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Characterization of Chromium-Reinforced Cadmium Oxide Thin Films Prepared by Thermochemical Decomposition Method for Gas Sensing Applications

Essam Karim Youssef[1] , Hanan Rida Abd[2]
[1,2]Tikrit University / Faculty of Education for Pure Sciences / Department of Physics
Email:essam.kareem202300@gmail.com

Abstract :

In this study, pure cadmium oxide (CdO) membranes inlaid with chromium ions in the proportions of 0%, 2% and 4% were prepared using the thermochemical spraying method and the effect of grafting on structural, morphological, optical and sensory properties towards NO_2 gas was studied.The X-ray diffraction results showed that all membranes possessed a polycrystalline cubic structure, with the average crystal size decreasing from 59.8 nm for the pure sample to 45.7 nm and 38.2 nm at grafting ratios of 2% and 4% respectively, as shown by the FESEM images . The grain size decreased from 52.6 nm in the pure sample to 41.8 nm and 34.5 nm with increased grafting, and was accompanied by a marked increase in surface roughness. Optically, the power gap decreased from 2.63 eV for the pure sample to 2.60 eV and 2.48 eV at 2% and 4% Cr, respectively, with a marked improvement in absorption. In gas sensing applications, the 4% Cr sample achieved the highest sensitivity of about 50% at 200°C with response and retrieval times of 19.4 s and 24.6 s, respectively. These results confirm that chromium CdO grafting within the 0–4% domain is an effective strategy to improve the structural, optical and sensor performance of CdO membranes.

Keywords: Cadmium Oxide (CdO) Membranes, Chromium Grafting, Thermochemical Spraying, Structural and Morphological Properties, NO_2 Gas Sensing.

توصيف الأغشية الرقيقة لأوكسيد الكادميوم المطعم بالكروم والمحضرة بطريقة التحلل الكيميائي الحراري لتطبيقات استشعار الغاز

عصام كريم يوسف[1] ، حنان رضا عبد[2]
[1,2]جامعة تكريت / كلية التربية للعلوم الصرفة / قسم الفيزياء

مستخلص:

تم في هذا البحث تحضير أغشية أوكسيد الكادميوم (CdO) النقية والمطعمة بأيونات الكروم بنسب ٠٪، ٢٪، و٤٪ باستخدام طريقة الرش الكيميائي الحراري ودراسة تأثير التطعيم على الخواص البنيوية و المورفولوجية و البصرية والاستشعارية تجاه غاز NO_2. أظهرت نتائج حيود الأشعة السينية أن جميع الأغشية تمتلك بنية مكعبة متعددة البلورات، مع تناقص متوسط الحجم البلوري من 59.8 nm للعينة النقية إلى 45.7 nm و 38.2 nm عند نسب تطعيم ٢٪ و ٤٪ على التوالي، كما بينت صور المجهر الإلكتروني الماسح (FESEM) تقلص حجم الحبوب من 52.6 nm في العينة النقية إلى 41.8 nm و 34.5 nm مع زيادة التطعيم، وترافق ذلك مع ازدياد واضح في خشونة السطح. من الناحية البصرية انخفضت فجوة الطاقة من 2.63 eV للعينة النقية إلى 2.60 eV و 2.48 eV عند نسب ٢٪ و ٤٪ Cr، على التوالي، مع تحسن ملحوظ في الامتصاصية. وفي تطبيقات استشعار الغاز، حققت عينة ٤٪ Cr أعلى تحسسية بلغت نحو ٥٠٪ عند 200 °C مع زمن استجابة واسترجاع قدره 19.4 s و 24.6 s، على التوالي. تؤكد هذه النتائج أن تطعيم CdO بالكروم ضمن المجال ٠–٤٪ يُعد إستراتيجية فعالة لتحسين الأداء البنيوي والبصري والاستشعاري لأغشية CdO.

الكلمات المفتاحية: أغشية أوكسيد الكادميوم (CdO)، التطعيم بالكروم، الرش الكيميائي الحراري، الخصائص البنيوية والمورفولوجية، استشعار غاز NO_2.

1- Introduction

Semiconductor oxide-based gas sensors are one of the most widely used types of sensors in industrial, environmental, and medical applications, due to their simplicity of manufacturing, low cost, and high sensitivity to various gases. The principle of action of these sensors is based on the change in the electrical properties of the semiconductor oxide layer as a result of the surface adsorption interactions between the gas molecules and the thin film surface [1,2].

Cadmium oxide (CdO) is an n-type semiconductor oxide with a cubic crystal structure, characterized by high electrical conductivity and a direct energy gap of 2.2–2.7 eV, making it a promising material for multiple applications including solar cells, conductive transparent coatings, and gas sensors [3,4]. Several studies have shown that CdO's performance in gas sensing is directly related to its structural, morphological, and optical properties [5].

In order to improve the properties of CdO membranes and increase their sensitivity and selectivity to gases, grafting with transition elements is used as one of the effective techniques, as grafting modifies the electronic structure, increases the concentration of carriers, and creates structural defects such as oxygen gaps, which contributes to improving the surface adsorption of gases [6,7]. Among these elements, chromium (Cr) is of particular interest due to its apparent effect on improving the electrical and structural properties of CdO membranes [8].

Chemical Spray Pyrolysis is one of the most widely used thinfilm preparation methods for the preparation of metal oxides, due to its simplicity of processing, control of membrane thickness and grafting ratio, and suitability for large-scale production. This method also allows for homogeneous membranes with good adhesion to the substrate when the appropriate preparation conditions are selected [9,10].

Recent studies have shown that chromium-inlaid CdO membranes prepared by thermochemical decomposition have a marked improvement in structural and morphological properties, such as reduced granular size and increased surface roughness, resulting in an increase in effective surface area and improved sensitivity performance to gases such as ammonia and formaldehyde [11,12].

