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INVESTIGATION OF THE EFFECTS OF ALKALIN MEDIUM ON CdO THIN FILMS PRODUCED BY USING ELECTRO DEPOSITION METHOD UNDER MAGNETIC FIELD

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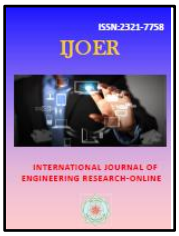


ABSTRACT
Electrodeposition method was used for deposit CdO thin films. In all film growth, deposition solution pHs were varied from 8.7 to 11.2. The structural features of the CdO films were examined by using XRD. According to the XRD, it was found that the intensity of the peaks increased and the crystallization of CdO thin films were improved with decreasing deposition solution pH's. The CdO films thicknesses were decreased as the deposition solution pH's were increased. Furthermore, micro strains, lattice constants, average stresses, crystallite sizes and dislocation densities were ascertained from the XRD results. In accordance with them, the micro strain and average stress of the film significantly high for M2 sample. Surface characteristics of the CdO thin films were searched by using SEM. According to optical results, while the deposition solutions pHs were decreased from 11.2 to 8.7, the energy band gap of the CdO films also decreased from 2.66 eV to 2.25eV proportionally

I. Introduction

Among the various nanostructured metal oxides, CdO is an important n-type semiconductor metal oxide with almost metallic conductivity [1], that belongs to the II–VI group [2,3]. Cadmium oxide (CdO) is in the visible light region of the electromagnetic spectrum [4]. CdO is a transparent conductive oxide with high electrical conductivity and high optical transparency (> 80%). It has a direct energy band gap (Eg) of ~ 2.3 eV and two indirect passages at lower energies [5].For the past few years, the characteristics of CdO film for the research on the growth of nano particle CdO films has increased for its technological various nanodevice applications have been studied such as photovoltaic cells, transparent electrodes, photodiodes, phototransistors, photovoltaic cells, transparent electrodes, liquid crystal displays, IR detectors, and anti-reflection coatings [6-11].

CdO films can be obtained by several techniques such as magnetron sputtering [10,12,13], chemical vapor deposition (CVD) [14], reactive pulsed laser deposition technique [15], thermal deposition [7,16], metal organic chemical vapor deposition [14], spray pyrolysis [17], thermal evaporation [18] sol gel [19], SILAR [20-22]and electrochemical deposition [23-27].The method of electrochemical deposition is presents a simple, cost, and quick method for the synthesis of CdO nanostructure.

The mechanisms of reactions were arranged bellow as is [29]:

$$NO_3^- + H_2O + 2e^- \rightarrow NO_2^- + 2OH^- \quad (1)$$

$$Cd^{2+} + 2OH^- \rightarrow Cd(OH)_2 \quad (2)$$

Subsequently deposition, annealing reasons cadmium hydroxide turns into to cadmium oxide [28].

$$Cd(OH)_2 \rightarrow CdO + H_2O \quad (3)$$

In this paper, we report the producing of CdO thin films by using a electrodeposition method under variable magnetic field on ito-coated glass substrate in alkaline medium. In all the experiments, deposition solutions placed in variable magnetic field. Besides, deposition solution pH's were varied between 8.7 and 11.2 and the effects of pH were examined in detail. It is realized from the energy band gaps that the values of energy band gap were increased as pH's were reduced.

II. Procedure

Thin films deposition

ITO-coated glass slides and deposition bath were degreased in with 5 % (w/w) hydrochloric acid and after than were washed with deionized water, ultra-sonicated in a mixture of acetone and double distilled deionized water for 10 min and then dried in air. Chronoamperometry method was made use of to deposit CdO thin films. Saturated calomel electrode, ITO-coated glass substrate, platinum wire and were rendered as the reference, working and counter electrode. The mechanism demonstrate in Figure 1 was used in the depositions [30].The magnetic field was applied perpendicular to the surface of the ITO-coated glass during the depositions. The frequencies of the magnetic field were adjusted to 50Hz.

The deposition solution include 0.015 M $Cd(NO_3)_2$ and 0.12 M KCl. KCl was used for the supporting electrolyteand depositions were served out 2700 s. Depositions temperatures were hold at 82 ± 2°C pending the deposition. The alkalinity is set using, sodium hydroxide (NaOH) .Deposition solutions were mixed at 500 rpm. After the deposited thin films was annealed in air at 300°C for 1h to transform the hydroxide phase to the oxide. The depositions contexts are shortened in Table 1.

