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Synthesis of ZnO-CdO Nanocomposites

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Abstract

The ZnO-CdO nanocomposite was prepared by sol-gel method by using their respective nitrates. It is a simple and low cost method to prepare nanocomposites. The drying temperature and drying period of prepared gel was varied during the synthesis process. The prepared samples were characterized by using scanning electron microscope (SEM), particle size analysis (PSA), X-ray diffraction (XRD) and photoluminescence spectroscopy (PL) to get surface morphology, idea of getting particle of nanosized range so that further characterizations can be done, to study the optical property of synthesized nanocomposite and measure the band gap . The grain size determined by Scherrer's formula was found to be between 30-50 nm.

Introduction

A nanoparticle of semiconductor is a very popular topic in the ongoing research activity across the world. As the semiconductor particles exhibit size-dependant properties like scaling of the energy gap and corresponding change in the optical properties, they are considered as the front runners in the technologically important materials.

Zinc oxide (ZnO) has a wide direct band gap (3.37 eV) and a relative large excitation binding energy (60 meV) compared to thermal energy (26meV)[1-3]. It is also called as II–VI semiconductor, because Zn belongs to II group, and O_2 belongs to VI group in the periodic table. ZnO is a first metal oxide to use as chemical sensors, modulating it by mixing with with CdO make useful for gas detection sensors[4] like CO, NO_2, and CH_4 as reported by R.Ferro et al.[5] Thus coupling of different semiconductor oxides can reduce the band gap, extending the absorbance range to visible region leading to electron-hole pair separation under irradiation and consequently, achieving a higher photocatalytic activity[6]. Among various elements cadmium (Cd) incorporation into ZnO serves the purpose of band gap narrowing efficiently because of smaller band gap of CdO as compared to ZnO. Furthermore, the low dimensional structures of ZnO-CdO also show very good gas sensing[7] properties and increased conductivity due to their high surface to volume ratio[8]. Moreover after incorporation a ZnO-CdO heterojunction and super lattice which is a key element in ZnO based light emitters and detectors[9]. Ayman et al.[10] prepared ZnO-CdO nanocomposite nanofiber by electrospinning method by using their respective acetates and test it for effective visible light photocatalyst for waste water treatment. Sarvana et al.[6] prepared by thermal decomposition method and test its photocatalytic activity by decomposition of Methylene blue dye. On combining ZnO and CdO the band gap of ZnO reduces. S. Meenakshi et al.[11] prepared by solvothermal method by using zinc acetate, cadmium acetate and urea along with ethyl alcohol. The prepared nanocrystals have different colors varying between pure white (for ZnO) to dark yellow (for CdO) depending upon the amount of CdO and the yield obtained decreases with increasing cadmium content in the sample.

In the present work ZnO-CdO nanoparticles are prepared by using sol-gel method and characterize the prepared samples. The results are discussed.

Experimental

Materials

Chemicals used in the present work are shown in Table 1.

Table 1: Used chemicals

S. No.	*Material*	*Wt %*
1.	$Zn(NO_3)_2.6H_2O$	2 gm
2.	$Cd(NO_3)_2.4H_2O$	2 gm
3.	PVA (Poly vinyl alcohol)	8 gm
4.	Alcohol(ethanol)	55 ml
5.	Distilled Water	45 ml

Method

The ZnO-CdO nanocomposites were synthesized by using sol-gel method. Zinc nitrate hexahydrate and cadmium nitrate tetrahydrate was used as source of zinc oxide and cadmium oxide respectively while ethanol and water served as a solvent to dissolve these salts and PVA is used to give rigidity to the gel network where the dispersed ions cannot alter their positions. The different drying temperatures (90^0 C, 110^0 C, 130^0 C,150 C) and time for drying (8,10,14,16,18 hours) were used to prepare nanocomposites. During synthesis the solvent was mechanically agitated using a hot plate / magnetic stirred and teflon tablet coated magnetic paddle.

Firstly solution is prepared by mixing ethanol into distilled water in 250ml beaker and mixed with magnetic stirred up to 15 minute to get homogeneous solvent. Requisite amount of different chemicals i.e. $Zn(NO_3)_2.6H_2O$, $Cd(NO_3)_2.4H_2O$, PVA were

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weighted separately in electronic balance. When the solvent is completely formed then the zinc nitrate was added and stirred after that cadmium nitrate was added and stirred continuously after that PVA was added and dissolve until a clear transparent viscous mixture was obtained. Then this mixture was heated to 70-75^0 C to form a hard homogeneous sol solution the thermometer was used to measure the temperature in interval of 4-5 minutes. The obtained sol was slowly heated to evaporate the solvent and form a hard homogeneous gel. Then the gel formed was kept in oven for different time intervals and for different temperatures. After evaporating the solvent, brown colour crunchy mass was obtained. Then it was taken out from the beaker with the help of spatula into the mortar pestle and then crushed into fine powder. Hence we get the ZnO-CdO nanocomposite powder.

