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Araştırma Makalesi Research Article

The Dependence of The Nickel Concentration of ZnO Thin Films for Gas Sensors Applications

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Keywords

Gas Sensor
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Abstract

Undoped ZnO and Ni-doped ZnO thin films were synthesized on glass substrate using the SILAR method. The aim of this work is the analysis of NO gases using $Zn_{1-x}Ni_xO$ sensors concentrations in the range from 100 ppb to 25 ppm. The gas sensing properties of the films for low NO gas concentrations were carefully investigated within a temperature range from 35 to 135ºC. The gas measurement results revealed that the doping process was strongly affected by the response of Ni-doped ZnO thin films. The $Zn_{0.75}Ni_{0.25}O$ sensor exhibited higher sensitivity, faster response, and recovery times for NO gas at low (100 ppb) concentration. It was concluded that the Ni dopant enhanced the properties of ZnO films for gas sensor applications by changing the microstructure, morphology, and bandgap of ZnO material.

Cite

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1. INTRODUCTION

Nowadays, there is two basic ways to improve the properties of metal oxide semiconductor-based (MOS) sensors, which widely used in the development of gas sensors. The first one is the using of thin ZnO and the second is doping with transition or noble metals into ZnO. The first method based on that the response of MOS-based sensors depends on the surface area, grain size, morphology, and structural properties of sensing material (Moon et al., 2010). These properties can be tuned via choosing the suitable synthesis method and conditions, thus the properties of gas sensing material with different structures and morphologies have been studied intensively in (Wang et al., 2016). Also, low-temperature synthesis in an aqueous solution is convenient and is considered to be a chemical alternative with practical advantages (Nwanya et al., 2015). The synthesis of ZnO thin films using the SILAR method in low temperature is preferred in recent years because it is cheaper, simpler, and less time-consuming in semiconductor film synthesis than expensive vacuum equipment. The SILAR is an aqueous solution method involving a series of successive reactions between a substrate and a solution, in which the compound semiconductor to be grown on the underlying material is submerged in a series of aqueous solutions containing ions of each species (Salunkhe & Lokhande, 2008; Yıldırım et al., 2016). It is possible that the number of the SILAR cycles can be determined for the control of the thickness of the material and this is one of the

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most important parameters in the SILAR method. As the number of cycles increases, the thin film thickness increases and a more stable structure is obtained. However, too many SILAR cycles are not suitable because when the thin film thickness reaches a certain value, the ions will now accumulate on the surface in the form of sediment, and as a result, the breaks will become easier and the thin film quality will decrease (Çorlu et al., 2018). The second method, the doped MOS thinsensors have become an attractive subject in the various fields (Chen et al., 2016). The physical and structural properties of MOS sensors have been studied and enhanced by doping with either the noble metals (Pt, Pd, Ag and Au) or by the transition metals (Mn, Al, Cu, and Ni) (Sun et al., 2017). Transition metal-doped MOS-based sensors have intrinsic donor defects which effect the electrical and optical properties of gas sensors (Keskenler et al., 2012). Besides, it also improves the ferromagnetic and sensor properties (sensitivity and selectivity). Thus, transition metal-doped MOS-based nanostructure material is an important role in the gas sensing application and other optoelectronic devices. Nickel (Ni) is one of the transition metals, which shows higher response and better selectivity for the gas at low concentrations (∼0.1-100 ppm). Ni doped ZnO nanostructure material for gas-sensing applications enhances the parameters of sensor such as sensitivity and selectivity. Due to the similarities between the valence of Zn^{2+} and Ni^{2+}, the effective ionic radii of Ni^{2+} (0.69 Å) is closer to that of Zn^{2+} (0.74 Å), thus there becomes the possibility of exchanging Zn^{2+} in ZnO lattice by Ni^{2+}. The exchanging of Zn^{2+} by Ni^{2+} produces high donor defect and makes the charge separation and transport in the ZnO easy (Cheng et al., 2008; Gao et al., 2017; Gu et al., 2017; Wang et al., 2017). So, Ni can be used as the efficient doping transition metal into host ZnO nanostructure. Therefore, Ni doped ZnO nanostructures may be an appropriate candidate for MOS-based gas sensors. In this study, Ni doped ZnO thinfilms were synthesized on glass substrate by the SILAR method at room temperature. The sensitivity changes of gas sensors were observed depending on the concentrations of Ni dopant and gas-sensing properties to NO gas for low gas concentrations were carefully investigated.

