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PREPARATION OF CADMIUM OXIDE THIN FILMS BY SPRAY TECHNIQUE USING PERFUME ATOMIZER AND EFFECT OF SOLVENT VOLUME ON THEIR PHYSICAL PROPERTIES

G. Shanmugavel[1] --- A.R. Balu[2] --- V.S. Nagarethinam[3]

[1] *Research Scholar, PG and Research Department of Physics, A Veeriya Vandayar Memorial Sri Pushpam College, Tamilnadu, India*

[2,3] *Assistant Professor, PG and Research Department of Physics, A Veeriya Vandayar Memorial Sri Pushpam College, Tamilnadu, India*

ABSTRACT

Cadmium oxide (CdO) thin films were deposited from starting solutions having different values of solvent volume (30 – 60 ml) by employing a simplified spray technique using perfume atomizer. X-ray diffraction studies showed that all the films exhibit cubic structure with preferential orientation along the (1 1 1) plane. The crystallinity increases as the solvent volume increases. Film coated with 60 ml solvent volume has minimum value of strain (3.972×10^{-3}) and dislocation density (1.062×10^{15} lines/m^2) which confirm the improved crystallinity of that film. The sheet resistance (R_{sh}) of the film coated with 30 ml solvent volume was found to be minimum (14.69×10^3 ohm/sq.) and there was a sharp increase in R_{sh} for higher values of solvent volume. The Energy dispersive X-ray analysis confirms that the number of oxygen vacancies governs the electrical resistivity of the CdO films. The films were found to have good optical characteristics which make them suitable for optoelectronic devices.

Keywords: X-ray diffraction, Preferential orientation, Crystal structure, Thin films, Optical band gap, Electrical resistivity.

Contribution/ Originality

This study is one of very few studies which have investigated CdO thin films based on solvent volume. The paper contributes the first logical analysis on CdO thin films based on solvent volume. This study documents the effect of solvent volume on some physical properties of CdO thin films.

1. INTRODUCTION

Conducting metal oxide thin films such as zinc oxide, indium oxide, cadmium oxide, etc. are widely used for various applications such as transparent electrodes, solar cells, phototransistors, liquid crystal displays, optical heaters and gas sensors [1-3] because of their high electrical conducting and optical transmittance. Among these conducting metal oxides, CdO has attracted notable attention for various applications such as solar cells, photodiodes, etc., because of its low electrical resistivity, high carrier concentration and high optical transmittance in the visible region of the solar spectrum. CdO is an n-type semiconductor having band gap of 2.27eV [4] The high conductivity of undoped CdO ($10^2 - 10^4$ S/cm) is because of defects of oxygen vacancies and cadmium interstitials [5]. Different techniques such as SILAR [6], sol-gel [7], spray pyrolysis [8], ion beam sputtering [9], magnetron sputtering [10], chemical laser deposition [11], chemical vapor deposition [5] and pulsed laser deposition [12] have been used for deposition of pure and doped CdO films. Among these techniques, spray pyrolysis technique has the following advantages: low cost, simple and possibility of large area coating [13]. Also, spray parameters such as spray rate, substrate temperature, spray nozzle distance, solution concentration, and pressure of carrier gas can tune the film properties well. In the present work, spray pyrolysis technique using perfume atomizer is used to deposit CdO thin films from the starting solution having different solvent volumes. The effect of solvent volume on the structural, morphological, optical and electrical properties of the films was studied.

2. EXPERIMENTAL DETAILS

Chemical used (Cadmium acetate ($Cd(CH_3COO)_2$)) for the deposition of CdO thin films was of analytical reagent grade (99 % purity procured from Sigma Aldrich). Different sets of starting solutions were prepared by dissolving 0.1 M cadmium acetate with different volumes of (30, 40, 50 and 60 ml) doubly deionized water. The solutions thus got are sprayed manually by a perfume atomizer on preheated glass substrates with dimensions of 75 x 25 x 1.35 mm^3 kept at 400°C. A temperature controller with chromel-alumel thermocouple is used to keep the substrate temperature constant. The substrates were pre cleaned ultrasonically with organic solvents and doubly deionized water for degreasing and to remove the contaminations if any on the surface.

