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Characteristic Analysis on the Suitability of CdO Thin Films Towards Optical Device Applications – Substrate Temperature Effect

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Abstract: Cadmium oxide thin films were synthesized by spray pyrolysis technique using perfume atomizer at different substrate temperature on (225, 275, 325 and 375°C) glass substrates. Effect of substrate temperature on the growth mechanism and physical properties of the films was investigated in order to find the suitability of them towards optical device applications. All the films were polycrystalline in nature having cubic structure with a preferential orientation along the (1 1 1) plane irrespective of the substrate temperature. Film transparency increases from 78% to 89% with increase in substrate temperature. Optical band gap decreases from 2.55eV to 2.38 eV with increase in substrate temperature. The refractive index dispersion of the films obeys the single oscillator model. The dispersion parameters, oscillator and dispersion energy of the films were calculated to analyze their choice in designing optical devices. A minimum resistivity of 3.57 x 10^{-4} Ω cm was obtained for the film coated at 375°C

Keywords: Thin films; X-ray diffraction; Texture coefficient; Optical band gap energy; Electrical studies.

1 Introduction

Transparent conducting oxide (TCO) thin films have great importance in electronic device applications and among these TCOs, cadmium oxide (CdO), an n-type semiconductor with band gap of 2.5 eV [1], has attracted considerable attention for various applications such as solar cells and photodiodes, due to its low electrical resistivity, high carrier concentration and high optical transmittance in the visible region of the solar spectrum [2]. It also finds applications in phototransistors, liquid crystal displays and IR detectors due to its low band gap [3]. It is well know that the electrical properties of CdO films can be improved by the movement of interstitial cadmium atoms and oxygen vacancies. Flores-Mendoza et al. [4] reported that the electrical conductivity and optical transparency of CdO thin films depend on the nature, number, atomic arrangements of metal cations, morphology and on the presence of intrinsic or intentionally produced defects. Lokhande et al. [5] obtained a low resistivity value of 10^{-6} Ω cm for the CdO films fabricated by spray pyrolysis technique at 400°C. They observed that this low resistivity value is due to the increment of oxygen vacancies because of the high substrate temperature adopted. CdO thin films have been deposited by techniques such as dc magnetron sputtering [6], spray pyrolysis [7], chemical bath deposition [8], SILAR [9], pulsed laser deposition [10], sol-gel dip coating [11], etc. of all these techniques, spray pyrolysis has some advantages when compared with other methods. It is quite simple, flexible for process modification, capable of preparing thin films over larger area. Moreover spray parameters can be adjusted to active enhanced film properties. In this work, substrate temperature is varied from 225 - 375°C in steps of 50°C while preparing CdO thin films and the effect of substrate temperature on the physical properties of the films was investigated. Although, there are earlier reports on the electrical and optical properties of CdO thin films based on substrate temperature, studies on the luminescent properties of the films are very scarce in the literature. Also substrate temperature effect to find the suitability of CdO films towards optical devices applications is less evident in the literature. So in the present study, luminescent studies were performed on the CdO films and also the suitability of them towards optical device application was justified.

2 Experimental Details

2.1. Preparation of CdO thin films

Thin films of CdO were prepared by spraying an aqueous solution (50ml in volume) of 0.1 M cadmium

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acetate [Cd $(CH_3COO)_2$] on to ultrasonically cleaned glass substrates (dimensions : 76 mm x 25 mm and 1.4 mm) at different substrate temperatures. The precursor solution was sprayed intermittently on preheated substrates using a perfume atomizer. The use of perfume atomizer has several advantages over the conventional spray method which uses compressed air such as: low cost, lesser substrate temperature fine atomization and improved wettability between the sprayed micro particles and previously deposited layers [12]. When the precursor is sprayed over hot substrates, pyrolytic decomposition of the solution takes place and orange colored films of CdO are formed.

