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Transparent Conducting CdO Thin Films Fabricated by Low Cost Simplified Spray Technique Using Perfume Atomizer

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ABSTRACT

Highly crystalline transparent conducting cadmium oxide thin films were fabricated at 325°C by employing an inexpensive, simplified spray technique using perfume atomizer. X-ray diffraction peaks depict that the films have a polycrystalline structure of cubic phase, with a (1 1 1) preferred orientation. The microstructural parameters such as strain, dislocation density and number of crystallites were found to be equal to 1.003×10^{-3} , 8.163×10^{14} lines/m² and 1.294×10^{22} / unit area respectively. The dislocation density is very low, indicating the good crystallinity of the films. The crystallite size was found to be equal to 35 nm. The band gap energy was found to be equal to 2.25 eV. The refractive index and packing density of the film were 1.81 and 2.07 respectively. M_1 and M_3 moments of the optical spectrum were found to be equal to 2.178 and 237×10^{-3} (eV)⁻² respectively. The resistivity was found to be in the order of 10^{-5} ohm-cm and the activation energy is equal to 0.05 eV. The films were found to have desirable figure of merit (1.194×10^{-3} (Ω/\square)⁻¹) suitable for solar cell applications and low temperature coefficient of resistance (-1.434×10^{-3} / K). Hence this simplified spray technique may be considered as an alternative to the conventional spray technique to coat device quality cadmium oxide thin films over large area.

KEYWORDS: X-ray diffraction; Preferential orientation; Crystal structure; Optical properties; Figure of merit

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1. INTRODUCTION

Transparent conducting oxide (TCO) films have high technological potentials in the field of optoelectronic and other solid state devices. Cadmium oxide (CdO) is one of the promising transparent conducting oxides from II to VI group of semiconductors having high absorption and emission capacity of radiation in the energy gap ¹. Its special features i.e. high conductivity, high transmission, low band gap made it applicable in photodiodes ², phototransistors ³, photovoltaic cells ⁴, transparent electrodes ⁵, liquid crystal displays, IR detectors and antireflecting coatings ⁶, solar cells ⁷, gas sensors ⁸, low-emissivity windows, wear resistant applications, flat panel displays and thin film resistors ⁹. CdO is an n-type semiconductor, with a direct band gap at approximately 2.5 eV. The transmittance of CdO in the visible region of the spectrum is low compared to ZnO ¹⁰, however it is characterized by a much lower resistivity. Conductivity in TCOs can be enhanced either by increasing the charge carrier concentration or by improving the mobility of those carriers. Increasing carrier concentration can be achieved by heavy doping of the TCO materials. This will, however, degrade the transparency due to increased free carrier absorption ¹¹, and reduce the carrier mobility due to an increase in carrier scattering from ionized impurities. Increasing the mobility of charge carriers in TCOs will allow the conductivity to increase without compromising the transparency, thereby enhancing the overall performance of the TCO material. The defect structures of oxides based upon CdO play a major role in determining their unique electrical and optical properties, which qualify them to serve as transparent conductors in a variety of well known applications under specific conditions.

To improve chemical and physical properties of CdO thin films, researchers are trying to modify the synthesis procedure. CdO thin films have been obtained by different techniques such as dc reactive magnetron sputtering ¹², reactive vacuum evaporation process ¹³, chemical bath deposition ¹⁴, spray pyrolysis ¹⁵, successive ionic layer adsorption and reaction (SILAR) ¹⁶, sol-gel ¹⁷, and electro deposition ¹⁸. Among these techniques, spray pyrolysis has been proved simple and inexpensive ¹⁹. In the present work, a simplified spray procedure using a perfume atomizer is employed to deposit CdO films. This simplified version of spray technique has several advantages over the conventional spray method: low-cost, lesser substrate temperature, no need for carrier gas, fine atomization and improved wettability between the sprayed micro particles and previously deposited layers ^{20, 21}. The coated films have been characterized by different techniques such as X-ray diffraction, optical absorption and electrical resistivity for further investigation, which is essential to make full use of their active properties.

2. MATERIALS AND METHODS

CdO thin films were deposited on glass substrates (dimensions – 76 mm x 25 mm x 1.4 mm) by employing a simplified spray technique using perfume atomizer from aqueous solution of cadmium acetate $[Cd(CH_3COO)_2]$ – (Merck made). The deposition parameters adopted to coat CdO films are given in Table 1.

