investigated by Chaure and co-workers [39]. The bath composition was 0.02 M CdCl₂, 0.07 M NH₄Cl and 0.14 M thiourea. The film thickness was found to be 0.1–0.2 µm after deposition of 15 minutes by maintaining the bath at pH 9.5 and temperature 900°C. Synthesis of flowerlike CdS nanostructured films have
been reported by Dongre and co-workers [40-41]. They studied their application in photoelectrochemical solar cells. A CBD technique has been used to grow CdS films over metal/glass substrates using precursors CdSO₄, thiourea and NH₄OH. They observed that the flower like morphology enhances the PEC properties as compare to planer morphology. Dongare et al. also reported nanowires like morphology of CdS thin films but with fewer short circuits current. It explains that, for the applications of solar energy harvesting and conversion, the morphology with complex nanostructures is more interesting.

5.2 Synthesis and characterization of Cadmium Sulphide thin films by chemical bath deposition (CBD).

5.2.1 Preparation of CdS thin films by chemical bath deposition:

All chemical were purchased from s. d. fine-chemicals and used without any further purification. The cadmium sulfate (CdSO₄·H₂O) and thiourea (H₂N×CS×NH₂) were used as cadmium (Cd) and sulphur (S) precursors. Ammonia (NH₃) was used as complexing agent for the deposition of nanostructured CdS thin film.

Cadmium sulfide films were prepared from cadmium sulphate and thiourea by CBD in alkaline solution. The typical procedure for the film growth is described as follows. 25% NH₄OH solution was added drop wise into 500 ml beaker containing 150 ml of 1 mM CdSO₄ solution until the initially formed white precipitate was dissolved completely. The cleaned substrates were mounted vertically in the bath using Teflon holder, such that the substrates were separated by 2 cm distance from each other & the wall of bath. Then 150 ml of 1mM thiourea [CS (NH₂)₂] was added in the bath solution. The temperature was gradually increased to 60°C under magnetic stirring for all samples. The films removed from the bath after 20 min. were highly transparent and uniform with well adhesion to the glass substrate and therefore used for further characterization. The deposited CdS films were rinsed with double distilled water and alcohol and allowed to dry at room temperature, in ambient air. Figure 5.2 show the schematic diagram of experimental setup used for chemical bath deposition method for the preparation of Cadmium sulphide thin films.
\[
\begin{align*}
CdSO_4 + NH_4OH & \leftrightarrow Cd(OH)_2 + (NH_4)_2SO_4 \\
Cd(OH)_2 + 4NH_4OH & \leftrightarrow Cd(NH_3)_2^{2+} + 2OH^- + 4H_2O \\
H_2N - C - H_2N - C & = H_2N - C = NH \\
H_2N - C - H_2N + OH^- & \leftrightarrow H_2N - C = NH + SH^- \\
Cd(NH_3)_2^{2+} + SH^- & = CdS \downarrow + NH_4^+ + 3NH_3
\end{align*}
\]

Figure 5.2 Schematic of Chemical Bath Deposition
5.2.2 X-ray Diffraction studies of CdS thin films Prepared by chemical bath deposition

Figure 5.3 Enlarge view of XRD spectra of Cadmium Sulphide (CdS) thin film prepared on glass substrate by CBD method.

Figure 5.4 XRD spectra of Cadmium Sulphide (CdS) thin film prepared on FTO substrate by CBD method
Figure 5.4 shows enlarge view of XRD spectra recorded for CdS sample deposited on FTO (conducting Glass). Well defined reflections belonging to CdS cubic structure along (111), (220), (311) planes were observed at angles 26.5°, 43.9°, 52.8°. Figure 5.5 shows XRD spectra recorded for CdS sample on glass, it is observed that the glass is having amorphous structure and the CdS (111) peak and glass hump exists at the same θ values and therefore the intensity of (111) peak gets reduced.

