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NON-THERMAL PLASMA EFFECT OF LI DOPED NIO THIN FILMS PREPARED BY THE SPRAY PYROLYSIS TECHNIQUE FOR SENSOR APPLICATIONS

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Abstract

In, this research, pure nickel oxide NiO and Lithium-nickel oxide NiO:Li films were prepared on to glass substrates at a temperature of 450°C, by using the spray pyrolysis technique to develop the gas sensor, utilizing nickel and lithium chlorides as precursors. The effect of non-thermal plasma at (3,5,7sec) on the structural, optical, and electrical properties for NiO:Li thin films were studied. XRD pattern shows all films are polycrystalline belonging to cubic structure and the intensity was around the plane of (111).In addition, using SEM images, all samples exhibit porous with nanoparticles in the range (16-33nm) as size. Optical measurements revealed that pure NiO and NiO:Li films have direct energy gaps (Eg) and decrease (from 3.65eV to 3.60eV), respectively. The optical energy gap was reduced after exposure to non-thermal plasma for 3,5,7 seconds (3.50, 3.45, 3.30eV) respectively. The results of Hall effect showed that the electrical conductivity of all films were of the p-type. The gas sensing performance of the prepared films towards NO2 gas was studied at various working temperatures, including (RT, 100 oC, 200 oC, and 300 oC). The current study found that pure NiO and NiO:Li films deposited can detect nitrogen dioxide gas at low work temperatures, where showed pure NiO, NiO:Li films sensitivity towards nitrogen dioxide NO2 gas at an operating temperature RT about 26.1% , 36.8% respectively. After

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exposure to non-thermal plasma at (3,5,7sec), the sensor NiO:Li showed an increase in sensitivity, and the best sensitivity was towards NO2 gas at room temperature for the sample exposed to non-thermal plasma at (7 sec) (49.9 percent). The same sample showed a maximum sensitivity towards NO2 gas at 200 oC about (80.2%) with moderate response and recovery time. The influence of non-thermal plasma leads to good sensing performance for NiO:Li films, as shown in the study.

Keywords: Non-Thermal Plasma; Nio Doped Li; XRD; Spray Pyrolysis Technique, NO2 Gas.

Introduction

In recent decades, plasma physics and plasma-based technology applications have made significant development [1]. It's a crucial part of a lot of today's materials processing. These include sputtering, etching, nitriding, and photolithography, among many other things . Plasma research will have a significant impact on the development of deposition technologies, since technology needs finer scales. These processes have many implications for future development in fusion research, thin film solar technology, materials processing and semiconductor fabrication [2]. Scientists have shown a great interest in non-thermal plasma because its advantages such as low temperature, scalable size, low operation cost, flexible operation, and high electron [3]. Plasma is a gas in an ionized state that contains positive charged particles (ions), negative charged particles (electrons), and neutral charged particles. The motion of charged particles in the plasma generates electric and magnetic fields. The main process in plasma is ionization [4]. The word "plasma" has Greek origins and meaning "something molded" or "jelly" [5]. Plasma was initially used in 1929 by Tonks and Langmuir to describe the inner region of highly ionized gases formed by electric discharge in some tube, where the ionized gas remained electrically neutral [6]. Air quality is a serious problem for people all around the world. Our health and the ecosystem both require clean air. Gas sensors are needed to detect varying amounts of harmful and flammable gases in order to reduce pollution and control disasters[7]. Due to its low cost, ease of fabrication, small size, and simple sensing idea, semiconductor metal oxide gas sensors have aroused interest.. [8]. Nickel oxide (NiO), Due to its unusual physicochemical properties, In a variety of cutting-edge research disciplines, one of the few p- type metal oxides stands out as one of the most promising prospects. [9]. nickel oxide has a wide band gap of (3.6-4.3eV) and has good chemical stability, durability, low toxicity, high ionization energy, and low manufacturing costs, in addition to fascinating electrical, optical, and magnetic characteristics[10]. NiO has been investigated as a potential gas sensing material. It's a model semiconductor since it can be manufactured using a range of physical and chemical methods [11]. Sputtering [12], thermal evaporation [13], the sol–gel spin coating method [14], and spray pyrolysis method [15]. Among these approaches, spray pyrolysis was chosen in this study because it allows for a large production area and good adhesion of thin films to glass substrates. [16].

