

## A Study of NO<sub>2</sub> Gas Concentration on Response of CdO Thin Films Prepared by Novel Reflux Method

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In present study, using simple and inexpensive novel reflux method, Cadmium Oxide (CdO) thin films were successfully deposited on glass substrate. Here to deposit CdO on glass substrate, Cadmium Chloride (CdCl<sub>2</sub>) was used as a source of Cd<sup>+</sup> ions, while ammonia was taken as complexing agent. Structural analysis and surface morphology of prepared CdO thin film was analyzed by X-ray diffraction and scanning electron microscopy respectively. Also wettability test was done by using goniometer which showed hydrophilic nature of deposited CdO thin film. Optical properties of CdO thin film was completed by using UV-Visible spectroscopy which revealed that deposited CdO thin film has direct band gap about 2.01 eV. NO<sub>2</sub> gas sensing properties like sensitivity, response recovery & response time of prepared CdO sensor was determined by using Keithley gas sensing unit. In present work effect of concentration of NO<sub>2</sub> gas on response of CdO sensor was studied and it was cleared that as NO<sub>2</sub> gas concentration increases (25 ppm – 100 ppm), the response of CdO sensor was also increases & it becomes maximum i.e. (57 %) for 100 ppm of NO<sub>2</sub> with optimized temperature of 200 °C. Variation of response and recovery time with NO<sub>2</sub> gas concentration was studied and it was concluded that as concentration of NO<sub>2</sub> gas increases, response time increases while recovery time decreases.

**Keywords:** CdO thin film, Reflux method, Gas sensor.

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### 1. INTRODUCTION

The deposition of thin films of nanometer thickness is very much applicable because of their role in many applications in the various fields of science and technology such as electronics, space science, industry area and optics [1]. There are number of physical and chemical methods are used to deposit thin films of various kinds, out of which transparent conducting oxides (TCO) thin films have been paid much more attention due to its crucial role in gas sensor, solar cell, supercapacitors and smart windows [2]. TCO are very much attracted in recent years because of their chemi adsorption property and changing its conductivity in the presence of numerous gases [3]. Previously published results also showing that when TCO or MOS exposed by same gases, their electrical properties are changes and this phenomenon is much useful in gas sensing application [4]. In recent years, detection of hazardous and harmful gases is very important and serious issue in environmental monitoring chemical control and safety in home and society [5]. The designing and development of efficient devices for monitoring hazardous and toxic gases present in air is very challengeable in gas sensor field. The air pollution in environment which arises due to increase in the concentration of harmful and toxic gases like carbon monoxide (CO), sulphur dioxide (SO<sub>2</sub>), carbon dioxide (CO<sub>2</sub>), nitrogen dioxide (NO<sub>2</sub>) which affect very badly on human health. Out of

all these toxic gases NO<sub>2</sub> is common pollutant which affect on human respiratory system and causes of heart diseases [6]. Therefore for detection of NO<sub>2</sub> gas at its low level concentration, the preparation of small sized quick response sensors with long lifetime is necessary thing [7]. There are different types of TCO gas sensors are investigated for NO<sub>2</sub> gas sensing such as ZnO, SnO<sub>2</sub>, CuO, TiO<sub>2</sub>, Nb<sub>2</sub>O<sub>5</sub>, MoO<sub>3</sub> [8-11]. Among all these metal oxides cadmium oxide is well known and famous TCO with direct band gap having n-type semiconducting nature. CdO has attracted interest because of its excellent sensitivity and selectivity towards many harmful gases such as LPG, NO<sub>2</sub>, CO<sub>2</sub> [12]. There are different physical & chemical routes are used to deposit TCO thin films such as Hydrothermal [13], Sol-gel [14], spray pyrolysis [15] but in present work to prepare CdO thin films we used simple inexpensive aqueous based unique reflux method. In present work we have reported the synthesis and characterization of CdO thin films via reflux method. Here thin films fabricated on glass substrate and then prepared films after annealing at 400 °C were characterized using XRD, SEM, UV-Vis, contact angle techniques. After this material characterization of CdO thin films were carried out towards host of gas (i.e. NO<sub>2</sub>) at optimized temperature of gas was 200 °C. During this study additionally response time, response value of CdO films were systematically studied and explored. Here our attempt to depositing CdO thin film by novel reflux method which

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is simple & inexpensive & to explain sensing mechanism of CdO sensor with varying concentration of NO<sub>2</sub>.