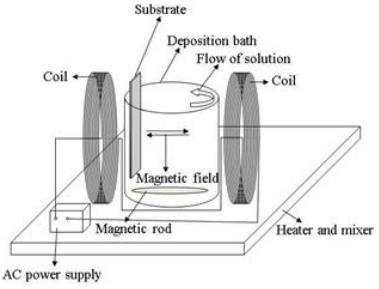


Fig.1 Schematic illustration of electrodeposition mechanism [30]

Table 1. Contexts of the prepared CdO thin films

Experiments	Concentration of $Cd(NO_3)_2$ (M)	Deposition time (sec)	Cathodic Potential (V)	Magnitude of Magneticfield (mT)	Depositiontemperature (°C)	pH
M1	0.015	2700	-0.73	3.25	82 ± 2	11.2
M2	0.015	2700	-0.73	3.25	82 ± 2	10.7
M3	0.015	2700	-0.73	3.25	82 ± 2	10.2
M4	0.015	2700	-0.73	3.25	82 ± 2	9.7
M5	0.015	2700	-0.73	3.25	82 ± 2	9.2
M6	0.015	2700	-0.73	3.25	82 ± 2	8.7

Characterizations of thin films

The prepared CdO thin films were described for their structural and morphological characteristics. Film thickness was measured and calculated using an optical reflectometer (Filmetrics F20) and gravimetric method respectively.

A PAN alytical Empyrean XRD (X-ray diffractometer) were exploited for the analysis of the structural analysis in the range of 20–80 with Cu Ka of 1.5410 A°. The prepared CdO thin films of surface morphology wereperused by using a Zeiss SUPRA 40VP scanning electron microscope (SEM). JASCO V–530 UV-vis was used for analysing the optical characteristics of the CdO films.

III. Results

A. XRD of the CdO thin films

The characteristics of the CdO films examined by XRD technique such as crystal structure, crystallite size, preferential orientation, strain and stress acting on the unit surface have been determined. The diffraction peaks of the CdO thin films observed at diffraction angles 2θ of 32.99 °; 38.1 °; 55,3 °; 65,9 ° and 69.2 ° correspond to the (111), (002), (022), (113) and (222) planes of the cubic CdO type structure. M1, M2, M3, M4 and M5 are confirmed by the ASTM number (98-002-9289), (98-002-9290), (98-002-9290), (98-002-9290), (98-002-9290) and ((98-002-9289), respectively. It has been observed that the intensity of the peaks increased and the

crystallization of CdO thin films were improved with decreasing deposition solution pH's. XRD analyses of CdO thin films produced in M1, M2, M3, M4, M5 and M6 are demonstrated in Figure 2 and they betrayed that all films formed in cubic monteponite structure.

Film thicknesses were calculated by using gravimetric method and results were demonstrated in Table 2. While the thicknesses of the CdO films were being varied between 597 nm and 376 nm. The film thicknesses were decreased as the pH's were increased.

The texture coefficients, given in Equation 4[31], of the films were utilized to determination of preferred orientation.

$$TC = \frac{I_{(hkl)}/I_{0(hkl)}}{\frac{1}{N}\sum_N(\frac{I_{(hkl)}}{I_{0(hkl)}})} \quad (4)$$

where I(hkl) is the measured relative intensity of a plane (hkl),I_0(hkl) is the standard intensity of the plane (hkl) given in ASTM card. The calculated texture coefficients were offered in Table 2. It has been assigned that all films have 3 texture coefficient values larger than 1 concern with different planes. The preferred orientation for all films produced is thought to be random. According to the texture coefficients although the films produced at all pH, the peak intensity of (111) plane of the film was higher than that of the (002) plane. This result is an expected result according to the previous study [26].

The average crystallite sizes of samples were estimated Debye Scherrer equation served in Equation 5.

$$cs = \frac{0.089*180*\lambda}{314*\beta*\cos\theta_C}\text{nm} \quad (5)$$

where λ was the radiations wavelength (1.54056 Å), βwas the full width half maximum, $2\theta_C$ was the peak centre, β and $2\theta_C$were calculated by fitting the XRD peak profile [32]. The estimated crystallite sizes of the films were presented in Table 3. When Table 3 is examined, it can be seen that crystallite size of all CdO thin films are same and varies from 26 nm to 31 nm.