2. MATERIAL AND METHOD

Zn1-xNixO thinfilms (pure ZnO, $Zn_{0.75}Ni_{0.25}O$, $Zn_{0.50}Ni_{0.50}O$, $Zn_{0.25}Ni_{0.75}O$ and NiO) were synthesized on glass substrate using the SILAR method. To synthesize thin films, aqueous $[Zn(NH_3)_4]^{2+}$ and $[Ni(NH_3)_4]^{2+}$ complexes were chosen for the cation precursors, in which $ZnCl_2$ (99.9%) of 0.1M, $NiCl_2$ (99.9%) of 0.1M and NH_3 (28%) were used. The obtained complexes were mixed in appropriate proportions according to the composition for $Zn_{1-x}Ni_xO$. Glass substrates were prepared and cleaned to growing $Zn_{1-x}Ni_xO$ thin films. After cleaning, solutions were prepared at the desired doping rate. $Zn_{1-x}Ni_xO$ thin films were growth by the SILAR method. The chemical reactions taking place during the growing process are given below;

$$ZnCl_2 + 2NH_4OH \leftrightarrow Zn(OH)_2 + 2NH_4^+ + 2Cl^-$$

$$Zn(OH)_2 + 4NH_4^+ \leftrightarrow [Zn(NH_3)_4]^{2+} + 2H_2O + 2H^+$$

$$NiCl_2 + 2NH_4OH \leftrightarrow Ni(OH)_2 + 2NH_4^+ + 2Cl^-$$

$$Ni(OH)_2 + 4NH_4^+ \leftrightarrow [Ni(NH_3)_4]^{2+} + 2H_2O + 2H^+$$

When $ZnCl_2$, $NiCl_2$ and NH_3 solutions are mixed, the above reactions take place and $[Zn(NH_3)_4]^{2+}$ and $[Ni(NH_3)_4]^{2+}$ (pH≈10) complex is formed. The glass substrate was kept in a solution of $[Zn(NH_3)_4]^2$and $[Ni(NH_3)_4]^{2+}$ for 20 seconds and the aqueous thin film was coated on the glass substrate surface. The glass substrate was removed from the solution and kept in distilled water at 90°C for 7 seconds. the main goal here is to achieve the $Ni(OH)_2 \rightarrow NiO$ transformation. substrates taken out of hot water were kept in air environment for 60 seconds. It was then kept in pure water at room temperature for 30 seconds. Thus, a SILAR cycle was completed.

After the preparation of $Zn_{1-x}Ni_xO$ thin films by the SILAR method, the morphological and structural analyses were analyzed and investigated with XRD, SEM and UV absorbance analysis. The gas sensing performance of prepared the $Zn_{1-x}Ni_xO$ thin films via a computer-controlled gas measurement system was included a Keithley 2400 sourcemeter, a LakeShore 325 temperature controller, mass flow controllers and

a humidity sensor. The gas measurements of sensors were carried out to NO gas at low concentrations (100 ppb - 25 ppm) and different temperatures range (35 to 135°C) by observing the resistance changes of samples. During all gas measurements, the relative humidity (RH) was kept stable about 25%.

3. RESULTS AND DISCUSSION

Figure 1 shows the SEM results of $Zn_{1-x}Ni_xO$ thin films. All thin films have dense surface morphology and very well covered on the glass substrates. The uniformity and smoothness of the films decreased with increasing Ni doping as seen in Figure 1 (b, c, d). $Zn_{0.75}Ni_{0.25}O$ have nameplates structure and localized clusters on the surface morphology (Figure 1b). Also, $Zn_{0.75}Ni_{0.25}O$ film has interconnected nano-network structure and smoothness very high compared with other films. Due to the increase of concentration of Ni ions in the solution, the nucleation in the ZnO structure becomes easier to the lower activation energy barrier of heterogeneous nucleation. This assists to grow ZnO in a different direction as a result to form nano-network structure morphology (Kumar Rana et al., 2016).

