

Deposition of CdO thin films by dip coating technique and the effect of concentration on gas sensor applications

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ABSTRACT In this study, CdO thin film coated glass samples were used as sensors for the measurement of butane gas. The deposition process was applied by dip coating technique using sol-gel solutions prepared at different concentrations. The samples were annealed at 500 °C after 10 times of dipping. The effects of the concentration on the structural, surface, optical properties of the produced thin films were investigated. Gas sensor measurements have been tested with the gas chamber that can be placed on the sensor and the created gas sensor control system. The effect of concentration on CdO gas sensors was investigated under gasless and butane gas conditions. As a result of the tests, it was determined that the CdO thin films produced at 0.1 M concentration showed the optimum gas sensor value.

KEYWORDS CdO thin film; Semiconductor; Sol-gel; Gas control system; Butane

1. Introduction

Metal-oxide semiconductor thin films are deposited on the surfaces of different materials such as glass, plastic, and ceramics in order to both benefit from their own properties and increase their functionality by adding new functions to their structures. Thus, semiconductor thin films allow the use of materials in different areas. Metal oxide thin films in gas sensors are used with their high sensitivity to gases (Nikolic et al., 2020), suitability for electronic devices (Gao and Zhang, 2018), low cost (Manorama et al., 2003), low power consumption (Zhang et al., 2019), excellent gas selectivity (Salunkhe and Lokhande, 2008; Patil et al., 2015), and short recovery time. Gas sensors can detect flammable gases such as hydrogen (Hübert et al., 2011; Sayago et al., 2005), butane, propane (Lee et al., 2003), suffocating gases such as CO (Liu et al., 2015), explosive gases such as LPG (Jain et al., 2006), acetylene (Austin et al., 2009), methane (Massie et al., 2006) and poisonous gases such as nitrogen dioxide (Lee et al., 2018; Navale et al., 2017), sulfur dioxide (Guo et al., 2016) hydrogen sulfide (Yadav and Indurkar, 2021). They also provide monitoring and control of environmental pollution.

CdO thin films are used in electrodes (More et al., 2019), gas sensors (Othman et al., 2021; Faris et al., 2022; Hassan and Salih, 2021), optoelectronic devices (Chandiramouli and Jeyaprakash, 2013), phototransistor (Srivastava et al., 2008), diodes and transparent electrodes (Ristić et al., 2004). They are sensitive to butane, CO₂ (Bari and Patil, 2014), N₂ (Salunkhe et al., 2009a), LPG (Bulakhe and Lokhande, 2014), ethanol (Kamble et al., 2011) gases and they are usually deposited on glass surfaces due to their smooth structure. CdO thin films are coated on glass substrates by techniques such as spray pyrolysis deposition (SPD) (Kasirajan et al., 2022; Shaker

Ahmed et al., 2020), sol-gel spin coating (Ceviz Şakar et al., 2021; Soylu, 2020; Rajput et al., 2018; Kumari and Kumar, 2020), SILAR (Salunkhe et al., 2009b; Beevia et al., 2010; Shameem et al., 2017; Gokul et al., 2013), chemical bath deposition (CBD) (Jassim and Nassar, 2020, 2021) and sol-gel dip coating in literature (Paulraj et al., 2018).

In this study, CdO thin films prepared at 0.1 M, 0.3 M and 0.5 M concentrations were deposited on glass surfaces by dip coating technique. They were characterized by Scanning Electron Microscopy, X-Ray Diffraction Device and UV-Vis spectrophotometer. Then, the characterized samples were placed as sensors in the designed gas chamber. The response measurements of the CdO gas sensor to Butane gas were made with the gas control and detection system that was originally created. In addition, the effect of concentration on semiconductivity and sensitivity to butane gas was also investigated.

2. Methods

2.1 Preparation process

In this study, cadmium acetate dihydrate salt (Cd(CH₃COO)₂ · 2 H₂O) (Sigma-Aldrich, ≥ 98.0 % purity) as chemical reagent, 2-methoxyethanol (CH₃OCH₂CH₂OH) (Sigma-Aldrich, ≥ 99 % purity) as solvent and monoethanolamine (NH₂CH₂CH₂OH) (Sigma-Aldrich) as stabilizer were used for CdO thin film deposition by sol-gel dip coating technique. In the experimental processes, 3 different solution concentrations were chosen as 0.1 M, 0.3 M and 0.5 M. The cadmium acetate dihydrate salt was prepared for 3 different concentrations and dissolved in 100 mL of 2-methoxyethanol. 2 drops of monoethanolamine stabilizer were added to the CdO solution. The solutions were stirred continuously for 2 hours at room temperature with a magnetic stirrer and left to rest for 24 hours.

