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Conference Paper

Structural, Optical and Electrical Characterization of CdSe Nanorods Synthesized by Solvothermal Process

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CdSe nanorods are synthesized by solvothermal process in a mixed solvent of hydrazine hydrate [H_6N_2O], ammonia [NH_3] and deionized water. Cadmium acetate [$(CH_3COO)_2Cd{\cdot}2H_2O$] and sodium selenite [$Na_2SeO_3$] are used as precursors for cadmium and selenium ions, respectively. FESEM is used to study the morphology of the synthesized nanorods. XRD measurement shows wurtzite phase of CdSe. SAED pattern and TEM measurement are also used to study the structure and morphology of the particles. UV-Vis_NIR measurement shows strong absorption around 730 nm. Nanorods thin films are prepared using doctor blade technique. Conductivity of the thin film samples is measured in vacuum using two-probe method.

1. Introduction

CdSe is a direct band gap II–VI compound semiconductor, which can exist in both zinc blende phase and wurtzite phase. Its direct band gap of 1.7 eV divides the solar spectrum into two parts, namely, the "thermal" part with $h\nu < E_g$ and the "optical" one with $h\nu > E_g$, both parts with practically equal radiation energy. It is almost optimum for certain opto-electronic applications. Therefore, CdSe has been extensively studied as potential active media for optical and optoelectronic devices in the form of thin film and nanostructure. CdSe has now been widely used in various devices including thin film solar cells, solar coatings, LEDs, sensors [1–4]. Various low cost manufacturing processes like physical thermal evaporation, chemical bath deposition, hydrothermal, solvothermal, and so forth, have been utilized to successfully prepare the CdSe materials: thin film and nanostructure [5–8]. Among the preparation methods mentioned above, solvothermal process is one of the most efficient techniques for synthesizing nanomaterials of good crystalline quality. There are several solvothermal chemical reaction routes reported in the literature using various solvents and reaction precursors. In this paper, we report the CdSe nanostructure synthesis by solvothermal routes using mixed solvent of ammonia, hydrazine hydrate, and deionized water and the study of structural, optical, and electrical properties of the synthesized materials.

2. Experimental Section

2.1. Synthesis of CdSe Nanorods by Solvothermal Method. A Teflon lined stainless steel closed cylindrical chamber with 110 mL capacity is used for the synthesis. Cadmium acetate {$(CH_3COO)_2Cd{\cdot}2H_2O$} and sodium selenite ($Na_2SeO_3$) are used as precursors for the chemical synthesis. Appropriate precursor salts with 1 : 1 molar ratio (0.005 mol each) are taken into separate cylindrical beakers. The metal salt (group II element precursor) is dissolved in 10 mL of deionized water and then slowly mixed with 8 mL of ammonia solution ($NH_3{\cdot}H_2O$). The precursor salt of group VI element is mixed with 10 mL of hydrazine hydrate ($N_2H_4{\cdot}H_2O$) before the two solutions are transferred to the autoclave. The autoclave is then filled with deionized water up to 80% of its volume. After

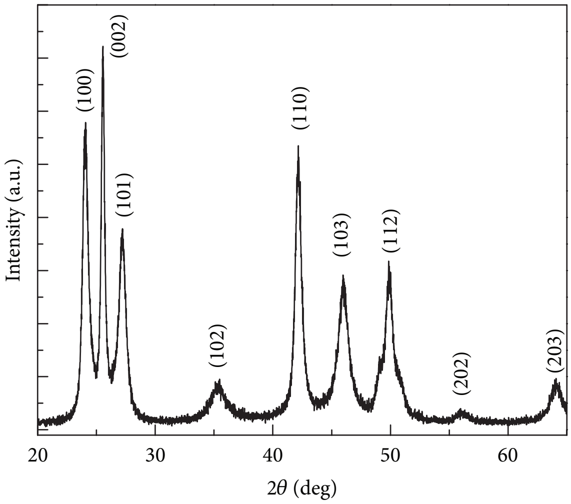
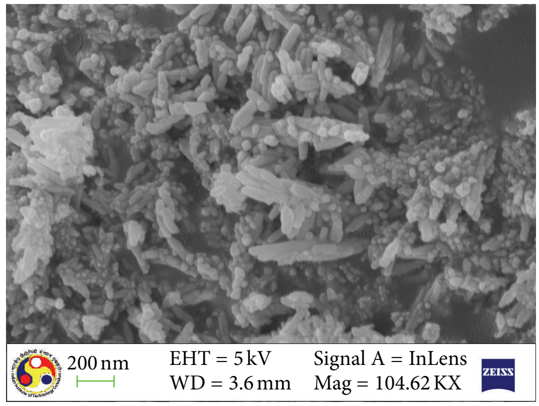


Figure 1: XRD pattern of CdSe powder.

10 minutes of stirring, the closed chamber is placed inside a muffle furnace at a preset temperature of 180°C for 5 hrs. The precipitates are filtered off and washed several times in distilled water and ethanol. The final products are dried at room temperature for several hours to get the nanopowder samples.

