STRUCTURAL ANALYSIS OF CdO THIN FILM BY SILAR METHOD

S.Anna Swathi

Department of Physics, St. Mary's College (Autonomous), Thoothukudi,

Affiliated to Manonmaniam Sundaranar University, Tirunelveli, Tamil Nadu, India

ABSTRACT:

Cadmium Oxide (CdO) thin films have garnered significant attention for their optoelectronic and semiconductor applications. In this study, CdO thin films were successfully synthesized using the Successive Ionic Layer Adsorption and Reaction (SILAR) method. The structural properties of the deposited films were analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray analysis (EDAX). The XRD patterns confirmed the polycrystalline nature of the CdO films with a cubic phase, and the crystallite size was estimated using Scherrer's equation. SEM images revealed a uniform and well-defined surface morphology, while EDAX confirmed the elemental composition of Cd and O in appropriate stoichiometric ratios. The films exhibited excellent structural stability upon annealing, with the best crystalline quality observed at 300°C. These findings demonstrate that the SILAR technique is an effective and cost-efficient method for fabricating high-quality CdO thin films with potential applications in photovoltaic cells, sensors, and transparent conductive electrodes.

KEYWORDS: CdO thin films, SILAR method, structural analysis, XRD, SEM, EDAX.

INTRODUCTION:

Cadmium oxide (CdO) is an n-type semiconductor with a wide band gap, high transparency, and good electrical conductivity, making it suitable for applications such as transparent conductive electrodes, gas sensors, and photovoltaic devices. Its excellent optical and electrical properties make it a promising material for various optoelectronic applications [1].

This study aims to synthesize and characterize CdO thin films, focusing on their structural properties. The work includes analyzing crystallinity and phase composition using X-ray diffraction (XRD), examining surface morphology and elemental composition via scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDAX), and measuring film thickness. These analyses are essential for understanding and optimizing the performance of CdO thin films in technological applications.

MATERIALS AND METHODS:

MATERIALS USED:

In this project, cadmium nitrate ($Cd(NO_3)_2$) is used as the Cd^{2+} precursor, a highly soluble inorganic salt that provides cadmium ions for film formation and Hydrogen peroxide (H_2O_2) which serves as the oxidizing agent, promoting the conversion of cadmium ions into cadmium oxide (CdO) . [2] The substrate used is glass, chosen for its optical transparency, chemical stability, and smooth surface, which aids in uniform film deposition. The deposited CdO thin films exhibit semiconducting properties, with a direct bandgap that varies based on synthesis conditions. CdO is known for its high electrical conductivity, optical transparency in the visible region, and n-type conductivity due to inherent oxygen vacancies. These properties make CdO suitable for applications in optoelectronic devices, sensors, and transparent conductive coatings.[3]

METHODOLOGY:

The Successive Ionic Layer Adsorption and Reaction (SILAR) method was choosen for preparing CdO thin films due to its simplicity, cost-effectiveness, and ability to control film thickness and uniformity. The SILAR method allows for the deposition of thin films at room temperature, which is beneficial for preserving the substrate's properties. Additionally, the SILAR technique enables the precise control of deposition parameters, making it an ideal method for achieving high-quality CdO thin films with desired properties.

For the deposition of CdO thin films, the cation solution was prepared using cadmium nitrate, while the anion solution was made with hydrogen peroxide (H_2O_2). The films were deposited onto a glass substrate under controlled conditions, with the deposition cycles being repeated for a total of 40 cycles. The concentration of cadmium nitrate and H_2O_2 was optimized to ensure uniform film growth. After deposition, the films were annealed at 200°C, 300°C, and 400°C, with the best coating achieved at 300°C. Following annealing, the properties of the films are studied using X-ray diffraction (XRD), scanning electron microscopy (SEM) with energy-dispersive X-ray analysis (EDAX), to analyze their structural and morphological characteristics. The results indicated that annealing at 300°C produced the most optimal properties for the CdO thin films. The CdO thin film prepared by SILAR method and annealed at varius temperature is given in the fig.1.