Film thicknesses were calculated using gravimetric weight difference method. Diffractometer (PANalytical-PW 340/60 X'pert PRO) working at 40 kV and 30 mA using CuKα radiation (λ = 1.5406Å) is used to find out the structure, crystallinity and phase of the CdO thin films. The surface morphology of the films was characterized by scanning electron microscope HITACHI model S-3000H. To find out the band gap energy of CdO in thin film form, the optical transmission studies were carried out using UV-vis-NIR double beam spectrophotometer (Perkin Elmer LAMBDA – 35). Measurements of electrical resistivity were carried out with a four-point probe set up.

3. RESULTS AND DISCUSSION

3.1. Film Formation

When aqueous solution containing the precursor salt is atomized over hot glass substrates using perfume atomizer, pyrolytic decomposition takes place resulting in the deposition of orange colored CdO thin films. Fig.1 shows the variation of the weight of mass deposited and thickness of the CdO films coated at different solution volumes. As seen, the weight of CdO deposited on substrate surface increases with increase in solvent volume which reflects on the film thickness. For spray deposited films, amount of mass deposited on glass substrates per unit area depends on the reaction kinetics occurring at the substrate surface in one hand and the quantity of reactive species Cd and O on the other hand. The reaction kinetics in this work does not contribute to film thickness as the substrate temperature is kept constant as 400°C. Therefore the quantity of reactive species (Cd and O) alone contributes the films thickness in the present study. The quantity of reactive species deposited depends on i) molarity of the precursors used, and ii) volume of the solution sprayed. So in the present case, solution volume alone contributes for the quantity of reactive species. Therefore because of high amount of reactive species deposited with increased solution volume the thickness increases. The terminated thickness at which the highest amount of CdO was deposited on the substrate was 0.1039 mg/cm^2 for 60 ml solvent volume.

3.2. Structural Studies

The XRD patterns of CdO thin films grown from starting solution having different solvent volumes (30ml, 40ml, 50ml and 60ml) are show in Fig.2 (a – d). These patterns show sharp and narrow diffraction peaks suggesting the films display good crystallinity. The X-ray diffraction profiles reveal that the films are polycrystalline in nature with cubic structure. All the films have preferential orientation along the (1 1 1) plane irrespective of the solvent volume. Our previous work on CdO thin films prepared by the SILAR technique showed a similar structure with a preferential orientation along the (1 1 1) plane [6]. The intensity of the peaks (1 1 1), (2 0 0) and (2 2 0) increases as the solvent volume increases, showing the crystalline nature of the samples. The other prominent peaks in the XRD pattern corresponds to the planes (2 0 0), (2 2 0), (3 1 1) and (2 2 2) according to the JCPDS card No. 65-2908. The preferential orientations of the films were evaluated by calculating the preferential orientation factor f(hkl) from the X-ray data by a method reported earlier [14]. The variation in preferential orientation factor f(h k l) for the (1 1 1) and (2 0 0) planes as a function of solvent volume is shown in Fig. 3. As seen from Fig. 3, the f(1 1 1) increases as the solvent volume increases whereas f(2 0 0) decreases. The results revealed that solvent volume of the starting solution influences the structural properties of the CdO films. Scherrer's formula [15] is used to calculate the crystallite size (D) of the as deposited samples, $D = \frac{0.9\lambda}{\beta \cos\theta}$ where β is the full width at half maximum, θ is the Bragg's angle and λ is the wavelength of the X-ray used (1.5406Å). The calculated crystallite size values are found to be in the nano-range and are presented in Table 1.

Table-1. Structural parameters of CdO thin films

Solvent volume (ml)	**Crystallite size, D (nm)**	**Grain size (nm)**	**Lattice constant, 'a' (Å)**	**Strain, ε x 10^{-3}**	**Dislocation density, δ x 10^{15} lines/m^2**
30	20.2	250	4.690	6.026	2.451
40	23.01	178	4.694	5.293	1.89
50	26.72	131	4.692	4.563	1.401
60	30.68	89	4.691	3.972	1.062

It is observed that the crystallite size increases as the solvent volume increases. This may be because of the tendency of high agglomeration among smaller crystallites to form larger crystallites.