Table No.1: “Deposition parameters applied in this work”

Precursor molar concentration	0.2 M
Substrate temperature	325°C
Substrate to spray nozzle distance	28 cm
Spray angle	45°
Spray interval	2 sec
Spray rate	6ml / min.

B.J. Lokhande et. al ²² and C.H. Bhosale et. al ²³ reported that sprayed CdO films deposited at 400°C have desirable electrical and optical properties. But in this work, CdO films with desirable physical properties were coated at 325°C which is comparatively lesser than the conventional spray technique. The starting solution is sprayed intermittently on pre-heated substrates using a perfume atomizer. Each spray cycle has – a spray and a 2 sec wait so that the desired temperature is maintained throughout the deposition process. Before the start of the deposition, the substrates were cleaned thoroughly with organic solvents using an ultrasonic agitator. X-ray diffraction pattern, SEM and AFM images, optical transmission and electrical resistivity values were obtained using X-ray diffractometer (PANalytical – PW 340/60 X’pert PRO), Scanning electron microscope (HITACHI S-3000H), and Atomic force microscope (Veeco CP - II), PerkinElmer UV-Vis-NIR double beam spectrophotometer (LAMBDA – 35), and four point probe apparatus respectively. X-ray diffractometer was operated at 40 kV and 30 mA with $CuK\alpha$ radiation of wavelength 1.54060 Å. Transmission spectra were recorded in the range 300 – 1100 nm. The thickness of the films was measured by employing a profilometer (Surftest SJ – 301).

3. RESULTS AND DISCUSSION

3.1 Film Formation Process

Aqueous solution of cadmium acetate, when sprayed over the hot substrates kept at 325°C, pyrolytic decomposition of the solution takes place and yellowish colored films of CdO are formed. The volatile byproducts that evolved during thermal decomposition of the initial gradient were expelled out from the deposition chamber by an exhaust system attached to the spray unit. The thickness of the film measured was found to be equal to 0.56 μ m.

3.2 Structural Analysis

Fig. 1 shows the XRD pattern of the CdO films grown at 325°C by the simplified spray technique.

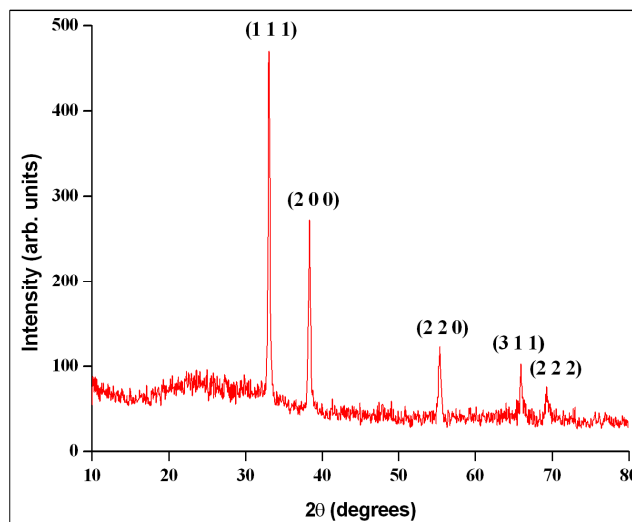


Fig. 1

Fig.1 XRD pattern of CdO film fabricated by the simplified spray technique

The diffraction peaks depict that the films have a polycrystalline structure of cubic phase, with a (1 1 1) preferred orientation. The preferred orientation factor $f(h\ k\ l)$ of the prominent peaks ((1 1 1), (2 0 0), (2 2 0) and (3 1 1)) were calculated and is presented in Table. 2. The preferential orientation factor of the (1 1 1) plane is high (0.5083) indicating a strong orientational growth along that plane. This is in accordance with the reports on CdO thin films prepared by d.c magnetron sputtering²⁴ and by SILAR method²⁵. The observed ‘d’ values are in good agreement with standard ‘d’ values taken from ASTM data file²⁶ as shown in Table. 2.

Table No.2: “d-spacings (d) and calculated preferential orientation factor $f(h\ k\ l)$ of the CdO film coated at 325°C using simplified spray technique”

Standard ‘d’ values (Å)	Observed ‘d’ values (Å)	(h k l) plane	$f(h\ k\ l)$
2.712	2.7093	(1 1 1)	0.5083
2.349	2.3471	(2 0 0)	0.2754
1.661	1.6591	(2 2 0)	0.1191
1.416	1.4152	(3 1 1)	0.0697
1.356	1.3566	(2 2 2)	0.0275

The lattice parameter ‘a’ of the cubic structure of CdO film is calculated using the following formula:

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2} \quad (1)$$

The lattice constant 'a' was found to be equal to 4.694 Å ± 0.002 Å. The obtained 'a' value is in good agreement with other results^{24,30}.