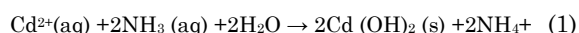
## 2. EXPERIMENTAL DETAILS

### 2.1 Materials & Chemicals

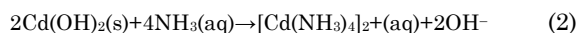
Cadmium chloride (CdCl<sub>2</sub>·2H<sub>2</sub>O) purchased from Sigma Aldrich, Ammonia solution (NH<sub>3</sub>) purchased from Sigma Aldrich, and glass substrates purchased from Star India. This stainless-steel plate SS 304 grade was cut into pieces of 1 × 5 cm<sup>2</sup> and polished with sic paper and then degreased with double distilled water. The SS pieces were kept in ultrasonic bath for 30 minutes to remove surface oxides. Afterwards they were rinsed with double distilled water and dried in air to use as substrates.

### 2.2 Preparation of CdO Thin Films

In present work nanostructured CdO thin films are prepared by the reflux method. The solution was prepared by adding ammonia solution drop wise into the aqueous solution of 0.03 M CdCl<sub>2</sub> with constant stirring during this process there was formation of precipitate of Cd(OH)<sub>2</sub>. This process of formation of white colored Cd(OH)<sub>2</sub> precipitated is given by,



Now after adding excess ammonia drop wise, precipitation of Cd(OH)<sub>2</sub> was vanished and we get clear solution when PH of solution was adjusted to 12. This process is cleared by following reaction,



Now this cleared solution was transferred into well cleaned round bottom flask containing ultrasonically cleaned glass substrate and fix it to reflux unit at 60°C temperature about 1 hr. 30 min. To get Cd(OH)<sub>2</sub> on glass substrate & schematic of reflux unit is shown in Fig. 1a.

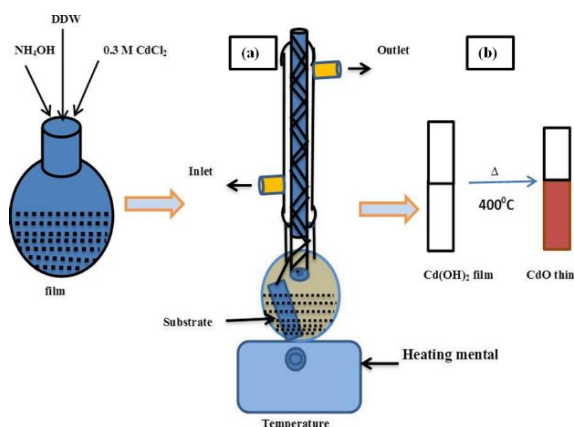
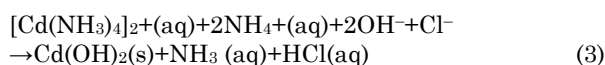


Fig. 1 – (a) Schematic of reflux setup (b) CdO thin film after annealing



Further to remove hydroxide phase & to obtain CdO thin film Cd(OH)<sub>2</sub> film was annealed at 400 °C for 1 hr & then we get brown coloured CdO thin film shown in Fig. 1b.



### 2.3 Characterization

Many characteristics properties of CdO thin film such as crystal structure, surface morphology, wettability test, optical band gap were studied by using X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Contact angle measurement, UV-Vis spectroscopy techniques.

## 3. RESULTS & DISCUSSION

### 3.1 XRD Analysis

XRD pattern of synthesized CdO thin film prepared by novel reflux method which was annealed at 400 °C is shown in Fig. 2 which conclude that number of sharp peaks of diffraction are obtained which are well matched with JCPDS card no. 05-0640 of cubic FCC crystal structure of CdO thin film. The lattice parameter such as values of *a*, *b*, *c* are calculated by using the relation [16],

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}, \quad (5)$$

From this relation calculated values of lattice parameters are *a* = 4.706 Å, *b* = 4.706 Å, *c* = 4.560 Å which are good agreement with above mentioned JCPDS card no. From this result we conclude that *a* ≈ *b* ≈ *c* so prepared CdO thin film shows cubic crystal structure. From Fig 2 it was confirmed that, no any impurity peak was observed in XRD pattern which revealed that highly pure CdO material is synthesized by this method [17]. The crystallite size (*D*) of prepared CdO thin film was calculated by using Scherrer formula which is given by,