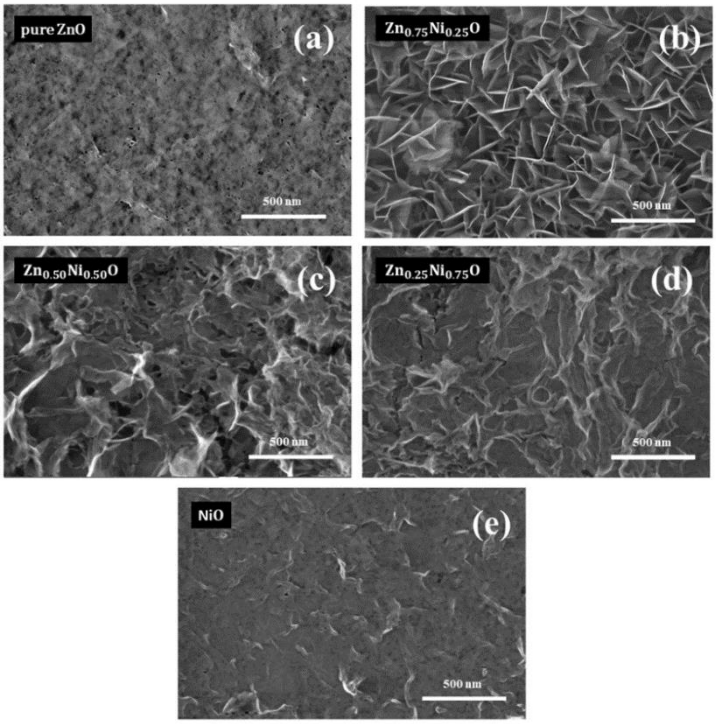

Figure 1. *The SEM Images of* ***(a)*** *Pure ZnO,* ***(b)*** *$Zn_{0.75}Ni_{0.25}O$,* ***(c)*** *$Zn_{0.5}N0i_{0.50}O$,* ***(d)*** *$Zn_{0.25}Ni_{0.75}O$,* ***(e)*** *NiO*

Figure 2a shows XRD patterns of pure ZnO, $Zn_{0.75}Ni_{0.25}O$, $Zn_{0.5}N0i_{0.50}O$, $Zn_{0.25}Ni_{0.75}O$, and NiO thin films. The fims have well-defined peaks belonging to ZnO and NiO, as well as polycrystalline nature. As shown in Figure 2a, ZnO film has a hexagonal wurtzite phase (Çorlu et al., 2017), when the pure NiO film has a cubic phase (Karaduman et al., 2017). The diffraction peaks of pure ZnO corresponding to (100), (002), (101), (102), (110), (103) and (112) are good agreement with the standard JCPDS data (Card No. 36-1451). The intensity of the diffraction peaks of pure ZnO decreased, as well as (100), (002), and (101) peaks start disappeared in high Ni dopant ($Zn_{0.5}N0i_{0.50}O$, $Zn_{0.25}Ni_{0.75}O$), whereas the diffraction peaks of NiO began to appear and the intensity of (200) and (111) peaks increased with increasing Ni doping concentration. This changes of the parameters can be attributed to the replacement of larger Zn^{2+} (0.60 Å) ions with smaller Ni^{2+} ions (0.55 Å). The exchanging of Zn^{2+} by Ni^{2+} produces high donor defect and makes the charge separation and transport in the ZnO easy. Ni doping within ZnO films caused the crystallinity to degenerate (Cheng et al., 2008). Therefore, the change from hexagonal wurtzite structure to cubic structure with increasing Ni dopant was seen from Figure 2a. The bandgap energy values of thin films increased from 3.14 to 3.48 eV according to the doping rate, as shown in Figure 2b. The bandgap energy of pure ZnO was 3.37 eV and NiO was 3,6 eV. So, With the increase of Ni contribution, the rising bandgap can be associated with this difference (Akaltun & Çayır, 2015). Moreover, an increase in the

bandgap energy with the doping element is explained by changes in morphological, crystallinity, atomic distances, and grain sizes of the base material (ZnO, in an actual case).

