

Synthesis and Characterization of Nanostructured Copper Oxide Thin Films Prepared by Home-Made DC Magnetron Sputtering for Gas Sensor Applications

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Abstract. In this research, high-quality nanostructured copper oxide (CuO and Cu₂O) thin films were prepared by home-made dc magnetron sputtering technique. The properties and characterizations of the prepared nanostructure films have been determined by the ratios of gases (argon and oxygen) inside the discharge chamber, and heat treatment (annealing) of these films around 400 C°. These parameters were effectively realized important to control the structural characteristics of the prepared nanostructures, especially the energy band gap E_g which was determined by using uv-visible measurements, and particles size. The phase structures of cupric and cuprous were examined by X-ray diffraction to compare between two phases. The type of copper oxide semiconductors two distinct phases: cuprous oxide (Cu₂O), and tenorite (CuO) was determined through Hall effect measurement which was indicated in P-type binary copper oxide. The phases of copper oxide nanostructures were successfully verified for gas sensing applications and they exhibited reasonably high sensitivity with increasing temperature (up to 96% at 350°C). This work can be good attempt to use copper oxide nanostructures in such important application.

Highlights:

1. Nanostructured CuO and Cu₂O thin films were successfully fabricated using a home-made DC magnetron sputtering technique under varying Ar:O₂ gas ratios.
2. XRD and FTIR analyses confirmed the formation of distinct CuO and Cu₂O phases with controlled crystallinity and optical band gaps of 2.33 eV and 2.9 eV, respectively.
3. The prepared films exhibited high gas sensitivity, reaching 96% at 350 °C, particularly toward NO₂, demonstrating strong potential for gas sensor applications.

Keywords: Copper oxide; Nanostructures; Magnetron sputtering; Gas sensing; Reactive sputtering

Introduction

Due to various advantages presented by copper oxide nanostructures, the interests in fabrication and characterization of such structures have recently increased to support their use in existing applications. Copper oxide semiconductors with different morphologies and copper oxidation states have two significant phases: cuprous oxide (Cu_2O) and cupric oxide (CuO) [1,2]. They have unlimited application potential in thin film devices like solar applications and thin film lithium-ion battery [2-3]. Several struggles were prepared to understand the physical properties in theoretical [1,4,5] and experiments calculations [6–9]. The symmetries of Cu_2O , and CuO vary from cubic to monoclinic, resulting in the diversity of optical and electronic properties.

The energy band gap structure of Cu_2O , about (2.1-2.6 eV) [7,10–12], have been established. While Cu_2O have the advantages of good transparency in the visible light range, large resistivity leads to reduced performances [3,10]. The second phases CuO have two opinions for the type of its band gap; in some studies, its band gap is suggested to be direct [16–18], but it is considered that its band gap is indirect in other studies [1,19,20], and its accurate band gap value is still a greater challenge for electronic structure calculations.

Many methods and techniques have been used to prepare copper oxides thin film nanostructures. Such as thermal oxidation [21,22], pulsed laser deposition [21,22], spray-coating [23], electrochemical deposition [26], and reactive sputtering [9,11,12,14]. Among those methods, magnetron sputtering at room temperature is desirable for the growth of thin films with good physical properties. Moreover, one can easily deposit copper oxides films or their mixed phases by merely tuning the oxygen partial pressure during depositions [9,14,15].

The investigation of the thin film nanostructures based gas sensors was taking place over 50 years ago [12], however, there is a continuous need for developing novel materials with improved parameters such as sensitivity, selectivity and stability for gas sensing applications.

In this work, copper oxide thin films including Cu_2O and CuO were prepared by DC magnetron sputtering under different gas mixing ratio (argon-oxygen). The crystal structures of those binary copper oxide films were studied using XRD and FTIR; band gaps were measured by introducing a UV–visible measurements; and a gas sensor was fabricated from copper oxide nanostructures. The characteristics and surface topography of these nanostructures as well as their sensitivity to gases as a function of temperature were studied.