5.2.3 Scanning electron Microscopy (SEM) studies of CdS thin films Prepared by chemical bath deposition

Figure 5.5 (a), (b), (C) and (d) Scanning Electron Microscopy (SEM) micrograph of Cadmium Sulphide (CdS) thin films prepared on glass by CBD method

Figure 5.5 (a), (b), (C) and (d) shows scanning electron micrographs of CdS thin films prepared by CBD method. From figure it is clearly observed that the sample
exhibits nanocrystalline CdS platelets like morphology. At low magnification structure seems like cabbage. The density of these platelets is uniform throughout the sample. The platelets are having size of 1µm and thickness 10-15 nm. The films are having throughout uniform structure and no cracks are observed in the film.

5.2.4 UV-VIS absorption spectroscopy studies of CdS thin films prepared by chemical bath deposition

![UV-VIS absorption spectra of Cadmium Sulphide (CdS) thin films prepared on glass by CBD method](image)

**Figure 5.6 UV-VIS absorption spectra of Cadmium Sulphide (CdS) thin films prepared on glass by CBD method**

Figure 5.6 shows absorption spectra recorded for CdS sample in the wavelength range 300 nm and 1000 nm. Figure clearly shows that the absorption initiates at 524 nm. This gives rise to band gap of ~ 2.36 eV for CdS sample.
Figure 5.7 UV-VIS transmission spectra of Cadmium Sulphide (CdS) thin films prepared on glass by CBD method

Figure 5.7 shows transmission spectra recorded for CdS thin film in the wavelength range 300 nm and 1000 nm. From figure it is clearly observed that the transmittance of CdS film deposited by CBD method is approximately 99% above 400 nm.

5.3 Preparation of Zn$_2$SnO$_4$/CdS thin films electrodes and their photoelectrochemical solar cell testing

5.3.1 Introduction

Within last couple of years wide band gap semiconductors such as TiO$_2$ and ZnO have shown remarkable enhancement in their photoelectrochemical (PEC) performance when coupled with low band gap chalcogenides such as CdS, CdTe, CdSe. The improvement in PEC performance is mainly attributed to extended visible light absorption which facilitates electron hole pair generation and charge injection into conduction band of host semiconductor. In the present work we investigate a new interesting system.
Because of wide band gap of 3.2 eV, TiO$_2$ nanotubes cannot absorb sunlight in the visible region efficiently. A hopeful solution to this difficulty is to use narrow band gap semiconductors along with TiO$_2$. A care should be taken that, there should be alignment between energy band levels of TiO$_2$ with that of semiconductors which facilitates photoinduced electrons transfer to the collector electrode. In order to increase the photoelectric response of TiO$_2$, semiconductors such as CdS [81–84], PbS [42, 46], Bi$_2$S$_3$ [42, 47], CdSe [48, 49], CdTe [50, 51] and InP [52] have been extensively used in the visible region. It has been reported that CdS quantum dots(QDs)[44] and CdTe QDs[51] can be implemented into TiO$_2$ NTs and the performance of the TiO2 NT array based photoelectrochemical [PEC] solar cell can be improved. Since cadmium chalcogenide semiconducting nanocrystals (CdX; X= S, Se and Te) belonging to II–VI semiconductors have size-tunable optical properties (Peng and Peng [53]; Bilgin et al [54]; Robel et al [55]), they can be used effectively as sensitizers for TiO$_2$.

5.3.2 Preparation of Zn$_2$SnO$_4$ thin films by spray pyrolysis technique,
Cubic phase Zn$_2$SnO$_4$ thin films were prepared by simple and inexpensive spray pyrolysis technique, using Stannic chloride (SnCl$_4$.5H$_2$O) and zinc Chloride as precursors of Sn and Zn respectively. The solvent used was de-ionized water. With a mole ratio of Zn: Sn as 2:1 the precursor salts were dissolved in DI water and further pneumatically sprayed over FTO coated glass substrates maintained at 400°C.
5.3.3 X-ray Diffraction Studies of Zinc Stannate (Zn$_2$SnO$_4$) thin films

Figure 5.8 XRD spectra of as prepared Zn$_2$SnO$_4$ thin film at 400°C

Figure 5.8 shows XRD pattern of as Zn$_2$SnO$_4$ prepared thin film at 400°C. The existence of well defined reflection along (311) plane at 2 theta = 34.3° and well matching of 2 theta and ‘d’ values with JCPDS card No. 73-1725 depicts the formation of phase pure Zn$_2$SnO$_4$ thin film with cubic face centered crystal structure.