For film coating with CdO sol-gel solution, 1 mm thick glass substrates were cut evenly in dimensions of 10 mm × 25 mm. Glass substrates were cleaned in an ultrasonic bath with ultra pure water and alcohol, then dried

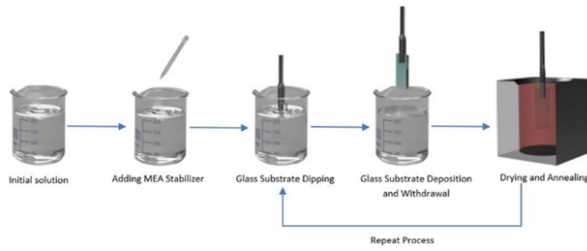


Figure 1: Process of dip coating technique.

at 150 °C. It is aimed to deposit CdO particles homogeneously by dipping coating technique on glass substrates with smooth surface.

2.2 Thin film deposition by dip coating technique

In Fig. 1, the process of Dip Coating Technique is shown. In the dip coating technique, sol-gel initial solution is prepared at the selected concentration. Stabilizer is added to the prepared solution with a dropper. The dip coating process is done by manually or mechanically dipping the substrate into the prepared solution. After the dipping process, the substrate is removed from the solution and taken to the drying process after waiting for a while. This process is repeated according to the desired number of floors. Finally, the thin films are annealed.

In this study, 3 different concentrations of CdO solutions were prepared. Thin films were deposited on the surface of glass substrates by the dip coating technique. Glass substrates were dipped in the prepared solution, and after waiting for 60 seconds, they were removed from the solution at a constant speed. Then, the substrates were washed with ultrapure water to create an even distribution on the surface and dried at 150 °C for 90 seconds. Dipping was done mechanically. Dipping and withdrawing were done at a constant speed of 10 mm/s. This process was done with 5, 10 and 15 repetitions. In the 5-layer coating, it was observed that the CdO structure was not evenly distributed on the surface of the glass substrates and there were many voids in the structure. It was found that 5 layers were insufficient for the coating. In the 15-layer coating, it was determined that the CdO structure over accumulated and agglomerated on the surface of the glass substrates. The transparency of the surface decreased and 15 layers were determined to be too much for the coating. The homogeneous coating was observed on the surface of substrate materials deposited 10 layers. In the 10-layer coating, it was determined that the CdO structure did not over accumulate on the surface and the surface voids were less. The coating layer was determined as 10. CdO coated glass substrates, whose dipping process was completed, were taken to the annealing. For annealing, experiments were first carried out at 150 °C. It was observed that the temperature was not sufficient for crystallization. It was determined that crystallization started at 300 °C, but it was not sufficient for glass substrates. As a result of annealing processes on glass substrates, the optimum temperature for the crystallization of the CdO structure on the surface was determined as 500 °C. CdO

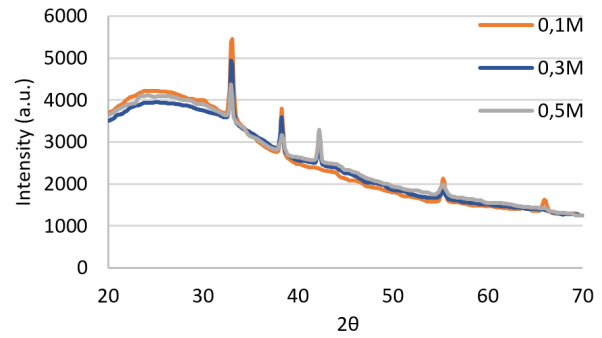


Figure 2: Comparative XRD analysis spectrum of CdO thin films at different concentrations.

Table 1: Crystallographic properties of CdO thin films.

Series	2θ (°)	Miller Index (hkl)	FWHM(°)	a (Å)	D (nm)
0.1 M	32.99	111	0.36	4.6988	23.014
	38.27	002	0.27	4.6997	31.1432
	55.27	022	0.36	4.697	24.9084
	65.93	113	0.45	4.6951	21.041
0.3 M	32.97	111	0.2716	4.7016	30.503
	38.23	002	0.2716	4.7044	30.956
	55.25	022	0.5432	4.6986	16.5062
0.5 M	32.91	111	0.2716	4.7099	30.4983
	38.3	002	0.4526	4.6962	18.5802
	55.23	022	0.7242	4.7002	12.3797

coated glass samples were annealed at 500 °C for 2 hours.