Experimental work

Chemical Spray Pyrolysis technique was used to prepared nickel oxide thin films using a solution of nickel chloride ($NiCl_2.6H_2O$) salts, which is a green solid material with a molecular weight of (237.69g/mol) and the solution was made in (0.1M), where the (1.1885gm) nickel chloride was dissolved in 50 ml distilled water. This solution was on magnetic stirrer for (15minute), to ensure complete dissolution of the substance, as a homogenous green solution was obtained. Glass bases were used after cleaning and drying, and their dimensions were (2.5*2.5) cm. The solution was then sprayed on the bases, which had been heated to ($450^{\circ}C$). The thin films are left on the surface of the heater after the

deposition procedure is completed to allow the prepared films to complete the oxidation and crystal formation process. The mass of salts was calculated by the equation [17]:

$$W = \frac{Mw \times M \times V}{1000} \quad \text{....(1)}$$

Where:

W: mass of salt, M_w: molecular weight of salt, M: solution molarity, V: volume of distilled water.

In order to prepared lithium-infused nickel oxide films Lithium molar percentages of one percent were used to create doping samples. The spray solution contained lithium chloride (LiCl) as a lithium source.It is a white crystals with a molecular weight of (42.39g /mol) with concentration of molarities (1%) , distilled water was used as the solvent. To prepare the denaturation solution, a mass of lithium chloride) 0.0021g) was added to the previous nickel chloride solution and then the solution was placed on the magnetic stirrer for a period (15) minute to obtain a well-homogeneous solution, then the solution was sprayed on heated glass bases at the temperature ($450^{\circ}C$). Due to the heat the NiO:Li films remain on the base. When the deposition process is completed, the thin films is left on the surface of the heater after it is extinguished until it cools down without attempting to lift it for two reasons: first, to ensure that the thin films are completely oxidized (the reaction is complete), and second, to avoid film breakage when attempting to lift it abruptly from the heater's surface due to temperature differences (rapid cooling). Then the doped samples were exposed to non–thermal plasma at (3,5,7sec). The films prepared were of good consistency, strong adhesion to the base and free of cracks and. The optimum conditions for preparing films were pure NiO and NiO: Li on the base by Spray pyrolysis technique as follows:

Table.1 Deposition Parameters applied in study

Substrate temperature	450± 5˚C
Pyrolysis rate	2 ml/min
Distance between Nozzle to substrate	(29 ± 1)cm
Pyrolysis Time	5 sec
Stop Time	55 sec
Pressure of Carrier Gas	3.5 bar

Thickness Measurement

Film thickness measurement is important since it affects the physical properties of the film. The thickness of the films was measured by the optical interference method, in which a (He-Ne) laser with wavelength (632.8 nm) was used. The thickness of the film is estimated by using the following equation [18] and the interference of the cilia created as a result of the laser beam reflection in front and behind the film:

$$t = \frac{\Delta x}{x} \cdot \frac{\lambda}{2} \quad \text{.... (2)}$$

Where:

x: is fringe width, Δx : is the distance between two fringes, λ: is wavelength (632.8nm)

Table.2 Values of thickness films

Samples	Thickness (nm)
Pure NiO	120
NiO:1%Li	155
NiO:1%Li after exposure (3sec)	160
NiO:1%Li after exposure (5sec)	170
NiO:1%Li after exposure (7sec)	185