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (6)$$

Where,

*λ* – Wavelength of X-ray radiation

*θ* – Diffraction angle

*β* – FWHM in radiation

Using this formula the average *D* value was calculated using highly preferred (111) peak which is equal to 25 nm this result well matched with *D* value given by JCPDS card no. 05-0640. Using obtained XRD pattern other structural parameters like stacking fault (SF), dislocation density (*δ*) and microstrain (*ε*) were also calculated to find all these parameters, the following relations were used,

$$\text{S.F} = \frac{2\pi^2\beta}{45(3\tan\theta)^2}, \quad (7a)$$

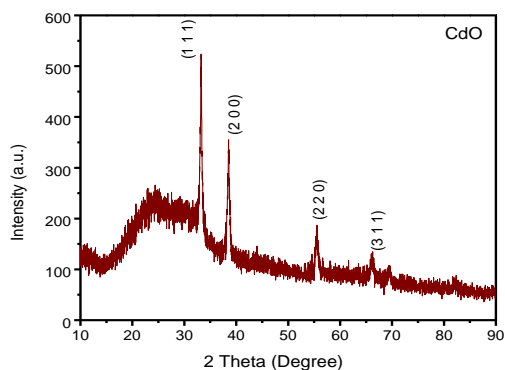
$$\delta = \frac{1}{D^2}, \quad (7b)$$

$$\epsilon = \frac{\beta \cos\theta}{4}. \quad (7c)$$

Table 1 represents values of SF, *δ*, *ε* for high intense (111) plane of prepared CdO thin film. From previous obtained results on CdO thin films it was confirmed that the values in table (1) are good agreement with previous performed results [18].

**Table 1** – Structural parameters of CdO thin film

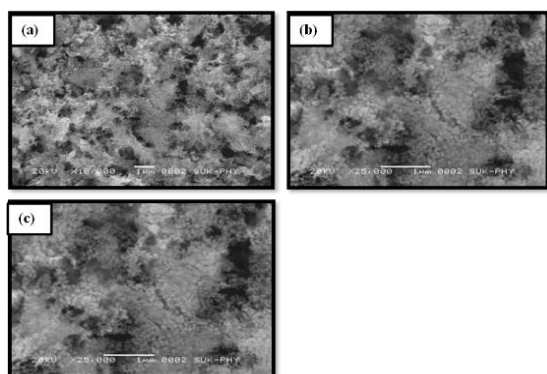
Annealing temperature (°C)	D-value(nm)	Dislocation density( $\delta$ ) lines/m <sup>2</sup>	Microstrain ( $\epsilon$ ) m <sup>4</sup> /lines <sup>2</sup>	S.F(J/m <sup>2</sup> )
400	20.85	2.300 × 10 <sup>15</sup>	1.6625 × 10 <sup>-3</sup>	3.366 × 10 <sup>-3</sup>



**Fig. 2** – XRD pattern of CdO thin film

**3.2 Surface Morphology Studies**

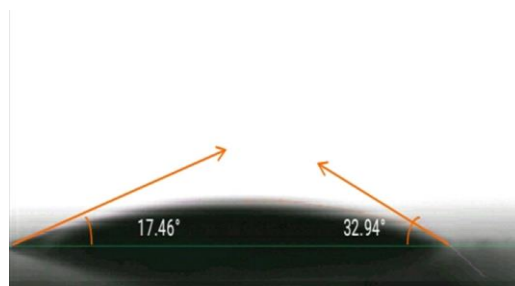
The SEM micrographs of CdO thin films at different magnification on glass substrate are shown in Fig. 3 (a), (b), (c). All these figures showed porous morphology of prepared CdO thin film which provide large surface to the volume ratio and so increase the active adsorption sites for NO<sub>2</sub> gas molecules this property of CdO thin film increases sensing properties of CdO sensor [12].



