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Synthesis and Characterization of Cadmium Oxide thin film using Chemical Bath Deposition method

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Abstract: In the present investigation, low cost and effective CBD method was used to deposit the CdO nanostructured thin films on glass substrate. This thin films were characterized by the XRD, FTIR,UV-Vis Spectroscopy, FESEM, EDAX and AFM analysis. The thickness of CdO thin film was reported 182 nm. The X-ray diffraction patterns revealed that, the CdO film has nano-crystalline in nature having face centered cubic structure and highly oriented along (111) plane. The average crystallite size was 82.93 nm. The direct band gap energy of the film was 2.2 eV .The Field Emission scanning electron micrograph images of films shows that there was uniform distribution of CdO nanoparticles deposited onto entire glass substrate. Further EDAX analysis confirms elemental composition of Cd and O on the surface of substrate and the surface topography was determined by the AFM.

Keywords: Thin film, Cadmium oxide, CBD.

Introduction:

As a rapidly growing sector in materials science, nanotechnology and nanoscience deal with materials that have particles within a size range of 1 to 100 nm and a high surface-to-volume ratio [1]. However, these unique features create opportunities to use them in different sectors such as electronics, optoelectronics, agriculture, communications, and biomedicine [2, 3]. The metal oxide nanomaterial is easily involved to change the above mentioned properties. The oxide nanomaterials like ZnO, SnO2 and CdO are played a remarkable role in wide range of device applications such as solar cells, optical communications, photo-transistors, gas sensors, low emissive windows, and thin film resistors [4]. Among these metal oxide semiconductor nanoparticles the CdO shows the vital support in the development of device fabrications due to the establishment of oxygen vacancies as well as interstitial cadmium [5]. CdO not only possess high electrical conductivity [6] but also exhibits high optical transparency in the visible region of the spectrum [7].CdO found in nature either a crystalline or randomly installed, the crystalline structure of CdO is cubic (FCC), which has many important properties, such as the large band gap (2.16-2.6) eV, and a refractive index (n=2.75)., high density (8150 kg/m^3)[8-10].Thin film of the CdO are deposited by various methods including sol-gel, successive ionic layer adsorption and reaction (SILAR), pulsed laser deposition, sputtering and chemical spray pyrolysis [11,12], mechano chemical method [13], electrochemical deposition [14] and chemical bath deposition (CBD) [15]. In this present of work, a low coast and simplified CBD method is used for deposition of CdO thin films. The aim of work is to study the physical properties of CdO thin film.

Experimental methods:

In the present work, a chemical bath deposition method is used to deposit nanocrystalline cadmium oxide thin film onto well cleaned glass substrate at room temperature using 0.1 M $CdCl_2$:H_2O and 0.1 M NaOH as cationic and anionic precursors respectively. Since the well clean glass substrate provide good platform to deposit the thin film. Before the deposition, glass substrates were boiled in chromic acid for 2hr and then clean thoroughly by using soap solution, hydrochloric acid and deionized water respectively, so as to remove any dusty and oily contamination from the substrate surface. For the preparation of cadmium oxide thin film, initially 80 ml solution of 0.1 M cadmium chloride is kept in 100ml beaker without complexing agents. Further the 0.1M NaOH solution was added drop wise to it till the faint white colored solution with pH 10 is achieved at room temperature. Afterward, well cleaned glass substrate was rinsed in it for 3h with help of thin film holder and the solution is stirred using magnetic stirrer. After 3h, the substrate was removed from the solution and it was then washed with distilled water and dried in air at room temperature. White film of cadmium hydroxide that was formed on the substrate. Further grown film was annealed at 500^0C for 100 min to obtain the pure phase of CdO.