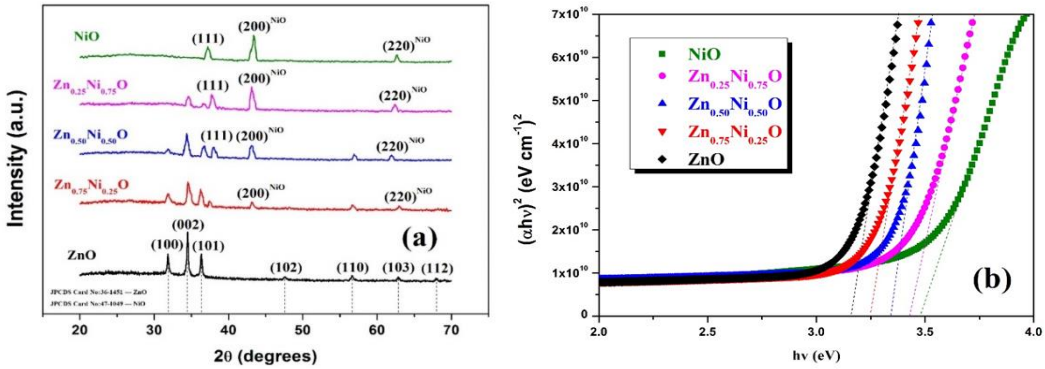

Figure 2. *XRD Patterns* ***(a)*** *and* $(ahv)^2$ *Versus Light (hv) Energy Plots* ***(b)*** *of* $Zn_{1-x}Ni_xO$

Generally, the sensing performance of MOS-based gas sensors depends on operating temperature. The operation temperature is the temperature at which MOS-based gas sensors obtain an optimal response, the response will increase first and decrease next with the increase of the temperature (Wang et al., 2008). Starting from the accepted oxygen adsorption theory, modulating the operating temperature is a possible way to realize the dual gas selectivity of MOS, because during the gas sensing process, the gas diffusion and adsorption, the formation of chemisorbed oxygen, and the surface redox reaction are all closely related with the operating temperature. In order to estimate the optimum operating temperature of the gas sensor, the sensing response of $Zn_{1-x}Ni_xO$ gas sensors for 25 ppm NO was measured at different temperatures range (35-135ºC). The operating temperature of $Zn_{1-x}Ni_xO$ gas sensors was found at 85°C. As seen in Figure 3, the gas sensing responses of $Zn_{1-x}Ni_xO$ gas sensors began to fall when the operating temperature exceeded 85°C. Maximum response has been achieved in the $Zn_{0.75}Ni_{0.25}O$ sensor. While 40% response was achieved in the $Zn_{0.75}Ni_{0.25}O$ sensor, the responses of $Zn_{0.50}Ni_{0.50}O$, $Zn_{0.25}Ni_{0.75}O$ and NiO sensors 31%, 24% and 33% were achieved, respectively. This phenomenon has been attributed the distribution and the adsorption of oxygen species (O^-, O_2^- and O^{2-}) on the sensor surface which depends on the operating temperature (Wang et al., 2008). For operating temperatures less than 50°C, the response of the sensor is very low because the adsorbed target gas is not activated enough to react with the adsorbed oxygens on the sensor surface. An increase in operating temperature by 85°C contributes to overcoming the activation energy barrier of the reaction and an increase in the response of the sensor. However, above 85°C the decrease in target gas adsorption is not compensated by the increase of surface reaction, and the response of the sensor decreases.

