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RESEARCH PAPER

SnO_2:NiO nano Composite Thin Films for Gas Sensing Applications

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ABSTRACT

Thin films of SnO_2 thin films doped with different NiO atomic ratios were deposited by spray pyrolysisfrom chloride solutions. The prepared thin films were measured by different technique tostudy their structural, optical and electrical properties. The X-ray diffraction shows polycrystalline structures of mixed phases SnO_2 and SnO in addition to a newphase of NiOat 20and 30% NiO percentage, with phase ratio depends on the started material ratio. The optical absorbance decreased while the energy bandgap increased from 3.85 to 4eV with increasing the NiO percentage from 10 to 30%. The charge carrier concentration increased while their mobility decreased with increasing the NiO ratio. These variations in some fundamental properties of the deposited thin films by varying components percentage in the started solution show the simply controlling the properties of the prepared thin films for use in gas sensing application. The gas sensitivity against NO_2 gas was exponentially dependent on the gas concentration. The best sensor that prepared with a 30% NiO percentage.

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INTRODUCTION

The principle of resistive gas sensor mechanism depends on the adsorbed oxygen atoms on the surface of the sample, which turns into ions due to the extract of electrons from the conduction band near the surface of the sample forming depletion region [1]. Usually, the change in the resistance of this type of sensor occurs due to the interaction of the target gas molecules with the oxygen ions adsorbed on the surface, and this interaction leads to the return or extract of additional electrons from the semiconductor[2].To obtain a sensor with good specifications, it is necessary to know the mechanism of this reaction and the efficiency of converting this reaction into an electrical signal such as the change of resistance or current proportional to the gas concentration [3].

Tin oxide (SnO_2) is a semiconductor with a wide bandwidth of 3.6 eV and is usually n-type. There are many methods for depositing SnO_2 thin films such as chemical vapour deposition [4], spin coating [5] and spray pyrolysis [6] etc. SnO_2 has attracted a lot of interest in the field of gas sensors and for many gases such ashydrogen [7] and NO_2 [8].

Exposing the n-type semiconductor to an oxidizing gas leads to an increase in oxygen ions adsorbed on the surface of the sample, and more electrons are attracted from the conduction band.This process leads to an increase in the resistance of the sample for two reasons: either because of reducing the concentration of the charge carriers or restrictingtheir mobility [9]. The sensitivity of the sensor device is calculated using the equation $(R_g-R_o)/R_o$, where R_o and R_g are the sample resistance in air and its resistance when

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exposed to a given gas concentration, respectively [10]. SnO_2thin films can be doped with different materials to improve their properties to use as gas sensors [11,12].

In this paper, we study the effects of composite thin formation between SnO_2 and NiO at different percentageconcerning structural, optical, and electrical properties of the SnO_2 thin films prepared by spray pyrolysis. Also, the effects of NiOpercentage on the NO_2 gas sensitivity were studied.