2.2. Thin Film Preparation by Doctor Blade's Technique. Doctor blade's technique is a popular technique for preparation of nanoparticle thin films. It is a room temperature process that involves flattening of a paste containing colloidal particles onto a substrate using a sharp knife, which results in a smooth thin film. It does not destroy the structural and morphological features of the nanomaterials. For film preparation, appropriate amounts of CdSe nanorods, ethyl cellulose (binder), and terpineol are mixed in a mortar by grinding until a uniform concentrated gel is formed. A drop of the paste is added to the Corning 1737 and ITO coated glass substrates and flattened with a sharp knife followed by drying for several hours at room temperature.

2.3. Characterizations. X-ray diffraction (XRD) analysis was carried out on both the synthesized powder and thin film samples using CuKα radiation and was measured in 2θ range of 20°–65°. The dimension and morphology of the prepared samples were studied using field emission scanning electron microscope (FESEM). Energy dispersive X-ray analysis (EDX) was performed for composition analysis. Absorbance spectrum was recorded in the range 200 nm to 800 nm using JASCO V-650 UV-Vis spectrophotometer with integrating sphere. Thicknesses of the prepared thin film samples were measured using DekTak 150 Stylus Profilometer. The conductivity of the films was measured by two probe method in vacuum using Keithley 6430 source meter. Silver paste was used for making ohmic electrical contact to the thin film sample.



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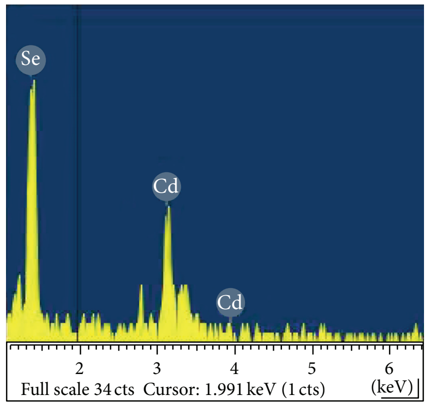


(b)

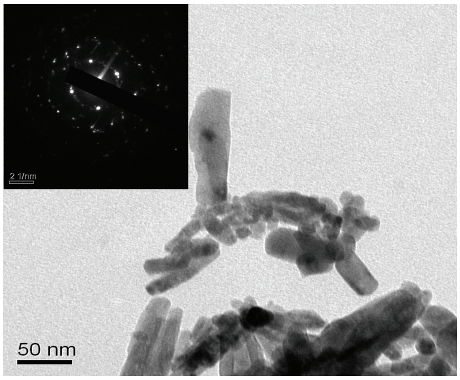
Figure 2: (a) FESEM image of CdSe powder. (b) EDX spectrum of CdSe powder.

3. Results and Discussion

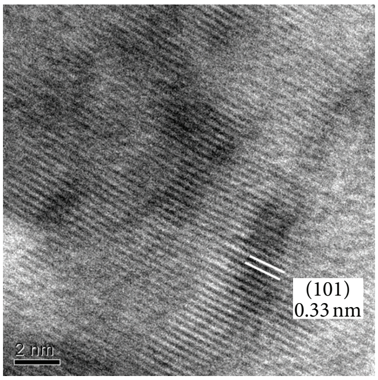
XRD pattern of the synthesized powder is shown in Figure 1. There are nine characteristic peaks of CdSe wurtzite structure in the 2θ range of 20° to 65°. The grain size is calculated from the full width at half maxima (FWHM) of (002) peak using Debye Scherrer's formula. It is obtained as 25.5 nm. The peaks shown in the figures are slightly shifted to higher 2θ values by about 0.2°, which is an indication of structural defects. The strain and dislocation densities are calculated from the XRD data and are found to be 1.4×10^{-3} and 2.25×10^{15} lines per m^2, respectively.

FESEM image of the synthesized CdSe nanoparticles is shown in Figure 2(a). The particles growth is not uniform, but mostly they have elongated rod shape feature. There are bigger nanorods with cross-sectional diameter of almost 50 nm and smaller elongated particles with less than 25 nm diameter. The EDX spectrum is shown in Figure 2(b). The atomic percentage composition obtained from the measurement is shown in Table 1. The synthesized nanorods are slightly cadmium rich.

TEM image shown in Figure 3(a) also shows rod shape nanostructure. The SAED pattern in the inset of Figure 3(a) shows a good diffraction pattern indicating the material with good crystalline quality. The HRTEM image in Figure 3(b)



(a)



(b)

Figure 3: (a) TEM image and SAED pattern of CdSe nanoparticles. (b) HRTEM image of CdSe nanoparticles.