**Fig. 3** – SEM images of CdO thin film at magnification (a) – ×5 000, (b) – ×15 000, (c) – ×25 000

**3.3 Surface Wettability**

It is most important test to determine the quality of deposited material (i.e. either hydrophobic or hydrophilic). Fig. 4 shows the contact angle image of prepared CdO thin film which was annealed at 400 °C. Fig. 4 shows the values of contact angle of CdO film with water was different on the both sides of water drop. The value of contact angle at left side was 17.460 while at right side of water drop 32.940 and this result conclude that obtained value of contact angle is less than 90 so CdO thin film shows hydrophilic nature of surface behaves property of completed wetting surface.



**Fig. 4** – Angle of contact of CdO thin film with water drop

This complete wet surface can be very effective for gas adsorption on CdO surface which enhance sensing property of CdO [19].

**3.4 Optical Properties Study**

Optical absorption spectra for CdO thin film annealed at 400 °C was studied in Fig. 5. This plot shows variation of  $(\alpha h\nu)^2$  with photon energy  $(h\nu)$ . From Fig. 5 it was cleared that plot of  $(\alpha h\nu)^2$  Vs  $(h\nu)$  is almost linear nature of optical transition of CdO film is direct nature. Extrapolation of this curve to the  $h\nu$  axis gives the value band gap of CdO thin film which is 2.01 eV. This value can be calculated by using Tacus relation which is given by,

$$(\alpha h\nu) = A(h\nu - E_g)^P \tag{8}$$

Where,

$A$  – Constant

$P$  – Discrete value  $\frac{1}{2}$  or 2 depends on direct or indirect transition

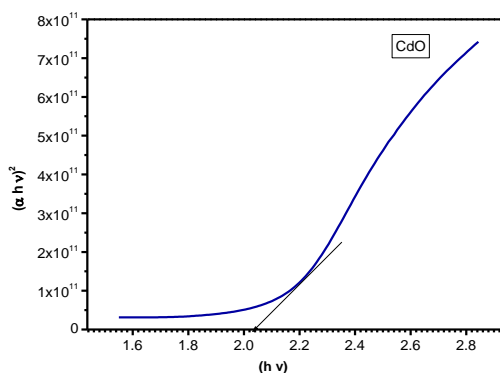
$\alpha$  – Absorption coefficient

$h$  – Planck's constant

$\nu$  – Frequency of light used

$E_g$  – bandgap energy

As obtained bandgap is narrow electrons are easily available for the conduction which is beneficial in gas sensing.



**Fig. 5** – Optical absorption spectra of CdO thin film

**3.5 Gas Sensing Study**

It is well familiar that the gas sensing mechanism of metal oxide gas sensors ( $N$ -type or  $P$ -type, here in CdO- $N$ -type) is mainly depends on the adsorption and desorption of oxidizing or reducing gases (here NO<sub>2</sub> – oxidising gas) and its effect on change in electrical resistance or conductivity [21]. Gas sensing properties

of prepared CdO thin film was studied by using help of custom fabricated gas sensing measurement unit containing two probe system in it. To study the response of CdO sensor to the NO<sub>2</sub> gas with varying concentration of NO<sub>2</sub> was measured and examined by using programmable electrometer (Model: Keithley 6514 system) as shown in figure (6) [20].

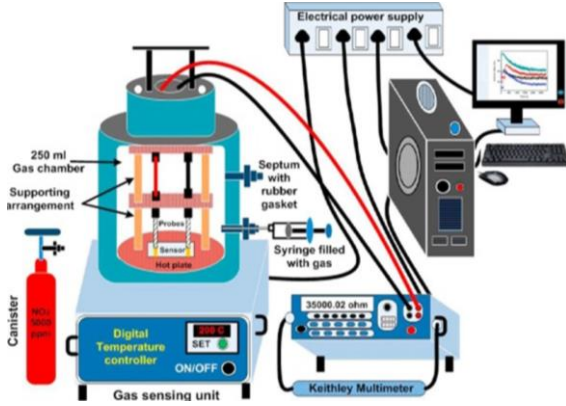


Fig. 6 – Schematic of Gas sensing setup

The gas sensor unit consist of 250 ml gas chamber surrounded by insulated cylindrical wall as shown in Fig. 6. The response of CdO sensor to corresponding NO<sub>2</sub> gas and fresh air respectively as a function of time (i.e. response curve)was recorded in computer which is attached to the system of electrometer for the measurement of effect of gas concentration on the resistance of sensor, initially two silver electrode separated by distance 10 mm apart from each other were attached on the CdO thin film for contact and then corresponding measurement of response in terms of change in resistance were taken using kiethley 6514 system electrometer [20].