The constant of the lattice for cubic rock salt structure is given bellow as is [33].

$$a = d\sqrt{(h^2+k^2+l^2)} \quad (6)$$

where h, k and l are the Miller indices and d is the interplanar distance. In addition to micro strain and average stress were calculated by using Equation 7 and 8 respectively and they are given in Table 3.

$$\varepsilon = (a_0 - a)/a_0 \quad (7)$$

$$S = \varepsilon Y/(2\sigma) \quad (8)$$

where a and a_0 are the corrected value of lattice parameter and lattice parameter of the bulk sample of CdO thin films respectively. The Y and σ are the Young's modulus and Poisson's ratio of the bulk crystal respectively. The value of Y is 141,67GPa and σ is 0.33 for cubic CdO thin films [34].

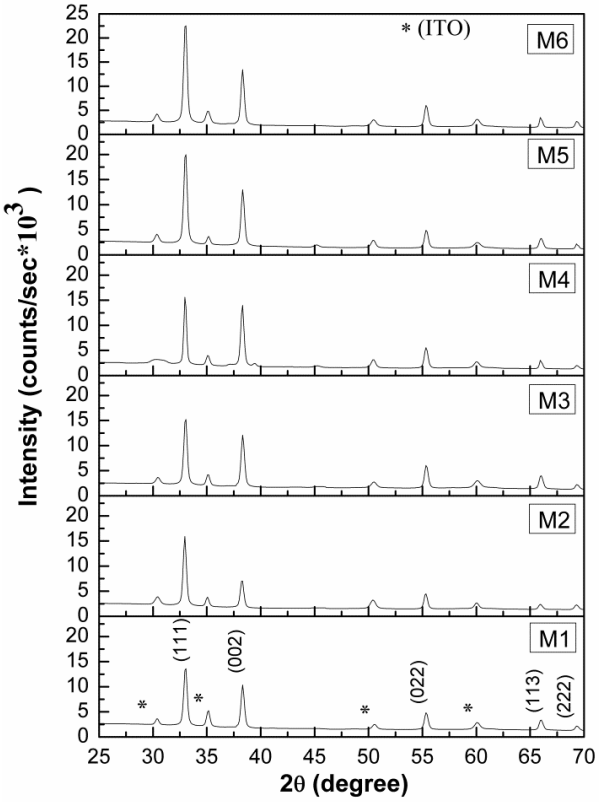


Fig.2X-ray diffractograms of CdO thin films according to deposition

Solution pH's

The Nelson–Riley plots presented that the corrected values of lattice parameters of a which is given in Figure 3, Figure 4, Figure 5, Figure .6, Figure 7 and Figure 8. The lattice parameters calculated from the peak positions of individual reflections are plotted against $F(\theta) = (cos^2\theta/2) * (\frac{1}{sin^2\theta} + \frac{1}{\theta})$ and the intercept of the linear plot at $(cos^2\theta/2) * (\frac{1}{sin^2\theta} + \frac{1}{\theta}) = 0$ at gives the corrected lattice constant [35]. The deviation of the corrected lattice

constant (a) from the strain of bulk sample (a_0 = 4.695 nm) indicates that the obtained films were under strain [36].

In generally, crystallite size is used to calculate dislocation density as given in Equation 9 and presented in Table 3[35].

$$\delta = \frac{1}{(cs)^2} \quad (9)$$

According to Table 4, the micro strain and average stress for the CdO samples are significantly is high for M2. The dislocation density is the same for all CdO films.

Table 2. The intensity, texture coefficient, and film thickness of the CdO thin films produced at pH of 8.7, 9.2, 9.7, 10.2, 10.7 and 11.2

EXPERIMENT	2θ	INTENSITY (COUNT/ SECONDS)	I / I_o	TC TC	(hkl)	FILM THICKNESS (nm)
M1	32,996	13585,61	100	4,67	(111)	597
	38,104	10335,86	52,99	2,48	(002)	
	55,310	4815,35	22,63	1,06	(022)	
	65,967	3283,45	12,15	0,57	(113)	
	69,299	2160,69	6,96	0,33	(222)	
M2	32,934	15902,66	100	4,61	(111)	556
	38,236	7000,08	43,9	2,02	(002)	
	55,261	4467,87	24,22	1,12	(022)	
	65,906	2374,40	8,77	0,40	(113)	
	69,248	2279,65	8,46	0,39	(222)	
M3	33,0724	15192,63	100	4,64	(111)	525
	38,3224	12083,79	79,97	3,71	(002)	
	55,3324	6049,756	36,75	1,70	(022)	
	65,9374	3886,18	22,3	1,03	(113)	
	69,2974	2217,144	7,85	0,36	(222)	
M4	32,9674	15583,16	100	4,76	(111)	463
	38,3224	13973,92	75,67	3,60	(002)	
	55,3324	5552,605	28,27	1,35	(022)	
	65,9374	3023,757	13,22	0,63	(113)	
	69,2974	1995,446	5,03	0,24	(222)	
M5	33,0724	19970,68	100	4,68	(111)	402
	38,3224	12970,56	60,57	2,84	(002)	
	55,3324	4899,245	20,94	0,98	(022)	
	65,9374	3154,595	12,77	0,60	(113)	
	69,2974	2186,231	6,73	0,32	(222)	
M6	33,0724	22519,62	100	4,67	(111)	376
	38,3224	13436,86	52,99	2,48	(002)	
	55,3324	6006,41	22,63	1,06	(022)	
	65,9374	3490,753	12,15	0,57	(113)	
	69,2974	2683,516	6,96	0,33	(222)	