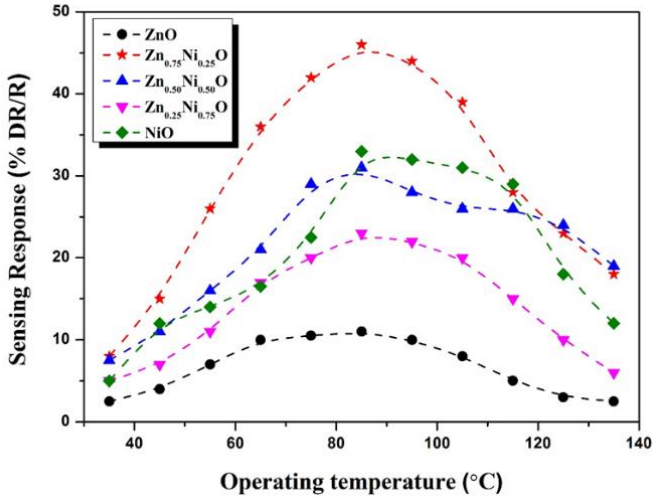

Figure 3. *The Sensing Responses of the Sensors versus Operating Temperature at Constant 25 ppm NO Gas Concentrations*

The dynamic gas measurements were carried out at 85°C. Figure 4 (a-e) shows the sensing responses of $Zn_{1-x}Ni_xO$ sensors as a function of gas concentrations from 100 ppb to 25 ppm NO gas. It is not difficult to deduce that $Zn_{0.75}Ni_{0.25}O$ have the maximum responses. Each measurement exhibits different response when the target gas is exposed, the sensors can distinguish each gas concentration. The sensing responses at 25 ppm NO gas were calculated 14%, 40%, 31%, 24% and 34% for ZnO, $Zn_{0.75}Ni_{0.25}O$, $Zn_{0.50}Ni_{0.50}O$, $Zn_{0.25}Ni_{0.75}O$ and NiO, respectively. ZnO sensor showed no response to 100 ppb NO gas. The responses of 100 ppb NO gas were calculated 7%, 5%, 4% and 5% for $Zn_{0.75}Ni_{0.25}O$, $Zn_{0.50}Ni_{0.50}O$, $Zn_{0.25}Ni_{0.75}O$ and NiO, respectively. For very low gas concentration like 100 ppb, maksimum response was obtained for $Zn_{0.75}Ni_{0.25}O$. As seen in Figure 5, the gas responses increased with increasing NO gas concentrations. It can be seen that Ni content considerably affects the response of sensor. In the absence of Ni doping, the ZnO sensor showed no response to 100 ppb NO gas. With Ni doping of 25%, the response of the sensor increased and exhibited a high response to NO gas. As Ni doping increased to 75%, the sensor response decreased. It can be related to the morphology of NiO.