Result and Discussion

1- Structural properties

❖ X-Ray Diffraction

Diffraction of X-rays the purpose of the analysis was to determine the structures of pure NiO and NiO:Li thin films, as well as the position of the peaks that emerge in all films prepared at 450°C.These films belonging to cubic structure, polycrystalline with a dominant sharp peak along (111) plane. This is consistent with researches [19,20]. Where the peaks correspond to (111), (200), (220), (311), (222), at 2θ= (37.37°), (43.58°) , (62.85°), (75°), (79.56°) respectively, which is fairly consistent with JCPDS Card(No:96-432-0488). The height of the crest represents the number of atoms scattered over specified dimensions, and the diffraction values indicate the locations of the atoms at which the reflections occur. From those peaks, we notice the displacement of the angles of nickel oxide from (37.37°) to(37.48°), and from(79.56°) to(79. 67°).The reason for this is due to the entry of impurities in to the crystal structure and their movement of some molecules from their original position, where the radius of the lithium atom is 152pm which is greater than the radius of the nickel atom is 124pm, this causes the angle to increase when doping. The XRD patterns of the pure nickel oxide and Li doped films are shown in figure 1. In the (200) plane, bulk NiO has the highest intensity. (JCPDS Card No:96-432-0488). The (111) plane, on the other hand, has the highest intensity in our case, indicating that the film has a favorable minimum surface free energy for oriented development in the (111) direction.on doping with Li [21]. Berzin et al. [22] found that (111) NiO thin films might be employed as buffer layers for depositing other oriented oxide thin films including perovskite ferromagnetic films and superconducting thin films. Because of their chemical stability and symmetry of the oxygen ion lattice and lattice constant, NiO and oriented oxide thin films have a lot in common. The X-ray diffraction spectra did not show the birth of new phases attributed to the diffraction peaks during exposure of the doped films to non-thermal plasma at (3, 5 ,7)sec. The NiO:Li films following exposure to non-thermal plasma at (3,5,7sec) were found to be polycrystalline with a cubic structure, according to X-ray diffraction (XRD) data. The higher intensity was found in the plane of (111), also is seen

from figure 2. that the intensity of the peaks increase after non-thermal plasma exposure at (3 sec) less intense of peaks indicates that increasing of localized state in the optical band gap consequently the energy gap decreased. The dimension (D) of NiO:Li films decreases following exposure to non-thermal plasma, while the full width half maximum (FWHM) increases. This is consistent with research [18].