Table 3. The crystalite size, dislocation densities, lattice parameter, micro strain values, and average stress values of the CdO thin films produced at pH of 8.7, 9.2, 9.7, 10.2, 10.7 and 11.2

EXPERIMENT	2θ	CRYSTALITE SIZE (nm)	LATICE PARAMETER a (VERIFIED) (Å)	MİKRO STRAİN *10^{-3}	DISLOCATION DENSITY (lines/m^2)*10^{15}	AVERAGE STRESS (10^8N/m^2)
M1	32,996	26	4,7020	1,51	1,43	3,23
	38,1041	27	4,7006	1,19	1,42	2,55
	55,31	29	4,6980	0,63	1,22	1,35
	65,9665	30	4,6968	0,38	1,10	0,83
	69,299	31	4,6972	0,47	1,05	0,99
M2	32,9674	26	4,7106	3,33	1,43	7,14
	38,2174	27	4,7077	2,71	1,39	5,82
	55,3324	29	4,7018	1,44	1,22	3,10
	65,9374	30	4,7006	1,20	1,10	2,57
	69,2974	31	4,7002	1,11	1,06	2,38
M3	33,0724	26	4,7017	1,44	1,43	3,08
	38,3224	27	4,6996	0,99	1,39	2,13
	55,3324	29	4,6974	0,52	1,22	1,12
	65,9374	30	4,6968	0,38	1,10	0,81
	69,2974	31	4,6967	0,37	1,05	0,79
M4	32,9674	26	4,7053	2,20	1,43	4,71
	38,3224	27	4,7041	1,94	1,39	4,17
	55,3324	29	4,6989	0,83	1,22	1,78
	65,9374	30	4,6979	0,61	1,10	1,31
	69,2974	31	4,6974	0,50	1,05	1,08
M5	33,0724	26	4,7019	1,49	1,43	3,20
	38,3224	27	4,6999	1,04	1,39	2,24
	55,3324	29	4,6973	0,50	1,22	1,07
	65,9374	30	4,6964	0,30	1,10	0,64
	69,2974	31	4,6972	0,47	1,05	0,99
M6	33,0724	26	4,7025	1,62	1,43	3,47
	38,3224	27	4,7013	1,35	1,39	2,91
	55,3324	29	4,6978	0,61	1,22	1,30
	65,9374	30	4,6976	0,57	1,10	1,22
	69,2974	31	4,6969	0,41	1,05	0,89