2.2. Characterization of CdO thin films

Film thickness was measured by means of weight gain method and compared with the vales determined by employing a profilometer (Surftest SJ 301). The structural analysis was made using X-ray diffractometer (PANalytical – PW 340/60 x'pert PRO) with CuKα radiation of wavelength 1.5406Å operated at 40 KV and 30 mA in the 2θ range of 10° to 80°. SEM images were obtained using a scanning electron microscope (HITACHI S-3000H). The optical transmission spectra were recorded in the 300-1100 nm wavelength range by using a Perkin Elmer UV-Vis-NIR doble beam spectrophotometer (LAMDA-35). The electrical resistivity was determined using four point probe set up.

3 Results and discussion

3.1 Structural analysis

Fig. 1 shows the XRD patterns of CdO thin films coated at different substrate temperatures using a perfume atomizer.

The XRD patterns revealed that all the films are polycrystalline in nature. The position of the diffraction peaks fit well with the cubic structure of pure CdO (JCPDS card No.65-2908) corresponding to (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) planes. It is seen that the preferential orientation is along the (1 1 1) plane for all the films irrespective of the substrate temperature. Our previous work on CdO thin films prepared by spray pyrolysis technique using perfume atomizer also showed a cubic structure with a preferred orientation along the (1 1 1) plane [13]. Shanmugavel et al. [14] also reported a similar cubic structure for CdO thin films synthesized by spray pyrolysis technique.

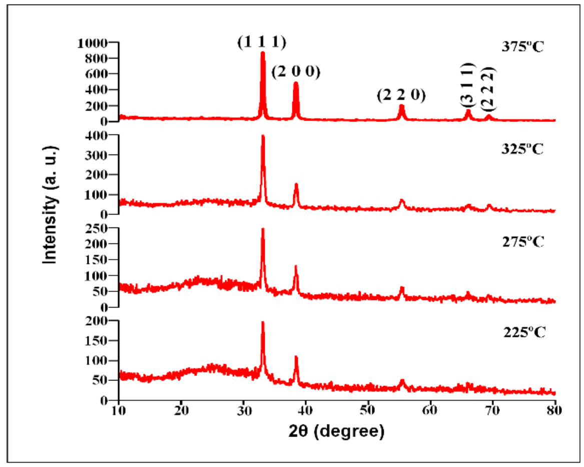

Figure 1: XRD patterns of CdO thin films coated at different substrate temperatures.

The observed 2θ values and calculated lattice constants 'a' (from Bragg's formula: $d = 2\lambda / \sin\theta$, where λ is the wavelength of X-ray used, 'd' denotes the crystalline plane distance for indices (h k l) and θ is the diffraction angle) of the films are presented in **Table 1**.

Table 1: Structural, optical and electrical parameters of CdO thin films

Substrate temperature (°C)	t (nm)	Structural parameters		Optical parameters	
		Observed 2θ of the (1 1 1) plane	Lattice constant 'a' (Å)	Optical band gap, E_g (eV)	Packing density (p)
225	298	33.160°	4.675	2.55	2
275	157	33.15°	4.677	2.48	2.3
325	105	33.132°	4.679	2.42	2.6
375	62	33.118°	4.681	2.38	3

It is seen from **Table 1**, that there is a slight shift of 2θ value of the (1 1 1) diffraction peak towards lower Bragg angle with increase in substrate temperature with induces the observed lattice expansion.

XRD patterns also revealed that the growth temperature induces variation of CdO film growth texture. The texture of a particular plane can be represented by the texture coefficient TC (h k l), which can be calculated from X-ray data using the formula [15]:

$$TC(hkl) = \frac{I(hkl)/I_o(hkl)}{N^{-1}\sum_N I(hkl)/I_o(hkl)} \quad (1)$$

Where I (h k l) is the measured relative intensity of the plane (h k l), I_0 (h k l) is the standard intensity of the plane (h k l) taken from the JCPDS data, and N is the reflection number. Any deviation of the calculated TC value from unity implies preferred growth. The variation of the texture coefficient of CdO thin films with substrate temperature for the diffraction peak (1 1 1) is shown in **Fig. 2**.