To examine the film growth nature, standard deviation (σ) is calculated using the equation [16]:

$$\sigma = \sqrt{\frac{\sum I^2_{hkl} - \left(\sum I_{hkl}\right)^2 / N}{N}} \qquad (1)$$

where I stands for the relative intensity of the (h k l) plane. The variation of standard deviation as a function of solvent volume is shown in Fig. 4. The high value of σ obtained for the film coated with 60 ml solvent volume suggests that the nucleation and absorption-desorption phenomenon is predominant because of heterogeneous nucleation taking place at that solvent volume.

The lattice constant 'a' for the CdO films is calculated from the equation:

$$a = \frac{d}{\left(h^2 + k^2 + l^2\right)^{1/2}} \qquad (2)$$

Where d is the interplanar distance. The calculated 'a' values are presented in Table.1. The 'a' value obtained for the film coated with 60 ml solvent volume exactly matches the standard value (JCPDS card No. 65-2908). Deviation in the lattice parameter values of the films coated with solutions having solvent volumes 30 ml, 40ml and 50 ml suggest that the films were under strain. The strain may be because of the oxygen vacancies and the interstitial Cd atoms incorporated in the CdO lattice. The strain present in the samples is calculated using the relation [17]:

$$\varepsilon = \frac{\beta \cos\theta}{4} \qquad (3)$$

Film coated with 60 ml solvent volume has the minimum value of strain which strongly favors for the improved crystallinity of this film. The minimum value of dislocation density (δ) obtained (1.062 x 10^{15} linear/m^2) for the film coated with 60 ml solvent volume strongly supports the discussion given above, as δ is the measure of amount of defects in a crystal. The lesser value of δ obtained for the film coated with 60ml solvent volume show the degree of crystallization of the film.

3.3. Surface Morphology

Fig. 5 (a – d) shows the SEM micrographs of CdO thin films deposited with different solvent volumes. The surfaces of the films are homogeneous (with no voids) and densely packed. Well-defined grains show the crystalline nature of the samples. The films deposited from solution having lesser solvent volume (30ml) has grains fused together (Fig. 5(a)). The SEM image of the film coated with 40ml solvent volume (Fig. 5 (b)) depicts densely packed patches of small sized grains fused together. For the film coated with 50 ml solvent volume, circularly shaped grains having different sizes are found scattered throughout the substrate surface (Fig. 5(c)). Traces of few grains fused together appear on the film surface. No fused grains appear for the film coated with 60ml solvent volume (Fig. 5(d)). Spherical grains with well define boundaries and with almost equal size found scattered throughout the surface, confirming the improved crystallinity of the film. Film morphology thus changes with solvent volume. The grain size of the films estimated from the SEM images is presented in Table 1. The XRD results (section 3.2) showed that the crystallite size increases with the increases in solvent volume, whereas the SEM images showed that the grain size of the film gradually decreases as the volume of the solvent increases. These results infer that crystallite size and grain size are not necessarily the same since a grain may be composed of multiple crystallites [18]. Crystallites are the coherently diffracting domains of crystals and grains may contain several of these domains. Deformation of a single crystal results in sub grains with different orientations. Now every sub grain having will be considered a crystallite. In other words, if a grain contains several sub grains, then each sub grain is a crystallite. As a special case, if a grain is made up of a single crystallite, the grain size and crystallite size have the same value. In the present work, the grain size (250 nm) is the largest for the film deposited from 30ml solvent volume while the crystallite size (20.2nm) is the smallest. It may be due to the fact that the grain is composed of nearly twelve number of smaller crystallites i.e., agglomeration of more crystallites takes place in this case. Losurdo, et al. [19] reported that agglomeration of crystallites results in increased grain size of indium tin oxide thin films. The grain of the CdO film prepared from 40ml solution is composed of lesser number of crystallites (eight). So, the grain size is lesser even though the crystallite size is slightly larger when compared with the previous case. This trend continues up to the film prepared from the solution having solvent volumes 50ml and 60ml respectively. The film coated with 50ml solvent volume has grain composed of nearly five crystallites whereas the film coated with 60 ml, it is composed of only three crystallites. So, obviously as the solvent volume increases, a stage will reach where the crystallite size equals the grain size. Now the grain has only a single crystallite. The variation in the grain size can be used to discuss the change in the electrical resistivity of the films (discussed in Section 3.5) as given below. When the grain size is larger, the charge transport is mainly intragrain which result in a substantial improvement in the electrical conductivity of the films. But for smaller grains, the fairly higher number of grain boundaries may act as low conductivity blockades making the resistivity higher.