Experiment

The copper oxide thin films had been prepared by DC reactive magnetron sputtering system in a mixture of $\text{Ar}:\text{O}_2$ using pure copper target 99.99%. The glass substrates have been thoroughly cleaned with ethanol and distilled water before placed inside deposition chamber. The total gas pressure inside chamber was 0.05 mbar, however, two phases CuO and Cu_2O samples were prepared at an inter-electrode distance of 2.5 cm and different ratios of $\text{Ar}:\text{O}_2$ have been used (1:1 and 2:1) by using a computerized mixing unit was fixed inside discharge chamber. The cathode, on which the copper target was maintained. The discharge current was maintained at 300 mA and the discharge voltage could be accurately varied (0-2 kV), while the operation discharge voltage was 1 kV. The deposition time was about (5-8 minute). The samples was annealed at $450\text{ }^\circ\text{C}$ and the time was 2 hours, to get more crystallinity and improve the cuprous oxide Cu_2O phase. The characterization measurements included x-ray diffraction (XRD) (Bruker, $1.54.5\text{\AA}$ $\text{CuK}\alpha$ radiation), Fourier-transform infrared (FTIR) measurement (Shimadzu FTIR-8400S). The prepared samples were tested in gas sensing experiments using a homemade setup. The sensitivity to ethanol was measured as a function to the different temperature and different gasses.

Results and Discussion

Figure 1 shows the XRD patterns for pure phase Cu_2O and CuO deposited at 0.2 mbar with the gas mixing rates ($\text{Ar}:\text{O}_2$) of (1:1) and (1:2), respectively. From the figure, one can notice that the peaks of the two samples are consistent with those characteristics of the cuprous oxide, and tenorite (cupric oxide) phases (JCPDS NO. 65-2388, 49-1830, and 65-2309), respectively. Two distinct peaks are observed at 2θ of 38.19° and 38.58° with h k l (111), (111) they are corresponding to the nanostructured cupric oxide CuO . And four peaks at 2θ of $29.51^\circ, 36.48^\circ, 36.55^\circ, 42.43^\circ$, with h k l (110), (111), (111), and (200), respectively, indicates to cuprous oxide Cu_2O . from XRD figures it can see that the increase of the oxygen ratios give rise to the deterioration of the film crystallinity of CuO and this results represents a consistent with previous results [23,24].

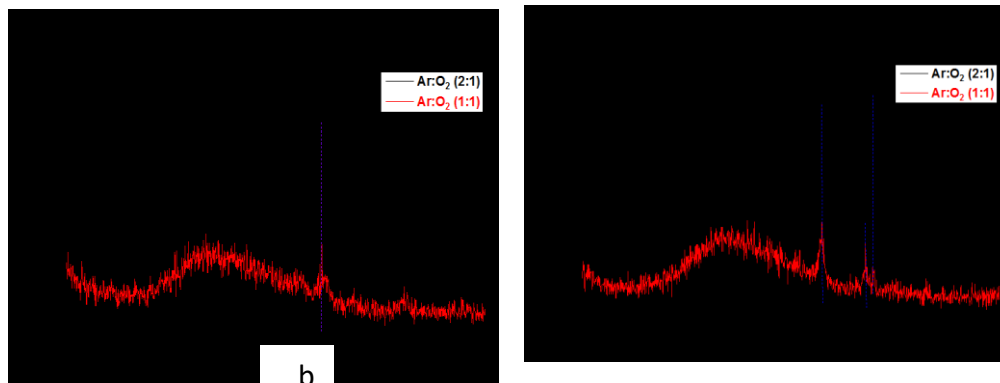


Figure (1): a-The XRD Patterns of CuO films prepared by magnetron sputtering method for a distance of (2.5 cm) between the electrodes at $\text{Ar}:\text{O}_2$ gas mixing ratios without heat treatment. b- The XRD of Cu_2O films prepared by magnetron sputtering method for a distance (2.5 cm) between electrodes at $\text{Ar}:\text{O}_2$ gas mixing ratios after heat treatment at 450°C for 2 h.

Figure (2) shows the FTIR spectra of Cu-O films as a function of R(O₂). The nature of peaks is presented in table 2. The peaks with wave numbers of $\sim 446.17\text{cm}^{-1}$ and $\sim 2376.71\text{ cm}^{-1}$ are characteristics of the stretching vibration of Cu(I)-O and Cu(II)-O bonds respectively [25,26].