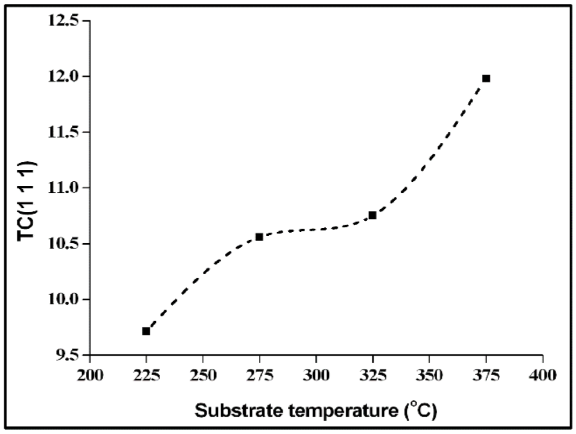

Figure 2: Variation of TC(1 1 1) with substrate temperature

The increment in the value of TC (1 1 1) with increase in substrate temperature indicates the improvement in the crystallinity of the film with substrate temperature.

The crystallite size (D) of the films for the (1 1 1) plane was calculated using the Scherrer formula:

$$D = \frac{0.94\lambda}{\beta\cos\theta} \quad (2)$$

Where β is the full width at half maximum in radians. **Fig. 3** shows the variation of FWHM and crystallite size of the films as a function of growth temperature.

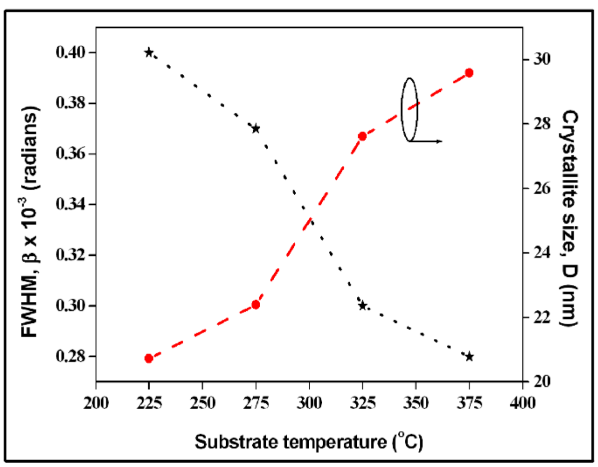

Figure 3: The variation of FWHM and crystallite size of CdO films with substrate temperature.

It can be seen from the figure that both β and D shows an opposite trend behavior. It is observed that the crystallite size increases with increase in substrate temperature confirming the fact that crystallization improves as the growth temperature increases.

In order to explain the growth mechanism, the standard deviation (σ) is calculated by using the equation [16]:

$$\sigma = \sqrt{\frac{I^2_{hkl} - \left(\sum I_{hkl}\right)^2 / N}{N}} \quad (3)$$

Where I stands for the relative intensity of the (h k l) plane. **Fig. 4** shows the variation of standard deviation of the CdO films as a function of substrate temperature.

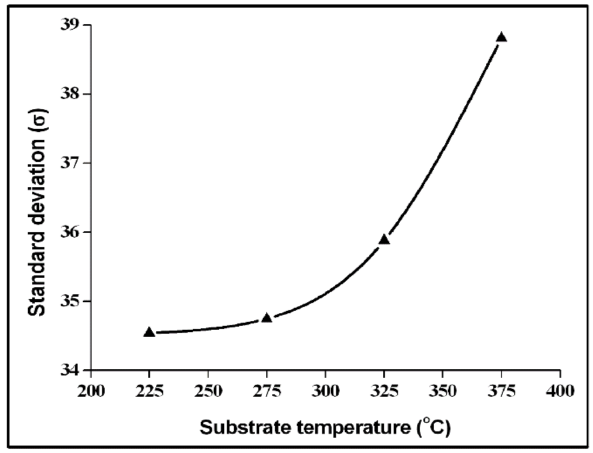

Figure 4: Standard Deviation versus substrate temperature of the CdO films

The increased values of σ observed for the films with growth temperature suggests that the growth nucleation improves with temperature and the high value of σ obtained for the film coated at 375°C suggests that the nucleation and absorption-desorption phenomenon is predominant, as a result of heterogeneous nucleation taking place at that temperature. This is in accordance with Suganya et al. [17] for spray deposited PbO thin films.