3.4. Elemental Analysis

The EDAX spectra of the CdO films deposited from starting solutions having different solvent volumes shown in Fig. 6, confirms the presence of Cd and O as expected.
The atomic proportion of the elements Cd and O present in the films are presented in Table 2.

Table-2. Elemental compositions of the CdO films

Solvent volume (ml)	Elements		O/Cd ratio
	Atomic %		
	Cd	O	
30	46.64	36.10	0.77
40	44.79	38.86	0.87
50	45.91	40.71	0.89
60	43.29	43.75	1.01

The composition of the elements confirms the presence of oxygen vacancies in the films from solution having lesser solvent volume (30 ml). Each oxygen vacancy creates a free electron in CdO lattice which increases the carrier concentration of the film which results in the minimum R_{sh} of this film. As the solvent volume increases, the atomic percentage of oxygen increases, and as a result the number of oxygen vacancies is decreased which causes a decrease in carrier concentration and consequently an increase in R_{sh}. The O/Cd ratio of the film deposited with 60 ml solvent volume is equal to 1, which confirms that the film exhibit better stoichiometric nature, which may be the main reason for the improved crystallinity of this film as discussed in Section 3.2.

3.5. Optical Studies

Fig. 7 shows the optical transmittance spectra of the CdO films deposited from starting solutions having different solvent volumes in the wavelength range of 300-1100 nm.

The variations in the solvent volume affect markedly the optical properties. Film coated with 30 ml solvent volume show a high transmittance of 85 %. The transmittance gradually decreases for the films with increase in solvent volume. This might be due high thickness of the films and the optical scattering at the increased number of grain boundaries. Usually in transparent metal oxides, metal or oxygen ratio in the film decides the percentage of optical transmittance. Metal rich film usually shows less transparency [20]. Also the optical transmittance is known to strongly depend on the grain size of the films. Larger grain size marks to the high transmittance for the films coated with less solvent volume. When the grain size increases, the grain boundary scattering reduces, which causes an improvement in the transmittance. The surface morphology (Fig. 5) supports this fact. From the SEM images, grain size decreases as the solvent volume increases. The decrease in the grain size causes an increase in the grain boundary density of the film. It is also viewed that optical absorption edge slightly shifts towards blue region, as the solvent amount increases, suggesting a gradual increase in the optical band gap (E_g). The sharp absorption edge obtained for all the cases clearly show the good crystallinity of the films. The fundamental absorption which clearly shows itself by a rapid fall in the transmission can be used to find out the band gap of the as deposited samples. The absorption coefficient (α) can be calculated from the transmittance (T) values from the Lambert law, $\alpha = \frac{\ln(1/T)}{t}$. The variation of absorption coefficient with photon energy (hγ) takes the form $\alpha = A\left(h\gamma - E_g\right)^{n/2}$ [13], where E_g is the band gap, 'A' is a constant related to the effective masses associated with the bands and n is a constant which is equal to one for a direct- gap material and four for an indirect-gap material. To decide whether the CdO films deposited using the simplified spray technique have direct or indirect bang gap, $(\alpha h\nu)^2$ vs. (hν) and $(\alpha h\nu)^{1/2}$ vs. (hν) plots are drawn. Since better linearity is obtained in the $(\alpha h\nu)^2$ vs. (hν) plot, the direct band gap values are determined by extrapolating the linear portion of this plot to the energy axis (Fig. 8). The obtained band gap values are presented in Table 3.The optical band gap (E_g) is found to be in the range of 2.34 – 2.51 eV for the films. As the solvent volume increases, the optical band gap is blue shifted from 2.34 to 2.51eV. Thus increased solvent volume widens the optical band gap. This band gap broadening is due Moss-Burstein effect. The Moss-Burstein effect is associated with lifting the Fermi level into the conduction band of degenerate semiconductors, which leads to broaden the band gap.

3.6. Electrical Studies

CdO film is an n-type semiconductor which has good electrical conductivity even without any extrinsic doping. The factors that influence the resistivity of chemically sprayed CdO films are: i) presence of oxygen vacancies and ii) Cd incorporation at the interstitial sites [5]. The sheet resistance (R_{sh}) and resistivity values of CdO thin films are presented in Table 3.