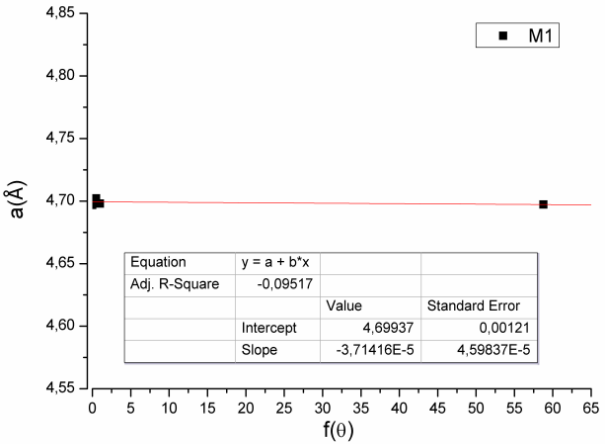


Fig.3 Nelson–Riley plots of CdO thin films acquired at pH 8.7

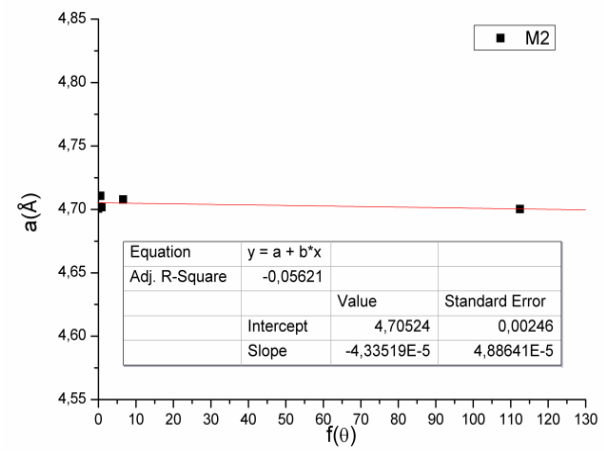


Fig.4 Nelson–Riley plots of CdO thin films acquired at pH 9.2

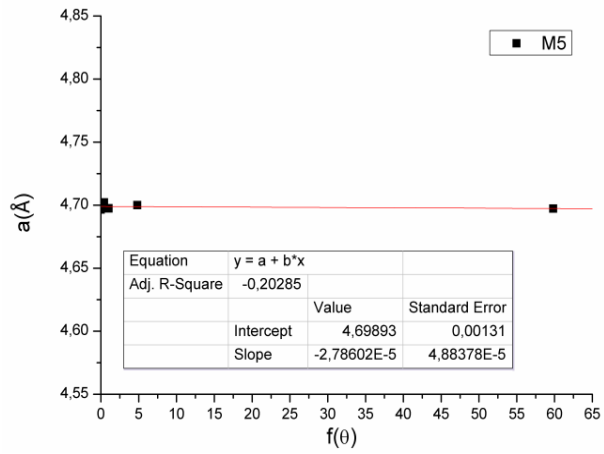


Fig.7 Nelson–Riley plots of CdO thin films acquired at pH 10.7

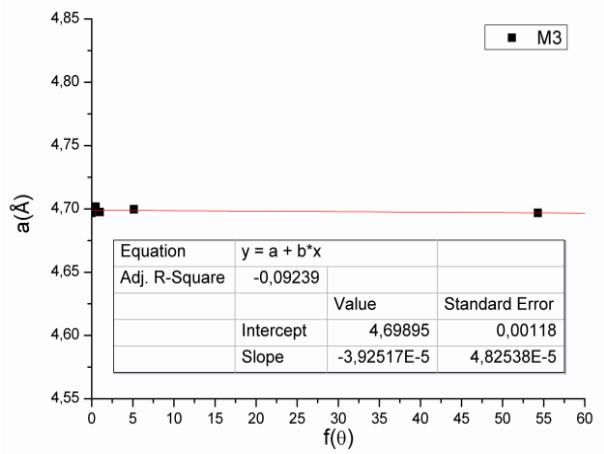