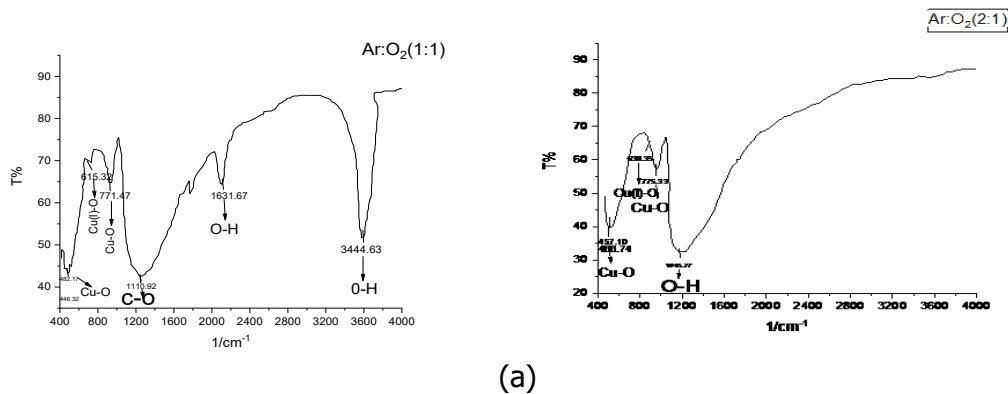


Figure (2-a) FTIR infrared spectrum of CuO films prepared by magnetron sputtering method for a distance of (2.5 cm) between the electrodes at Ar:O₂ gas mixture ratios without heat treatment.

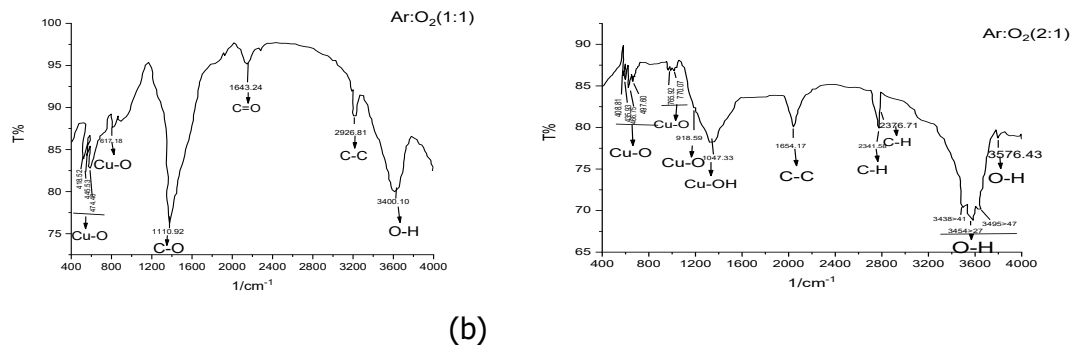


Figure (2-b) FTIR infrared spectrum of Cu₂O films induced by magnetron sputtering for a distance of (2.5 cm) between electrodes at Ar:O₂ gas mixture ratios after heat treatment at

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Table (1) FTIR infrared spectrum of CuO and Cu₂O films prepared by magnetron sput method for sputtering a distance of (2.5 cm) between the electrodes at the mixing ratios of Ar:O₂ gas without and after heat treatment at 450C° for a period of 2h.

sample	Peak of FTIR (cm) ⁻¹	Phase	Assignments Vibration Mode	[Ref.]
B ₁	482.17, 446.32 615 771 1110.92 1647.67 3444.63	Cu ₂ O Cu ₂ O Cu ₂ O Cu ₂ O Hydroxyls of absorbed water Hydroxyls of absorbed water	Cu-O Cu(I)-O Cu-O C-O Stretching vibration of OH- Stretching vibration of OH-	[27] [28,29]] [29] [31] [30] [32] [33]
B ₂	466.74 , 457.10 630.35 775.33 1045.77	Cu ₂ O Cu ₂ O Cu ₂ O Hydroxyls of absorbed water	Cu-O Cu(I)-O Cu-O Stretching vibration of OH-	[27] [34] [29] [35]
B ₃	418.52,445.53,474.46 617.18 1643.24 1110.92 1643.24 2926.81 3400.10	CuO OuO CuO CuO CuO CuO Hydroxyls of absorbed water	Cu-O Cu-O C=O C-O C-O C-C Stretching vibration of OH-	[36] [36] [36] [30] [31] [37] [29]
B ₄	408.81, 435.93 466.75,497.60 765.92,770.07, 918.59 1047.33 1654.17 2341.58, 2376.71 3438.41 3454.27, 3756.43	CuO CuO CuO Cu(OH) CuO CuO Hydroxyls of absorbed water Hydroxyls of absorbed water	Cu-O Cu-O Cu-O Cu-OH C-C C-H Stretching vibration of OH- Stretching vibration of OH	[36] [27] [29] [33] [35] [38] [39] [34] [40]