MATERIALS AND METHODS

SnO_2: NiO composite thin films were synthesized

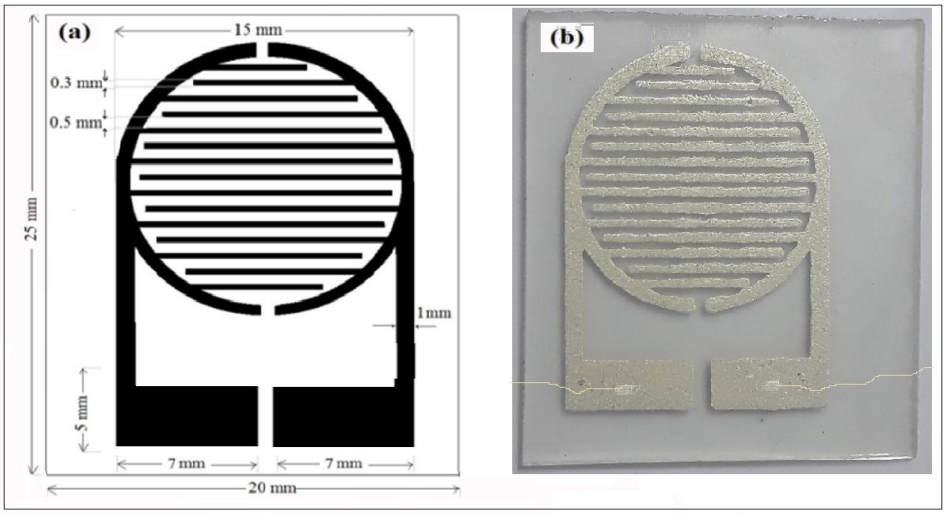

Fig. 1. Gas sensor device and the size of the sensor

Table 1. XRD peaks parameters for SnO_2NiO composite thin film at different atomic ratios.

NiO%	2θ (Deg.)	FWHM (Deg.)	d_{hkl} (Å)	C.S (nm)	hkl	Phase	Card No.
10	26.7574	0.4876	3.3291	16.8	(110)	SnO_2	96-900-7434
	28.5750	0.2659	3.1213	30.8	(112)	SnO	96-110-1036
	30.9690	0.4876	2.8853	16.9	(020)	SnO	96-110-1036
	33.7619	0.8423	2.6527	9.9	(101)	SnO_2	96-900-7434
	38.6384	1.2856	2.3284	6.6	(200)	SnO_2	96-900-7434
	47.8594	0.6206	1.8991	14.0	(115)	SnO	96-110-1036
20	26.5801	0.9520	3.3509	8.6	(110)	SnO_2	96-900-7434
	28.5750	0.5763	3.1213	14.2	(112)	SnO	96-110-1036
	31.1463	0.7536	2.8692	10.9	(020)	SnO	96-110-1036
	33.5845	1.0639	2.6663	7.8	(101)	SnO_2	96-900-7434
	43.3819	0.4877	2.0841	17.5	(200)	NiO	96-101-0382
	47.8151	0.6649	1.9007	13.1	(115)	SnO	96-110-1036
30	26.5801	1.4186	3.3509	5.8	(110)	SnO_2	96-900-7434
	28.5750	0.7093	3.1213	11.6	(112)	SnO	96-110-1036
	33.6289	1.1083	2.6629	7.5	(101)	SnO_2	96-900-7434
	37.3084	1.1083	2.4083	7.6	(111)	NiO	96-101-0382
	43.2489	1.4630	2.0902	5.8	(200)	NiO	96-101-0382
	51.9823	1.3743	1.7577	6.4	(211)	SnO_2	96-900-7434

on glass slides using spray pyrolysis technique from 0.1 M of mixedtin (II) chloride dihydrate ($SnCl_2$:$2H_2O$) with a purity ≥ 99% and Nickel(II) chloride ($NiCl_2$)of 99%purity from Sigma-Aldrich Co. with no further purification at the specific atomic ratios (NiO percentage= 10, 20, and 30%). The mixed salts powder was dissolved in distilled water using a magnetic stirrer at room temperature until entirely added powder was dissolved. Thin films were deposited on glass substrates by spray pyrolysis at a hot plate of the controlled temperature of 400 ◦C. The atomizer is set at a height of 30 cm. The solution is sprayed alternately, of 5 seconds open and 5 seconds closed, using compressed air at 4 bar pressure. The structural, optical and electrical properties were examined by x-ray diffraction (Shimadzu XRD 6000), UV-visible absorption (SP-8001 spectrophotometer), and Hall effect measurements.

Mesh electrodes were deposited onto the samples' surfaces using silver paste utilizing screen printing technique as illustrated in Fig. 1. The gas sensors were examined in a homemade vacuumed chamber on a hot plate of fixed temperature. The NO_2 gas mixed with air at 10 ppm concentration and flow over the sample at a specific time by two flow-meter. The change of sample resistance with time when exposed to the gas was detected using a multimeter connected to a personal computer.