After making necessary arrangement, mixture of NO<sub>2</sub> gas and fresh air introduced in chamber.In present study gas sensing characteristics of CdO sensor structure were studied as function of concentration (25 ppm – 100 ppm) with 200 °C optimized temperature of gas. In proposed work responses of CdO sensor with varying NO<sub>2</sub> gas concentration were calculated by using equation,

$$s\% = \frac{|R_a - R_g|}{R_a} \times 100. \tag{9}$$

Where,

R<sub>a</sub> – Resistance of CdO thin film in air

R<sub>g</sub> – Resistance of CdO thin film in presence of NO<sub>2</sub>

### 3.6 Gas Sensing Mechanism

Gas sensing mechanism of CdO sensor in the presence of air and oxidising gas is shown in Fig. 7. NO<sub>2</sub> gas sensing mechanism depends on change in the conductance of the CdO nanostructure metal oxide gas sensor, which is well and good agreement with previously recorded results for the oxidising gases on n-type MOS.

Initially there are several negatively charged oxygen such as O<sup>-</sup>, O<sub>2</sub><sup>-</sup>, O<sub>2</sub><sup>2-</sup> adsorb on the surface of CdO thin film and the corresponding reaction is given by,

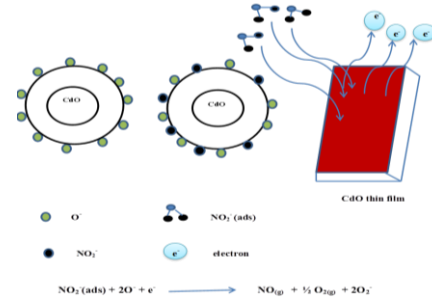
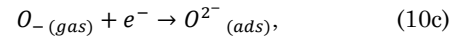
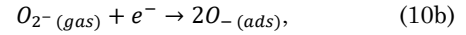
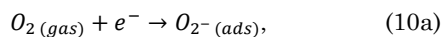


Fig. 7 – Gas sensing response of CdO thin film

Here CdO is N-type semiconducting materials so it contains electrons as majority charge carrier so when oxygen molecules are adsorbed at grain boundaries of CdO, they trap the electrons from the grains of CdO, so concentration of electron in CdO is decreased and so it produce barrier to the electron transport. This mechanism in terms of change in resistance due to adsorb oxygen was described by using schematic diagram as shown in Fig. 8a.

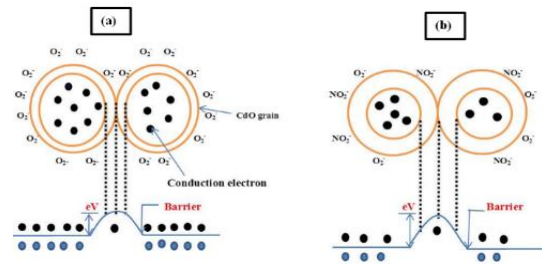
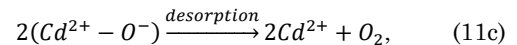
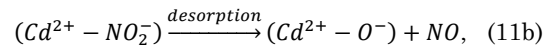
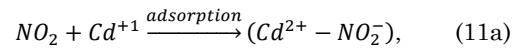


Fig. 8 – Schematic of band structure of CdO (a) when interact with O<sub>2</sub> (b) when interact with NO<sub>2</sub>

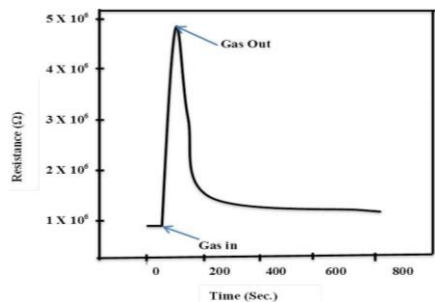
After formation of barrier, the movement of electron takes place when surface of CdO thin film interacts with NO<sub>2</sub> gas, then NO<sub>2</sub> molecules interact directly with the approachable Cd-sites and pullout electrons from the conduction band of CdO. The interaction of NO<sub>2</sub> with CdO takes place by following way



The lack of concentration of electron within the conduction band of CdO results into increase in resistance of CdO sensor shown in Fig. 8b. This change in resistance helps to find response of CdO sensor using equation (9). Plot of electrical resistance of CdO film after interaction with NO<sub>2</sub> is shown in Fig. 9.