The crystallite size (D) value is calculated using the Scherrer formula²⁷,

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (2)$$

where λ is the wavelength of the X-ray used (1.54060 Å), β is the full-width at half-maximum (FWHM) of the peak which has maximum intensity and θ is the Bragg angle. The grain size was found to be equal to 35 nm.

The dislocation density (δ), defined as the length of dislocation lines per unit volume, has been estimated using the equation²⁸,

$$\delta = \frac{1}{D^2} \quad (3)$$

δ is the measure of the amount of defects in a crystal and the value of dislocation density obtained in this work is found to be equal to 8.163 x 10¹⁴ lines/m². The small value of δ obtained in the present work confirms the good crystallinity of the CdO film fabricated by employing this simplified spray technique. The number of crystallites per unit surface area (N) and the strain (ε) of the film were determined using the formulae:

$$N = \frac{t}{D^3} \quad (4)$$

$$\varepsilon = \frac{\beta \cos \theta}{4} \quad (5)$$

The values of N and ε were found to be equal to 1.294 x 10²² / unit area and 1.003 x 10⁻³ respectively.

3.3 Surface morphological Studies

The SEM image of the CdO film fabricated at 325°C using the simplified spray technique is shown in Fig. 2.

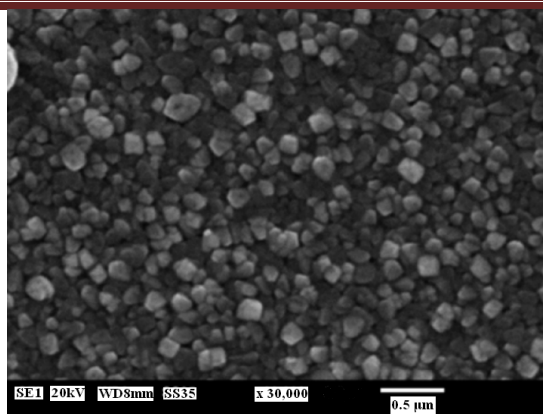


Fig. 2

Fig. 2 SEM image of CdO film coated at 325°C

The SEM image show uniform surface with well-defined grain boundaries. No cluster formation is observed and the grains appear homogeneous and uniform suggesting that there was a uniform nucleation throughout the surface. The micrograph depict that the grains of the as-deposited film are tightly packed. This can be correlated with the high value obtained for the number of crystallites per unit surface area from XRD analysis. The crystallite size calculated from the SEM image was found to be equal to 112 nm. Two-dimensional and three-dimensional AFM images of the as-deposited CdO films are shown in Fig 3(a, b).

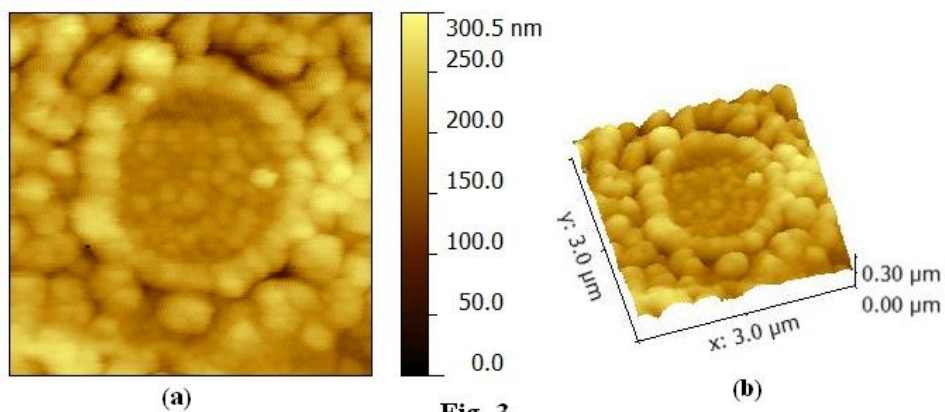


Fig. 3

Fig. 3 Two and three dimensional AFM images of CdO film coated by the simplified spray technique

AFM images show that the film consists of closely packed uniform spherical shaped grains without cracks. This indicates that the film is well adherent with substrate. The roughness of the film was found to be equal to 31.3 nm. The EDS spectrum of the CdO film is shown in Fig. 4 which shows that the film contains the elements Cd and O as expected.