CdO thin film formation:

Initially due to cadmium chloride and sodium hydroxide, $Cd(OH)_2$ cadmium hydroxide is formed as follows.

$$CdCl_2 + 2NaOH \rightarrow Cd(OH)_2 + 2NaCl$$

Further, to remove hydroxide phase thin films were annealed at 500^0C for 100 min to obtain the pure phase of CdO.

$$Cd(OH)_2 \xrightarrow{heat} CdO + H_2O \uparrow$$

CdO thin film thickness was determined by weighting method by using the formula which is found to be 182nm.

$$t = \frac{m}{A \times \rho}$$

Where t = thickness of film
W1 is the weight of cleaned substrate
W2 is the weight of cleaned substrate plus film
W2-W1= the weight of the film
ΔW=mg
m = ΔW/g A = area of deposited film, ρ = is the density of film material

The structural characterization of cadmium oxide thin film was carried out by analyzing the X- ray diffraction pattern obtained with Rigaku Table Top X-ray diffractometer with monochromatized Cu Kα radiation of wavelength 0.154 nm. The FTIR spectrum of the sample was studied by using PerkinElmer Spectrum IR Version 10.6.2 The film morphology was observed by field emission scanning electron microscopy (FESEM) and energy dispersive X-ray spectroscopy(EDX) (Model: 7610F Plus/JEOL).

Results and Discussions:

i) XRD Results:

Fig 1. Shows the XRD patterns of CdO thin film prepared by CBD method. The diffraction peaks are obtained at 31.58 and 66.12 correspond to the planes (111) and (311) respectively. These peaks were compared with JCPDS FILE NO (05-0640) which is good agreement with the standard data. The presence of two peaks indicated that polycrystalline with face centered cubic structure. The crystallite size D is calculated by Debye Sherer Formula

$$D = \frac{k\,\lambda}{\beta cos\theta}$$

Where k is the shaping factor which takes value from 0.89 to 0.94, λ is the wavelength of the Cu-kα line, β is the Full Width at Half Maxima (FWHM) in radians and 'θ' is the corresponding Bragg's angle. The average particle size is found to be 82.93 nm
Dislocation density δ is evaluated by using the relation.

$$\delta = \frac{1}{D^2}$$

The strain is calculated by from the following relation.

$$\epsilon = \frac{\beta cos\theta}{4}$$

The number of crystallites N_c per unit area was calculated by using the relation.

$Nc = \frac{t}{D^3}$ where 't' is the thickness of thin film.

Sr.No	Diffraction peaks '2θ' In degree	'd' lattice spacing		(hkl) planes	β (FWHM) In degree	Grain size 'D' in nm	Dislocation density $\delta \times 10^{14}$ per m^2	Strain $'\epsilon' \times 10^{-4}$	Number of crystallite per unit area 10^{15} per m^2
		Observed A^0	Standard A^0						
1.	31.58	2.832	2.712	(111)	0.1279	64.55	2.399	5.36	0.676
2.	66.12	1.411	1.416	(311)	0.0936	101.31	0.974	3.42	0.175

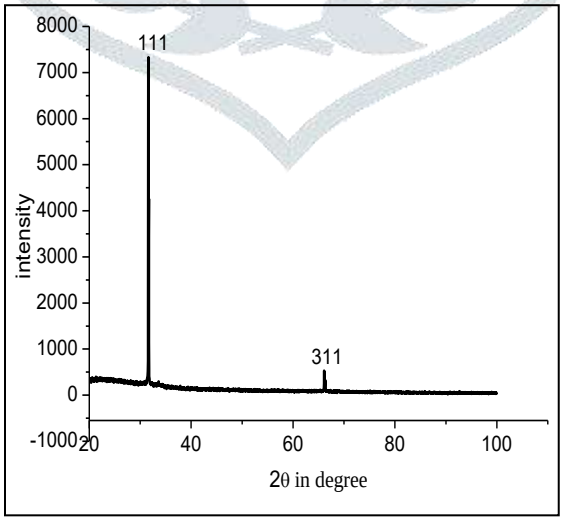


Fig 1. XRD spectra of CdO thin film

ii) FT-IR:

Fourier transform infrared spectroscopy (FTIR) is a technique which is used to obtain infrared spectrum of absorption, emission, and photoconductivity of solid, liquid, and gas. It is used to detect different functional groups present in the given compound. FTIR spectrum is recorded always between 4000 and 400 cm^{-1}.Fig 2 shows that FTIR spectra of CdO thin film which was carried out in the middle of IR region 400 to 4000 cm^{-1}.There are OH stretching at 3541.1 cm^{-1}, vibration band at 1650 cm^{-1} and CdO

bands. As CO_2 molecules are presented in the air, absorption peak at about 2360– 2390 (cm^{-1}) can be seen in the FTIR spectrum. The region from 500 to 1500 cm^{-1} is a fingerprint region. The peaks obtained in this region correspond to the symmetric and asymmetric vibration of frequencies of Cd-O band.