2- Experimental

2.1 Preparation of thin films by thermochemical spraying method

Pure CdO membranes inlaid with chromium (Cr) ions with atomic grafting ratios of 0%, 2% and 4% were prepared using the Chemical Spray Pyrolysis method, due to its simplicity, low cost, and ability to produce homogeneous films suitable for sensor applications.

The sprinkler system consists of a sprayer device connected to a solution tank and a compressed air source, in addition to an electric heater with a thermocouple to control the base temperature, and an electronic timer to regulate spraying intervals and stops.

2.2 Preparation of glass bases and chemical solutions

Glass bases with dimensions of (2.5 × 2.5) cm² and (1–1.2) mm thickness were used, where they were ultrasonically cleaned using distilled water, then ethanol, phacetone, and air-dried.

A 0.1 M cadmium base solution was prepared using dihydrous cadmium acetate and a 0.1 M chromium incubated solution of chromium nitrate was prepared. The two solutions were mixed in calculated volumes to obtain spray solutions with 0–4 at.% Cr grafting ratios with total solution volume constant.

2.3 Membrane Deposition and Thickness Measurement

The glass bases were placed on the electric heater and heated to 450° C before precipitation, then the solutions were sprayed vertically on the bases in regular cycles (10 seconds of spraying followed by a two-minute pause) until the desired thickness was reached. After completion, the membranes were left to gradually cool to room temperature to ensure complete oxidation and improved crystallization.

The thickness of the membranes was measured using the gravimetric method based on the pre- and post-precipitation weight difference, and the thicknesses of the prepared membranes ranged within the range of approximately 50–300 nm.

2.4 Equations Used

2.4.1 Crystallite Size Calculation (Debye–Scherrer Equation)

The average crystallite size (D) of the prepared thin films was calculated from the XRD patterns using the Debye–Scherrer equation [10–12]:

$$D = \frac{K\lambda}{\beta\cos\theta} \ldots\ldots\ldots.(1)$$

where D is the crystallite size, K is the shape factor (0.9), λ is the wavelength of Cu Kα radiation (1.5406 Å), β is the full width at half maximum (FWHM) of the diffraction peak in radians after instrumental correction, and θ is the Bragg diffraction angle.

2.4.2 Optical Band Gap Determination (Tauc Relation)

The optical band gap energy (E_g) was estimated using the Tauc equation [2,4,12]:

$$(\alpha h\nu)^n = A(h\nu - E_g) \ldots\ldots.(2)$$

where α is the absorption coefficient, $h\nu$ is the photon energy, A is a constant, and n depends on the nature of the electronic transition. For direct allowed transitions, $n=2$. The band gap energy was obtained by extrapolating the linear portion of the $(\alpha h\nu)^2$ versus plot to the photon energy axis. The photon energy was calculated using:

$$h\nu(eV) = \frac{1240}{\lambda(nm)} \ldots\ldots\ldots(3)$$

2.4.3 Gas Sensitivity Calculation

The gas sensing performance was evaluated in terms of sensitivity (S) using the following relation [6,13,16,18]:

$$S(\%) = \frac{R_g - R_a}{R_a} \times 100 \ldots\ldots\ldots(4)$$

where R_a is the electrical resistance of the sensor in air and R_g is the resistance in the presence of NO_2 gas.

3. Results and discussion

3.1 X-ray Diffraction

The results from X-ray diffraction (XRD) analysis show that the pure sample of cadmium oxide (CdO) exhibits sharp and narrow diffraction peaks, indicating good crystallinity. The diffraction peaks are observed at 2θ values of 33.0°, 38.3°, 55.3°, 65.9°, and 69.2°, corresponding to the crystallographic planes (111), (200), (220), (311), and (222), respectively, which confirm the formation of the cubic CdO phas., and (311), all of which correspond to the stereo-group cubic structure (Fm-3m).

The high intensity of these peaks and the small half-intensity width values (FWHM) indicate the formation of relatively large crystals with a regularity in internal structure, with a clear reduction in crystal defects and lattice stresses, reflecting the relative structural stability of pure cadmium oxide. Accordingly, this sample is a structural reference for comparing the grafting effect, with an average crystal size of about 59.8 nm.The results for the 2% chromium-grafted sample show clear changes in diffraction patterns compared to the pure sample, where an additional weak peak due to chromium ions appeared at level (111) near the angle of $2\theta \approx 44.3°$, indicating the beginning of the fusion of Cr^{3+} ions within the crystal network of CdO. A relative expansion of CdO peaks and a slight increase in FWHM values were observed, suggesting a decrease in the mean crystal size as a result of the generation of additional lattice stresses resulting from the difference in the ionic radius between Cr^{3+} (0.62 Å) and Cd^{2+} (0.95 Å). In addition, a slight shift of the peaks to higher angles, reflecting a limited contraction of the lattice constant, has decreased to about 45.7 nm.The results also show a marked increase in the width of the CdO peaks and greater visibility of the chromium crest (111)[13], indicating an increase in the density of crystal defects and grain boundaries, and an increase in internal reticular stresses resulting from the introduction of larger amounts of Cr^{3+} ions. Structurally, these results suggest that grafting limits the normal growth of the granules and reduces the regularity of the crystal reticle, leading to a further contraction in crystal size, with an average crystal size of about 38.2 nm.