This figure showed that in the presence of NO<sub>2</sub> gas resistance of CdO film increases with respect to time and flat region is produced in a few second, which showed fast response of CdO film to the NO<sub>2</sub> gas and to get recovery, in the chamber fresh air was allowed to pass so that NO<sub>2</sub> molecules desorbs and sensor again reach to its starting value of resistance.

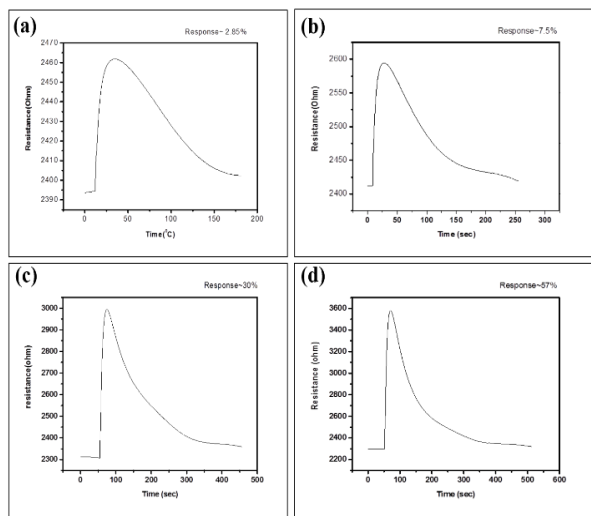




**Fig. 9** – General representation of CdO sensor response to NO<sub>2</sub> gas

**3.7 Response to Various NO<sub>2</sub> Concentration**

All the Fig. 10 (a), (b), (c), (d) showed the common characteristics of CdO sensor that, resistance of sensor increases with injection of NO<sub>2</sub> gas and the value of resistance goes to stable state and then again decreases after injection of fresh air. These results conclude that porous morphology is responsible for increasing resistance of CdO sensor by exposure of NO<sub>2</sub> gas molecules. The reason behind this conclusion is that, porous morphology provides more molecules on the CdO surface [20]. Fig. 10 (a), (b), (c), (d) showed that as concentration of NO<sub>2</sub> gas increase resistance of CdO sensor increase and therefore response of sensor was also increase. The maximum response of sensor is obtained at 100 ppm of NO<sub>2</sub> gas which is 57 % and minimum response is 2.85 % at 25 ppm.

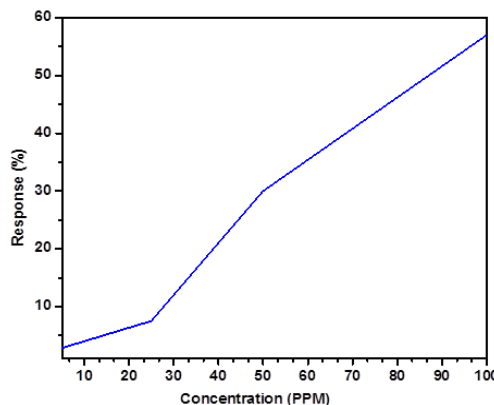


**Fig. 10** –Response of CdO thin film at (a) 25 ppm (b) 50 ppm (c) 75 ppm (d) 100 ppm concentration of NO<sub>2</sub> gas

This variation conclude that, lower or minimum concentration of NO<sub>2</sub> gas contains minimum amount of NO<sub>2</sub> gas molecules and so they covers only small surface of CdO thin film (sensor) and therefore resistance is minimum so corresponding response is also minimum, while at higher concentration of NO<sub>2</sub> gas molecules (i.e here 100 ppm) sensor response is maximum and it conclude that NO<sub>2</sub> gas molecules cover large surface area of CdO sensor. Fig. 10d showed fast response time nearly 75 second while recovery at 400 second for 100 ppm of NO<sub>2</sub> gas.