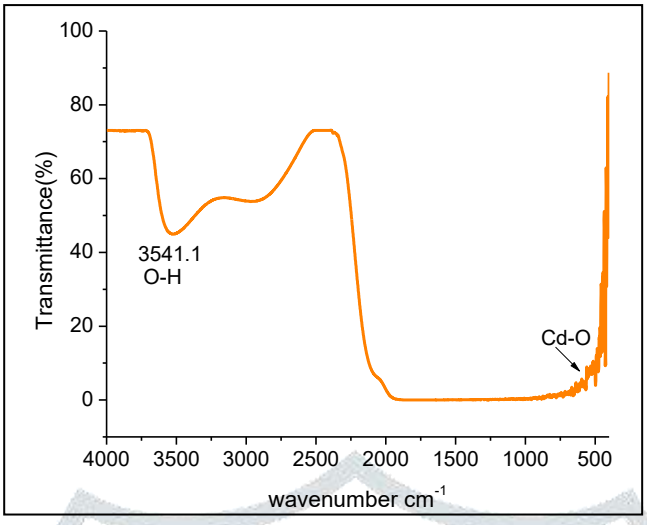


Fig 2. FTIR spectra of CdO thin film

iii) UV Spectroscopy Studies:

The absorption spectra of optimized CdO film was obtained from UV-Vis spectroscopy which is as shown in Fig. 3. This spectra was recorded in the wavelength region from 300 to 800 nm.. At about 320nm the maximum absorption was observed in the ultra violet region. The direct optical band gap energy (E_g) for the optical transitions was obtained by using the Tauc's relation,

$$\alpha h\vartheta = A(hv - E_g)^n$$

where, 'Eg' is optical band gap energy 'α' is absorption coefficient, , 'A' is a constant and 'n' is equal to 1/2 and 2 for direct and indirect transitions respectively. The band gap enrgy 'Eg' is determined by plotting a graph of (hv) against $(\alpha hv)^2$ as shown in fig 4. The obtained optical band gap is 2.2 eV which is less than to Bulk CdO (2.5 eV) [16].But this value approximately matched with this reported band gap (2.19 eV) [17].Since The optical band gap energy varies depending on synthesis method, thickness, deposition time and other growth parameters.