RESULTS AND DISCUSSIONS

Fig. 2 illustrates the X-ray diffraction patterns for the SnO_2: NiO composite thin film deposited on glass substrates. Polycrystalline structures appeared for all samples. The sample deposited with 10% NiO appeared as mixed of twophases, SnO_2 and SnO, identical to standard card No. 96-900-7434 and 96-110-1036, respectively. The

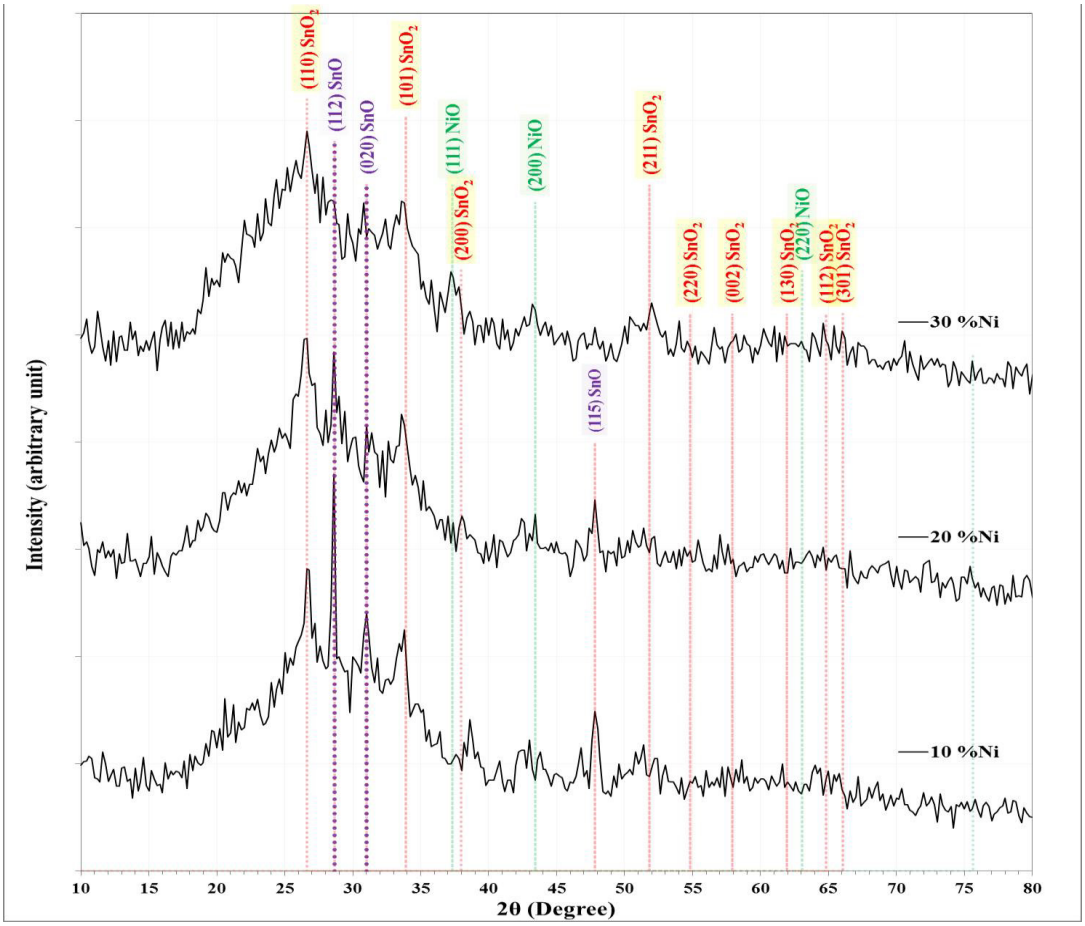

Fig. 2. XRD patterns for SnO_2:NiO composite thin filmsdeposited at different atomic ratios.