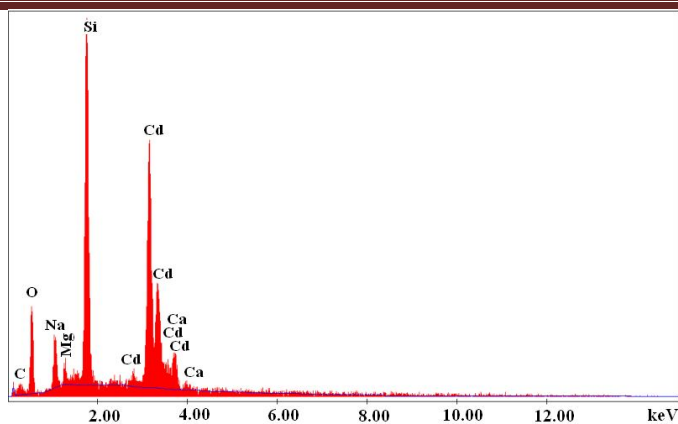


Fig. 4

Fig. 4 EDS spectrum of as-deposited CdO film

Other elements (Ca, Na, Si, Mg) present in the spectrum may be resulted from the glass substrates

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3.4 Optical Studies

The optical absorption of the coated film was studied in the wavelength range 300 – 1100 nm. The transmission spectrum in Fig. 5, shows a shift of transmission edges towards lower energies.

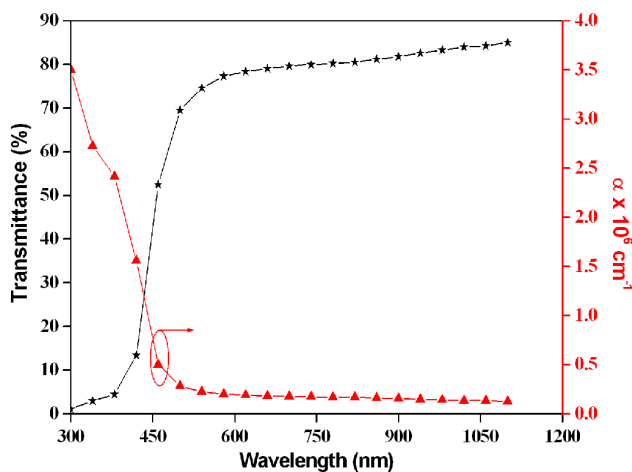


Fig. 5

Fig. 5 Variation of T% and Absorption coefficient of CdO film coated by the simplified spray technique

Such type of shift is found in the CdO films prepared by activated reactive evaporation method²⁹. The shift towards higher or to lower energies depends on the method of film preparation³⁰. The overall average transmittance in the visible region was found to be equal to 81 %. The variation of optical absorption coefficient (α) with wavelength λ which is in the order of 10^6 cm^{-1} , is shown in Fig. 5. It is observed at short wavelengths, a steep decrease occurs in the absorption coefficient around the absorption edge (at E_g). The variation of $(\alpha h\nu)^2$ versus $h\nu$ is shown in Fig. 6 which is a

straight line which on extrapolating the linear portion to the energy axis at $\alpha = 0$ gives the band gap energy value.

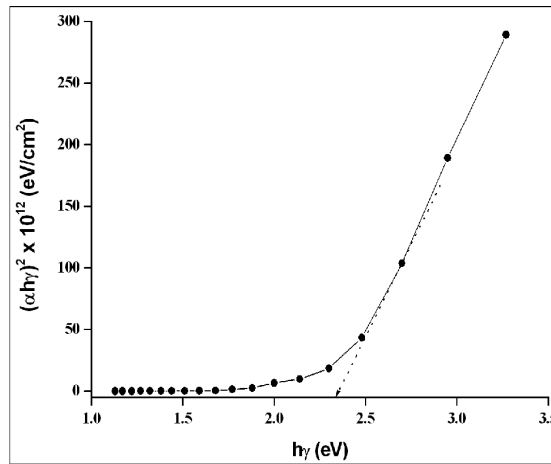


Fig. 6 Variation of $(\alpha h\nu)^2$ versus $h\nu$ of the as-deposited CdO film

The band energy for the as-deposited CdO film was found to be equal to 2.25 eV, which is near to the intrinsic value of the band gap 2.26 eV of CdO³¹.