3. Results

3.1 Structural properties

The structural properties of the obtained films were examined with the PANALYTICAL Empyrean X-Ray Diffraction (XRD) device. CuK α radiation ($\lambda = 1.5406 \text{ \AA}$) was used at a scanning speed of 2 degrees/min. The X-ray diffraction spectrum in the region of 2θ from 20° to 70° is given in Fig. 2. When XRD results were examined, three diffraction peaks observed at $2\theta = 32.9^\circ$, 38.2° and 55.2° corresponding to (111), (002) and (022) planes. Also, one peak observed at 65.9° , corresponding to (113) plane for the 0.1 M sample, is related to cadmium structure. An impurity peak was detected at a diffraction angle of 42.1° for 0.3 M and 0.5 M samples.

The crystalline sizes (D) obtained by Scherrer Formula, full width at half-maximum (FWHM) and lattice parameter "a" in CdO cubic structure (ICDD code: 98-002-4802) are given in Table 1. When the lattice parameter values are examined, it is understood that the CdO thin films deposited at all concentrations are cubic structure. With the increase in concentration, it is seen that crystalline sizes increase and FWHM values decrease.

3.2 Surface morphology

The effect of concentration on the morphological structure of CdO thin films deposited on glass substrates at 0.1 M,

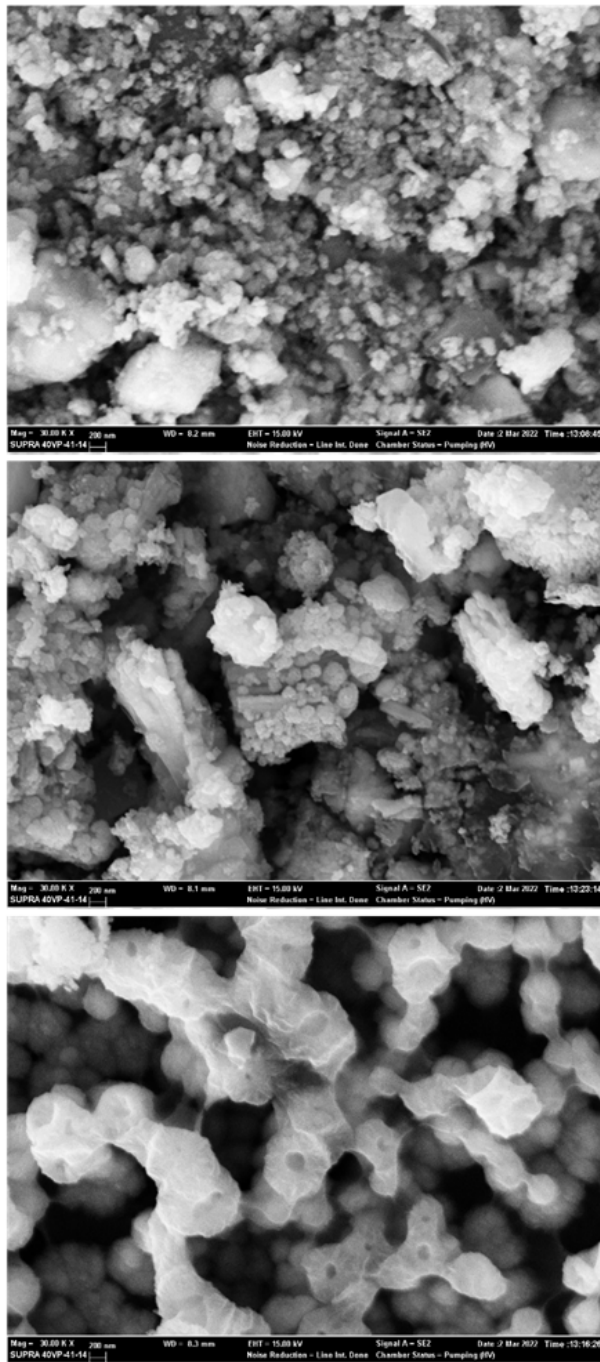


Figure 3: SEM images of 0.1 M, 0.3 M, and 0.5 M CdO thin film.