Table-3. Optical and electrical parameters of the CdO thin films

Solvent volume (ml)	**Transmittance (%)**	**Optical band gap, E_g (eV)**	**Sheet resistance, R_{sh} x 10^3 ohm/sq.**	**Resistivity, ρ x 10^1 ohm-cm**
30	85.123	2.34	14.69	0.101
40	82.54	2.4	27.41	0.234
50	80.13	2.46	182.06	1.1748
60	77.8	2.51	234.24	2.764

Fig. 9 shows the variation of sheet resistance with solvent volume. For CdO film coated with 30ml solvent volume, the R_{sh} is 14.69 KΩ/sq. and the value increases sharply to 234.24 KΩ/sq. (by nearly 29 times) for 60ml solution. The number of oxygen vacancies may be the reason for the above results. When the amount of solvent in the solution is low (say 30ml), the number of oxygen atoms offered by the solvent (water) is not enough for forming stoichiometric CdO structure. So, many oxygen vacancies are present in the deposited film. The representative EDAX spectrum shown in Fig. 4a and the quantitative results presented in Table 2 confirm the presence of oxygen vacancies in the cadmium oxide films prepared from solution having lesser solvent volume. The atomic percentage of oxygen atoms (36.10) is remarkably less than the value of the existing Cd atomic percent (46.64) suggesting the presence of oxygen vacancies for the film coated with 30 ml solvent volume. So carrier concentration is increased. As, R_{sh} is inversely proportional to the carrier concentration in the system, it has the low value in this case. Beside oxygen vacancies, Cd incorporation at interstitial sites also increases the carrier concentration with simultaneous increment in the electrical conductivity of the CdO films. Cd incorporation at interstitial sites provided conduction electrons because of an overlap of outer orbital when they can easily occupy the most spacious combination of interstitial lattice sites. This is accordance with the reports of Agashe, et al. [21] for SnO_2 thin films. But when the solvent volume increases, availability of oxygen atoms from the H_2O molecules increases correspondingly. As a result the amount of oxygen vacancies decreases which is obvious from the quantitative EDAX results of the films prepared with 50ml and 60ml volume (Table 2). The EDAX results reveal lesser oxygen vacancies as the solvent volume increases. This reduction in oxygen vacancies results in decreased carrier concentration with simultaneous increase in the R_{sh} as shown in Table 3.

4. CONCLUSION

The effects of solvent volume on the structural, morphological, optical and electrical properties of spray deposited CdO thin films were studied. The films are found to have good physical properties. The electrical studies revealed that a low resistivity can be achieved for CdO films by using solutions prepared from lesser amount of solvent, as it causes large number of oxygen vacancies in the CdO lattice. The resistivity is found to increase with the increase in solvent volume. The optical band gap is found to be 2.34 eV for film prepared from lesser volume (30 ml) of the solvent and the band gap is blue shifted as the solvent volume increases. The better optical and electrical properties make the films suitable for optoelectronic devices.

5. ACKNOWLEDGMENTS

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Illustrations

Fig-1. Variation of the weight of mass deposited and thickness of CdO thin films as a function of solvent volume.

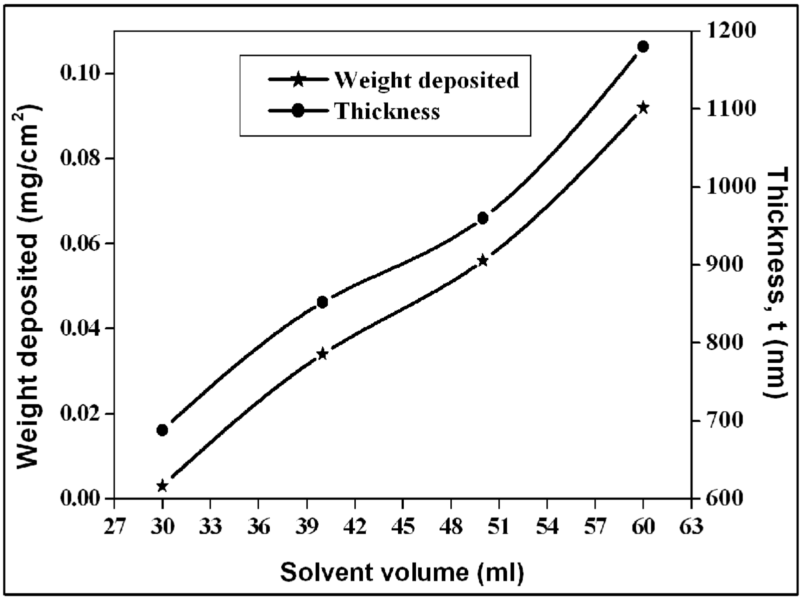

Fig-2.XRD patterns of the CdO films.