SnO_2peaks looked as broad features indicate its Nano size. Increasing the NiO percentage to 30% cause to presenta new phase of NiOcard No.96-101-0382. The peaks broadening was found toincrease with increasing the NiO percentage indicate on reducing the crystalline size, as revealed by Scherrer's formula [13].

Table 1 displaysBragg's angles, interplanarspacing (d_{hkl}) calculated using Bragg's law, full width at half maxima (FWHM), and crystalline size (C.S) for the corresponding Miller indices in the three samples. It seems that the calculated crystallite size,from (110) direction of the SnO_2 structure, reduced from 16.8 nm to 5.8 nm with increasing the NiO ratio from 10 to 30%.

Fig. 3 shows the absorption curves for the SnO_2:NiO composite thin films prepared at different ratios. The absorption edge appeared

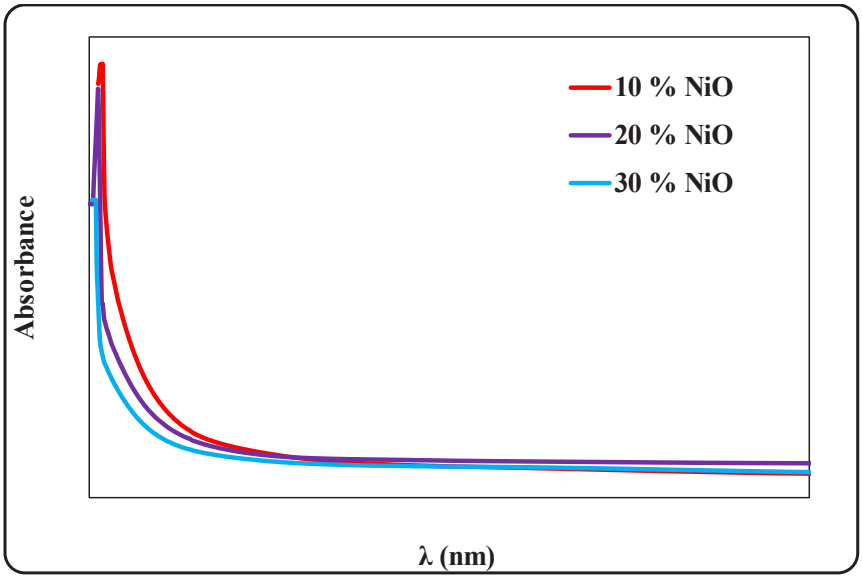

Fig. 3. Absorption curves for the SnO_2 thin films doped with different NiO ratios.

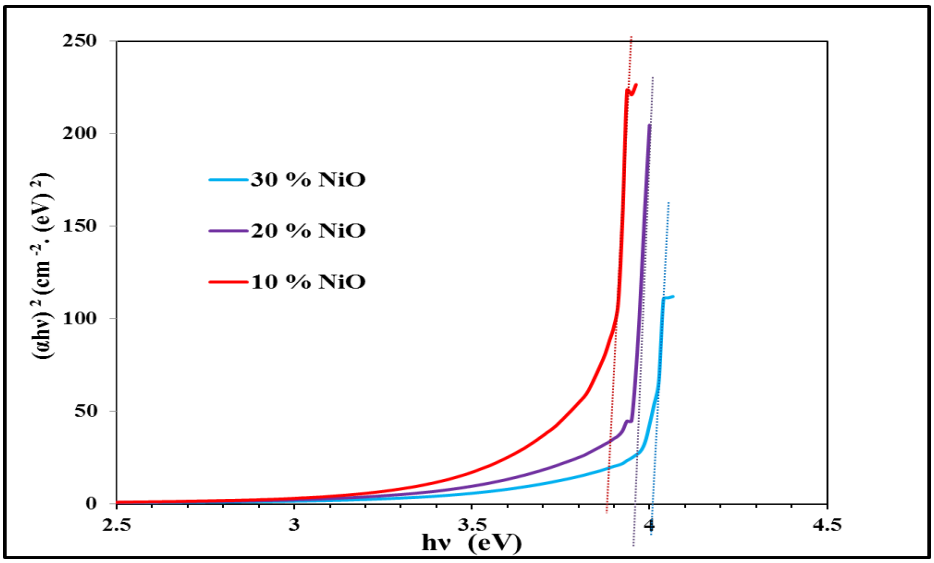

Fig. 4. $(\alpha h\nu)^2$ vs. hν for the SnO_2 thin films doped with different NiO ratios.