Optical parameters namely refractive index ‘n’ and extinction coefficient ‘k’, have been determined from the transmittance and reflectance measurements using the relation^{32,33}:

$$n = \frac{1 + R}{1 - R} \pm \left[\left(\frac{R + 1}{R - 1} \right)^2 - (1 - k^2) \right]^{\frac{1}{2}} \quad (6)$$

where $k = \frac{\alpha\lambda}{4\pi}$ is the extinction coefficient, and λ is the incident light wavelength. The

variation of extinction coefficient and refractive index ‘n’ with wavelength is shown in Fig 7.

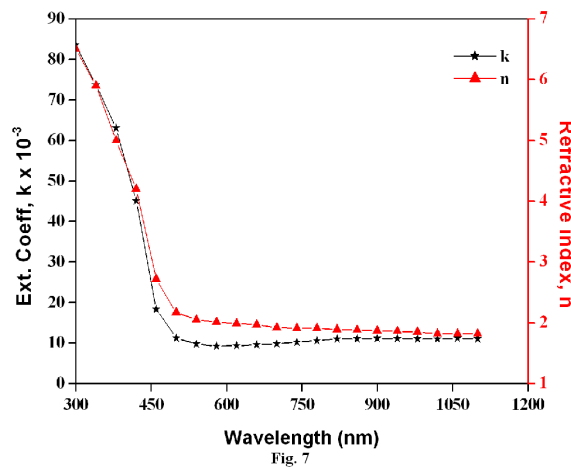


Fig. 7 Variation of extinction coefficient and refractive index of CdO films with wavelength

It is observed that the refractive index decreases with increasing wavelength which can be attributed to the decrease in the packing density of the film and the film might show normal

dispersion. The value of refractive index is found to be equal to 1.81 in the visible region. The obtained value of refractive index exactly matches with the value obtained by Hussein Abdel – Hafez Mohamed et. al³⁴ for the CdO films coated at 450°C. Thus CdO films with decreased packing density are obtained at a comparable lesser substrate temperature using this simplified spray technique which is an added advantage of this technique. On the other hand, the decrease in extinction coefficient could be correlated to the decrease in absorption due to the increased film transparency of the film. The packing density ‘p’ was calculated using^{35, 36}:

$$n^2 = \frac{(1-p)n_v^4 + (1+p)n_v n_s^2}{(1+p)n_v^2 + (1-p)n_s^2} \quad (7)$$

where n is the refractive index of the prepared film, n_s is the substrate refractive index and n_v is the void refractive index (equal to 1 for air). The value of packing density was found to be equal to 2.07. The decrease in the refractive index value with wavelength indicates that the as-deposited CdO films show a normal dispersion.

Refractive index dispersion plays an important role particularly in designing optical devices. The dispersion parameters of the as-deposited CdO films were calculated to analyze their choice in designing optical devices. According to the Wemple-DiDomenico single oscillator model, the refractive index (n) of a dielectric medium in the region of low absorption is given by³⁷.

$$(n^2 - 1)^{-1} = \frac{E_o}{E_d} - \frac{1}{E_o E_d} (h\gamma)^2 \quad (8)$$

where n is the refractive index, h is the Planck’s constant, γ is the frequency, hγ is the photon energy, E_o is the average excitation energy for electronic transitions (single oscillator energy) and E_d is the dispersion energy parameter, which is a measure of the strength of an interband optical transition. From the (n²-1)⁻¹ versus (hγ)² graph (Fig. 8), E_d and E_o can be directly determined from the slope (E_dE_o)⁻¹ and intercept E_o/E_d at the vertical axis.