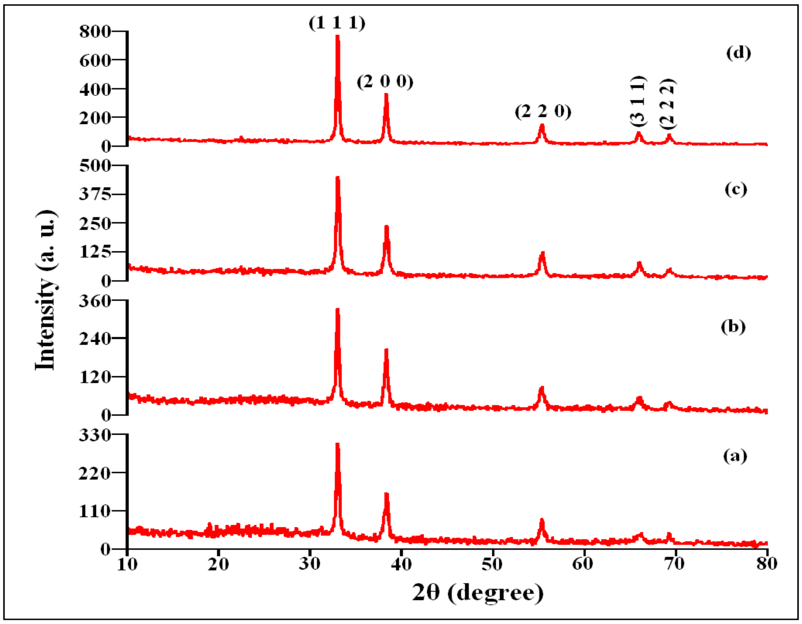

Fig-3. Variation of f(1 1 1) and f(2 0 0) as a function of solvent volume.

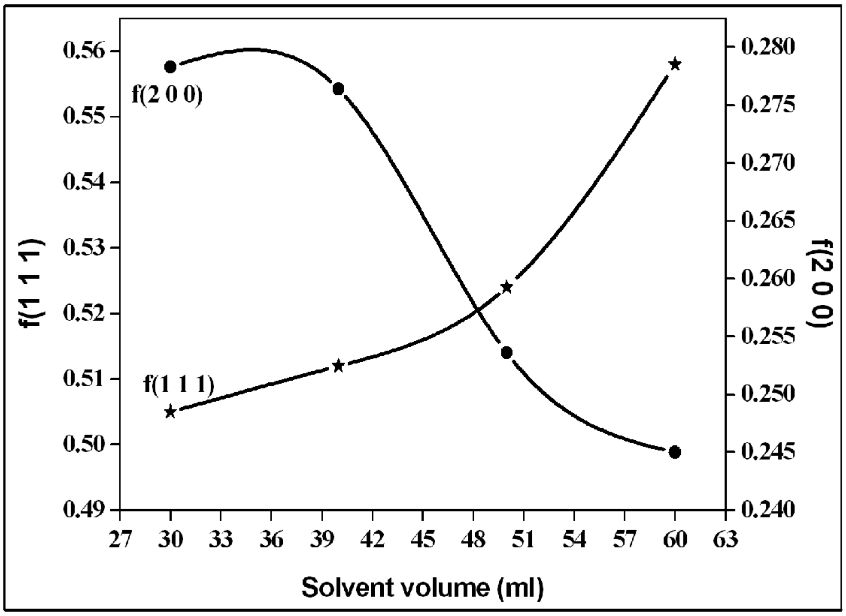

Fig-4. Standard deviation as a function of solvent volume.