0.3 M and 0.5 M concentrations were examined by SEM (ZEISS Supra 40VP Field Emission Scanning Electron Microscope) images. 0.1 M and 0.3 M CdO thin films have smooth and homogeneous surface morphology with nanocrystalline grains. Also, 0.1 M and 0.3 M CdO thin films are dense and adhered well to the glass substrate. It was observed that the grain size increased with increasing concentration. It was determined that the films formed on the surface for 0.1 M in Fig. 3a were smaller than those in Fig. 3b for 0.3 M. In Fig. 3c, which is 0.5 M, it was observed that the agglomeration increased. In addition, the CdO crystalline structure began to decompose at a

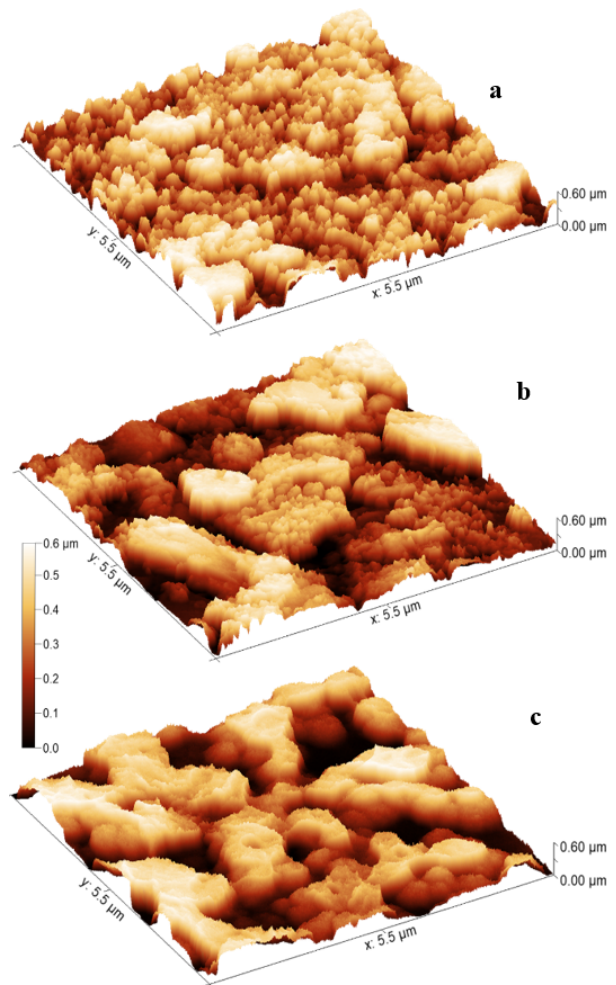


Figure 4: 0.1 M, 0.3 M and 0.5 M CdO thin film AFM images, converted by Gwyddion software of SEM images.

Table 2: Mean Roughness values of CdO thin films.

Series	0.1 M	0.3 M	0.5 M
Mean Roughness (nm)	119.7	87.9	66.8

concentration of 0.5 M. A tendency to transition from crystal structure to amorphous structure was observed. It was understood that the thin film crystal structure deteriorated with the increase of annealing temperature and concentration. It was determined that thin films produced at 0.1 M and 0.3 M concentrations formed cauliflower-shaped structures specific to the CdO crystal structure on the glass substrate surface.

SEM images of 0.1 M, 0.3 M and 0.5 M CdO thin films converted to AFM images with Gwyddion software are shown in Fig. 4. When the images are examined, it is seen that the roughness on the surface decreases with the increase in concentration. It is seen that the surface roughness of 0.1 M CdO thin films is high and there is agglomeration on the surfaces of 0.5 M CdO thin films.

The surface roughness of CdO thin films is calculated from the AFM images. Mean Roughness values of CdO thin films are given in Table 2.

Table 3: EDX results of CdO thin films.

Series	Cadmium (Atomic %)	Oxygen (Atomic %)
0.1 M	57.06	42.94
0.3 M	53.10	46.90
0.5 M	34.49	65.51

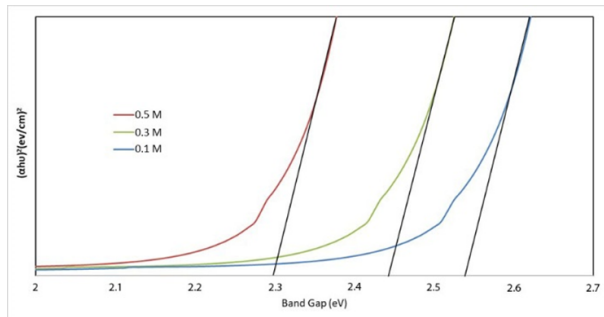


Figure 5: Band gap values of the CdO thin films.