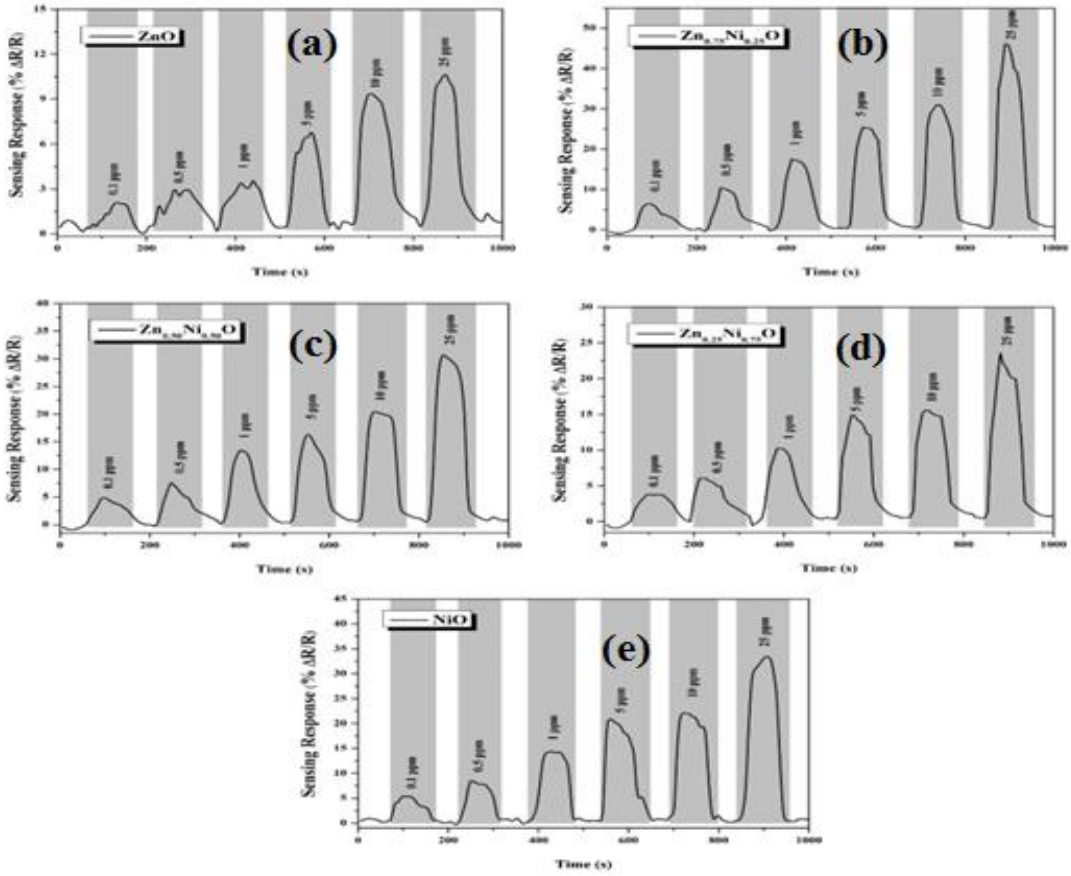

Figure 4. *The Dynamic Sensing Responses of $Zn_{1-x}Ni_xO$ for Different NO Gas Concentrations from 100 ppb to 25 ppm at 85℃*

The response and recovery times were calculated and were shown in Figure 6 (a-b). $Zn_{0.75}Ni_{0.25}O$ sensor has the fast response and recovery times compared to other sensors. The response and recovery times of 100 ppb NO gas were obtained 21 s and 17 s for $Zn_{0.75}Ni_{0.25}O$ sensor. As seen in Figure 6, the recovery time were faster than response time. The response and recovery times of 25 ppm NO gas were obtained 13 s and 10 s for $Zn_{0.75}Ni_{0.25}O$ sensor.

The gas sensing selectivity is another important parameter to evaluate the gas-sensing properties of the gas sensors and greatly desired in practical application. The selectivity of the sensors was analyzed for CO, NO, NH_3, H_2 and CO_2 at 85°C because the operating temperature was found 85°C from the previous measurements. The gas concentrations were taken from 100 ppb to 25 ppm, as shown in Figure 7. The responses were shown differences according to the doping concentrations. The responses of NH_3 and CO

gases increased with increasing Ni concentrations, whereas the response of NO decreased. No response was shown to the H_2 and CO_2 gases. Lin et al. (2017) showed that different nickel concentrations exhibit different responses to target gases. Ganesh Sanker et al. (2017) reported that there was an increase in response up to a certain nickel concentration and a decrease in response above optimal concentration. Shen et al. (2017) showed that optimal nickel concentrations exhibited higher response, have lower detection limit and better selectivity compared to other doping concentrations.