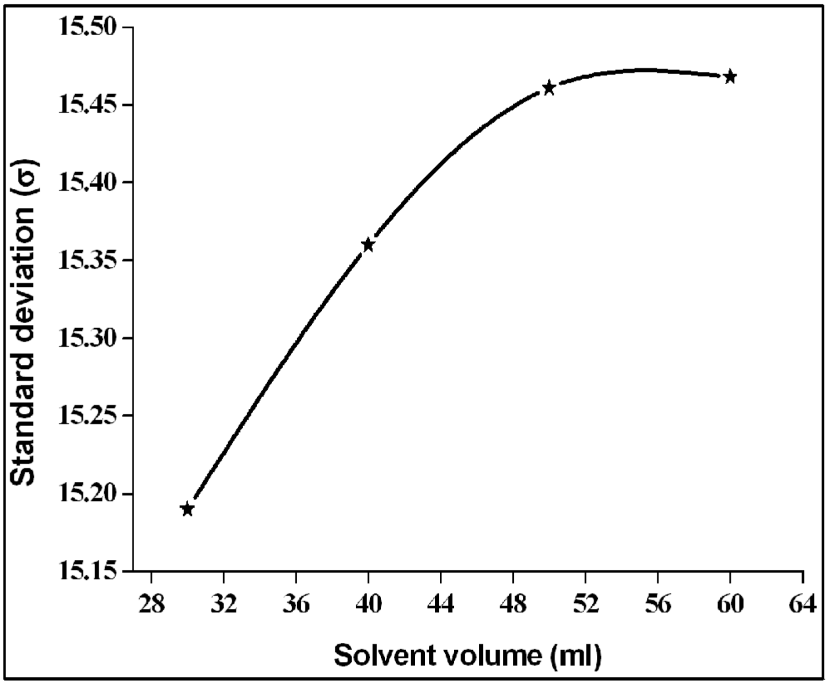

Fig-5.SEM images of the CdO films.

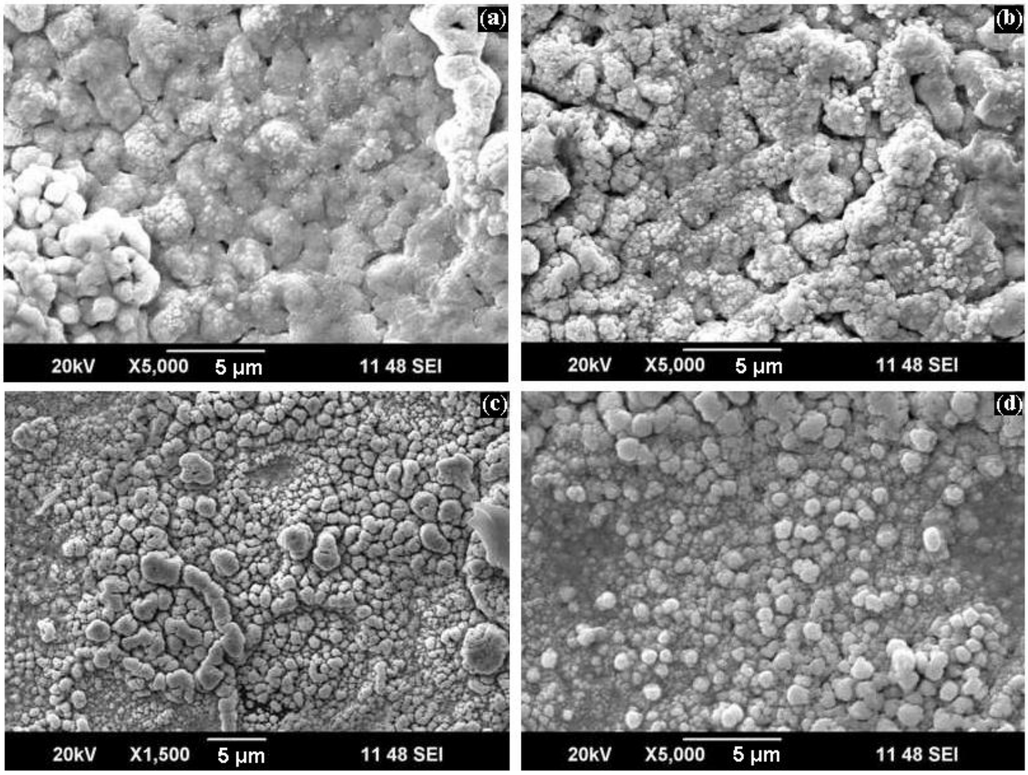

Fig-6.EDAX spectra of the films coated with different solvent volumes.