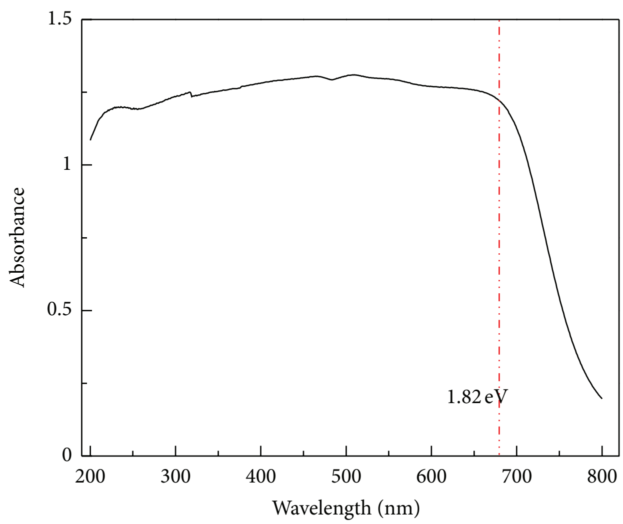
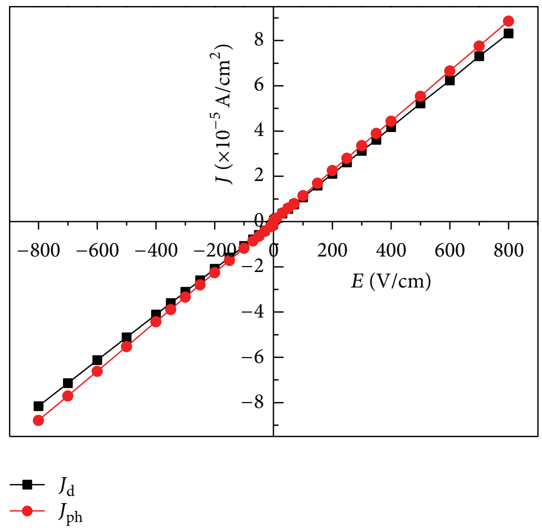
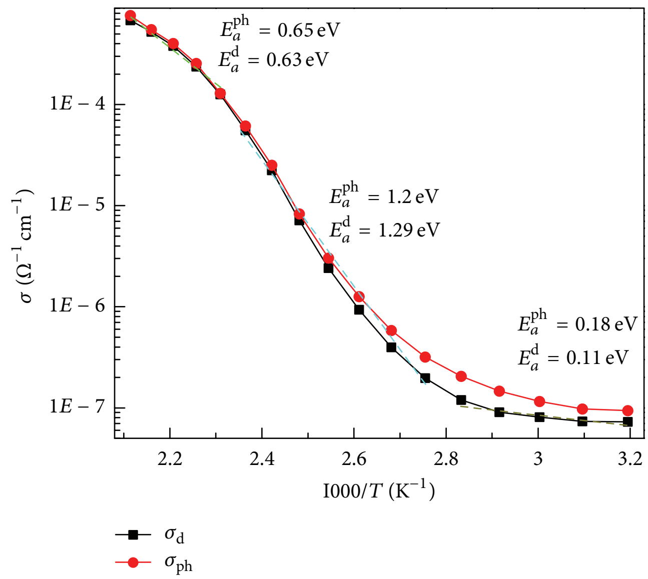


Figure 4: UV-Vis absorbance spectrum of CdSe nanoparticles.



(a)



(b)

Figure 5: (a) J versus E plot of CdSe nanoparticle thin film. (b) σ versus 10^3/T (K^{-1}) plot of CdSe nanoparticle thin film.

shows regular planes corresponding to (101) plane of wurtzite CdSe. The d-spacing obtained 0.330 nm is slightly greater than the corresponding standard value of 0.327 nm.

The recorded absorbance spectrum (range 200 nm to 800 nm) of CdSe nanorods is shown in Figure 4. The rough estimate of the band gap calculated from the absorption edge is 1.82 eV. This has been blue shifted from the bulk band gap of 1.75 due to quantum confinement effect in the nanostructure.

The conductivity of the nanoparticle thin film as prepared on Corning 1737 glass using doctor blade's technique is measured by two-probe method in coplanar geometry. The room temperature measurement for dark and photo conductivity is plotted in Figure 5(a). The dark conductivity is $1.033\times10^{-7}\ \Omega^{-1}\ \text{cm}^{-1}$, while the photo conductivity is $1.107\times10^{-7}\ \Omega^{-1}\ \text{cm}^{-1}$. The conductivity variation with temperature is studied for both dark and photo current. σ versus 1000/T plot for dark and photo current is shown in Figure 5(b). The conductivity increases with increase in temperature. The plot exhibits Arrhenius behavior in three different temperature ranges 313–353, 353–433, and 433–473 K. The activation

Table 1: Atomic percentage composition of CdSe powder.

Element	Atomic%
Se	48.02
Cd	51.98

energies in these regions are calculated and are shown in the figure for both dark and photo current. The calculated activation energies are higher than the corresponding activation energies for the reported thermally evaporated thin film [5].

4. Conclusions

CdSe nanorods with wurtzite structure are synthesized by solvothermal method. The nanorods diameter vary from 25 nm to 50 nm. The band gap obtained from the absorbance spectrum is 1.82 eV. The conductivity thermal activation follows Arrhenius behaviour. The prepared thin film by doctor blade's method exhibits high thermal activation energies in three different temperature ranges 313–353, 353–433, and 433–473 K.

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