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Nanostructured Zinc Selenide (ZnSe) Thin Films Deposited by Various Modes of Electrodeposition for Photovoltaic Application

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Abstract

Nanostructured zinc selenide (ZnSe) thin films were deposited from solutions containing zinc sulphate, selenium dioxide by galvanostatic (GS), potentiostatic (PS) and potentiodynamic (PD) modes of electrodeposition technique onto FTO coated glass substrates at 300 K. The films have been characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), optical absorbance and photoelectrochemical (PEC) cell. XRD study depicts nanocrystalline hexagonal crystal structure of deposited thin film by GS and PS mode, while amorphous nature by PD mode. The surface morphology studied by scanning electron microscope (SEM) shows nanocrystalline morphology with well adherence and uniform distribution of grains over the surface of substrate in GS and PS mode, while amorphous morphology confirmed in PD mode. Optical absorbance study shows variation in absorbance peak due to quantum size effect. Hence, band gap of ZnSe thin films deposited by various modes of electrodeposition shows variation in range from 2.1 eV to 2.7 eV. The PEC cell combination was n-ZnSe | 1M (Na_2S-NaOH-S) | Graphite. From the current-voltage (I-V) characteristics, it is concluded that films are of n-type conductivity. The I-V characteristics were used to calculate fill factor and to study performance of the PEC cells under the illumination condition of 1.5 air mass index (AM). PS mode deposited ZnSe thin film shows better performance with I_{sc} and V_{oc} values 1.16 mA/cm^2 and 218 mV, respectively.

Keywords: Electrodeposition; Thin film; XRD; SEM; PEC cell.
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1. Introduction

This Zinc selenide (ZnSe) is a promising material for optoelectronic semiconducting technology due to its wide band gap in the blue region of the visible spectrum.[1] ZnSe-GaAs heterojunction has attracted increased interest because of its potential applications in high speed and optoelectronic devices. Toxic cadmium sulphide (CdS) material in the buffer layer[2] of the solar cell can also be replaced by ZnSe as its band gap (~2.6 eV) is wider than that of CdS (2.4 eV) and it shows good lattice match with copper indium gallium sulphide (CIGS) thin film solar cell.[3]

ZnSe thin films have been deposited by various researchers with molecular beam epitaxy, vacuum evaporation, thermal evaporation, chemical vapor deposition, magnetron sputtering and electrodeposition from aqueous and nonaqueous solutions, as well as from molten salts,[1,4–10] on various substrates. Among these techniques electrodeposition is economical and suitable for large-scale applications. The quality of electrodeposited films depends on various parameters like deposition potential, pH of the solution bath, temperature of the bath and concentration of the species in the solution.[11] This technique also overcomes shortcomings due to high deposition temperature. Formation of ZnSe thin films by electrodeposition is difficult because of the wide difference in the reduction potentials of Zn and Se ions. Even after this complexity involved in optimization of various deposition parameters; significance of this compound in development of

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optoelectronics and solar cell maintains continued interest of researchers. We can apply the electric field in electrodeposition method by three different ways *i.e*. constant voltage, constant current and varying electric field.[12] These three different types of applied electric fields significantly affect the surface morphology, crystal structure and hence the optoelectronic property of deposited material, as performance is highly dependent on ZnSe morphologies as well as crystallographic forms,[10,13–15] Bhalerao *et al.*[16] reported effect of three modes of electrodeposition on PEC cell performance of ZnSe thin films deposited on stainless steel substrate. But thin films deposited on stainless steel substrate cannot be used as buffer layer or window layer in heterojunction solar cell.

Hence present work is directed to study PEC cell performance of ZnSe thin films deposited on transparent fluorine doped tin oxide (FTO) coated glass substrate by various modes of electrodeposition and to verify their suitability as various layers of heterojunction solar cell. In present work, ZnSe thin films are prepared on FTO coated glass substrate by potentiostatic (PS), galvanostatic (GS) and potentiodynamic (PD) modes of electrodeposition to achieve varied morphology. The effects of these three modes on structural, morphological and optical properties of thin films have been reported. Further, these thin films are used as photoelectrode in a photoelectrochemical (PEC) cell with configuration ZnSe / 1 M polysulphide / graphite. The PEC cell is used to study photovoltaic performance of thin films, so as to implement these films as different layers forming a heterojuntion solar cell.[17,18]