Results and Discussion

The micrograph of ZnO-CdO nanocomposite powder is showing (Fig. 1) that it is having polyhedron and some elongated grains which lie in nanometer range along with some clusters. The sample for which drying of gel was at 90^0 C and 110^0 C for 14 hrs. showing small irregular particles with agglomerates. Sample for 110^0 C for 18 hours shows polyhedron grains with small amount of longitudinal grains and with very less clustering. Whereas it has seen that at drying temperature 130^0 C for 18 hrs more clustering occurs as compared to other samples and 150^0 C for 8 hours are having smaller size .Only their morphology is suggested by the SEM that particles are irregular in shape. The bright colour in the pictures is due to charging of particles when electron light falls on it.

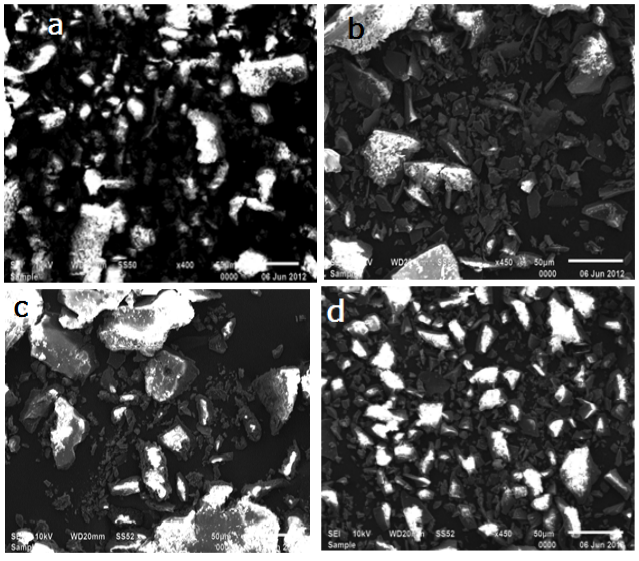


Figure 1: SEM micrographs of ZnO-CdO nanocomposite when drying of gel was done at (a) 90^0 C for 14 hours and (b) 110^{0}C for 18 hours (c) 130^0 C for 18 hours (d) 150^0 C for 8 hours

Figure 2 shows the XRD pattern of ZnO-CdO nanocomposites powder synthesized at different temperature. XRD was done for checking the formation, crystallite size and identification of present compound in obtained particles. The prominent peaks have been utilized to estimate the grain size of the sample with the help of Scherrer's equation:

$$B(2\theta) = \frac{0.94\lambda}{L\cos\theta} \qquad (1)$$

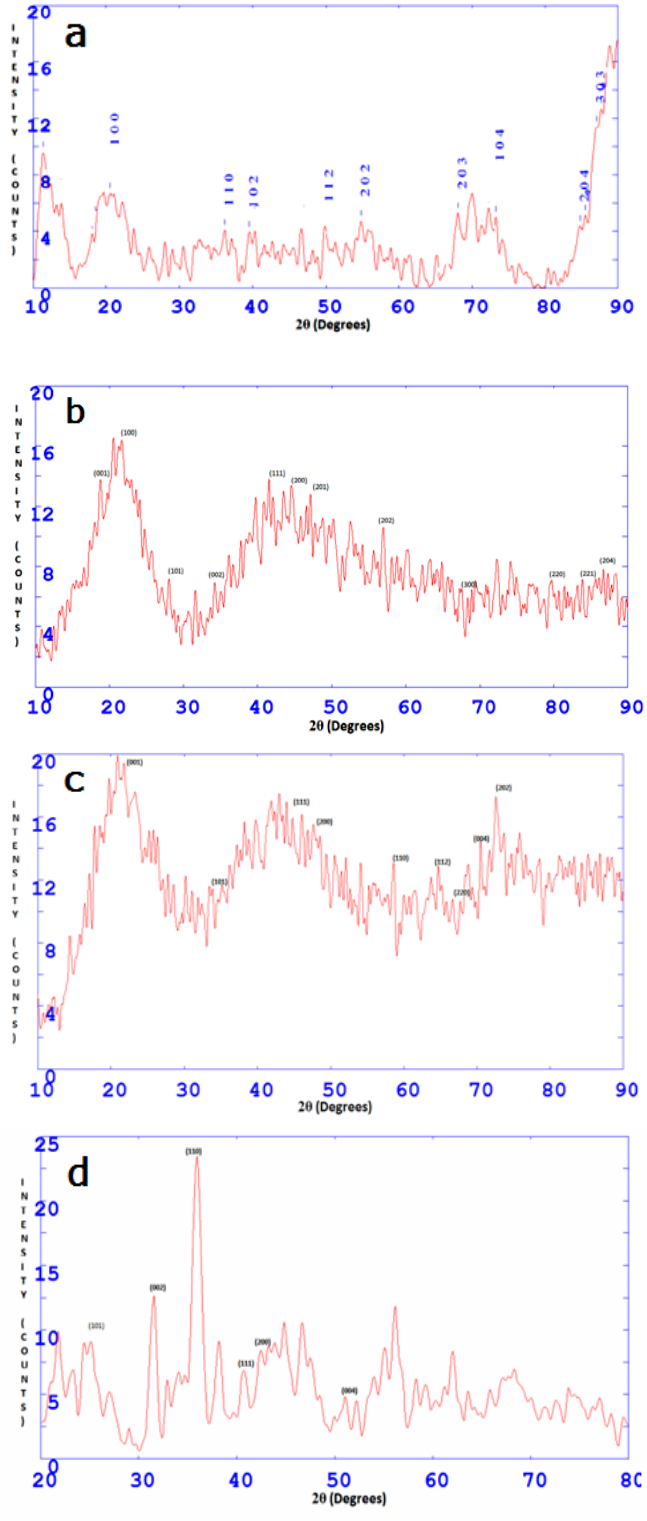
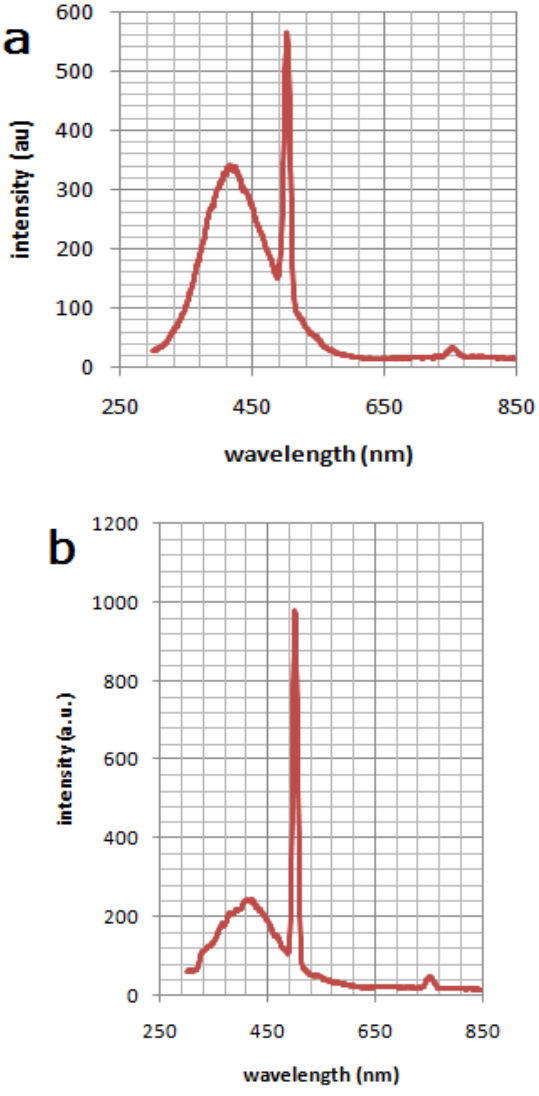


Figure 2: XRD of ZnO-CdO nanocomposite powder when drying of gel was done at (a) 90^0 C for 14 hr. (b) 110^0 C for 14 hrs. (c) 130^0 c for 14 hrs. (d) 150^0 C for 14 hrs.

Where B is peak width (full width half at maximum FWHM), applied to the principal diffraction peak corresponding to the plane and angle of diffraction. L crystallite size and "λ" is the wavelength used (1.5405 Å). Two or three diffraction peaks were chosen wherever possible and consistency in the grain size obtained from using their width. From the above diffractions patterns we see that mostly peaks observed are matching with hexagonal wurtzite

structure of ZnO and some are matched with CdO indexed to cubic structure. Some extra peaks are also observed because of mixture of ZnO-CdO, new planes area formed by combining different structures. There will be some lattice distortion occurs due to their combination. The ZnO and CdO peaks are matched with JCPDS data available. The ZnO peaks (101), (002), (102), (110), (112), (004), (202) etc and CdO peaks (111), (200), (222), (311) are seen. Mostly peaks in the pattern are shifted to left side of 2θ values of JCPDS data this may be due to slow drying temperature and time. The grain size was found to be in between 30-50 nm by using Sherrer equation.