Fig.5 Nelson–Riley plots of CdO thin films acquired at pH 9.7

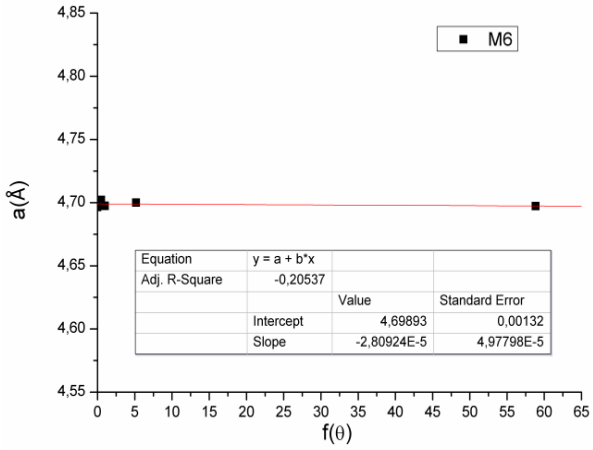


Fig.8 Nelson–Riley plots of CdO thin films acquired at pH 11.2

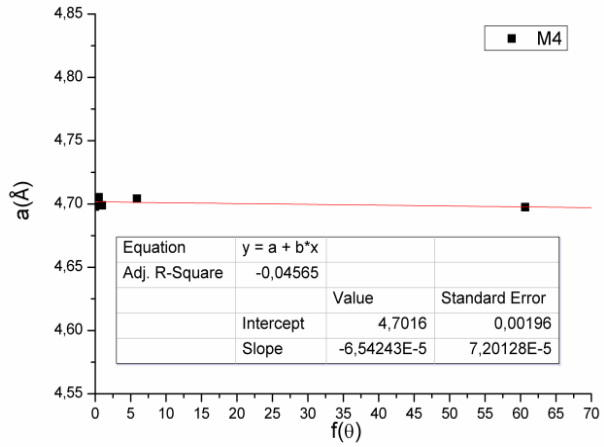
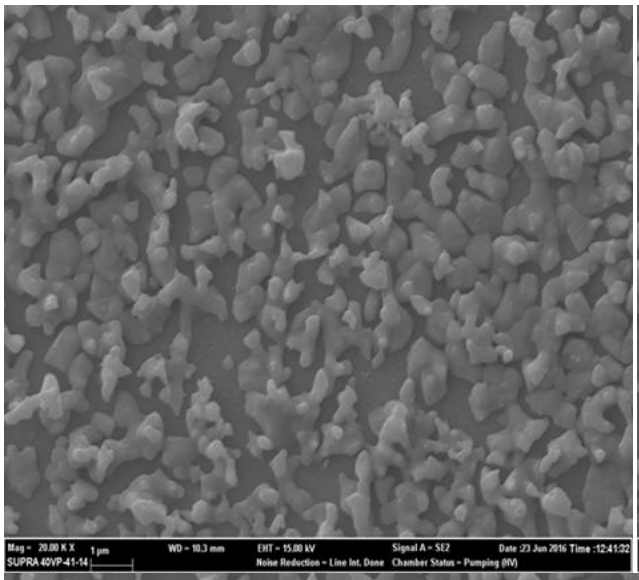
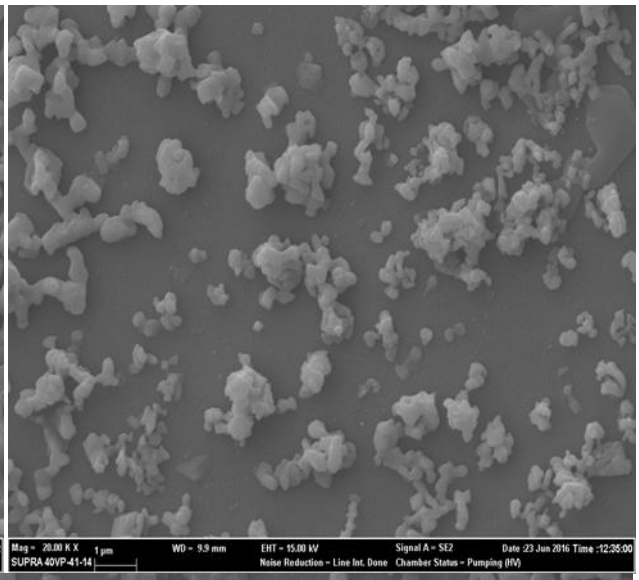


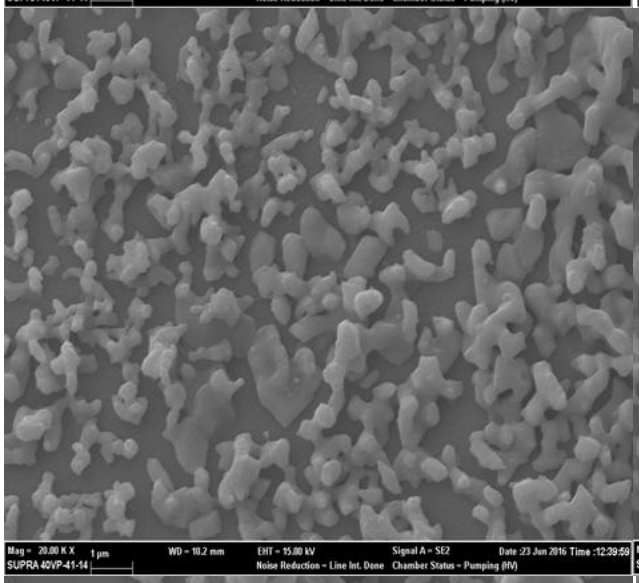
Fig.6 Nelson–Riley plots of CdO thin films acquired at pH 10.2