3.2. *SEM analysis*

Fig. 5(a – d) shows the SEM images of the CdO films coated at different substrate temperatures. It can be observed from the SEM images, that substrate temperature plays a vital role in the modification of CdO film morphology. Patches of grains interconnected is evident in the morphology of the film coated at 225ºC (**Fig. 5(a)**). This might be probably due to the lack of thermal energy for nucleation of deposited atoms and coalescence of grains at low substrate temperature. As the substrate temperature increases, the film surface modifies with grains having well defined boundaries (**Fig. 5(b)**). Patches of fused grains is minimized and the film surface appears tightly packed without any cracks or holes confirming an improvement in the film crystallinity with temperature.

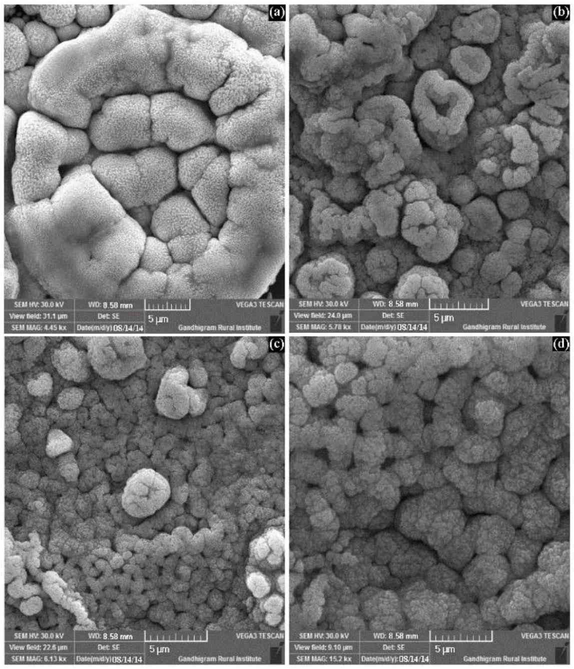

Figure 5: SEM images of the as deposited CdO thin films

For the film coated at 325ºC, the surface modifies with tightly packed grains of almost equal sizes (**Fig. 5(c)**) and only a minimal amount of fused grains are visible. Traces of cauliflower shaped structures appear to bloom at few sites of the film surface. As the substrate temperature is increased to 375ºC, film surface is completely packed with cauliflower shaped nano-structures (**Fig. 5(d)**), which confirms the improved crystallinity of this film. In nano-scale dimension, CdO thin films exhibit numerous structures like nanoparticles, nano-wires, nano-needles, and nano-crystals. Among these structures of this compound, cauliflower like structure emerges from the construction of nanorods bundles. This structure is of great importance due to its high specific area and potential applications in various fields.

3.3. Optical analysis

Optical transmission and reflectance spectra of the CdO thin films are depicted in **Fig. 6**.

It is observed that the film transparency increases with increase in substrate temperature. The low transmission observed for the film coated at 225°C is due to its non-stoichiometric nature which is probably due to the presence of excess un-reacted metallic cadmium ions.

With increase in substrate temperature, film stoichiometry improves due to the decrease in the density of defect centers. As the density of defect centers decreases, light loss by scattering decreases which enhances the transparency of the films. It is also observed from **Fig. 6**, that the overall specular reflectances of the films are very low. The high transmittance and low reflectance values obtained for the films makes them suitable as antireflection coating material for solar cell applications.