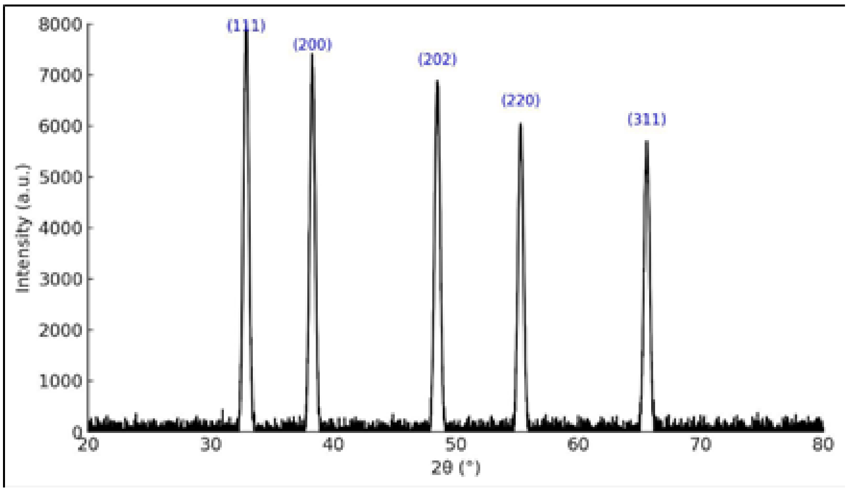

Figure 1: X-ray diffraction patterns of pure sample CdO

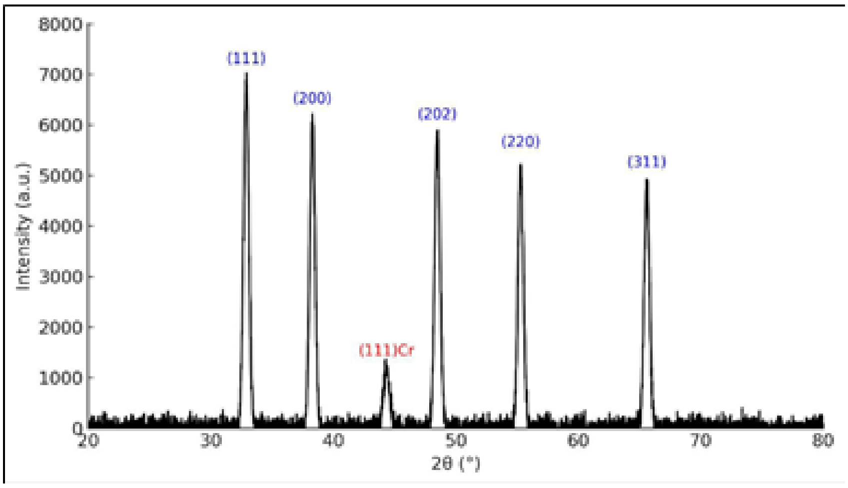

Figure (4-2) X-ray diffraction patterns of the 2%Cr sample

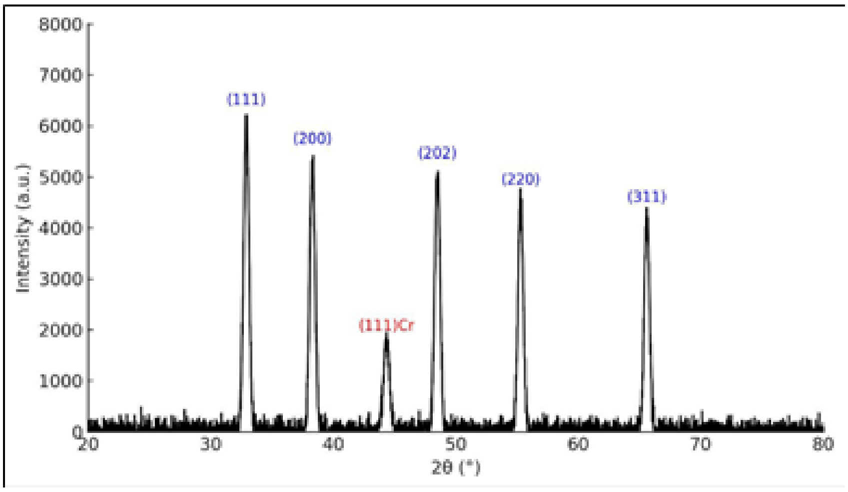

Figure 3: X-ray diffraction patterns of the 4%Cr sample

Table (1) XRD structural parameters of pure and Cr-doped CdO thin films

Sample	2θ (°)	FWHM (°)	d (Å)	Crystallite Size (nm)	(hkl)	Phase
Pure CdO	32.88	0.15	2.722	61.4	(111)	CdO
	38.29	0.14	2.349	58.6	(200)	CdO
	48.54	0.13	1.874	60.3	(202)	CdO
	55.33	0.16	1.659	59.0	(220)	CdO
	65.63	0.15	1.421	59.6	(311)	CdO
				Average = 59.8 nm		
Cr–CdO 2%	32.95	0.19	2.716	44.5	(111)	CdO
	38.35	0.21	2.345	46.0	(200)	CdO
	44.35	0.16	2.041	45.1	(111)	Cr
	48.60	0.22	1.872	45.9	(202)	CdO
	55.41	0.20	1.656	46.6	(220)	CdO
	65.90	0.21	1.416	45.3	(311)	CdO
				Average = 45.7 nm		
Cr–CdO 4%	32.99	0.23	2.714	39.2	(111)	CdO
	38.40	0.24	2.342	37.7	(200)	CdO
	44.32	0.21	2.042	38.1	(111)	Cr
	48.66	0.22	1.870	38.5	(202)	CdO
	55.47	0.25	1.655	37.0	(220)	CdO
	65.92	0.24	1.415	38.5	(311)	CdO
				Average = 38.2 nm		

3.2 **Scanning electron microscope**

The results from scanning electron microscopy (FESEM) images show that chromium grafting has a clear effect on the surface morphology of cadmium oxide membranes. In the Pure CdO sample, the images show the presence of relatively large grains and homogeneous distribution with an average size of about 52.6 nm, with a smooth and uniform surface, indicating stable crystal growth and a conspicuous absence of structural defects and sharp grain boundaries. This morphological behavior reflects the results of X-ray diffraction that Showed high crystalline degree for pure specimen. With the addition of 2% chromium, the results show the onset of a change in surface structure, with the average grain size decreasing to about 41.8 nm with a clearer granular boundary and higher structure cohesion. This behavior is attributed to the effect of chromium ions in hindering regular crystal growth and increasing the density of nucleation centers during the sedimentation process, resulting in the formation of a larger number of smaller grains compared to the pure sample. When the grafting percentage is raised to 4% chromium, FESEM images show a further grain size shrinkage to about 34.5 nm, with the surface becoming a rougher structure and filling with fine cavities. These appearances indicate an increase in the density of crystal defects and grain boundaries as a result of the introduction of larger amounts of chromium ions into the crystal lattice. This rough structure also contributes to an increase in the specific surface area, which is consistent It showed a significant increase in roughness at this grafting percentage, which is an important factor in improving membrane performance in gas sensing applications. 14] .