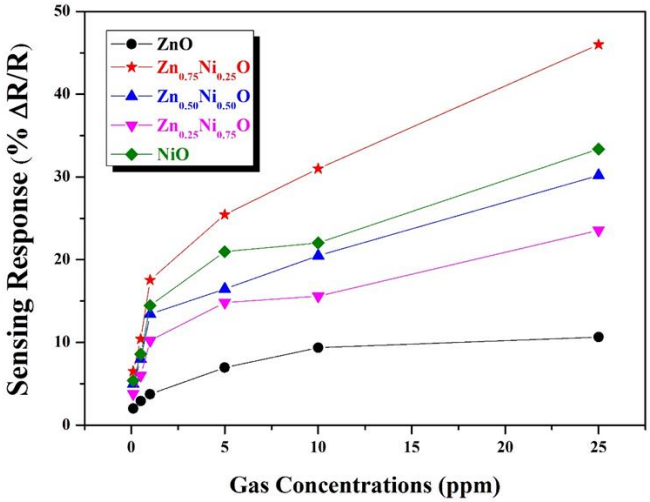

Figure 5. *The Sensing Responses of $Zn_{1-x}Ni_xO$ for Different Gas Concentrations from 100 ppb to 25 ppm at 85 ℃*

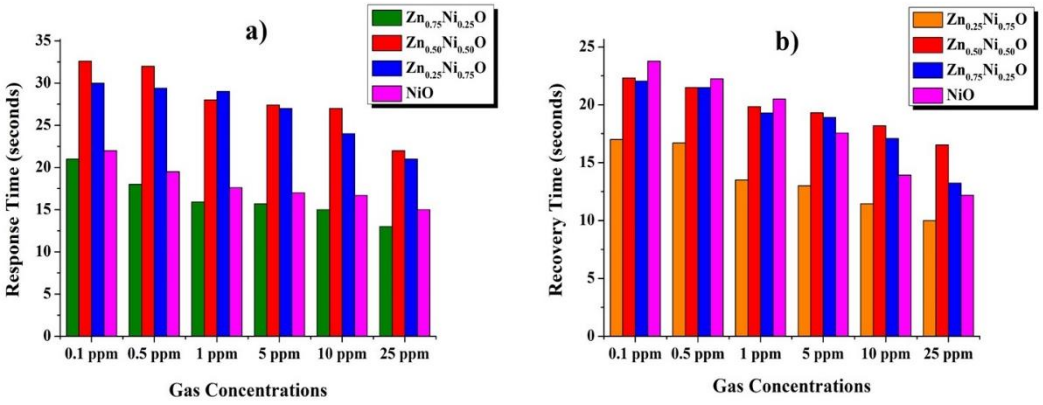

Figure 6. *The Response and Recovery Times of $Zn_{1-x}Ni_xO$ for 20 ppb-25 ppm*

The gas detection mechanism in metal oxide materials is based on the chemical reactions that occur between the surface of the metal oxide and the gas molecules in the atmosphere that cause changes in the electrical conductivity of the metal oxide (Rambu et al., 2013). The chemical absorption of oxygen plays an important role when examining the sensing mechanism in metal oxide gas sensors. Molecular oxygen is captured by the electron in the conductivity band of the metal oxide and clings to the surface. The charge transfer between chemically absorbed oxygen and the target gas changes the conductivity of the metal oxide (Karaduman Er, 2021). During this charge transfer, receiving or giving away electrons; differs according to whether the target gas is oxidizing or reducing and whether the metal oxide used as the sensing layer is p-type or n-type. Reactions may also differ, as the gas sent to the surface is reducing and oxidizing. After the oxygen adsorption reaction on the outer surfaces of the metal oxides sensor, an electron with higher resistance at the sensor surface depletion layer occurs (Karaduman Er, 2021). NO molecules, as a highly reactive species showing an oxidizing character, can be directly chemisorbed on the surface active sites and abstract electrons from the surface. When sensor exposed to NO gas, NO molecules capture electrons from the sensing surface and increasing the resistance.

In addition, nickel doping is a prominent effect on gas sensing. It accelerates the reactions and can drop the operating temperature (Rambu et al., 2013). Rambu et.al. (2013) showed that Ni-doped ZnO films exhibited high response and faster response-recovery times at low operating temperature. They found that the gas sensitivity was depend on the Ni concentration in ZnO films (Rambu et al., 2013). Wang et.al. (2013) reported that gas sensing properties of Ni-doped ZnO nanofibers were effectively improved by Ni doping.