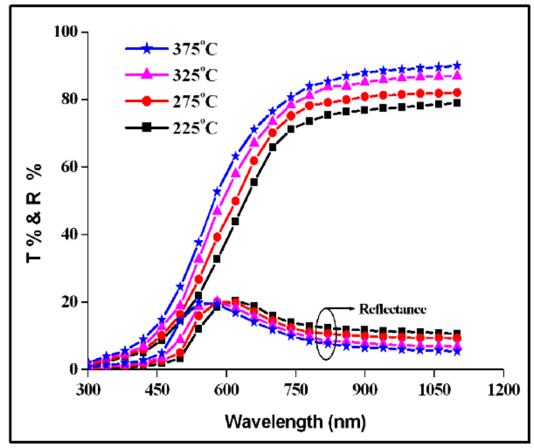

Figure 6: Transmittance and reflectance spectra of the CdO films

From the transmittance spectra, optical band gap of the films were calculated, using the fundamental absorption. The absorption coefficient α and the incident photon energy, hγ are related by the equation [18]:

$$\alpha h\gamma = A\left(h\gamma - E_g\right)^n \qquad (4)$$

where A is a constant, E_g is the band gap of the material and the exponent n depends on the type of transition, n = ½, 2 3/2 and 3 corresponding to allowed direct, allowed indirect, forbidden direct and forbidden indirect, respectively. Taking n = ½, the direct band gap from $(\alpha h\gamma)^{1/n}$ vs. hγ plot (**Fig. 7**) has been calculated by extrapolating the linear portion of the graph to hγ axis at α = 0.

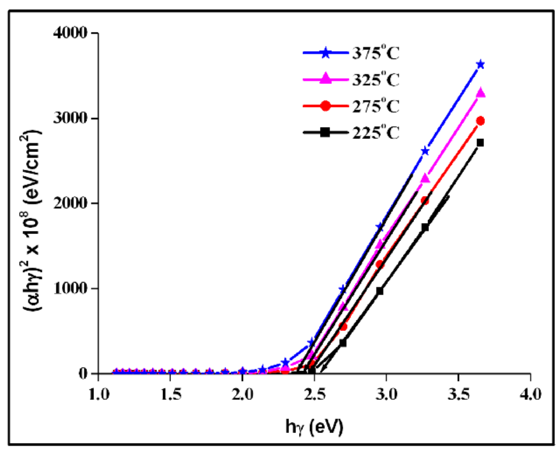

Figure 7: $(\alpha h\gamma)^2$ versus hγ graph of the CdO films

It is seen that direct optical band gap decreased from 2.55 eV to 2.38 eV with increase in substrate temperature (**Table 1**). The band gap value obtained here exactly matches with the value obtained by Dakhel [19] for Zn-incorporated CdO thin films prepared by sol-gel method. The decrease in band gap observed causes a strong red shift in the optical spectra and this is attributed to the increase of density of localized states in the energy gap. The decrease in the optical band gap of CdO with the increase in substrate temperature could be due to the structural modification in the CdO films which could be due to the increase of either substitutional or interstitial Cd^{2+} ions in the CdO lattice.

Refractive index, one of the fundamental properties of an optical material related to the electronic polarization of ions and the local field inside the material can be evaluated from the relation:

$$n = \frac{1+R}{1-R} \pm \sqrt{\frac{4R}{(1-R)^2} - k^2} \qquad (5)$$

Where K = αλ/4 is the extinction coefficient. From the refractive index data, the packing density (p), which is defined as the ratio of the solid volume to the total volume of the film, is estimated using the relation:

$$n = n_s p + (1-p) n_v \qquad (6)$$

where n is the refractive index of the film, n_s is the refractive index of the bulk material and n_v is the refractive index of voids, which are generally filled with ambient, i.e. air (n_v = 1). The calculated values of 'p' are presented in **Table 1**. It can be observed that the packing density increases with increase in substrate temperature and the high value obtained for the film coated at 375ºC indicates improved crystallinity of that film.