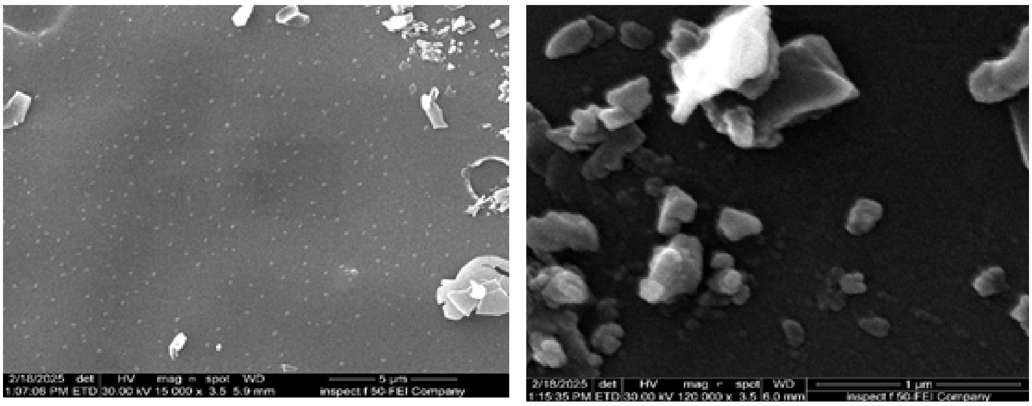

Figure (4) Scanning electron microscopy images at the pure specimen

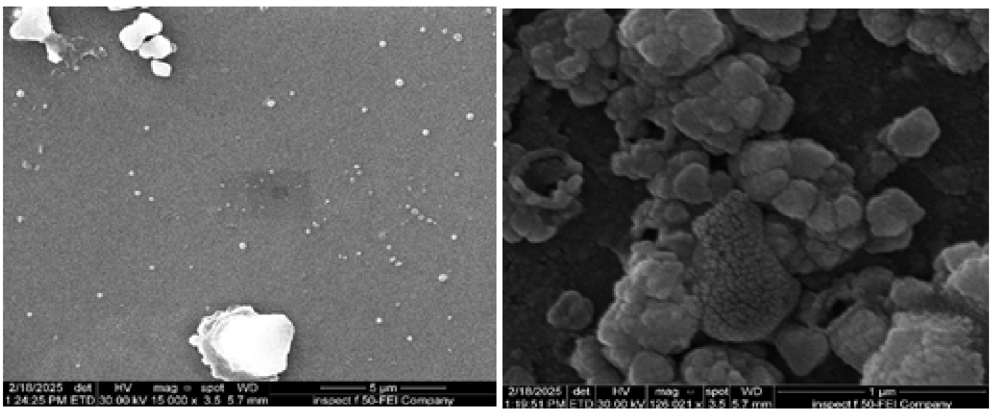

Figure (5) Images of the scanning electron microscopy at the sample 2%Cr

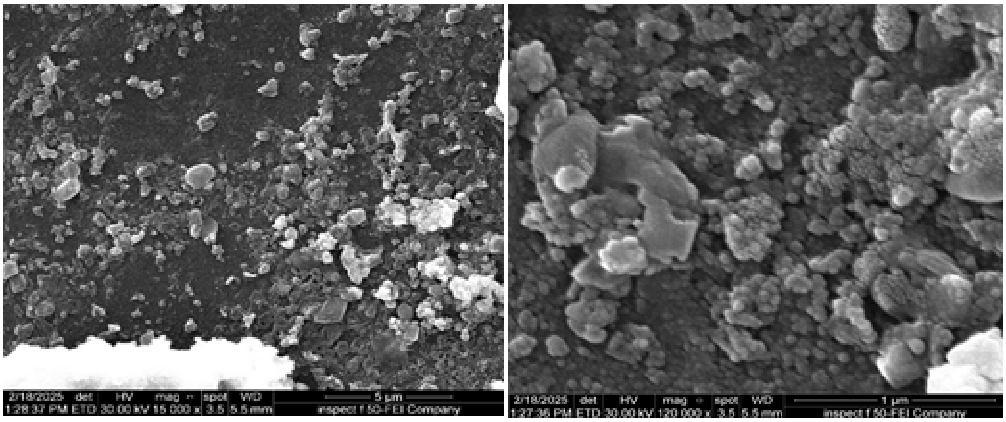

Figure (6) Scanning electron microscopy images at %C4

3.3Absorption

The results show that pure cadmium oxide membranes have the lowest absorption within the visible field, due to the large granular size and limited effective surface area. With the introduction of chromium ions, particularly starting at 2%, a gradual increase in absorption was observed as a result of the formation of additional energy levels within the energy gap that act as photon capture centers. This effect is clearly increased at grafting ratios of 4% where the decrease in grain size and the increase in surface roughness contribute to increasing the density of surface states and enhancing the likelihood of photon absorption.At the highest the absorption was maximized due to the formation of a nanostructure with a rougher surface and smaller grains, providing a greater number of active sites to react with light. Overall, chromium grafting resulted in a marked improvement in the optical properties of CdO membranes and their transformation from a material with limited absorption in its pure state to a material with a high and increasing absorption as the grafting concentration increased, which corresponds to the decrease in the energy gap derived from the Tauc curves [15].