2. Experimental details

2.1 Optimization of preparative parameters

Stability diagram, linear sweep voltammogram (LSV) and cyclic voltammogram (CV) are used to optimize deposition parameters like pH, potential, current density, and scan rate for zinc and selenium containing electrolytes, respectively. The films were grown at optimized deposition potential of -400 mV/ SCE for PS mode, at 0.5 mA/cm^2 current density for GS mode of electrodeposition and at 50 mV/S scan rate for PD mode within pH range selected from stability diagram.[16]

2.2 Preparation of ZnSe thin films

The ZnSe thin films were electrodeposited on FTO coated glass substrate. The FTO coated glass substrate functions as the cathode in three electrode cell with graphite as the counter electrode and saturated calomel electrode (SCE) as the reference electrode. The electrolyte was prepared by mixing solution of $ZnSO_4$ (0.1 M) and SeO_2 (0.001 M) in ratio of 1:1 respectively. The aqueous solution of above precursor chemicals were prepared using double distilled water. The pH of electrolyte solution was varied by dilute HCl. The distance of 1 cm was maintained between the working electrode and counter electrode during deposition. Before deposition the substrate were thoroughly cleaned with double distilled water in ultrasonic cleaner. The detailed growth kinetics was studied by changing the deposition parameters like deposition time or deposition cycles and the pH of solution. The ZnSe films are deposited from electrolytic bath maitained at pH~2.0, by different modes *i.e*. PS, GS and PD of electrodeposition.

2.3 Characterization techniques

Oxidation and reduction mechanism in the electrolyte bath was studied in potential range 0 to -1 V/SCE by CV technique. Battery Cycler unit of WonAtech model WBCS 3000 used to obtain all voltammetric curves. Further depositions were carried under chronoammpearometry, chonopotentiometry and potentiodynamic modes of the battery cycler. The structural characterization of optimized ZnSe thin films was carried out by X- ray diffraction (XRD) patterns obtained with Philips X- ray diffractometer model PW-1710 in scanning angle (2θ) range 20^0 to 80^0 with wavelength 1.5406 Å and voltage 40 kV. Surface morphological study carried out using scanning electron microscope JEOL-JSM model 6360, for the sample coated with platinum by sputter plasma technique. The optical absorption spectra studied using a UV-1800 Shimadzu spectrophotometer in the wavelength range 250 to 800 nm. The solid-liquid junction PEC cell is formed with ZnSe thin films as photoelectrode and graphite rod as counter electrode. PEC cell with configuration ZnSe | 1 M polysulphide | graphite, is used to check the photoresponse exhibited by ZnSe thin films using Princeton Applied Research Potentiostat model-273.

3. Results and discussion

3.1 Structural analysis by X-Ray diffraction

Figure 1 shows X-ray diffraction (XRD) pattern of the as deposited film electrodes. The XRD pattern shows crystalline texture growth with PS, GS and amorphous nature of PD mode of electrodeposition.

XRD study shows six peaks for as-deposited PS mode ZnSe film corresponding to interplanar distances 3.43, 2.36, 1.99, 1.70, 1.52 and 1.35 Å and five peaks for GS mode ZnSe film corresponding to interplanar distances 3.43, 2.36, 1.99, 1.70 and 1.52 Å. Both PS mode and GS mode electrodeposited film shows remarkable growth along (1 0 0), (1 0 2) and (1 1 2) planes, as evidenced by JCPDS data file no. [15-0105]. Some minor peaks are also observed at (1 1 0) and (2 0 2) planes. While in PD mode XRD pattern shows no peaks, indicating amorphous nature of material. Table 1 gives detailed structural analysis of the ZnSe thin films deposited by PS and GS mode

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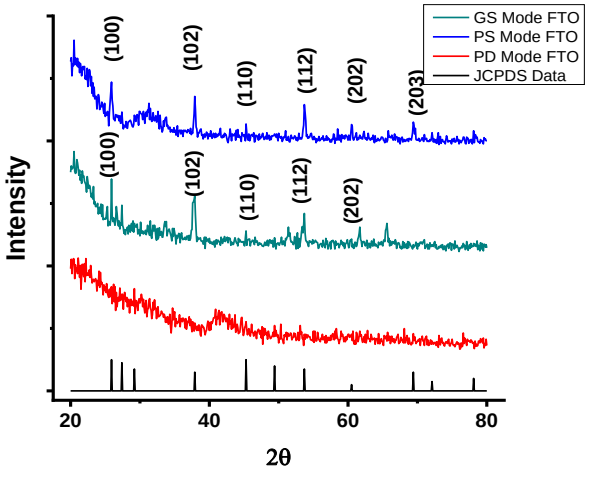