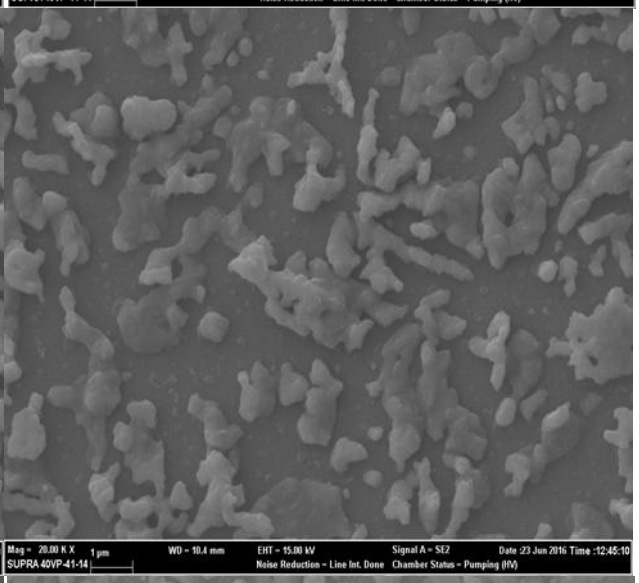
Surfaces of PbS thin films

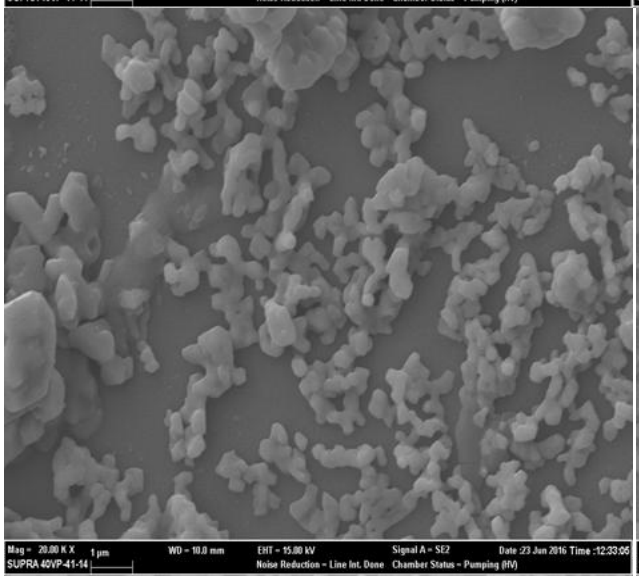
The 20000 times magnified surface images were given in Fig.9 and Fig.10.The films obtained at pH 8.7, 9.2and 9.7 given in Fig 9(a), Fig 9(b) and Fig (c) andpH 10.2, 10.7 and 11.2 given in Fig 10(a), Fig 10(b) and Fig 10(c) respectively. It is not seenon thesesurfacesthatcracks, voidsorpinholes. Besides, these surfaces are covered well with CdO crystals and are shown nearly compact, nearly smooth.Alsosurfacearenearlysamemorphologies in Fig 9 (a), and Fig 9(b) Fig 9(c). In the Figure 10, it is seen that the surface of the grain is dense than the Figure 9.











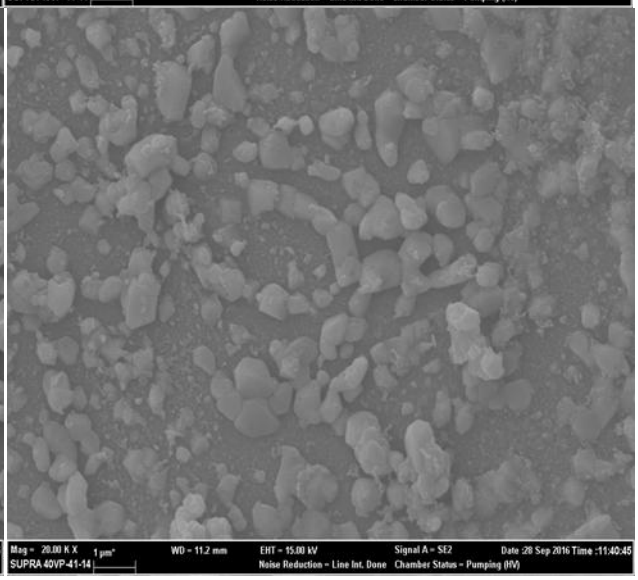


Fig.9 SEM images of the CdOfilms acquired at pH a) 8.7 b) 9.2 c) 9.7 magnified 20000 times.

Fig.10SEM images of the CdO films acquired at pH a) 10.2 b) 10.7 c) 11.2magnified 20000 times.

Optical studies of CdO thin films

UV-VIS spectra of CdO thin films are shown in Fig. 11 Absorbance measurementsis employed to calculate optical characteristics of the CdO films. Absorbance measurements were per-formed in a wavelength range between 350nm and 550nm.