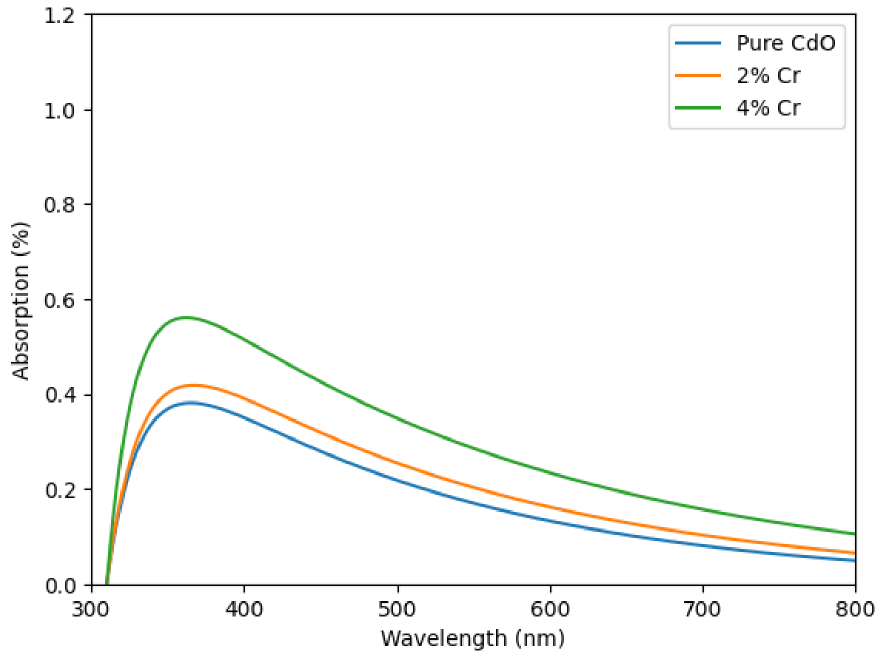

Figure (7) Absorption spectrum of prepared membranes

3-4 Optical Power Gap

The results show that the pure sample of cadmium oxide (CdO) has an optical energy gap of 2.63 eV, a value that is well consistent with the known reference values, demonstrating good crystal regularity and stability in the electronic structure with a marked absence of interstitial levels within the energy gap. This sample is therefore a primary reference for comparing the effect of grafting [18].

When the membranes were grafted with 2% chromium, the energy gap decreased slightly to about 2.60 eV, due to the entry of Cr^{3+} ions into the Cd^{2+} sites within the crystal lattice, generating internal stresses and minor structural distortions, resulting in additional energy levels close to the edge of the conduction band, thereby reducing the effective energy gap [16].As the grafting ratio increased to 4% of chromium, a more pronounced reduction in the energy gap was observed to reach about 2.48 eV, due to an increase in the density of structural defects and interstitial levels within the gap due to ion substitution and the difference in radii between Cd^{2+} and Cr^{3+}, as well as the effect of lattice stresses that reduce the energy required for electron transport.

This decrease contributes to enhanced absorption of low-energy photons and improved photoresponse of membranes.[17].