Fig. 1 X-ray diffraction pattern of ZnSe thin films deposited with different modes of electrodeposition.

of electrodeposition. According to Braggs law X-rays reflected from the successive atomic planes would interfere either constructively or destructively depending upon the path difference between the interfering rays.[19] The value of interplanar distance has been determined from Bragg's equation (Eq. 1)[19] and reported in Table 1.

$$2d\sin\theta = n\lambda \quad (1)$$

where, d is interplanar distance (lattice spacing), λ is the wavelength of the monochromatic X-ray; n is the order of diffraction and θ is angle of diffraction. The 'd' values are calculated using above relation for known values of θ, λ and n. The crystallite size has been calculated using Scherer's formula[39] as given in Eq. 2.

$$D = \frac{K.\lambda}{\beta.\cos\theta} \quad (2)$$

where, D is grain size, λ is wavelength of X-rays used, β is full width of half maxima of the peak (FWHM) in radians, θ is Bragg's angle and K is constant. Value of K varies from 0.89 to 1.39.

Dislocation density δ was calculated from $\delta = [1/(D)^2]$, which is about 2 nm^{-2}. Small δ value represents fewer amounts of defects in the deposited film. This indicates that crystallization levels of the films are good.[20,21] The strain value has been determined using Eq. 3.

$$\varepsilon = \left(\frac{\lambda}{\cos\theta} - \beta\right).\frac{1}{\tan\theta} \quad (3)$$

Among three deposition techniques comparatively broad peaks are observed in GS mode with respect to PS mode, indicating reduction in grain size. The keen observation of structural analysis from the Table 1 suggests that GS mode ZnSe thin layer deposits are nanocrystalline in nature with higher β value. Higher the β value, less strain is observed. This supports that, nanocrystalline ZnSe thin films have higher elastic properties.

The XRD pattern (Fig. 1) also shows that there is good matching between observed and standard interplanar spacing (d) values with hexagonal crystal structure and lattice parameters as (a=b=3.996 Å and c=6.55 Å), suggesting the formation of zinc selenide crystalline phase.[22]

3.2 Surface morphological study

Figures 2(a, b) shows scanning electorn micrograph (SEM) images of zinc selenide thin films deposited by three different modes of electrodeposition at magnifications 10,000 and 30,000. The SEM images of PS mode shows patterned granular crystalline morphology with dimensions of about 300 nm. Floral spike like morphology is observed for GS mode thin film with lentgth of spike about 600 nm and width about 100nm. This morphology provides higher surface area than geometrical area. The high surface area is useful for ion intercalation and deintercalation activity to enhance current in electrochemical or photovoltaic activity. The spike like morphology plays a crucial role in enhancing the electrochemical activity in comparison with bulk counterpart.[23] Lower magnification images of PS and GS mode shows uniform distribution of thin film on the surface of substrate. Well resolved grain growth with uniform film coverage is also observed in higher magnification images of PS and GS mode. While PD mode SEM image confirms

Table 1. Structural analysis of ZnSe thin films.

2θ	θ	Interplanar Distance 'd' observed. A^0	Crystallite Size 'D' nm	Dislocation Density 'δ' $\delta = 1/D^2\ (nm)^{-2}$	Strain 'ε'
A. Potentiostatic Mode Deposited ZnSe					
25.95^0	12.97^0	3.43	1.58	0.40	5.56
37.96^0	18.98^0	2.36	0.67	2.22	4.09
43.58^0	21.29	1.84	0.74	1.80	3.39
53.78^0	26.89^0	1.75	0.59	2.79	2.87
60.58^0	30.29^0	1.52	0.71	1.98	2.66
69.40^0	34.70^0	1.35	0.88	1.29	2.42
B. Galvanostatic Mode Deposited ZnSe					
25.95^0	12.97^0	3.43	1.347	0.55	6.40
37.96^0	18.98^0	2.36	0.44	5.16	3.77
43.58^0	21.29	1.84	0.74	1.80	3.40
53.78^0	26.89^0	1.75	5.01	3.98	2.80
60.58^0	30.29^0	1.52	0.981	1.03	2.77