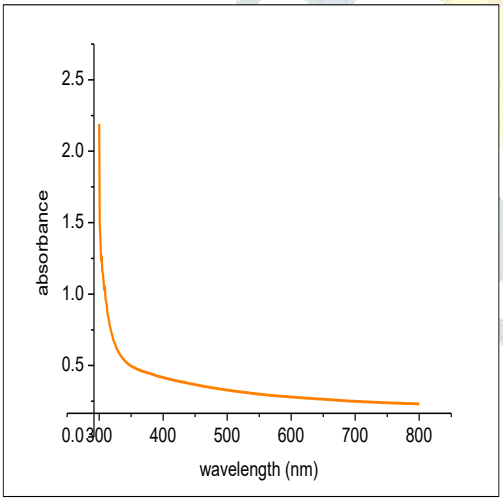


Fig 3. Absorption spectra of CdO thin film

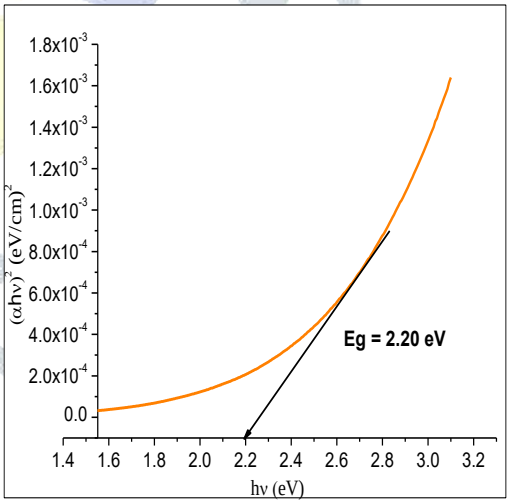
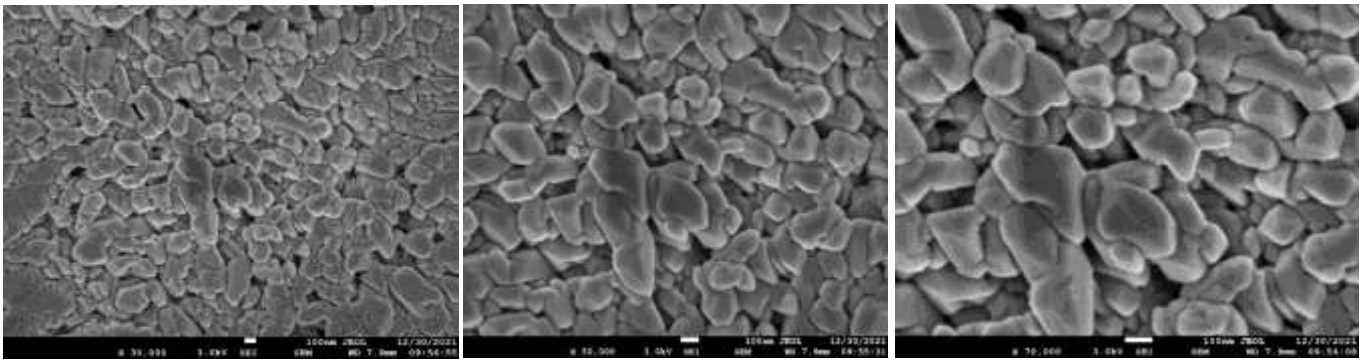


Fig 4. Tauc's plot for CdO thin film

iv) FESEM

Field emission scanning electron microscope is used to visualize very small topographic details on the surface or entire or fractioned objects. Researchers in biology, chemistry and physics apply this technique to observe structures that may be as small as 1 nanometer (= billion of a millimeter). Compared with convention scanning electron microscopy (SEM), field emission SEM (FESEM) produces clearer, less electrostatically distorted images with spatial resolution down to 1 1/2 nanometers – three to six times better. The surface morphology and elemental analysis of cadmium oxide thin film are investigated by Field Emission Scanning Electron Microscope (FESEM) as shown in Fig 5. The surface morphology of cadmium oxide thin film indicates presence of particles composed by the agglomeration of smaller crystallites. The FESEM image shows the uniform distribution of CdO particles on the surface of glass substrate. The range of these CdO nanoparticles are in the range of 28nm to 100nm.



v) EDX

In addition to FE-SEM, energy-dispersive X-ray (EDX) was conducted on the same specimens for further analysis. EDX is an X-ray technique used to identify the elemental composition of materials. These systems are attachments to electron microscopy instruments where the imaging capability of the microscope identifies the specimens of interest. Fig 6. Shows the Energy dispersive x-ray analysis. EDAX spectrum confirms the presence of both cadmium and oxygen levels of the CdO thin film. Other element present such as silicon is due to the glass substrate. The atomic percentages of Cd, O, and Si were recorded as 52.54%, 32.11%, and 15.35%, respectively. The results were indicated the presence of Cadmium oxide deposited on the Si substrate.