5.3.4 Scanning electron microscopy (SEM) studies of Zn$_2$SnO$_4$ thin film electrodes

Figure 5.9 shows the scanning electron micrograph (SEM) of as deposited Zn$_2$SnO$_4$ thin film at 400°C (at different magnifications). From the figure it is clear that film surface is smooth crack free and few small crystallites are observed on the surface of film.
5.3.5 UV-VIS absorption studies of Zn$_2$SnO$_4$ thin film electrodes

The band gap energy of Zn$_2$SnO$_4$ thin film was calculated from UV-VIS absorption measurement. The estimated value of band gap of Zn$_2$SnO$_4$ is 3.8 eV. [From figure 5.10.]

![UV-VIS absorption spectrum](image)

Figure 5.10 UV-VIS optical absorption spectrum for the Zn$_2$SnO$_4$ thin film.

5.3.6 Preparation of Zn$_2$SnO$_4$/CdS thin films electrodes

The as prepared (5.3.4) Zn$_2$SnO$_4$ electrodes were coated by
nanocrystalline CdS using chemical bath deposition. The thickness of the CdS coating on the glass was varied by removing the samples at the increased interval of 15 min. The photoelectrochemical properties of the samples coated for 15, 30 and 45 min have been studied. Hereafter the samples are referred as S₁, S₂ and S₃ respectively.

5.3.7 X-ray diffraction studies of Zn₂SnO₄/CdS thin films electrodes

![XRD spectra](image)

**Figure 5.11 Shows XRD spectra for Zn₂SnO₄/CdS thin film**

Figure 5.11 shows XRD spectra for Zn₂SnO₄/CdS thin film. The spectra shows X-ray reflection peaks at angles (2θ), 25.25°, 27.5°, 51.8° and 61.26° with planes (100), (101), (112) and (104). The well matching of observed ‘2θ’ values and typical values from JCPDS file no. 01-080-0006 confirms CdS cubic phase. The existence of well defined reflection along (311) and (222) plane at 2 theta = 34.3° and 36.1° and well matching of 2θ and ‘d’ values with JCPDS card No. 73-1725 depicts the formation of phase pure Zn₂SnO₄ thin film with face centered cubic crystal structure.
5.3.8 Scanning electron microscopy (SEM) studies of Zn$_2$SnO$_4$/CdS thin film electrodes

Figure 5.12 (a), (b) and (c) FESEM micrographs of Zn$_2$SnO$_4$/CdS thin film samples S$_1$, S$_2$ and S$_3$ respectively.

Figure 5.12 represents FESEM micrographs for samples S$_1$, S$_2$ and S$_3$ respectively. Figure clearly shows that all the three samples exhibit nanocrystalline structure with platelet like morphology. An approximate platelet thickness of 10-15 nm is observed for Sample S$_1$ while for Sample S$_2$, it is 40-50 nm with some budlike agglomerates initiated over these platelets.

5.3.9 UV-VIS absorption studies of Zn$_2$SnO$_4$/CdS thin film electrodes
Figure 5.13 UV-VIS absorption spectra recorded for Zn$_2$SnO$_4$-CdS thin film samples S$_1$, S$_2$ and S$_3$.

Figure 5.13 shows UV-VIS absorption spectra of Zn$_2$SnO$_4$-CdS sample. From figure it is clearly observed that the absorption edges are seen for all these samples. One of them is at higher wavelength around 500 nm which belongs to absorption due to CdS and the other is at lower wavelength at around 350 nm which belongs to absorption due to bottom Zn$_2$SnO$_4$. With increasing thickness from sample S$_1$ to S$_3$, absorption due to CdS is also increased and it is analogous to observations made by SEM.

5.4 Photoelectrochemical characterization

For the PEC measurement, a three electrode system was used which consists of a working electrode (CdS coated Zn$_2$SnO$_4$ film), a counter electrode (platinum foil) and Ag/AgCl as a reference electrode. To sweep the voltage across working and counter electrode, An Autolab PGSTAT 30-ECO-
Chemie instrument was used at scanning rate of 10 mV/S and the current measurement was made in dark and under 1 Sun (AM 1.5 condition using Newport solar simulator) illumination. The electrolyte used was prepared by combination of 0.24 M Na$_2$S and 0.35 M Na$_2$SO$_3$.