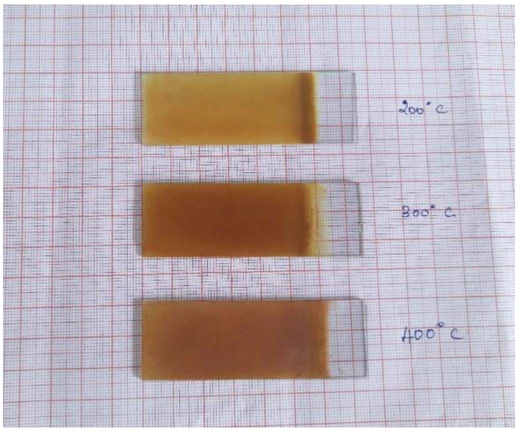

Fig. 1. CdO thin films prepared by SILAR and annealed at different temperatures.

RESULT AND DISCUSSION:

The CdO thin film prepared by SILAR method was investigated with XRD, SEM with EDAX and the thickness was measured.

X- RAY DIFFRACTION (XRD):

X-ray Diffraction (XRD) is a powerful analytical technique used to determine the crystalline structure, phase composition, and other structural properties of materials.

Principle of XRD:

XRD works based on Bragg's Law:

$n\lambda=2d\sin\theta$

where:

n = order of reflection (integer)

λ = X-ray wavelength

d = interplanar spacing

θ = diffraction angle

When X-rays hit a crystalline sample, they are scattered by atoms in specific directions based on the crystal lattice. The diffraction pattern is recorded, and the resulting peaks provide information about the structure.[4]

To study the structural properties of the deposited CdO thin films X-Ray diffraction (XRD) study was carried out using X-ray diffractometer D8 Advance ECO. Fig 1.1 shows the XRD pattern of CdO deposited by SILAR method with 40 deposition cycles and annealed at 300°c.The presence of four peaks (1 1 1), (2 0 0), (2 0 2), (3 1 1) indicates the polycrystalline with the cubic structure of the film and is well matched with the standard data (JCPDS File no. 96-900-6674). [5]

The crystalline size of CdO thin film is calculated from the XRD patterns by using scherrer's formula:

$$D = k\lambda / \beta\cos\theta$$

Where:

D = crystallite size (nm)

K = Scherrer constant

λ = X-ray wavelength

β = Full Width at Half Maximum (FWHM)

θ = Bragg's angle in degrees

The dislocation density has been calculated using crystallite size, by the equation,

$$\text{Dislocation} = 1/D^2$$

The microstrain has been calculated by the given formula,

$$\text{Microstrain} = \beta \cos\theta /4$$

The lattice parameter calculated for CdO by the given formula,

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

The lattice parameter has been calculated and reported. The grain size, dislocation density, micro strain and lattice spacing of CdO thin film for various deposition cycles have been tabulated in table 1.2.

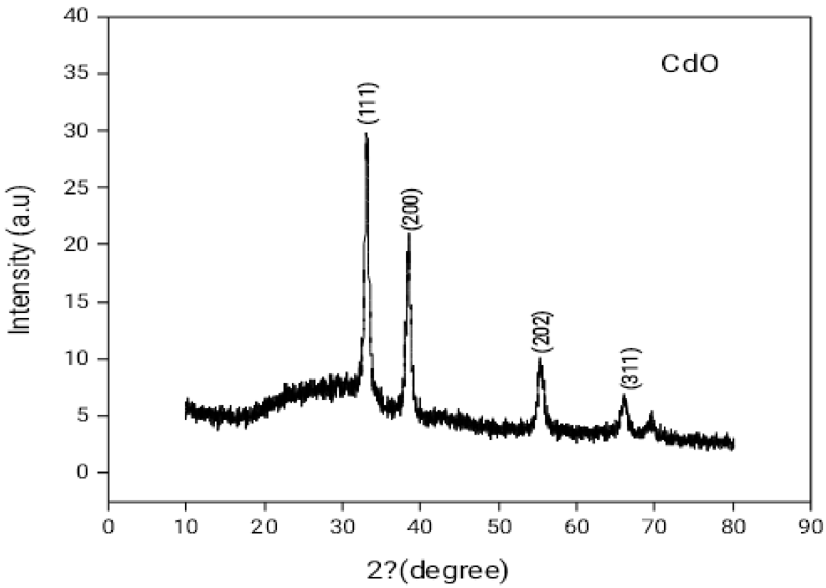

Fig. 1.1. XRD pattern of CdO thin film (40 cycles, 300 °C) showing cubic phase peaks at (111), (200), (202), and (311).