The optical band gaps of Cu₂O, and CuO also were analyzed. The transmission and reflectance spectra for distinct copper oxides formed under varied total pressures

are present in Figure3. The Tauc relation can be used to determine E_g values from transmission and reflectance:[8,42]

$$(\alpha h\nu)^n = A(h\nu - E_g) \dots\dots (1)$$

Where $h\nu$ denotes incident photon energy and A denotes a material constant. The measured values of n are (2, 1/2, 3, and 3/2), respectively, corresponding to authorized direct, permitted indirect, forbidden direct, and forbidden indirect transitions.

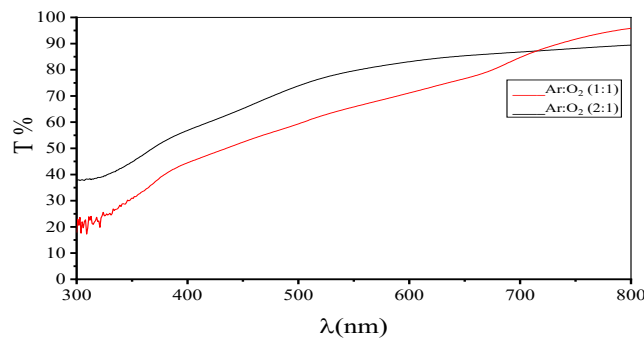


Figure (3) Transmittance as a function of the wavelength of CuO films prepared by magnetron sputtering method for a distance of (2.5 cm) between the electrodes at Ar:O₂ gas mixture ratios without heat treatment.

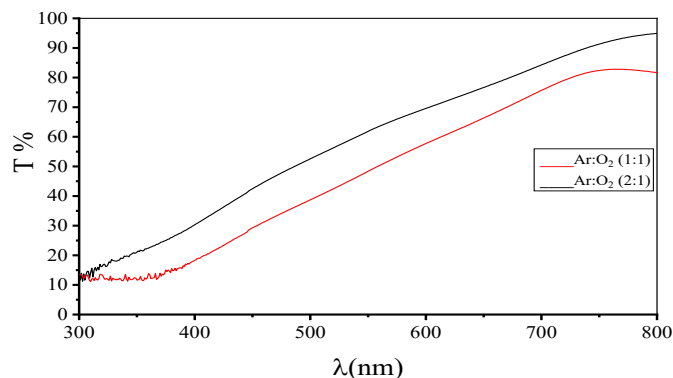


Figure (4) Transmittance as a function of the wavelength of Cu₂O films prepared by magnetron atomization method for a distance (2.5 cm) between electrodes at Ar:O₂ gas mixture ratios after heat treatment at 450C° for 2 h.

For CuO, the indirectly band gap is taken into account, hence $n = 1/2$. Furthermore, because Cu₂O is thought to be a straight transition, $n = 2$ is taken into account [1,43,44]. The absorption coefficient α may be calculated using the following formula:

$$\alpha = \frac{1}{d} \ln \left[\frac{(1-R)^2}{T} \right] \quad (2)$$

where d is the film thickness while R and T are the reflectance and transmission, respectively. The photon energy dependency of the $(\alpha h\nu)^n$ values is shown in Figure 5. For Cu₂O and CuO, the computed optical E_g values are 2.9 ± 2.83 eV, and 2.33 ± 2.21 eV, respectively. These findings are in line with prior findings [25,26,43,45,46, 47,]. Furthermore, although the morphologies of the films under various O₂ partial pressures fluctuate, the band gap value of each type of single-phase copper oxide remains nearly constant, according to the observed results of the band gap. This means that the ratio of Cu₂O /CuO in the mixed phase can be adjusted to modify the band gap of binary copper oxide films.