5.4.1 Result of photoelectrochemical measurements

Figures 5.14, 5.15(a, b) and 5.16(a, b) represents J-V characteristic for S$_1$, S$_2$ and S$_3$ samples measured using a standard three electrode system under AM 1.5 xenon arc lamp. Photocurrent densities for all the samples under chopping are also measured S1, S2 and S3 respectively. The dark current densities values 1.4, 16 and 20 μA/cm$^2$ were noted for samples S$_1$, S$_2$ and S$_3$ correspondingly. Sample S$_1$, S$_2$ and S$_3$ exhibits photocurrent densities 0.782, 1.22 and 1.71 mA/cm$^2$ respectively at -0.2 V versus Ag/AgCl as a reference electrode. The equation for calculating applied bias photon to current efficiency (ABPE) is

$$ABPE = \frac{J_{ph} \left( \frac{mA}{cm^2} \right) \times (1.23 - |V_b|)}{P_{total} \left( \frac{mW}{cm^2} \right)}$$

where $J_{ph}$ is the photocurrent density obtained under an applied bias $V_b$ between the working and counter electrodes and $P_{total}$ is incident light intensity (100 mW/cm$^2$). The sample S$_1$ shows ABPE value 0.008% whereas it increases upto 0.012 and 0.017% for sample S$_2$ and S$_3$. The observed increase in ABPE values can be attributed to increase in absorber thickness with respect to increase in CdS coating time, which improves number of photo-generated charge carriers.
Figure 5.14 (a) J-V characteristic for sample S₁ in dark and under illumination by AM 1.5 xenon arc lamp (b) J-V characteristic with chopping.
Figure 5.15 (a) J-V characteristic for sample $S_2$ in dark and under illumination by AM 1.5 xenon arc lamp (b) J-V characteristic with chopping.
Figure 5.16 (a) J-V characteristic for sample S₃ in dark and under illumination by AM 1.5 xenon arc lamp (b) J-V characteristic with chopping.

The stability of Zn₂SnO₄/CdS photoanodes was studied by potentiostatic [current (I) vs time (t)] measurement by chopping the illuminated light at -0.2 V
Vs Ag/AgCl electrode. Figure shows photoresponse curves of Zn$_2$SnO$_4$/CdS electrodes ($S_1$, $S_2$ and $S_3$), the photocurrent values increases as soon as the electrode is illuminated and decreases to zero when the illumination is blocked by the shutter. Figure 5.17 shows overlay of J-V characteristics for samples $S_1$, $S_2$ and $S_3$. The prompt switching of current under illumination indicates rapid charge transport in all the three photoelectrodes. The non-decayed photocurrent densities up to 300 sec. illuminations also prove higher stability of all the samples in the electrolyte (figure 5.18).

**Figure 5.17** (a) J-V characteristic for sample $S_1$, $S_2$, $S_3$ in dark and under illumination by AM 1.5 xenon arc lamp (b) J-V characteristic with chopping.
Figure 5.18 Transient photo-responses of Zn$_2$SnO$_4$/CdS thin films

5.5 conclusions

The zinc stannate thin films were prepared on FTO coated glass substrate using stannic chloride and Zinc chloride with mole ratio of Zn:Sn as 2:1 at 400 °C by spray pyrolysis method. The phase purity of Zn$_2$SnO$_4$ thin film was confirmed by XRD pattern. The as prepared Zn$_2$SnO$_4$ electrodes were coated with nanocrystalline CdS using chemical bath deposition method. The XRD pattern confirmed formation of CdS/ Zn$_2$SnO$_4$. The thickness of the CdS coating was increased by increasing deposition time to study their effect on PEC performance. The PEC performance was studied by using three electrode configurations. The applied bias to photon to current efficiency increases with increase in the thickness of CdS coating, alternately coating time of CdS. The potentiostatic measurements of CdS/ Zn$_2$SnO$_4$ photoanode confirmed their stability in the electrolyte.
References


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