The variation of refractive index, n with wavelength is shown in **Fig. 8.** It can be seen that the refractive index of the films decreases with wavelength, i.e., it exhibits refractive index dispersion.

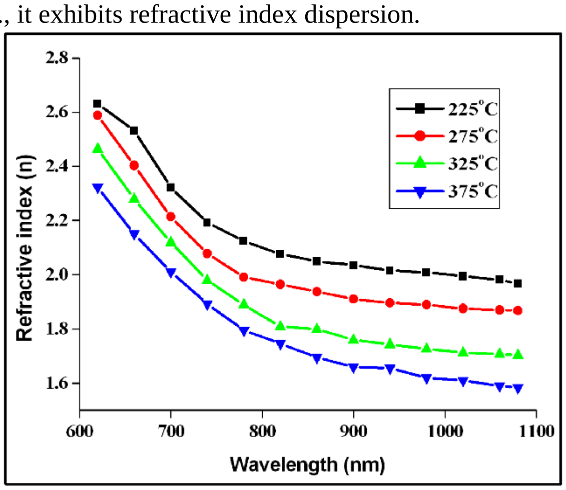

Figure 8: Variation of refractive index with wavelength

There are many dispersion formulae to fit the refractive index over a wide range of frequencies. Single oscillator model developed by DiDomenico and Wemple has been extensively used to analyze the dispersion parameters and this model is expressed by the relation [20]:

$$n^2 - 1 = \frac{E_d E_o}{E_o^2 - (h\gamma)^2} \qquad (7)$$

where n is the refractive index, h is Planck's constant, γ is the frequency, hγ is the photon energy, E_o is the average excitation energy for electronic transitions and E_d is the dispersion energy which is a measure of the strength of inter band optical transitions.

The dispersion parameters can be easily determined by plotting of $(n^2 - 1)$ vs. $(h\gamma)^2$ of CdO thin films. The E_d and E_o values were calculated from the slope $(E_dE_o)^{-1}$ and intercept (E_o/E_d) of **Fig. 9** and the calculated values are presented in **Table 2**.

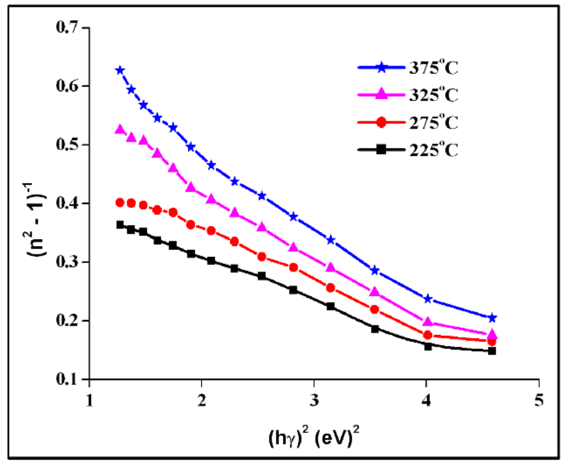

Figure 9: $(n^2 - 1)^{-1}$ versus $(h\gamma)^2$ graph of the CdO films

Table 2: Optical dispersion parameters of CdO thin films.

Substrate temperature (ºC)	Dispersion energy, E_d (eV)	Average excitation energy, E_o (eV)	E_d/E_o	n_∞	λ_o (nm)	S_o x 10^{13} (m^{-2})
225	8.81	3.4	2.591	1.894	364	1.659
275	5.5	2.5	2.2	1.788	357	1.823
325	4.72	2.43	1.942	1.715	345	1.86
375	4.05	2.41	1.674	1.635	305	2.03

The variation of refractive index with wavelength can be expressed by the relation [21]:

$$n^2 - 1 = \frac{S_o \lambda_o^2}{1 - \left(\lambda_o / \lambda\right)^2} \qquad (8)$$

Where λ is the wavelength of incident light, S_o is the average oscillator strength given by $S_o = \left(n_\infty^2 - 1\right) / \lambda_o^2$, and λ_o is an average oscillator wavelength. The parameters, n_∞, S_o and λ_o values were obtained from the slope and intercept of $(n^2 - 1)^{-1}$ vs. λ^{-2} curves (**Fig. 10**) and the obtained values are given in **Table 2**.