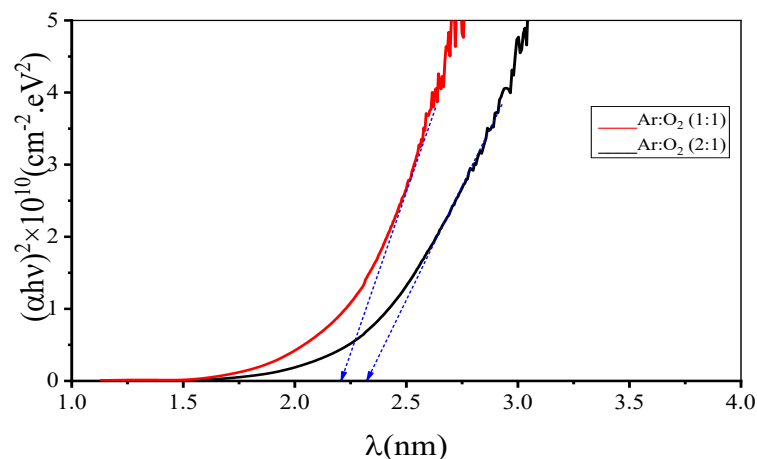


Figure (5-b) The allowed direct optical energy gap for Cu₂O films prepared by magnetron sputtering method for a distance of (2.5 cm) between the electrodes at Ar:O₂ gas mixture ratios after heat treatment at 450C° for 2 h.

In this work, phase copper oxide films CuO and Cu₂O prepared by magnetron sputtering method at a distance of 2.5 cm between the electrodes and at the mixing

ratios of Ar:O₂ gas without and with heat treatment at 450 C° for 2 h for the purpose of testing and using them as sensing elements for gas sensors were studied. Study the degree of gas sensitivity of these membranes when there is NO₂ gas and NH₃ gas, both separately, if it is found that the degree of sensitivity to NO₂ gas is higher than the degree of sensitivity to NH₃ gas, We could define gas sensing as the ratio in changing the conductivity of the sensor when exposing the target gas into sense conductivity in the air this lead to the equation:

$$S\% = \frac{G_g - G_a}{G_a} \quad (1)$$

Where G_a: the conductance of the sensor in air, G_g: the conductance in presence of target gas[48]. Through this study, it was found that the change in the electrical conductivity of the manufactured CuO,Cu₂O models with the operating temperature, where the operating temperature is one of the main factors affecting the responses of the oxide sensors and that the operating temperature for copper oxide is (30,100,200,300) C° and in the presence of NO₂, NH₃ gas Both separately, as the maximum value of the sensors was for the Cu₂O . S_g which is 114.83 at a temperature of 200° C for NO₂ and 16.6 for NH₃ at 200° C. The figer(6) represents the relationship between sensitivity and operating temperature for NO₂ and NH₃ gas