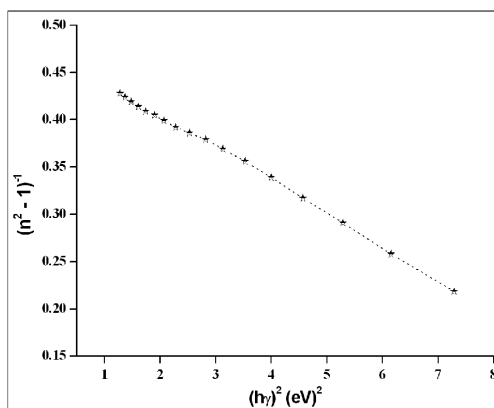


Fig. 8 (n²-1)⁻¹ versus (hγ)² graph

The values of E_o and E_d were found to be equal to 3.03 eV and 6.6 eV respectively. The obtained values of E_o and E_d suggest that the single-oscillator model is valid for the coated CdO films. The oscillator energy E_o , can be correlated with the optical gap by the empirical formula, $E_o \approx 1.5 E_g$. A measure of interband transition strengths can be provided from the M_{-1} and M_{-3} moments of the optical spectrum. The M_{-1} and M_{-3} moments are expressed as ³⁸:

$$M_{-1} = \frac{E_d}{E_o}, \quad M_{-3} = \frac{E_d}{E_o^3} \quad (9)$$

The calculated values of M_{-1} and M_{-3} are 2.178 and $237 \times 10^{-3} (\text{eV})^{-2}$ respectively.

It is well known that polarizability of any solid is proportional to its dielectric constant. The real and imaginary parts of the complex dielectric constant are expressed as ³⁹:

$$\varepsilon_1 = n^2 - k^2 \quad (10)$$

$$\varepsilon_2 = 2nk \quad (11)$$

where ε_1 and ε_2 are the real and imaginary parts of the dielectric constants, respectively. The variation of ε_1 and ε_2 values of the CdO films with wavelength is shown in Fig. 9.

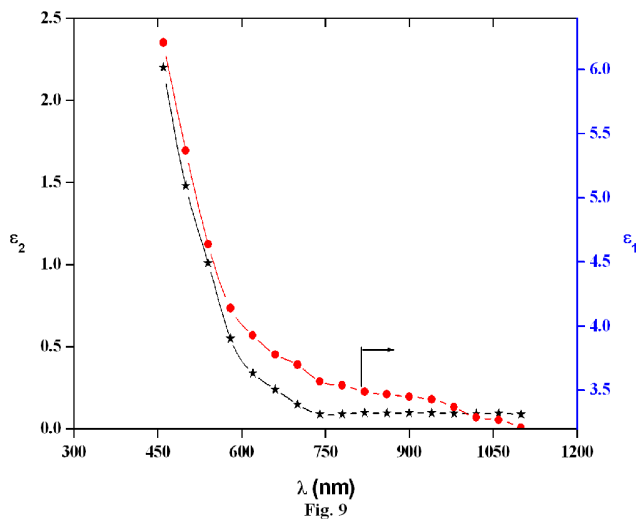


Fig. 9 Variation of real and imaginary parts of dielectric constant with wavelength

The real and imaginary parts of dielectric constants follow the same trend. The variation of the dielectric constants with photon energy indicates that some interactions between photons and electrons in the films are produced in this energy range. The imaginary part of dielectric constant is directly related to the density of states within the forbidden gap of semiconductor materials ⁴⁰.

The electronic states in materials can be studied from their optical conductivity, which can be determined by using the relation ⁴¹:

$$\sigma_{opt} = \frac{\alpha nc}{4\pi} \quad (12)$$

where c is the velocity of light, α is the absorption coefficient and n is the refractive index. The variation of optical conductivity with photon energy ($h\nu$) is shown in Fig. 10.

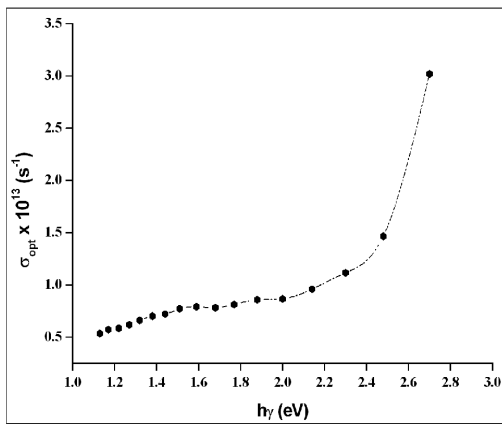


Fig.10 Variation of optical conductivity with photon energy of CdO films coated at 325°C

The optical conductivity increases sharply beyond 2.3 eV which might be due to the variations in the absorption coefficient of the films in that spectral range.

3.5 Electrical Studies

The electrical resistivity of the CdO films fabricated at 325°C using the simplified spray technique was measured as a function of temperature in the range 308 K – 348 K using the four point probe method. The variation of resistivity ρ with reciprocal of temperature is shown in Fig. 11.