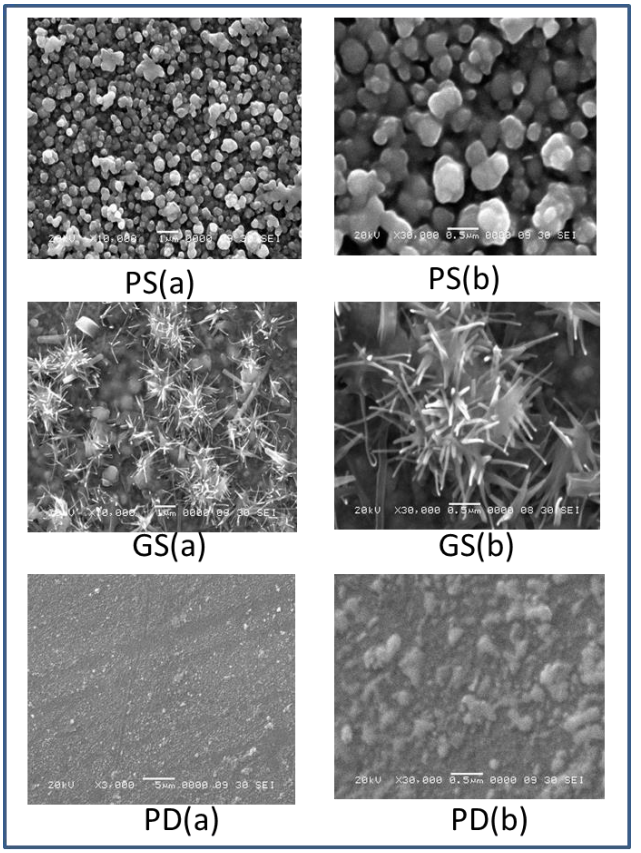

Fig. 2 Scanning electron micrograph of ZnSe thin films for PS mode, GS mode and PD mode at different magnifications (a) X 10,000 and (b) X 30,000.

amorphous nature of the deposit. Such varied morphology from different modes of electrodeposition is highly supportive for using ZnSe as either window layer or buffer layer in photovoltaic devices; even it is helpful in optical conductance for optoelectronic devices.

3.3 Optical absorbance study

The optical absorption spectrum of ZnSe films from three modes of deposition were studied in the wavelength range of 250-800 nm at room temperature for films of equal thickness of about 0.9 µm. Fig. 3 inset shows the variation of optical absorbance (αt) with wavelength. The PS and GS mode thin film shows good absorbance in visible region, while PD mode thin films show good absorbance in ultra violet region. Difference in absorbance coefficient value in three modes of deposition indicates size quantization in nanocrystalline semiconductors.[24] The reason behind this is electrons and holes are localized in a confined volume of semiconductor. The energy gap 'E_g' was calculated using the Eq. 4.

$$\alpha = A\,(h\nu - E_g)^{n}/h\nu \quad (4)$$

Where hν is the photon energy, 'E_g' the energy band gap, A and n are constants. For allowed direct transition n = 1/2 and for allowed indirect transition n = 2. Fig. 3 shows variation of $(\alpha h\nu)^2$ with hν for ZnSe films. The linear nature of the plot indicates the existence of the direct transition. The band gap 'E_g', was determined by extrapolating the straight portion to energy axis at α = 45°. The band gap value of PS, GS and PD mode is 2.1 eV, 2.25 eV and 2.7 eV, respectively. The energy band gap values of depositions developed by GS, and PD mode are in good agreement with earlier reported values,[25,26] while PS mode film shows red shift of 0.4 eV.

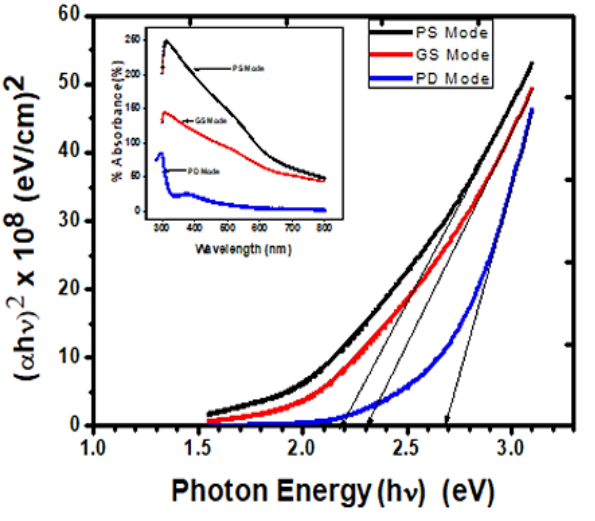

Fig. 3 The plot of $(\alpha h\nu)^2$ against the (hν) for ZnSe thin films to determine energy band gap in PS Mode, GS Mode and PD Mode. Inset shows the variation of absorbance (αt) with wavelength (λ) of ZnSe thin films.