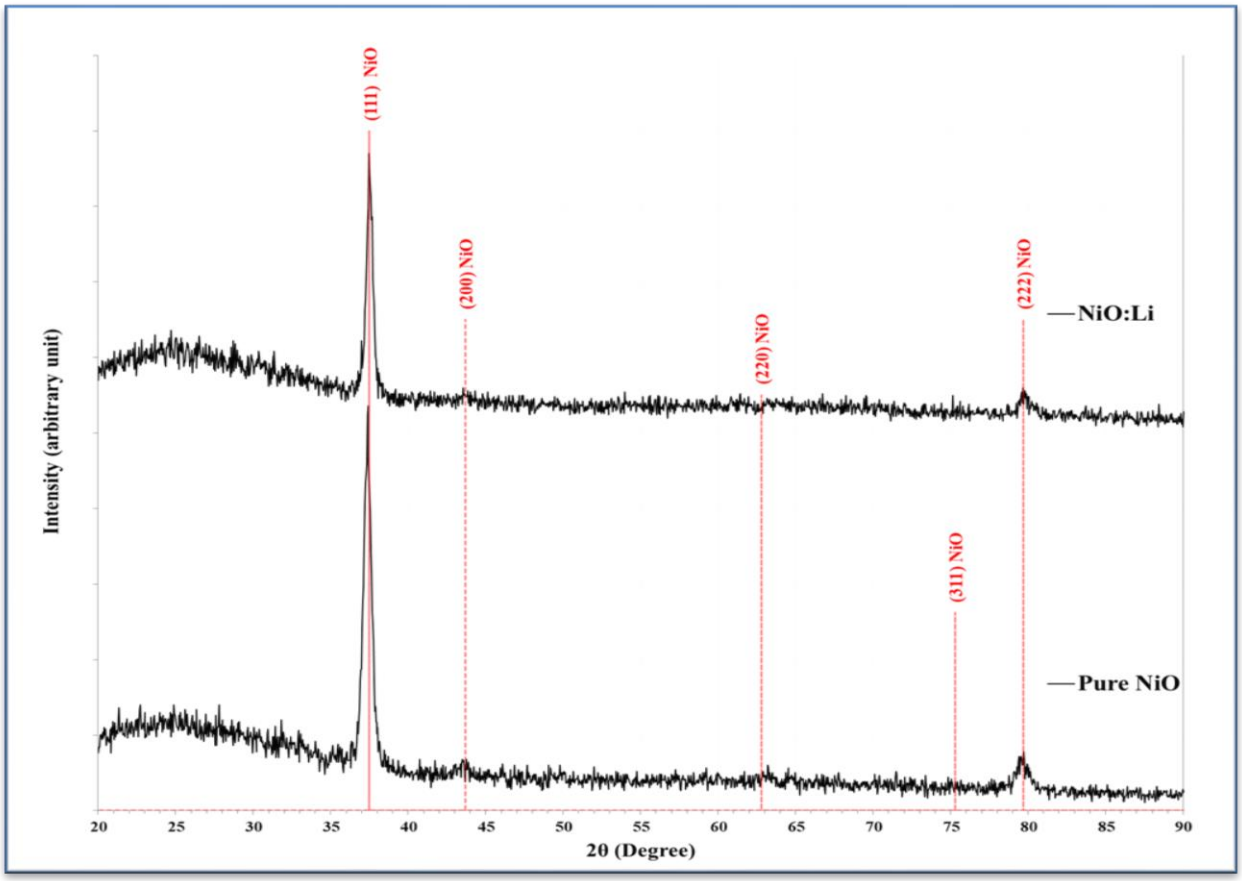

Figure. 1 X-ray diffraction patterns for pure NiO and NiO: 1%Li film

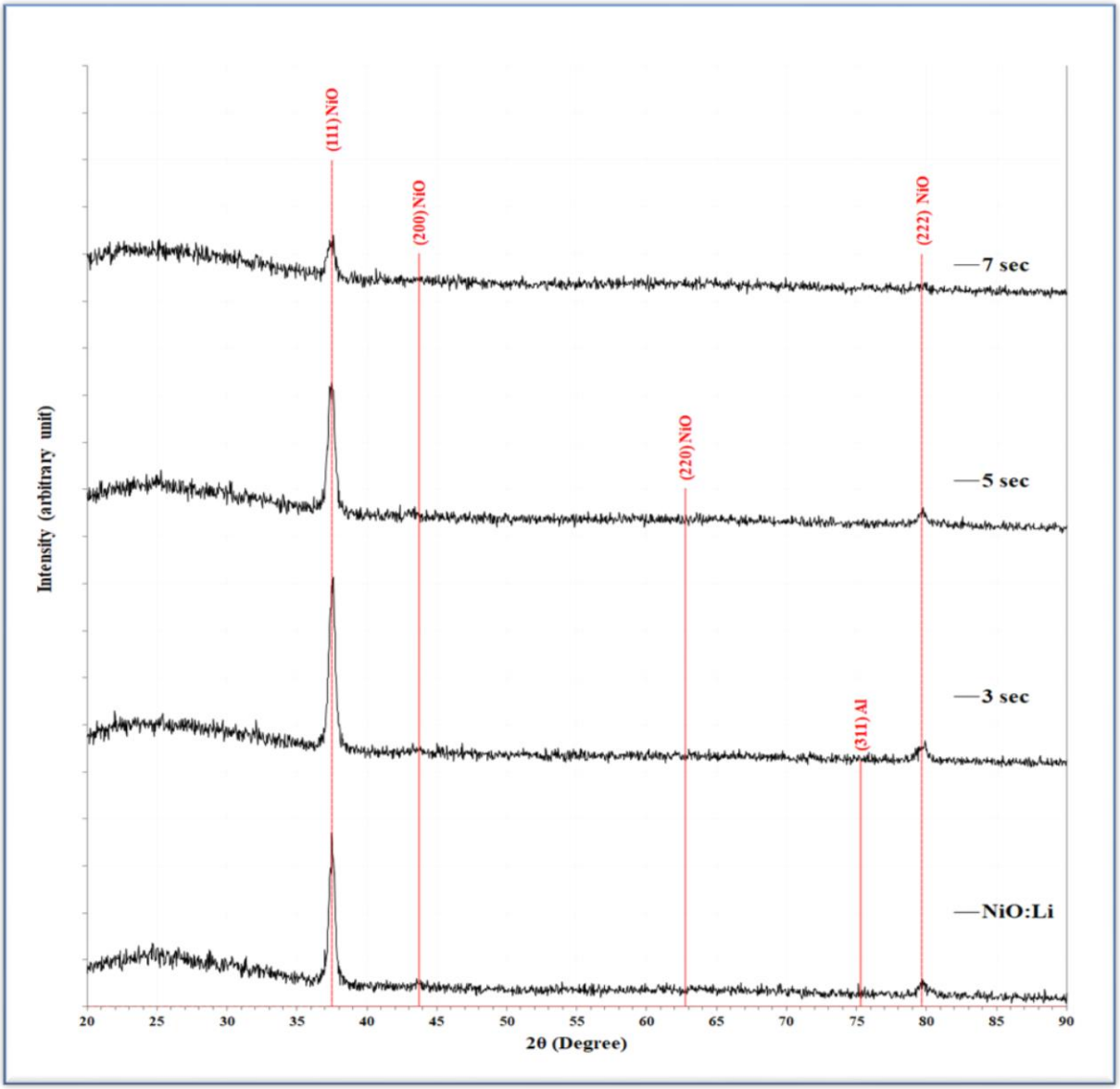

Figure. 2 XRD diffraction patterns for NiO: 1% Li films before and after to non-thermal plasma at (3,5,7) sec.

Table.3 The structural parameters for pure NiO thin film

Sample	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	C.S (nm)	d_{hkl} Std.(Å)	Phase	hkl	card No.
NiO Pure	37.3779	0.5590	2.4040	15.0	2.4148	Cub. NiO	(111)	96-432-0488
	43.5892	0.7328	2.0747	11.7	2.0913	Cub. NiO	(200)	96-432-0488
	79.5663	0.8723	1.2038	11.9	1.2074	Cub. NiO	(222)	96-432-0488
NiO:Li 1%	37.4826	0.4570	2.3975	18.4	2.4148	Cub. NiO	(111)	96-432-0488
	79.6710	0.6979	1.2025	14.8	1.2074	Cub. NiO	(222)	96-432-0488

Table. 4 The structural parameters of NiO:Li thin films before and after to non-thermal plasma exposure

Exposure time (s)	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	C.S (nm)	d_{hkl} Std.(Å)	Phase	Hkl	card No.
0	37.4826	0.4570	2.3975	18.4	2.4148	Cub. NiO	(111)	96-432-0488