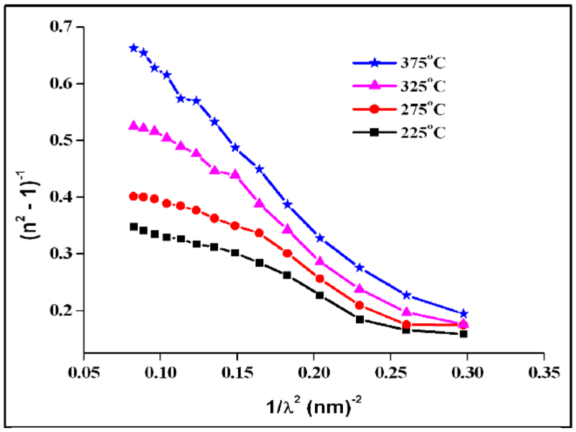

Figure 10: $(n^2 - 1)^{-1}$ versus $(\lambda)^{-2}$ graph of the CdO films

The obtained values of E_o and E_d suggest that the single oscillator model is valid for the CdO films and they can be used in optical device applications. It can be observed from **Table 2** that the value of E_d and E_o shows a decreasing trend with increase in substrate temperature. The decreased values of E_d with increased substrate temperature confirms that the strength of inter band transition decreases and hence the average excitation energy takes lesser values with increase in substrate temperature. This is well acknowledged by the low values of band gap obtained for the films with increase in substrate temperature.

3.4. Electrical studies

CdO is an n-type semiconductor which shows good electrical conductivity even without any doping. The oxygen vacancies and Cd interstitials are responsible for the high electrical conductivity of undoped CdO thin films [22]. Electrical characterization of the CdO films was investigated using the four point probe method. The electrical resistance 'R' was calculated using the relation:

$$R = k\frac{V}{I} \tag{9}$$

Where k is a constant equal to be 4.53, V is the applied voltage and I is the intensity of the dc current. All the films have resistivity in the range of 10^{-4} ohm-cm. **Fig. 11** shows the variation of electrical resistivity of the as deposited samples with substrate temperature.

The resistivity decreases with increase in substrate temperature and the film coated at 375ºC has a minimum resistivity value of 3.57 x 10^{-4} ohm-cm. The resistivity values obtained here exactly matches with the value obtained by Zheng et al. [23] for Sn-doped CdO thin films obtained by pulsed laser deposition. It is well known that substrate temperature governs the stoichiometric variation of sprayed CdO films. Higher substrate temperature leads to nearly stoichiometric films, which show high transmittance, and good uniformity. The minimum resistivity observed for the film coated at 375ºC might be due to the low value of thickness obtained for this film (**Table 1**). The least thickness might also be due to the removal of H_2O vapor which may resist conduction between CdO grains. This is in accordance with Salunkhe et al. [24] for SILAR deposited CdO films.