3.4 Photoelectrochemical study

PEC cell has been fabricated using ZnSe thin films as a photoelectrode, polysulfide as an electrolyte and graphite as a counter electrodel with configuration ZnSe| 1M Polysulfide| graphite. Dark voltage, V_d and dark current I_d has been obtained from PEC cell with ZnSe electrode as the negative and graphite electrode as the positive polarity ends. The origin of this dark voltage is attributed to the difference between two half-cell potentials in the PEC cells which can be written as [$E = E_{graphite} - E_{ZnSe}$].Where $E_{graphite}$ and E_{ZnSe} are the half-cell potentials when dipped in polysulphide electrolyte.[27] After illumination of the junction with light of intensity 100 mW/cm^2 (AM 1.5 irradiance: The ratio of the actual path length of the sunlight to its minimal distance (when the sun is at its zenith) is known as the optical air mass (AM).[5] AM1.5 is referred to as the standard spectral distribution. It corresponds to an angle of 48.20 between the sun's position and the zenith. At the irradiance of AM1.5 power density is 827 W/m^2. But, the value of 1000 W/m^2 is considered as a standard power density at AM1.5 irradiance, which is also expressed as peak watts.)[28] tungsten filament lamp; the photons having energy greater than band gap energy of thin film is absorbed in it to generate electron-hole pairs in the depletion region and in diffusion layer. The experimental set up for PEC cell performance study and illuminated cell using continuous simulator is as shown in Fig. 5. Electron-hole pairs are driven apart by the electric field present across the interface between semiconductor and polysulphide electrolyte, so as to generate photovoltage under open circuit (V_{oc}) and

Table 2. PEC performance of ZnSe thin films.

Sr. No.	Mode of Deposition	I_{sc} mA/cm^2	V_{oc} mV	I_m mA/ cm^2	V_m mV	Fill Factor %
1	PS	1.16	218	0.7	125	32.6
2	GS	0.59	205	0.4	90	29.76
3	PD	0.48	107	0.21	50	20.44

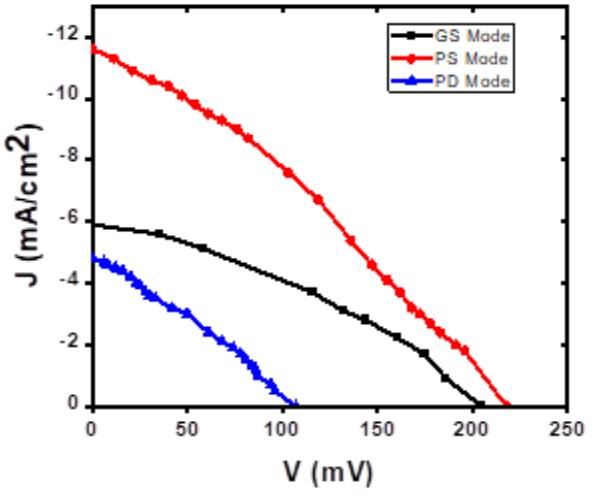

Fig. 4 The I-V characteristics curve showing photovoltaic output characteristics of PEC cell.

photocurrent under short circuit (I_{sc}) condition. Magnitude of voltage increases with negative polarity towards the ZnSe thin film and cathodic behaviour of semiconductor is observed which indicates that the conductivity of ZnSe thin films is of n- type; while polysulphide acts as the p-type semiconductor.