vi) AFM

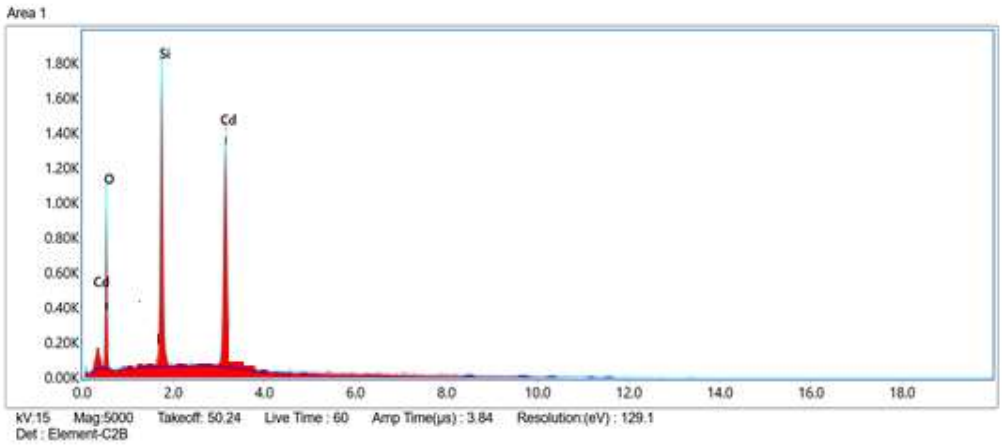
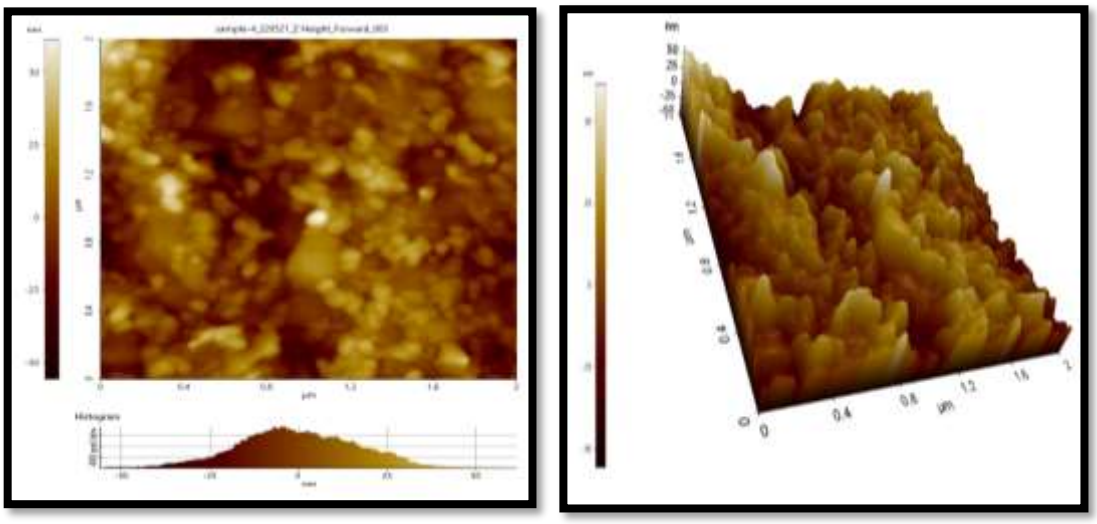


Fig 6. EDX analysis of CdO thin film

The structural details and three dimensional topographic images of CdO thin film are obtained using AFM [18]. Fig 7. 2D and Fig 8. 3D AFM image of CdO thin film synthesized by chemical route at room temperature .Within the scanning area 2 µm × 2 µm area AFM image shows that the particles are strongly agglomerate and arranged in a self-ordered on the glass substrate. AFM image of CdO thin film illustrate that the particles are closely packed arranged together in a vertical alignment and distributed uniformly without any pinholes. The root mean square roughness and average roughness for deposited film is 16.873 nm and 13.525 nm respectively. The flatness (Kurtosis) of CdO thin film was observed 2.966.



Conclusion:

In this work Nanostructured cadmium oxide thin film was successfully synthesized by CBD method on well clean glass substrate. The optimized thin film were characterized by different characterization techniques such as XRD, FESEM, EDX, FTIR UV-Visible spectroscopic AFM analysis. The thickness of the thin film is found to be 182nm. The sharp XRD peaks indicate that the CdO has a face centered cubic and polycrystalline structure. The average particle size is found to be 82.93 nm. The FE-SEM image shows the uniform distribution of CdO particles on the surface of glass substrate. The range of these CdO nanoparticles are in the range of 28nm to 100nm .EDX analysis confirms elemental composition of elements Cd and O on the surface of substrate. In the finger print region of FTIR spectra there are number of vibrations are observed which shows that these peaks are corresponding to preparation of metal oxide structure of CdO. The direct optical band gap energy 'Eg' of cadmium oxide thin film and is found to be 2.2 eV. AFM images show that the particles are strongly agglomerate and arranged in a self-ordered manner on the glass substrate.

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