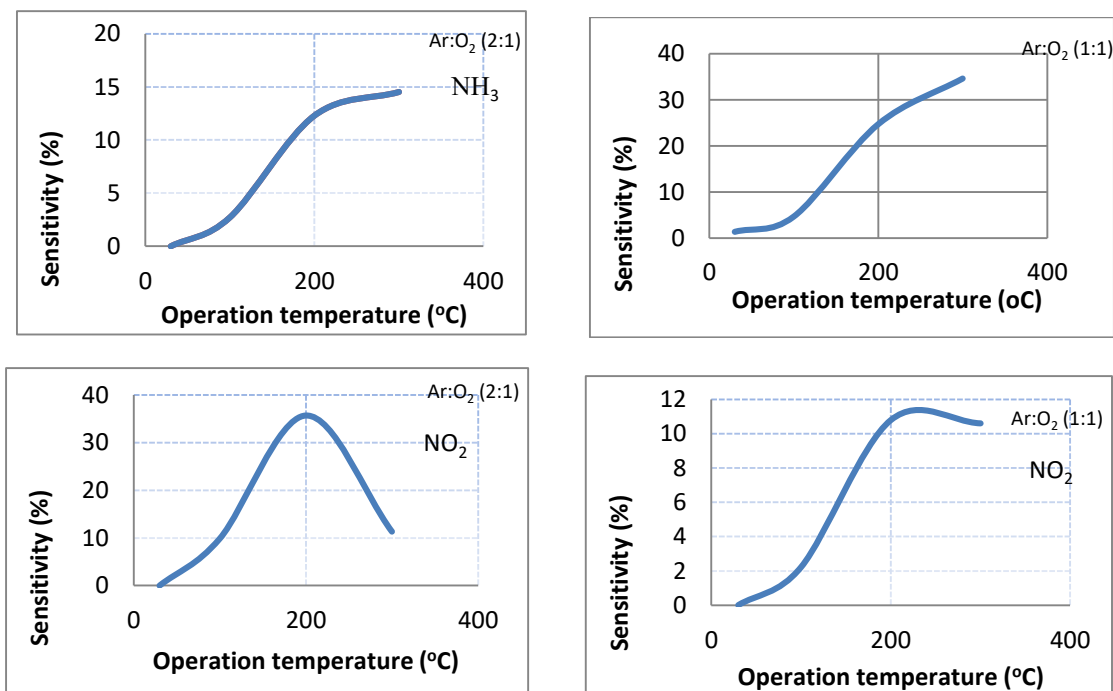


Figure (6-a) Sensor sensitivity as a function of operating time for CuO films prepared by magnetron sputtering method and for distance (2.5cm) at the electrodes at the mixing ratios of Ar:O₂ gas without and with heat treatment of NO₂ and NH₃ gas separately

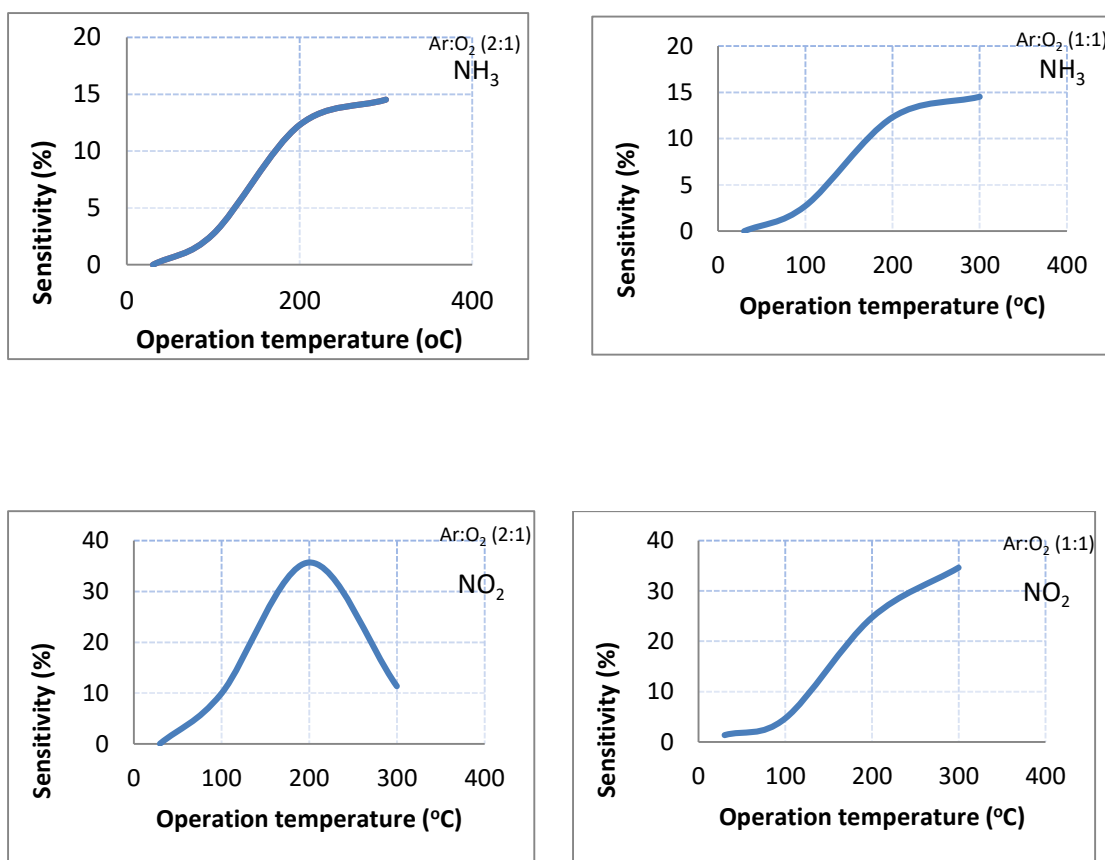


Figure (6-b) Sensor sensitivity as a function of operating time for Cu₂O films prepared by magnetron sputtering method and for distance (2.5cm) at the electrodes at the mixing ratios of Ar:O₂ gas without and with heat treatment of NO₂ and NH₃ gas separately.