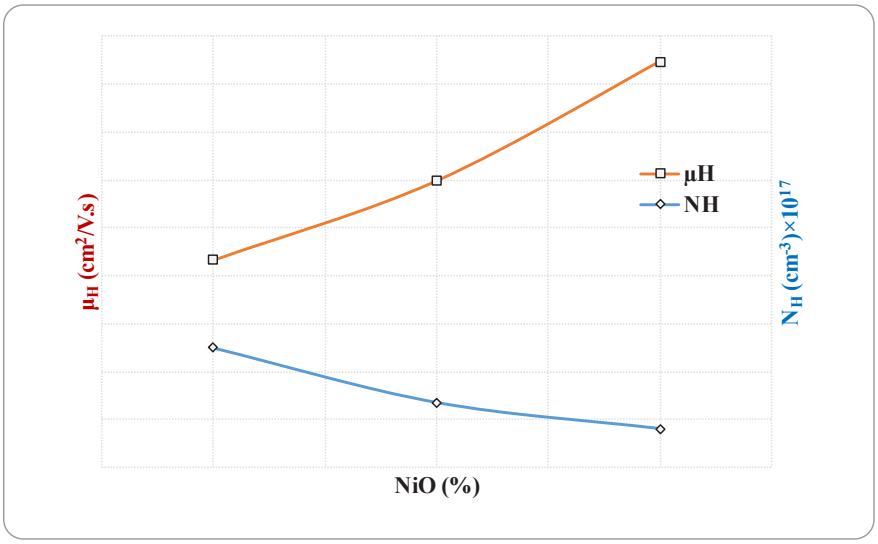

Fig. 5. Variation of charge carrier concentration andmobilitywith NiO percentage.

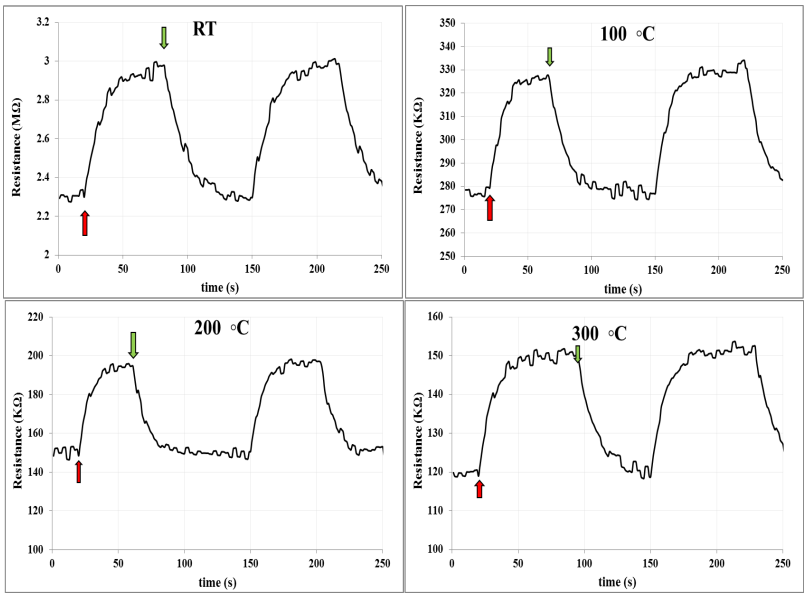

Fig. 6. Resistance fluctuation for SnO_2:NiO thin films sensor at 30% NiO atomic percentage against 10 ppm NO_2gas exposure at different working temperatures.

Table 2. Hall effect parameters for SnO_2:NiO composite thin films at different atomic ratios.