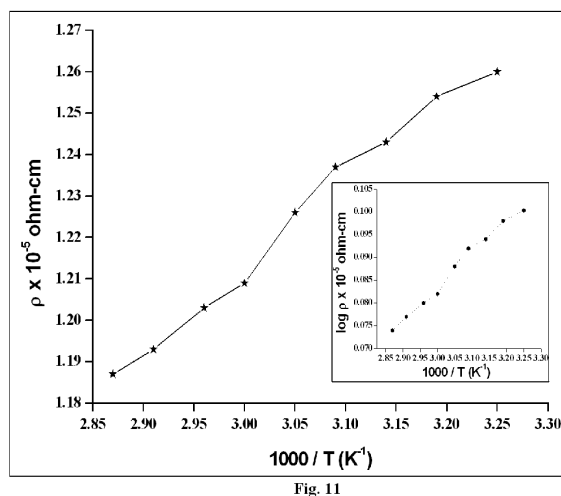


Fig. 11 Variation of resistivity and log resistivity with inverse of temperature

It is observed that the resistivity decreases linearly with increase in temperature indicating a semiconducting electrical behaviour of CdO. The electrical resistivity was found to be in the range of 10^{-5} ohm-cm. The resistivity value is very low which might be due to enhanced carrier

concentration which results in increased carrier mobility which can be related to the better crystallinity of the CdO film obtained, as evident from the XRD analysis. Another reason for the low resistivity value is the increase in oxygen vacancies which leads to the annihilation of oxygen acceptor states at the grain boundaries that act as traps for electrons⁴². The low resistivity value might also be due to the removal of H₂O vapour from the film surface, which may resist the conduction between CdO grains^{43, 44}. The defects of oxygen vacancies and cadmium interstitials are responsible for high conductivity of CdO thin films⁴⁵. The low resistivity and high transmittance values obtained for the as-deposited CdO films makes them as important TCO layers, especially suitable for solar cell applications⁴⁶. From the slope of log ρ versus inverse of temperature graph shown in the inset of Fig. 11, the activation energy is estimated and is equal to 0.05 eV. The obtained value of activation energy in the present work exactly matches with the value obtained by Uplane et. al¹⁵ for the CdO film deposited at 400°C.

From the resistance values recorded at different temperatures (35°C, 45°C, 55°C, 65°C and 75°C), the temperature coefficient of resistance of the as-deposited CdO film is evaluated. The value of TCR is found to be equal to $-1.43 \times 10^{-3} / \text{K}$. The suitability of the transparent conducting thin films for various applications is judged by their figure of merit, $\phi(\lambda)$, which can be calculated using Haacke's formula⁴⁷ as

$$\phi(\lambda) = \frac{T(\lambda)^{10}}{R_{sh}} \quad (13)$$

where T is the transmittance at $\lambda = 500 \text{ nm}$ and R_{sh} is the sheet resistance. The value of ϕ is in the order of $1.194 \times 10^{-3} (\Omega/\square)^{-1}$ which is desirable for solar cell applications.

4 CONCLUSIONS

Highly crystalline transparent conducting CdO thin films were fabricated at relatively low temperature by employing a simplified version of spray technique using perfume atomizer. The films have polycrystalline structure with preferential orientation along (1 1 1) plane. The grain size from XRD analysis was found to be equal to 35 nm. Various microstructural parameters such as strain, dislocation density and number of crystallites were calculated. SEM analysis showed no cluster formation with grains appearing homogeneous and uniform suggesting that there was a uniform nucleation throughout the surface. The coated films exhibit an overall average transmittance of 81% in the visible region and the obtained optical band gap value was found to be near the intrinsic value. Refractive index value decreases with increase in wavelength and films exhibit normal dispersion. The dispersion parameters were calculated to analyze the choice of the deposited CdO films in designing optical devices. Measurement of interband transition strengths was performed from the optical spectrum. The variation of the dielectric constants with photon

energy indicates that some interactions between photons and electrons in the films are produced in energy range studied. The electrical resistivity was found to be in the range of 10^{-5} ohm-cm. The activation energy was found to be equal to 0.05 eV. The coated films have desirable figure of merit suitable for solar cell applications and low temperature coefficient of resistance. Hence this simplified spray technique may be considered as a promising alternative to the conventional spray method for the fabrication of device quality CdO thin films on large area.

5. ACKNOWLEDGEMENTS

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