Response time, which is defined as the time needed to reach 90% of sensor resistance from the most sense resistance when exposed to the target gas. It was calculated in the samples prepared. As well as, the **recovery time** is defined as a time drop back to 10% of the resistance when the sensor is placed in clean air values in the air. **Figure (7)** shows the response time and recovery time of the sensors prepared from CuO, Cu₂O. CuO is considered a semiconducting material at a temperature of more than 200 °C. We note that there is an increase in conductivity where we can see a decrease in this response where we have the largest sensitivity at 200 °C, this may be due to a uniform increase in these samples.

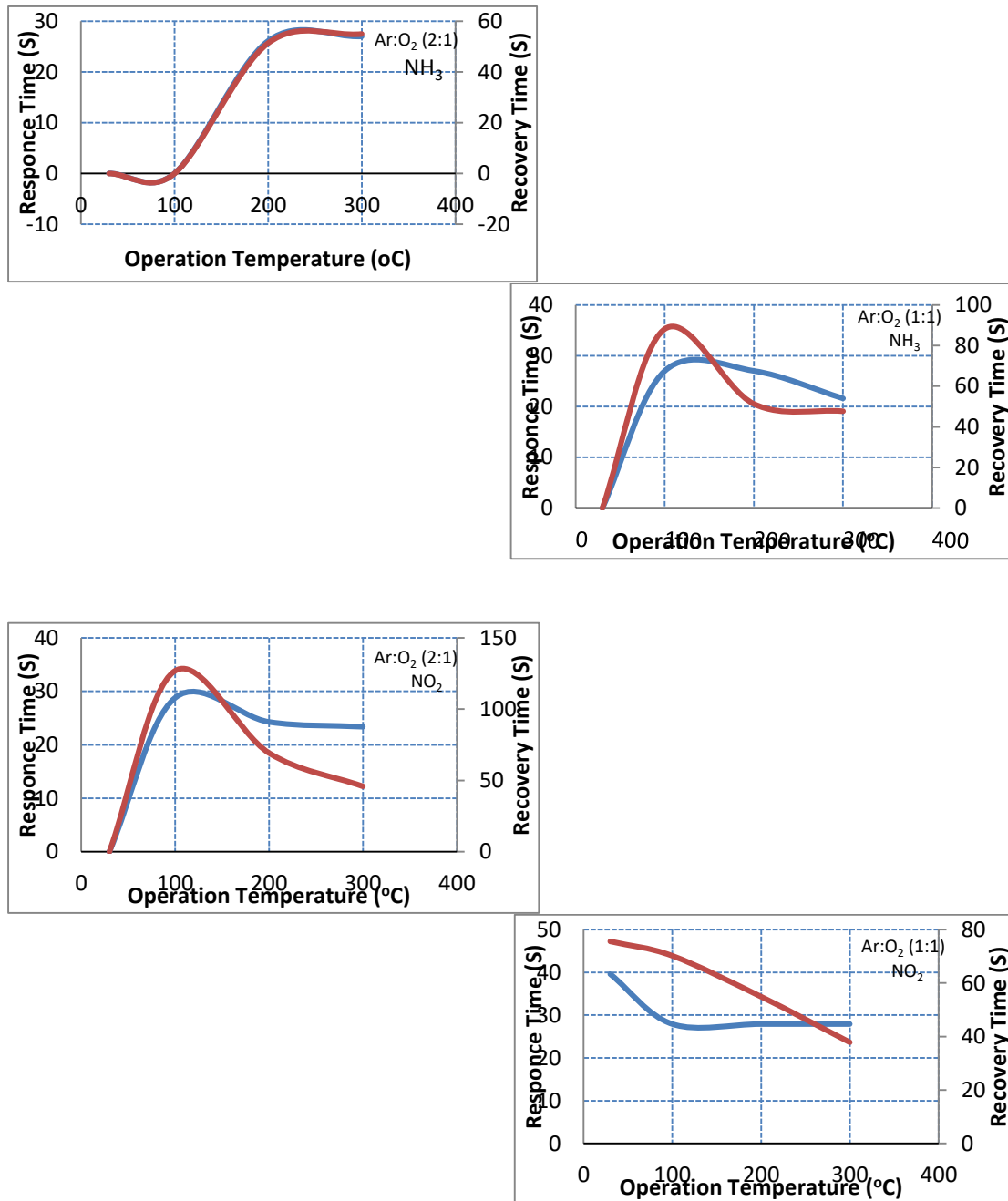
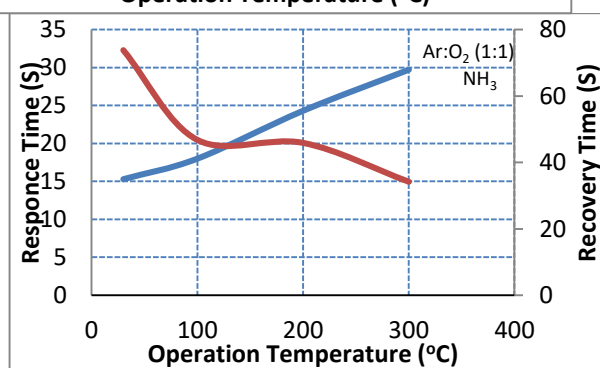
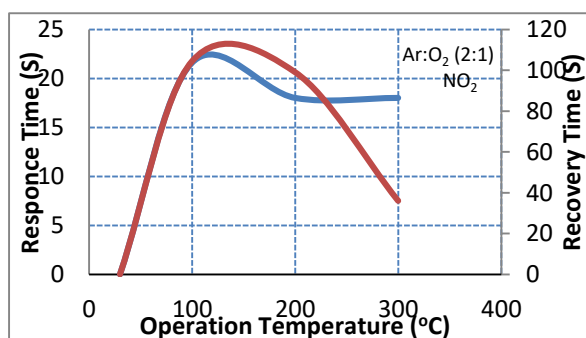
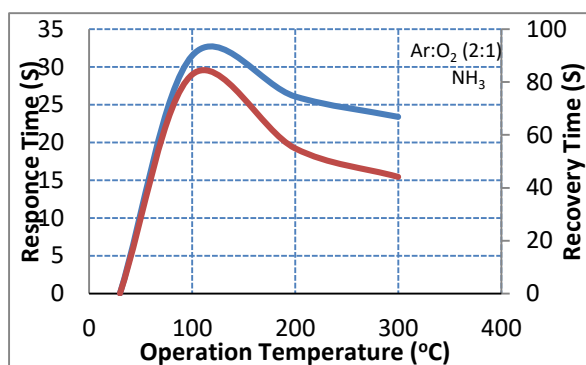