When pH was 11.2, the absorbance was nearly 0.35. Besides when pH decreased up to 8.7, the absorbance was nearly 1. The film thicknesses are related to these results. As a result, the absorbance depends on the film thickness and the film thicknesses depend on the pH of the deposition solution.

The Tauc plot was anticipated to band gaps of the CdO films and it is demostrated in Equation 10. The relationship between the absorption coefficient and photon energy for direct allowable transition can be denoted as [37,38];

$$\alpha h\nu = A(h\nu - Eg)^m \quad (10)$$

where, A is the absorption coefficient, m = 1/2 or 3/2 for direct allowable transmission, hν the photon energy [39,40]. The energy band gap of the CdO thin films were forecasted by extrapolating the linear portion of the plots of $(\alpha h\nu)^2$ vs. hν which is demonstrated in Figure 12.

In an earlier study, the pH value of the deposition solution and the variable magnetic field applied changed to the reaction rate [26,30]. Therefore, thinner films were produced. Energy band gap of the films produced ranged from 2.66 to 2.25. As a result, if the deposition solution pHs increased, the energy band gap of the produced films was reduced. The energy band gap of the bulk CdO was almost 2.3eV. In this study, CdO thin films which energy band gaps were below and above of 2.3eV could be produced.

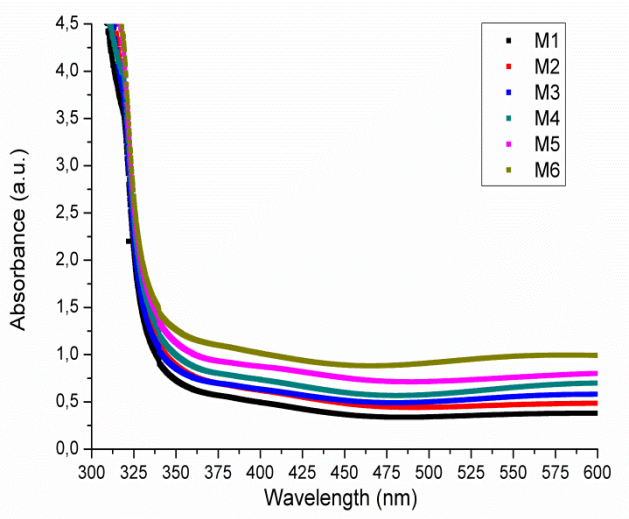


Fig 11. Absorption spectra of CdO thin films acquired at various pH values

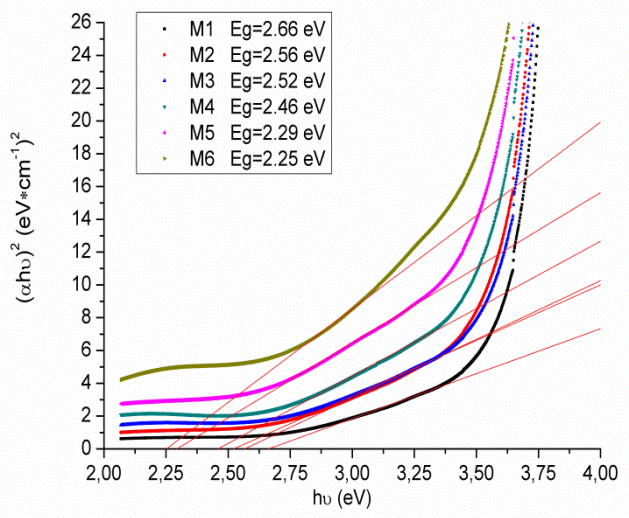


Fig.12 Tauc plots and band gaps of CdO thin films at various pH values.

IV. CONCLUSIONS

In this work, CdO thin films were growth by electro deposition. Six different deposition solution pH's were applied in the productions. XRD analysis showed that the intensity of the peaks increased and the crystallization of CdO thin films were developed with decreasing deposition solution pH's. All CdO films formed in cubic structure and their thickness decreased as depending on the deposition solution pH's decreased.

Dislocation densities, micro strain, and average stress values of all films were calculated. The micro strain and average stress of the film is significantly high for M2 sample.

The surface morphologies were explored by using a SEM. All SEM images are seen typical same morphology. It is noteworthy that theyhave not pinholes, cracks and voids.

Optical studies have bright to lighted very interesting results such as band gap depends on deposition solution pH's. When pH of the solutions decreased from 11.2 to 8.7, the energy band gap of the CdO films also decreased from 2.66 eV to 2.25eV proportionally.

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