NiO %	σ(Ω cm)$^{-1}$	N_H cm^{-3} ×10^{17}	μ_H cm^2/V.s	Type
10	0.173	2.500	4.33	n
20	0.130	1.359	5.98	n
30	0.110	0.812	8.47	n

at about 350 nm and blue-shifted with increasing the NiO ratio. The optical bandgap was measured using theTauc formula as shown in Fig. 4 which is larger than that for bulk SnO_2, that shown by previous studies, due to the oxygen vacancies [14]. The bandgap increased from3.85 eVto 4eV with increasing the NiO ratio from 10 to 30%. Due to reducing the crystalline size asshown by the XRD measurements, which affected by quantum confinement [15].

The Hall effect is used to investigate the electrical properties of semiconductors such as the concentration of charge carriers (N_H) and their mobility (μ_H). Table 2 and Fig. 5 show the variation of N_H and μ_H, where N_H decreased while μ_H increased with the increase of the percentage of NiO, where the mobility may inversely depend on the concentration of the carriers due to variation

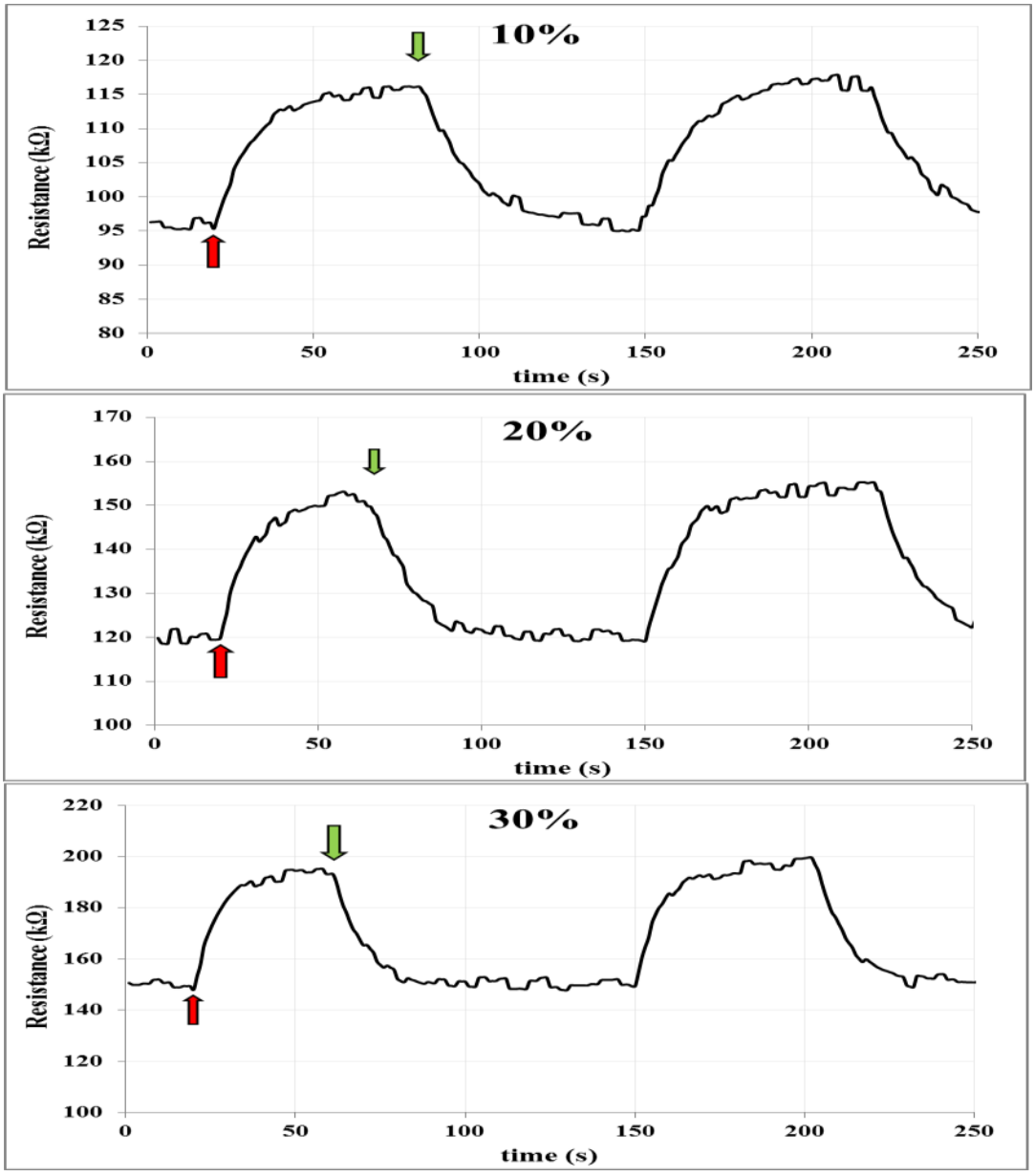

Fig. 7. Resistance fluctuation for SnO_2:NiO thin films sensor at different NiO ratio against 10 ppm NO_2gas exposure at 200 ◦C working temperatures.

Table 3. Sensitivity, response time, and recovery time for SnO_2:NiO thin-film sensor at 30% NiO atomic percentage against 10 ppm NO_2gas exposure at different working temperatures.

Operating temp (◦C)	Sensitivity (%)	response time (s)	recovery time (s)
RT	28.5	44.0	48.0
100	18.0	35.0	39.0
200	30.0	28.0	30.0
300	25.0	38.0	45.0

Table 4. Sensitivity, response time, and recovery time for SnO_2:NiO thin films sensor at different NiO ratio against 10 ppm NO_2gas exposure at 200 ◦C working temperatures.

NiO (%)	Sensitivity (%)	response time (s)	recovery time (s)
10	20.8	43.0	45.0
20	26.7	33.0	38.0
30	30.0	28.0	30.0

of scattering probability [16]. Decreasing N_H value and increasing μ_H may enhance gas sensitivity as shown in Li *et al* [17].