Table 1.2 XRD data

Deposition cycle	**h k l**	**2θ degree**	**Grain size(nm)**	**Dislocation density(line/ m^2)**	**Microstrain (10^{-4})**	**Lattice constant(A°)**
40 (300°c)	(1 1 1)	33.135	14.5586	4.718	0.00238	a = 4.682
	(2 0 0)	38.434	12.9042	6.005	0.00254	
	(2 0 2)	55.372	11.6891	7.31	0.00263	
	(3 1 1)	65.666	13.5412	5.455	0.00215	

MORPHOLOGICAL AND COMPOSITIONAL ANALYSIS:

SEM (Scanning Electron Microscopy) provides high-resolution images of a material's surface morphology and structure.EDX (Energy Dispersive X-ray Spectroscopy), also called EDAX, is an analytical technique used along with SEM to determine the elemental composition of a sample.

The SEM electron beam interacts with the sample, producing secondary electrons (for imaging) and characteristic X-rays (for elemental analysis).The EDX detector collects these X-rays and identifies elemental peaks, allowing for qualitative and quantitative analysis. [6]

Scanning Electron Microscopy (SEM) combined with Energy Dispersive X-ray Analysis (EDAX) is an essential technique for studying the surface morphology and elemental composition of thin films. The SEM images and EDAX spectra of CdO thin films are analyzed to understand their structural characteristics and elemental composition using the Energy dispersive X-ray spectrometer quantax 200 with X flash 6130.

The SEM images (Fig. 2 and Fig. 2.1) show closely packed particles with a smooth surface and no visible pores. The clustered nanostructures indicate that the film has polycrystalline grains, with some regions showing agglomeration.

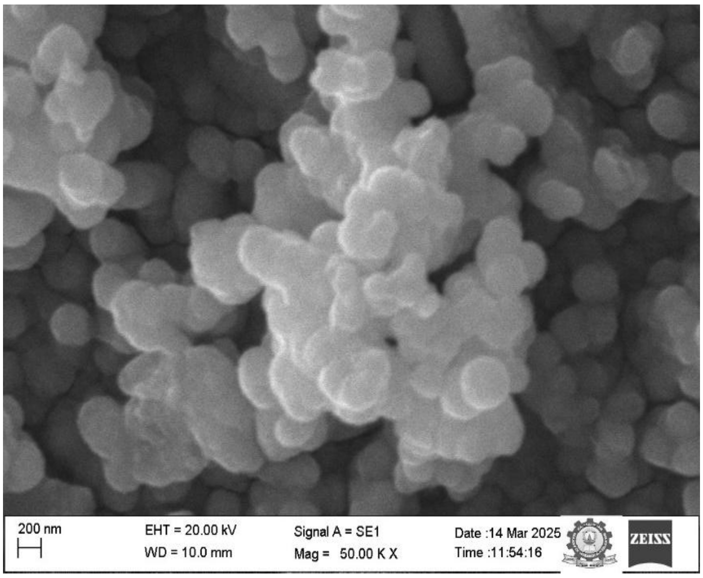

Fig. 2. SEM image of CdO thin film with closely packed particles and no visible pores.