The interactions between the gas and CdO films mainly take place at the surface of films. It is thought that the gas holding capacity of the films increases with the increase in surface roughness.

The elemental composition of 0.1 M, 0.3 M and 0.5 M CdO films according to the energy dispersive X-ray (EDX) analysis system are given in Table 3. With the increase of the concentration, a decrease was observed in the atomic cadmium percentage, while an increase in the oxygen percentage was observed. As the active sites on the Cd surface react, oxidation has occurred in these regions. Therefore, the amount of oxygen increases in EDX results. At 0.5 M concentration, the difference between the percentage of cadmium and oxygen became increasingly evident.

3.3 Optical properties

The produced CdO thin films were measured by UV-vis spectrophotometer (Perkin Elmer Lambda 25) in the wavelength range of 300–1100 nm. The plots of $(\alpha h\nu)^2$ vs. $h\nu$, calculated according to the Tauc method, are given in Fig. 5. The band gap values of thin films are determined from where the linear portion of the curve intersects with the hu axis.

The band gap of CdO thin films was calculated as 2.54 eV for 0.1 M, 2.43 eV for 0.3 M and 2.29 eV for 0.5 M. When the literature is examined, the band gap values of CdO thin films at 0.1 M, 0.3 M and 0.5 M concentrations are between 2.21 eV and 2.60 eV (Soylu, 2020; Shameem et al., 2017; Güney et al., 2022; Nakate et al., 2019; Rajput et al., 2017). The band gap values calculated within the scope of this study were found to be compatible with the studies in the literature.

3.4 Gas sensor measurement system

The use of CdO thin films produced in this study as a gas sensor was tested in the gas sensor application and control unit given in Fig. 6. As a control card; simple to use, easy to access and reprogrammable Arduino Uno

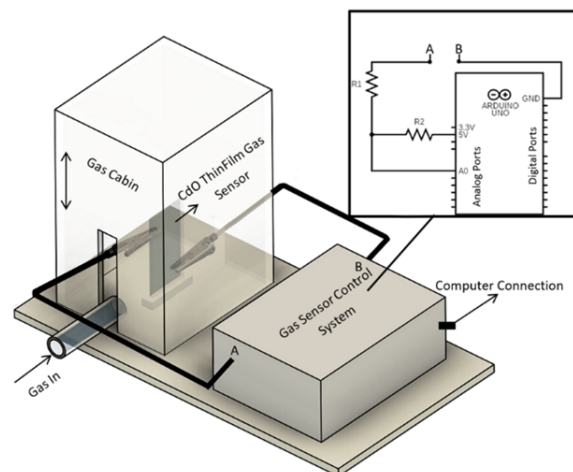


Figure 6: Schematic illustration of Gas Sensor and Control System.

card was preferred. CdO thin film gas sensor trials were carried out with the sensor control system installed on this card. The circuit diagram of the gas sensor control system is shown in Figure 6. The gas sensor control system is composed of the control card, two resistors and a sensor module. 5 V DC constant current was applied to the CdO thin film gas sensor. The voltage change on the resistor was measured. Analog data received from resistor was converted into digital data and monitored. CdO thin film coated glass substrate was connected between two conductive crocodiles and used as a sensor. The gas cabinet designed to make gas sensor measurements was placed on the sensor. After the gas sensor was placed in the cabin, gas entry was provided to the medium. Thus, sensor detection and response tests were carried out.

3.5 Gas sensor response

In this study, butane gas was chosen for gas sensor experiments. The response graph of the produced gas sensors according to time in butane gas and non-gas medium conditions was given in Figure 7. The voltage on the resistor in the system increases as the sensor interacts with the oxygen in the medium under room conditions. When the gas enters the medium, it is seen that the oxygen that the gas sensor interacts with is replaced by butane gas, reducing the voltage on the resistor.