Fig. 6 shows the variation of sample resistance when exposed to NO_2 gas. Electrons can be easily removed from the conduction band of the n-type semiconductor by oxidizing molecules on the sample surface. This process forms oxygen ions on the surface, and a depletion layer of thickness depends on the amount of adsorbed gas. This depletion layer creates an energy barrier in the way of charge carriers in the interparticle contact areas [18] and thus an increase in the resistance of the sample upon exposure to the gas. The gas sensitivity highly dependent on working temperature due to the variation of reactions speed between the target gas and different species of oxygen on the sample surface.The maximum sensitivity appeared at 200 ◦C working temperature[11].

Fig. 7 and shows the variation of the resistance of the samples prepared at different percentages when exposed to 10 ppm NO_2 gas. We note that the sensitivity of the gas depends largely on the properties of thin-film, where the best one was the 30% ratio due to having the smallest crystalline size as indicated by the XRD measurements [19], as well as it may also be due to this sample having the lowest concentration of carriers and greater mobility. The gas sensor specifications were shown in Table 4.

CONCLUSION

In summary, thin films of SnO_2:NiO composite with different ratios of NiO has been successfully deposited with a good specification for gas sensing application by a simple and low-cost method, spray pyrolysis technology. The research showed the possibility of controlling the basic properties of the deposited films easily by changing the percentage of the substance. The XRD showed polycrystalline structures for a mixture of phases whose proportion depends on the percentage of the starting substance component. The energy gap increased, the charge carrier concentration decreased, and their mobility increased with the increase of Ni ion percentage from 10 to 30%. The nitrogen dioxide gas sensors were manufactured based on composite thin films. The best working temperature was 200 °C and the sensitivity increases with an increase of Ni to 30%. The results were good compared to the gas sensor results of the literature on SnO_2 sensitivity to NO_2 gas.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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