Table 2: Energy Gaps (Eg) of Prepared Membranes

SAMPLE	Eg (eV)
Pure CdO	2.63
2% Cr	2.60
4% Cr	2.48

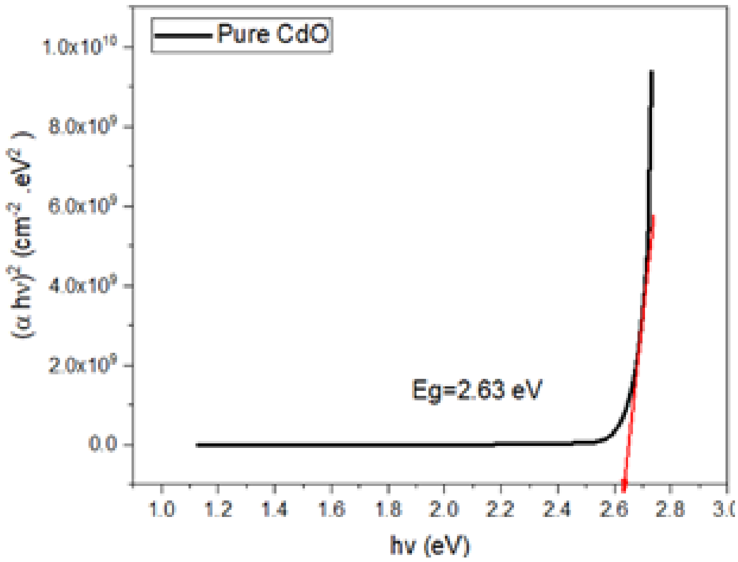

Figure 8: Energy Gap of Pure Sample CdO

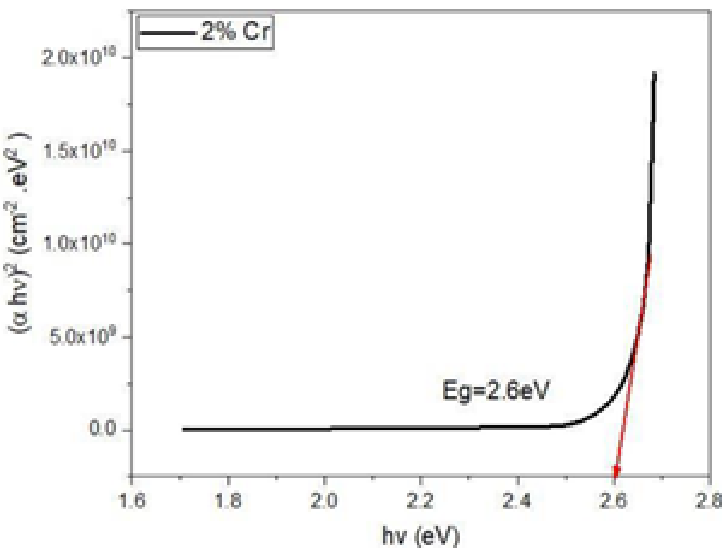

Figure 9: Energy gap of the sample 2%Cr

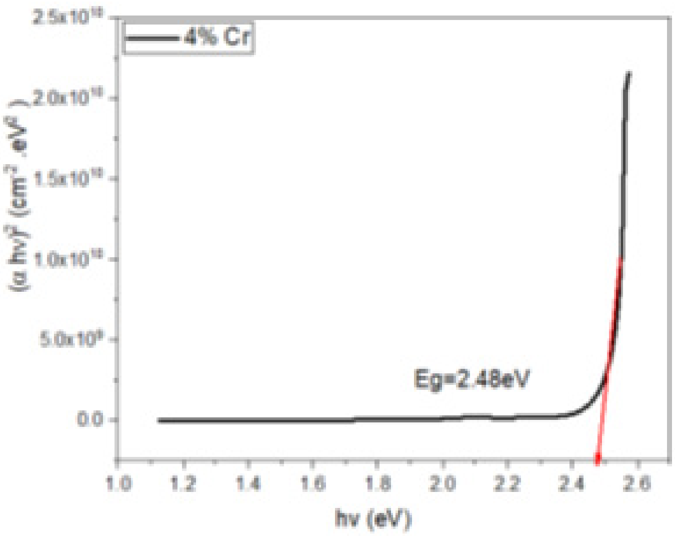

Figure (10) Energy Gap for Sample 4%Cr

3.5 sensitivity

The results show that the sensitivity of cadmium oxide membranes to NO_2 gas is clearly dependent on both temperature and grafting ratio. The pure sample (0% Cr) showed a relatively limited sensitivity, rising from about 20% at 50 ° C to about 34 at 200 °C, due to the large crystal size and limited effective surface area, which reduces the number of active centers capable of reacting with gas molecules.

With 2% chromium grafting, a marked improvement in allergy was observed, reaching about 41% at 200°C. This improvement is due to a decrease in crystal size and an increase in the density of the granular boundary, as well as a modification in the electronic distribution resulting from the replacement of Cd^{2+} ions with Cr^{3+} ions, which has enhanced the surface adsorption process of NO_2 molecules.

When the grafting percentage was raised to 4% chromium, the sensitivity increased more pronounced, reaching about 50% at 200°C, which corresponds to increased surface roughness and decreased granular size as shown by the results FESEM. This structural change led to an increase in the specific surface area and the number of active sites, making this sample more efficient and stable compared to the pure and grafted sample by 2%., results within the 0–4% Cr grafting domain show a gradual and systematic improvement in the sensitivity of CdO membranes with an increase in chromium content and temperature, confirming the positive role of grafting in enhancing the sensory performance of NO_2 gas [32]. The results of the response and retrieval time show that the pure sample of CdO has a relatively slow response, at 200 °C it was about 21.0 sec for response and 26.0 sec for retrieval. This is due to the lack of active centers and large crystal size, which limits the adsorption and deadsorption process.When the sample was grafted with 2% chromium, the response dynamics were clearly improved, with the response time reduced to approximately 20.1 sec and the retrieval time to 25.2 sec at 200°C. This improvement is due to the increased density of the effective centers and the higher granular boundaries resulting from vaccination. As the vaccination rate increased to 4% Cr, there was a further improvement, with the response time reduced to about 19.4 sec and the latency to 24.6 sec at 200°C. This behavior is due to the decrease in crystal size and the increase in surface roughness, which has enhanced the adsorption speed and deadsorption of NO_2 molecules.These results suggest that chromium CdO grafting within the 0–4% domain results in a significant improvement in response speed and retrieval, and makes membranes more suitable for NO_2 gas sensing applications. [18] .

Table 3: Sensitivity at different temperatures

Sample	Temperature (°C)	Sensitivity (%)	Response Time (sec)	Recovery Time (sec)
Pure CdO	50	20.0	22.8	27.9
	100	26.0	22.0	27.1
	150	31.0	21.4	26.6
	200	34.0	21.0	26.0
2% Cr–CdO	50	24.0	22.0	27.2
	100	31.0	21.3	26.5
	150	37.0	20.6	25.8
	200	41.0	20.1	25.2
4% Cr–CdO	50	29.0	21.3	26.6
	100	38.0	20.6	25.9
	150	45.0	19.9	25.2
	200	50.0	19.4	24.6