When the system was started, the semiconductor properties of the CdO gas sensors were measured by first waiting for 100 seconds under room conditions. The voltage values on the resistor were balanced. Then, butane gas was introduced into the gas cabin in the system for 80 seconds. The response values of the CdO gas sensor to butane gas were measured. The sensor values were determined after the butane gas output. 0.1 M and 0.3 M CdO thin films showed semiconductor properties under room conditions, but data could not be obtained for 0.5 M CdO thin films. Therefore 0.5 M CdO thin films were not used in gas sensor applications. The voltage on the resistor is between 4.5–5 V without including the sensor in the system. In the 0.1 M sample, the voltage on the resistor

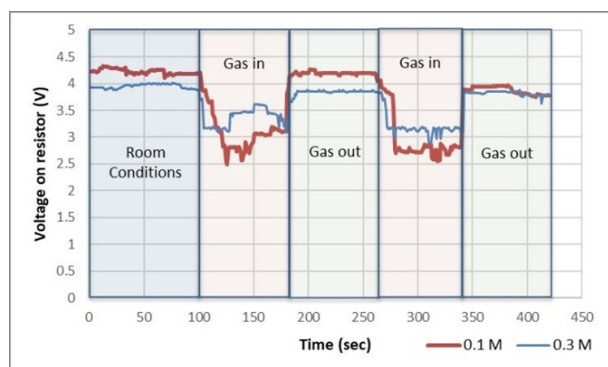


Figure 7: Response graph of gas sensors by time.

Table 4: Response and recovery times of CdO thin films in butane gas applications.

Series	First		Second	
	Response (sec)	Recovery (sec)	Response (sec)	Recovery (sec)
0.1 M	22	3	12	5
0.3 M	6	10	12	4

was determined the range of 4–4.5 V at room conditions. When butane gas was included in the medium, the voltage on the resistor decreased to the range of 2.5–3 V. When butane gas was released, the voltage on the sensor resistor returned to values close to the initial state. In the 0.3 M sample, the voltage on the resistor was measured close to 4 V at room conditions. These values decreased to the range of 3–3.5 V at the butane gas in. The response and recovery times of 0.1 M and 0.3 M CdO thin films in butane gas sensor applications are given in Table 4.

At the first butane gas inlet, the conductivity change in the 0.1 M sample was measured as 1.51 V and the response time as 22 seconds. The conductivity change in the 0.3 M sample was determined as 0.82 V and the response time was 6 seconds. At the 2nd gas inlet, the conductivity change was measured as 1.65 V and 0.67 V in the 0.1 M and 0.3 M samples, respectively, and the response times were 12 seconds. When similar studies were examined, it was observed that the response time was 12 seconds and the recovery time was 15 seconds for the CdO gas sensors deposited by the magnetron sputtering method (Abed et al., 2022), the response time was 18 seconds and the recovery time was 32 seconds for the CdO gas sensors deposited by the spray pyrolysis method (Nakate et al., 2019).

In the 0.1 M CdO film, its surface to volume ratio is the largest owing to the smallest grain size, which is helpful to increase the adsorption of oxygen or butane gas on CdO films, resulting in the optimum gas sensor properties. It was observed that the responses of the 0.1 M sample were higher than the 0.3 M sample under room conditions and butane gas in. For the repeatability of the system, butane gas in and out trials were made for the second time and values similar to the first trials were measured. In the

gas sensor measurements, it was observed that the sensor returned to values close to its state at room conditions each time, which indicates the repeatability of the CdO gas sensor. When the gas sensor experiments were examined, it was determined that the glass samples coated with 0.1 M CdO thin film showed better semiconductivity and the gas responses were clearer than the other samples

4. Conclusions

CdO thin films were deposited on glass substrates by the sol-gel dip coating technique. The effect of three different initial concentrations (0.1 M, 0.3 M and 0.5 M) on thin film coating was investigated. When the XRD spectrum was examined, the diffraction peaks observed (111), (002) and (022) planes of the CdO crystal structure in all series. When the SEM images of CdO thin films were examined, the specific cauliflower shape of the CdO crystal structure was detected in the 0.1 M and 0.3 M samples. The initial concentration of 0.1 M was determined as optimum for CdO thin films. In the gas sensor tests for butane gas, it was determined that the 0.1 M and 0.3 M samples had gas sensitivity and the 0.5 M sample had no sensor response. It was observed that the gas sensitivities decreased as the CdO concentration increased, and it was determined that the semiconductor thin films produced at 0.1 M concentration showed optimum gas sensor properties. CdO thin films coated on various substrates at low concentrations can be used as an alternative gas sensor for the detection of different gases. It is understood that the produced CdO gas sensor will contribute to the development of sensors with high sensitivity at room temperature.

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