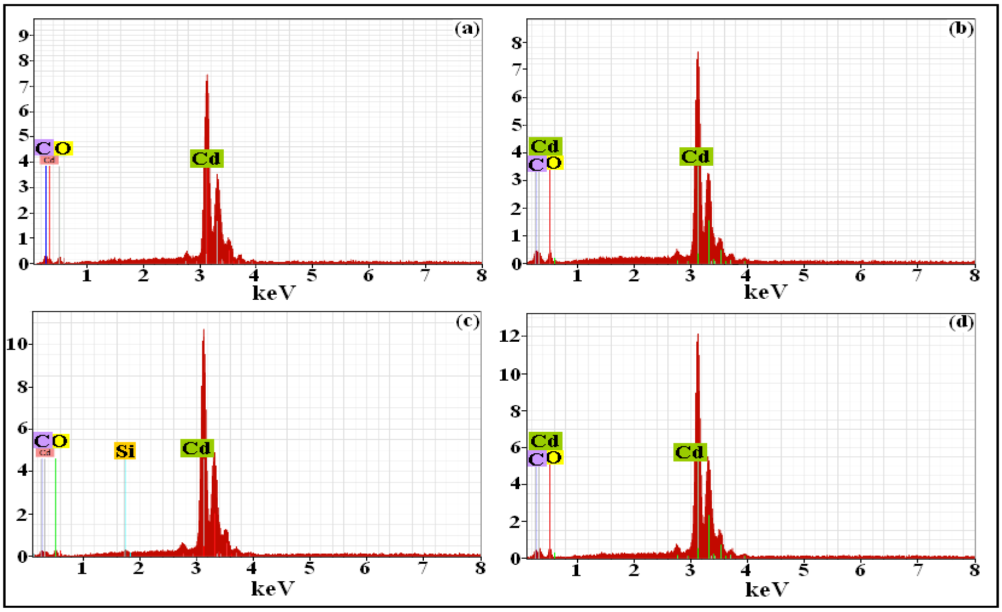

Fig-7. Transmittance spectra of the CdO films.

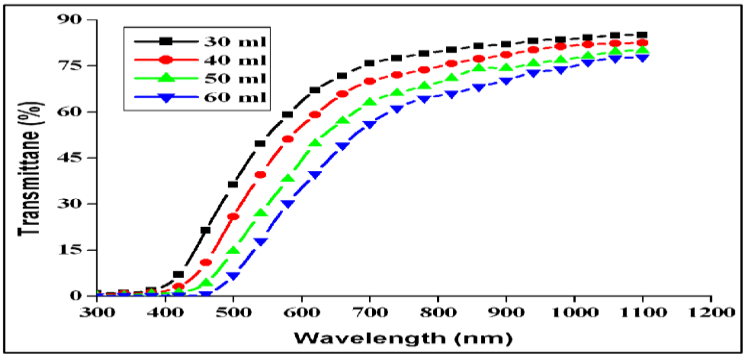

Fig-8. Plots of $(\alpha h\nu)^2$ vs. $(h\nu)$.

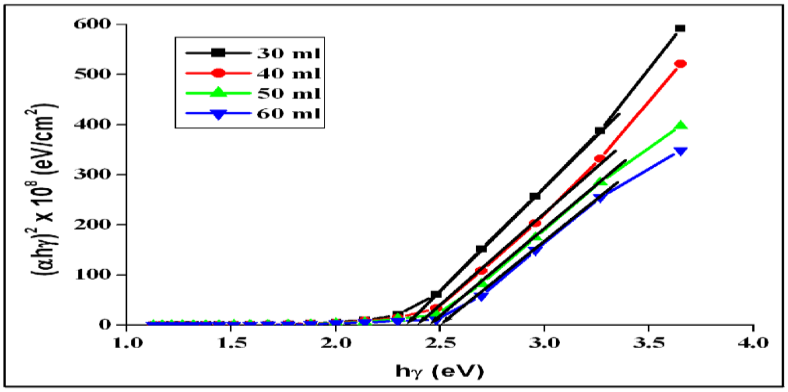

Fig- 9. Variation of sheet resistance as a function of solvent volume.