Figure 3 shows Photoluminescence characterization. Photoluminescence is a powerful characterization technique to detect the electronic structure and energy level of semiconductor materials. The PL spectra of samples were measured with an excitation wavelength of 250 nm. The characteristic emission peak as observed at 420nm, 415nm, 404nm for sample drying at 110^0 C for 18 hrs., 130^0 C for 18 hrs. , 150^0 C for 8 hrs. (temperature and time). All samples show UV emission centred at peaks with corresponds to near band edge emission of Zno. PL peaks changes by adding CdO because of intrinsic band gap due to replacement of Zn atoms by Cd atoms. The pure ZnO peaks are at approximately at 382 nm known from literature survey 8. Thus we are getting peaks at greater wavelength. This means band gap is changes from its original value of ZnO.



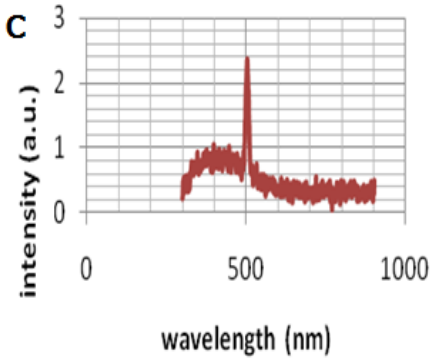


Figure 3: PL spectra of (a) 110^0 C at 18 hours shows emission peak at 420 nm. (b) 130^0 C at 18 hours shows emission peak at 415 nm. (c) 150^0 C at 8 hours shows emission peak at 404 nm.

The spectral distribution of PL from a semiconductor can be analyzed to nondestructively determine the electronic band gap. This equation is used to determine band gap via PL spectrum[8] :

$$E_{(ev)} = 1240/\lambda_{(nm)} \quad (2)$$

Table.2: Change in band gap after forming ZnO-CdO nanocomposite

Sample	*Emission peak(nm)*	*Band gap (eV)*
Sample (110^0 C -18hrs.)	420	2.9523
Sample (130^0 C-18 hrs.)	415	2.9879
Sample (150^0 C -8 hrs.)	404	3.0693

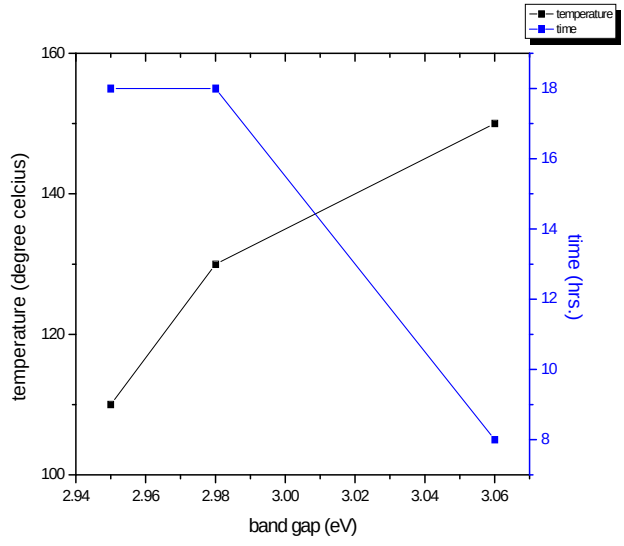


Figure 4: Plot between variation of band gap as temperature and time.

Figure 4 shows that on decreasing temperature, the band gap energy of the ZnO-CdO nanocomposite is decreases. This means more CdO is diffused and dispersed into the surface of ZnO on decreasing temperature. Band gap is the major factor for determining the electrical conductivity. Here it is decreasing this

means ZnO band gap (3.37eV) decreases, electrons can freely move within material thus electrical conductivity increases. And it is also seen by plot that as the drying time increases corresponding to these particular temperatures, band gap decreases.

Conclusions

ZnO-CdO nanocomposites have been prepared by sol-gel method. The optimum drying/ heating temperature of gel to form nanocomposite is 110^0C and time is 18 hours. Where combined and improved properties of ZnO and CdO are observed, which is the motive of synthesizing a nanocomposite.

SEM analysis showed that synthesized powder is having irregular/polyhedron shape of grains and some are having with very few elongated grains. Some clusters are also present.PSA was done to get the conformity of forming a ZnO-CdO powder is in nanometer range. So that further characterization can be preceded. The XRD pattern reveals the presence of both ZnO and CdO present in powder by showing both the peaks of both. CdO peaks are less intense peaks. The particle size is calculated from XRD by using scherrer's formula and it comes in the range of 30-50 nm for different temperature and time. The presence of CdO improves the band gap of Zno is shown by photoluminescence results. The band gap of ZnO is 3.37 eV and CdO is 2.3 eV, the composite band gap is tuned in the range of 2.952 to 3.069 eV . This means Cd^{2+} ions replace Zn^{2+} ions and consequently nanocomposite is prepared. Thus decreasing the temperature the band gap reduces is concluded from PL spectroscopy.

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