Figure (7-a) Response time and recovery time of CuO films prepared by magnetron sputtering method for a distance of (2.5cm) between electrodes at Ar:O₂ mixing ratios without and with heat treatment of NO₂ and NH₃ gas separately

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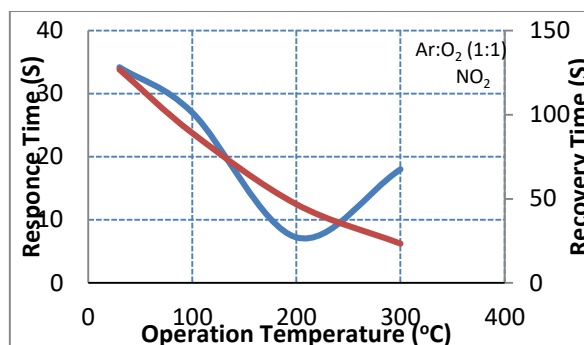


Figure (7-b) Response time and recovery time of Cu_2O films prepared by magnetron sputtering method for a distance (2.5cm) between electrodes at $\text{Ar}:\text{O}_2$ mixing ratios without and with 450°C heat treatment for NO_2 and NH_3 gas separately.

Conclusions

High-quality nanostructured copper oxide thin films can be prepared by reactive DC magnetron sputtering technique. The properties of the prepared structures are sensitively determined by the ratios of gases (argon and oxygen) in the discharge gas mixture. This parameter was effectively seen important to control the structural characteristics of the prepared nanostructures, especially surface roughness and particle size. The reactive dc magnetron sputtering technique can be reliably used to prepare copper oxide nanostructures in two phases to serve some certain applications, such as gas sensing. The prepared nanostructures were successfully tested as a gas sensor and they exhibited reasonably high sensibility with increasing temperature (up to 96% at 350°C). This value can be considered as high and the work can be good attempt to employ copper oxide in different phases nanostructures in such important application.

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