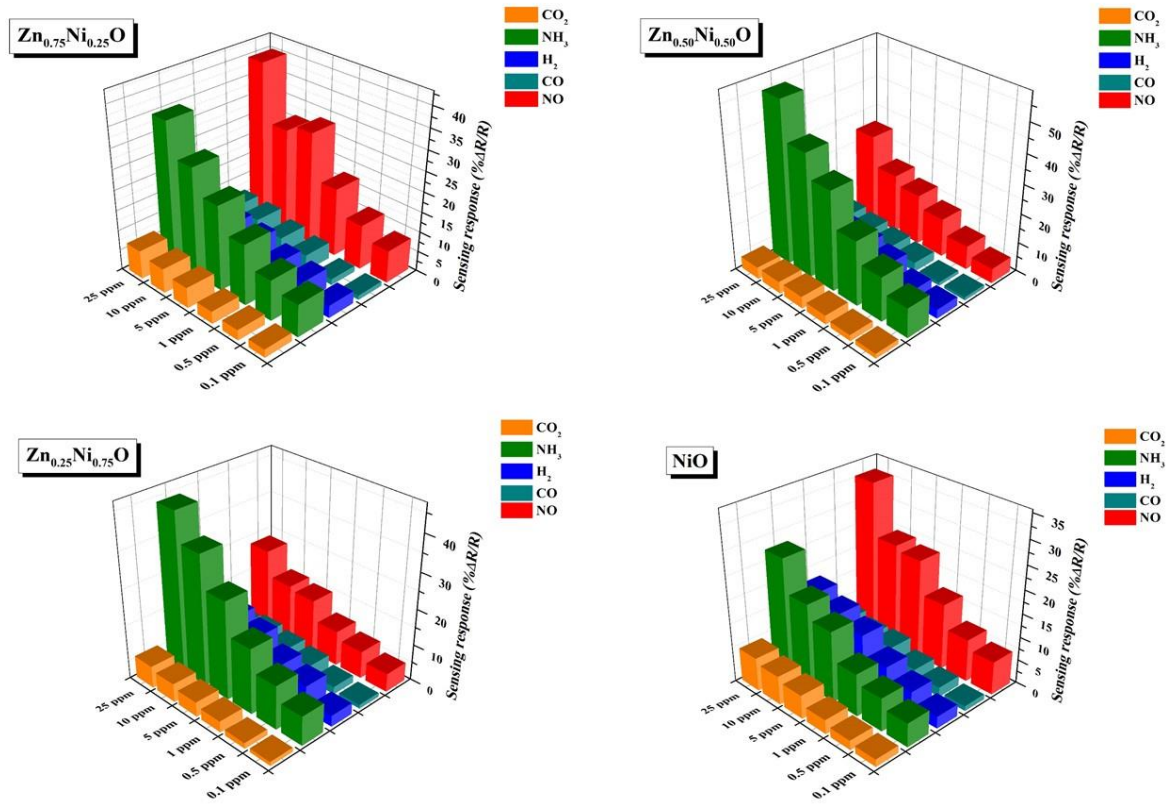

Figure 7. *The Selectivity of the Sensors Exposed to NO, CO, NH_3, H_2 and CO_2 Gases for Different Gas Concentrations at 85 ℃*

4. CONCLUSION

$Zn_{1-x}Ni_xO$ based sensors were synthesized by SILAR method and the gas sensing properties of the films were studied with the effect of operating temperature and doping concentrations. The maksimum sensing response of $Zn_{0.75}Ni_{0.25}O$ sensor to 100 ppb NO gas was 7% at 85°C. The responses began to decrease as concentration increased. It was proposed that the catalytic effect of Ni, the concentration of Ni doping, and also the morphology of thin films affected the response of gas sensors. ZnO based gas sensors have exhibited high gas sensing performance through Ni doping and this modification seems very encouraging to develop high performance, low cost, compact NO gas sensors.

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CONFLICT OF INTEREST

This manuscript has been written by the stated authors who are all aware of its content and approve its submission. There is no conflict of interest exists about this submission.

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