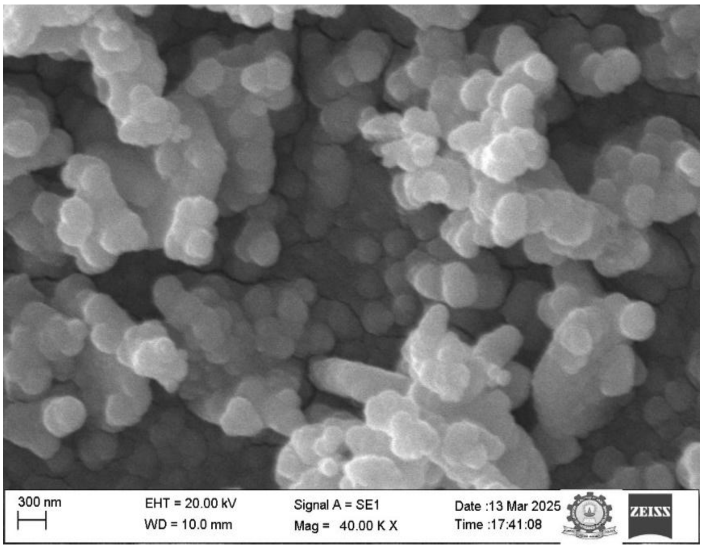

Fig. 2.1. High-magnification SEM image showing uniform grain structure.

The film shows a peak corresponding to Cd and O elements(fig 2.2). The atomic percentages of Cd and O were recorded as 68.6% and 31.4% respectively. Hence the result indicates the presence of cadmium oxide thin film. The quantitative result of CdO is given in the table 2.3.

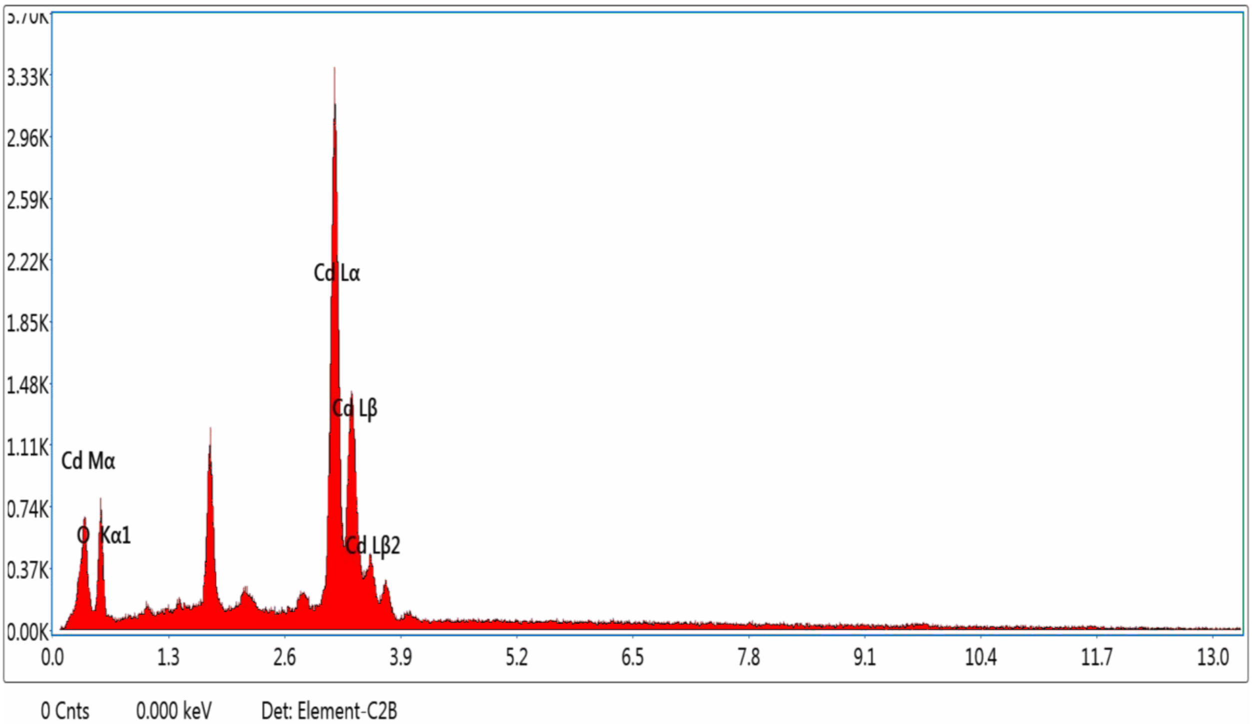

Fig. 2.2. EDAX spectrum showing cadmium (Cd) and oxygen (O) peaks.