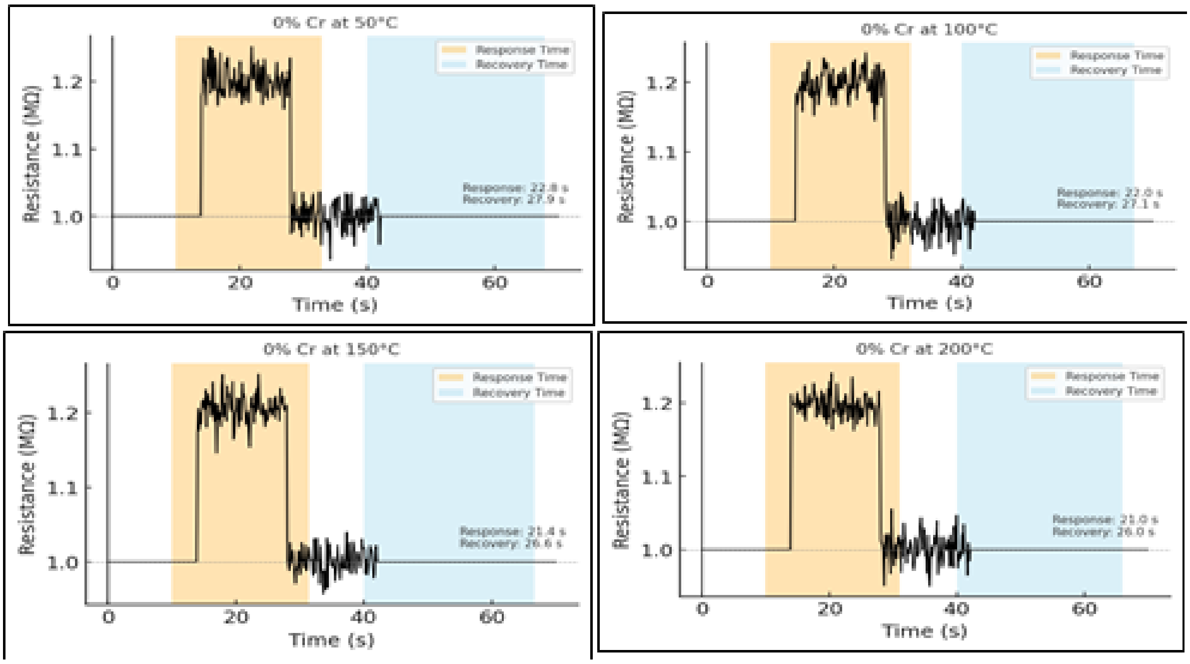

Figure (11) Response Time and Return of Pure Sample CdO

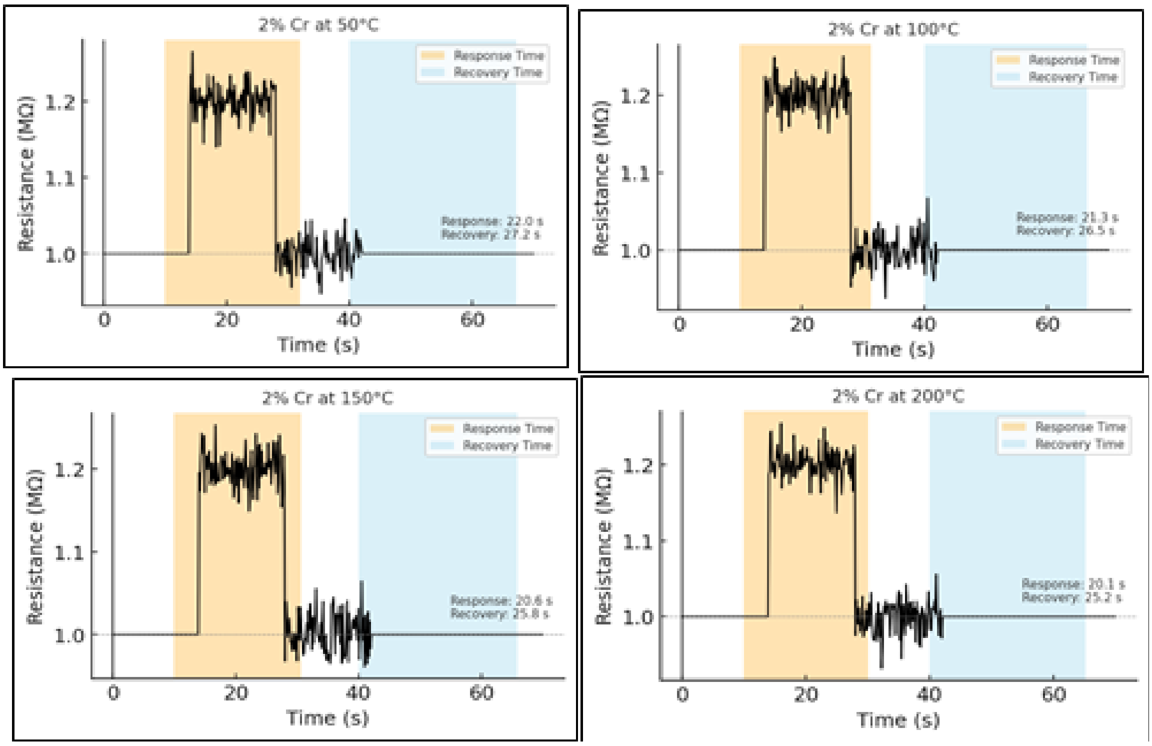

Figure (12) Response Time and Return Time for Sample 2%Cr

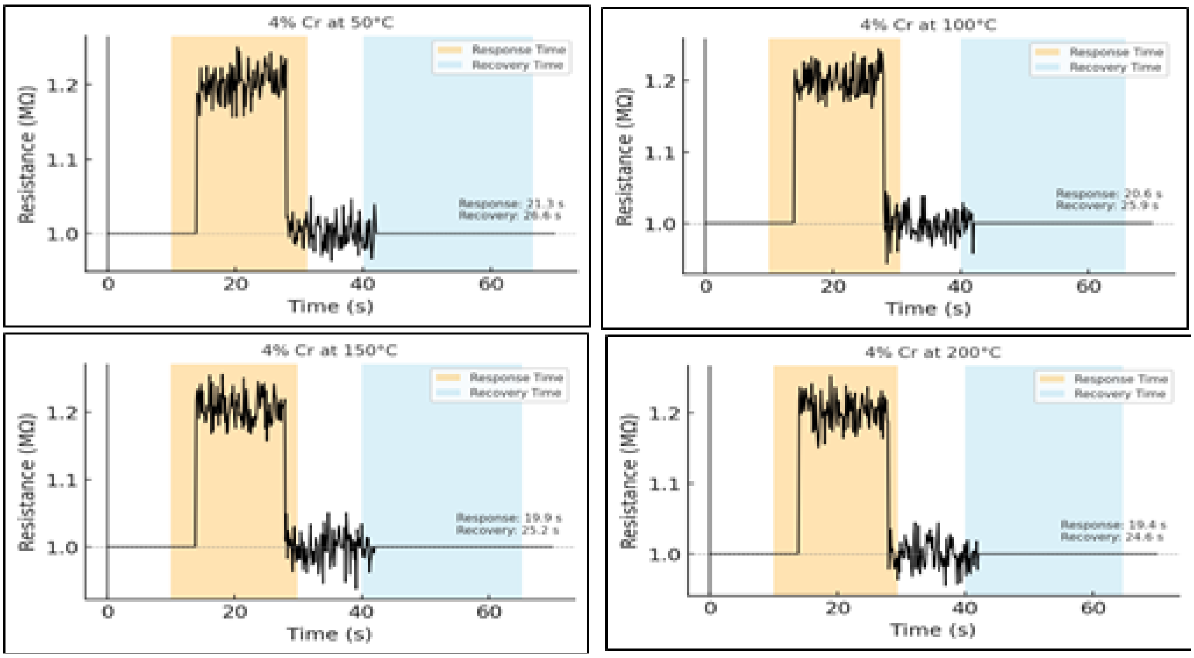

Figure (13) Response Time for Sample 4%Cr

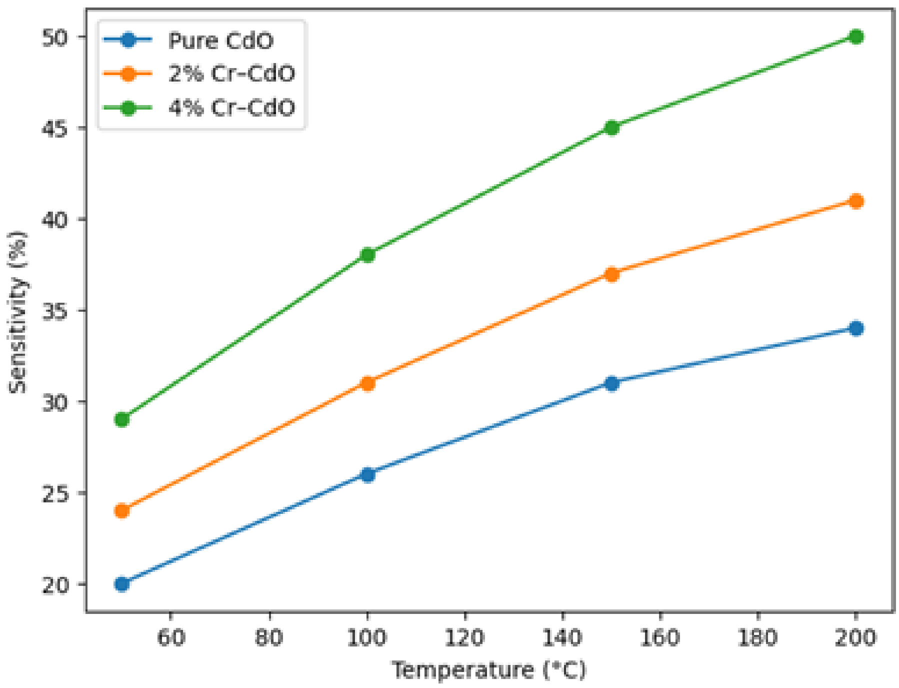

Figure (14).Variation of gas sensitivity as a function of operating temperature for pure and Cr-doped CdO sensors.

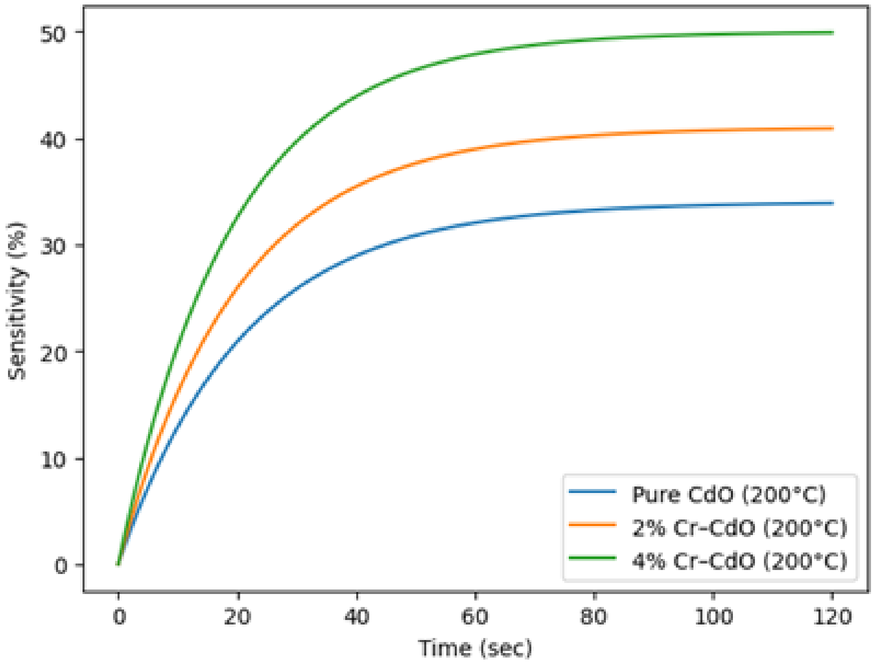

Figure (15)Dynamic response curves (sensitivity versus time) of pure and Cr-doped CdO sensors toward NO_2 gas at 200 °C.

Conclusions

The results showed that the grafting of the cadmium oxide membranes with chromium resulted in a clear improvement in structural and morphological properties by reducing the crystal size and increasing the roughness of the surface. The grafting also contributed to enhancing the optical properties by increasing the absorption and reducing the energy gap. In addition, the grafted films showed better sensor performance towards NO_2 gas compared to the pure sample, confirming their suitability for gas sensing applications.

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