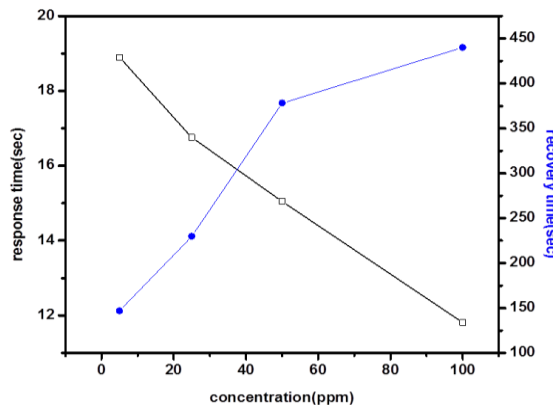
Fig. 11 showed variation of NO<sub>2</sub> gas response of

CdO sensor as function of NO<sub>2</sub> concentration at 200 °C. It concludes that as concentration of gas increases, response of sensor increases due to increasing active sites for adsorption of NO<sub>2</sub> gas molecules.



**Fig. 11** – Response of the CdO gas sensor as a function of the NO<sub>2</sub> concentration at 200 °C

In Fig. 12 it was cleared that, recovery times and response times are completely opposite to each other, they showed inverse relation with each other with respect to various concentration of NO<sub>2</sub> gas.



**Fig. 12** – Variation of response and recovery time of CdO sensor with variation in concentration of NO<sub>2</sub> gas

This figure confirmed that, response time of CdO sensor is decreasing from 19 second to 12 second while recovery time increases from 12 second to 19 second with increase in concentration from 5 ppm – 10 ppm.

**4. CONCLUSION**

CdO thin film was successfully deposited by the Reflux method. Contact angle shows surface is hydrophilic in nature. The XRD analysis confirmed cubic lattice parameters. UV visible spectroscopy showed that CdO has direct band gap of 2.01 eV. SEM morphology showed that CdO thin film is spherical shaped grains spread over surface hence it is porous morphology that provides a large surface area to enhance gas sensitivity of sensor. From gas sensing study of CdO sensor it was concluded that as concentration of NO<sub>2</sub> gas increases, resistance of CdO sensor increase and therefore response of sensor was also increase. The maximum response of sensor is obtained at 100 ppm of

NO<sub>2</sub> gas which is 57 % and minimum response is 2.85 % at 25 ppm.

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## Дослідження концентрації газу NO<sub>2</sub> на реакцію тонких плівок CdO, отриманих за новим методом зворотного флюсу

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У цьому дослідженні, використовуючи простий і недорогий новий метод зворотного холодильника, тонкі плівки оксиду кадмію (CdO) були успішно нанесені на скляну підкладку. Тут для нанесення CdO на скляну підкладку як джерело іонів Cd<sup>+</sup> використовувався хлорид кадмію (CdCl<sub>2</sub>), а як комплексотворювач – аміак. Структурний аналіз і морфологію поверхні підготовленої тонкої плівки CdO аналізували за допомогою рентгенівської дифракції та скануючої електронної мікроскопії відповідно. Також тест на змочуваність проводили за допомогою гоніометра, який показав гідрофільну природу осажденої тонкої плівки CdO. Оптичні властивості тонкої плівки CdO було завершено за допомогою УФ-видимої спектроскопії, яка виявила, що осаждена тонка плівка CdO має пряму ширину забороненої зони близько 2,01 eV. Властивості чутливості газу NO<sub>2</sub>, такі як чутливість, відновлення віддуку та час віддуку підготовленого датчика CdO, було визначено за допомогою датчика газу Keithley. У цій роботі було вивчено вплив концентрації газу NO<sub>2</sub> на відгук датчика CdO, і було з'ясовано, що зі збільшенням концентрації газу NO<sub>2</sub> (25 ppm – 100 ppm), відгук датчика CdO також збільшується і стає максимальним, тобто (57 %) для 100 ppm NO<sub>2</sub> з оптимізованою температурою 200 °C. Було вивчено зміну реакції та часу відновлення залежно від концентрації газу NO<sub>2</sub> і було зроблено висновок, що зі збільшенням концентрації газу NO<sub>2</sub> час віддуку збільшується, а час відновлення зменшується.

**Ключові слова:** Тонка плівка CdO, Флегмовий метод, Газовий датчик.