Table 2.3

Element	Weight%	Atomic%	Error%	Kratio
O k	23.7	68.6	12.0	0.0370
CdL	76.3	31.4	2.2	0.7098

THICKNESS MEASUREMENT:

The thickness of thin films is a crucial parameter that influences their optical, electrical, and structural properties. In this study, the thickness of CdO thin films deposited by the SILAR method was determined using the Weight Gain Method. This technique is based on measuring the mass difference before and after deposition and using the film's area and density to estimate thickness.

The thickness (t) of the CdO thin film was calculated using the formula:

$$t = m / (A\rho)$$

where,

m = mass difference before and after deposition (g),

A = area of the deposited film (cm^2),

ρ = density of CdO (g/cm^3).

The film, deposited over 40 cycles and annealed at 300°C, exhibited a thickness of 416 nm which is in accordance with other reports. [7]. This indicates a uniform and controlled deposition process.

CONCLUSION:

The successful deposition of CdO thin films using the SILAR (Successive Ionic Layer Adsorption and Reaction) method demonstrates the potential of this technique for producing high-quality thin films for optoelectronic and sensor applications. CdO thin films, deposited with 40 cycles and annealed at different temperatures, showed the best structural properties at 300°C, as confirmed by X-ray diffraction (XRD), SEM-EDAX analysis. The XRD results matched the standard JCPDS file 96-900-6674, confirming the crystallinity of CdO. SEM images revealed a uniform surface morphology, and EDAX confirmed the elemental composition. The structural analysis showed that the mean crystalline size of the CdO thin films was approximately 13.17 nm, with a mean dislocation density of 5.8725×10^4 line/m^{2}and a mean microstrain of 0.00243×10^{-4}. The lattice constant was found to be 4.682 Å and the thickness is measured to be 416 nm indicating good agreement with standard CdO values.

CdO thin films have potential applications in solar cells, gas sensors, and electronic devices. Future research can focus on investigating electrical properties, optimizing deposition parameters, and exploring CdO-based heterojunctions for enhanced device performance. Additionally, doping or composite thin films could further improve their efficiency in various technological applications. This study demonstrates the effectiveness of the SILAR method for depositing CdO thin films and provides a strong foundation for future research in thin-film technology.

REFERENCE:

[1] Ali, K. H., & Ahmed, N. M. (2017). Effects of concentration on CdO films grown by electrodeposition. Applied Nanoscience, 7(8), 801–808.

[2] Hadi, A. H., & Abbas, S. M. (2015). Effect of pH on the structural and optical properties of CdO thin films prepared using the SILAR method. Iraqi Journal of Physics, 13(26), 74–81.

[3] Azzouz, N,et.,al.,(2019). Synthesis and study of some physical properties of cadmium oxide CdO thin films. Materials Research Express, 6(11), 116434.

[4] Ehsani, M. H,et.,al.,(2019). Effect of substrates on the structural, morphological, and optical properties of sprayed CdO thin films using a nebulizer. Journal of Sol-Gel Science and Technology, 91(3), 517–528.

[5] R.A. Ahmed,et.,al.,(2019) ,synthesis and characterization of cdO for solar cell aplications,vol 14,p15-22

[6] Larki, A,et.,al.,(2025). Chemometric optimization of the CdO superhydrophobic thin film coating process: DOE-based modeling and structural analysis. Journal of Materials Science: Materials in Electronics.

[7] M.Anusuyab,et.,al.,(2024),characterization of CdO thin films preparedb y silar deposition technique.

[8] A. Shameem,et.,al.,(2017) ,Preparation and Characterization Studies of Nanostructured CdO Thin Films by SILAR Method for Photocatalytic Applications,Volume 27, pages 692–699.

[9] M Anitha,et.,al.,(2017),Investigations on the structural, electrical and optical properties of thin films of CdO,Volume 28, pages 17297–17307

[10] M Soylu,et.,al.,(2017),CdO thin films based on the annealing temperature differences prepared by sol–gel method and their heterojunction devices ,, Volume 4, Number 12.