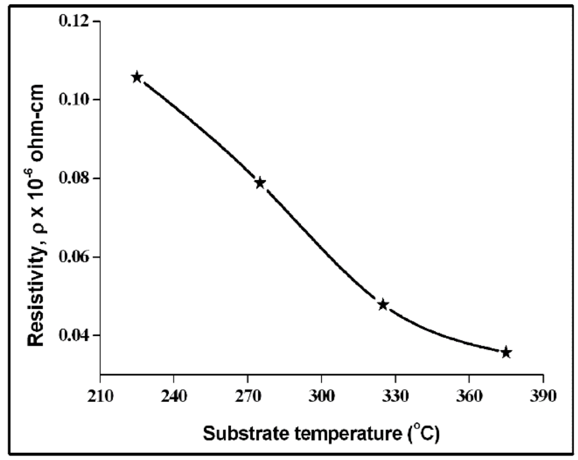

Figure 11: Variation of the electrical resistivity with substrate temperature

3.5. PL studies

Photoluminescence (PL) is an important tool to investigate the quality of a thin film, which depends on size of the crystallites, morphology and chemical environment. Photoluminescence spectroscopy can be used to determine the band gap of semiconductors since the most common radiative transition in semiconductors occur between states at the bottom of the conduction band and the top of the valence band [25]. PL spectroscopy in the band edge emission region is one of the versatile techniques to study the lattice defects in thin film samples. When samples are excited by radiation, characteristic broad luminescence bands are observed with peaks corresponding to the F and V centers. The PL spectra of CdO thin films coated at different substrate temperatures excited at λ = 350 nm are shown in **Fig. 12**.

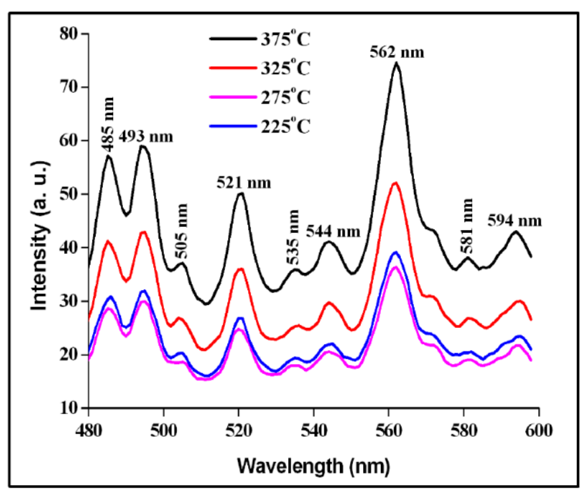

Figure 12: PL spectra of the CdO films

Clearly, it could be seen that there are emission peaks at 485, 493, 505, 521, 535, 544, 562, 581, 585 and 594 nm respectively. The peak at 493 nm is attributed to the excitonic transitions which is size-dependent and excitation wavelength-independent in certain wavelength range. Zhong et al. [26] observed a similar peak at 493 nm (2.48 eV), for ZnO thin films which they treated as donor related luminescence peak. They attributed this green peak to electron transition from the bottom of the conduction band to the O_{zn} level. The weak green emission peak centered at 505 nm may be attributed to the excitonic recombination corresponding to the near-band-edge (NBE) emission. Pan et al. [27] observed this peak for ZnO thin films which they attributed to defects such as oxygen vacancies and interstitial Zn. The peak at 521 nm can be ascribed to the deep trap emission and surface-state emission that is less size dependent [28]. The low intensity peak observed at 535 nm indicates the minimum concentration of surface defects in the films. The peak at 562 nm (2.21 eV) arises from the oxygen vacancies of CdO films because of recombination of photo generated holes in the valence band with electrons in the conduction band. The small width peaks at 485, 544, 581 and 594 nm could not be assigned to any emission process but may be regarded as noise. These PL observations confirm that CdO films deposited here can be used for developing luminescent devices.

4 Conclusion

CdO thin films were deposited on glass substrates at different substrate temperatures. The effect of substrate temperature on the crystal structure, morphology, optical band gap and electrical resistivity of the as-deposited films was investigated. XRD patterns suggested that the films were polycrystalline in nature with cubic crystal structure with a preferential orientation along the (1 1 1) plane irrespective of the substrate temperature. Optical studies revealed that the optical transparency increases and band gap value decreases from 2.55 eV to 2.38 eV with increase in substrate temperature. The high transmittance and low reflectance values obtained for the films makes them suitable as antireflecting coating materials for thin film solar cells. The dispersion parameters, oscillator and dispersion energy for the films were calculated to analyze their choice in designing optical devices. Electrical studies showed that the film coated at 375ºC exhibit a minimum resistivity of 3.57 x 10^{-4} ohm-cm.

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