Figure 4 shows I-V characteristics study of PEC cells prepared with ZnSe thin films of PS, GS and PD mode of electrodeposition. Table 2 shows detail values of I_{sc}, V_{oc}, I_m, V_m, and fill factor of PEC cells at AM1.5 irradiance, with ZnSe thin films deposited by various modes of deposition. Where I_m and V_m are maximum useful current and potential, respectively which is obtained by determining maximum power of I-V coordinates of curve. The comparative photoresponse study from Table 2 indicates that, I_{sc} and V_{oc} response and fill factor shown by PS mode deposited thin film is quite higher than GS and PD mode. This may be attributed to the comparatively smaller energy band gap value of 2.1 eV in PS mode than GS and PD mode for effective absorbance of photons. This indicates that, well resolved nanocrystalline granular morphology of PS mode ZnSe thin film is helpful in using this thin layer as absorber layer in solar cell. The floral spike like morphology of GS mode ZnSe thin film with little higher band gap can be used as buffer layer. As buffer layer is used to favour charge collection and extraction in heterojunction solar cells, floral spikes will serve this task more efficiently.[29,30] While PD mode thin film with still higher band gap and less photoresponse is useful for window layer of solar cell.

4. Conclusions

ZnSe thin films have been synthesized on FTO coated glass substrate by PS, GS and PD modes of electrodeposition. XRD patterns of PS and GS mode deposited films revealed formation of hexagonal crystalline structure of thin films with remarkable growth along (1 0 0) and (1 0 2) planes corresponding to interplanar distances 3.43, 2.36 Å. PD mode deposited film shows amorphous nature. Significant effect on the surface morphology of ZnSe films was observed through SEM study. GS mode film shows rarely observed floral spikes like morphology for easy conduction of carriers in buffer layer.

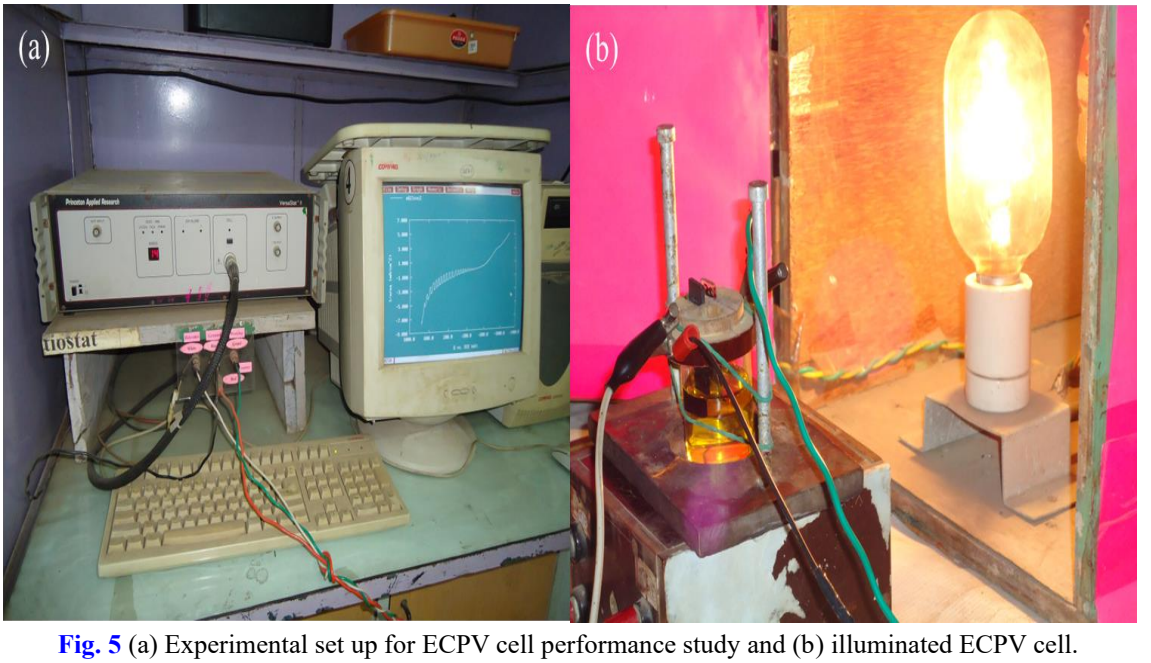

Fig. 5 (a) Experimental set up for ECPV cell performance study and (b) illuminated ECPV cell.

The variation in optical absorption coefficient results in variation of energy band gap indicating quantum confinement of particle size. I-V characteristics study by PEC cell shows applicability of these thin films as different layers of hetero junction solar cell. The values of Isc and Voc for PS mode thin film are greater than GS and PD mode, suggesting their applicability as absorber layer, buffer layer and window layer, respectively.

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Conflict of Interest
There is no conflict of interest.

Supporting Information
Not applicable.

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