	79.6710	0.6979	1.2025	14.8	1.2074	Cub. NiO	(222)	96-432-0488
3	37.5940	0.5000	2.3906	16.8	2.4148	Cub. NiO	(111)	96-432-0488
	79.7948	0.7522	1.2009	13.8	1.2074	Cub. NiO	(222)	96-432-0488
5	37.5104	0.6250	2.3958	13.4	2.4148	Cub. NiO	(111)	96-432-0488
	79.8120	0.6686	1.2007	15.5	1.2074	Cub. NiO	(222)	96-432-0488
7	37.4269	0.7970	2.4009	10.5	2.4148	Cub. NiO	(111)	96-432-0488
	79.8448	0.6269	1.2003	16.5	1.2074	Cub. NiO	(222)	96-432-0488

❖ **Surface Morphology**

Figure.3 showed surface morphology of pure NiO and Li doped thin films using SEM images. NiO:Li films are made up of small grains, as may be seen. Nanoparticles with diameters ranging from 16 to 33 nm were created. The porous nature of the nanostructures is also noted. These types of porous nanostructures are expected to play a key role in the performance of the sensor because they control the rate of gas diffusion due to the big surface area. Grain sizes grow as a result of exposure to non-thermal plasma. Also, increased roughness and some pores with increasing to non-thermal plasma exposure.

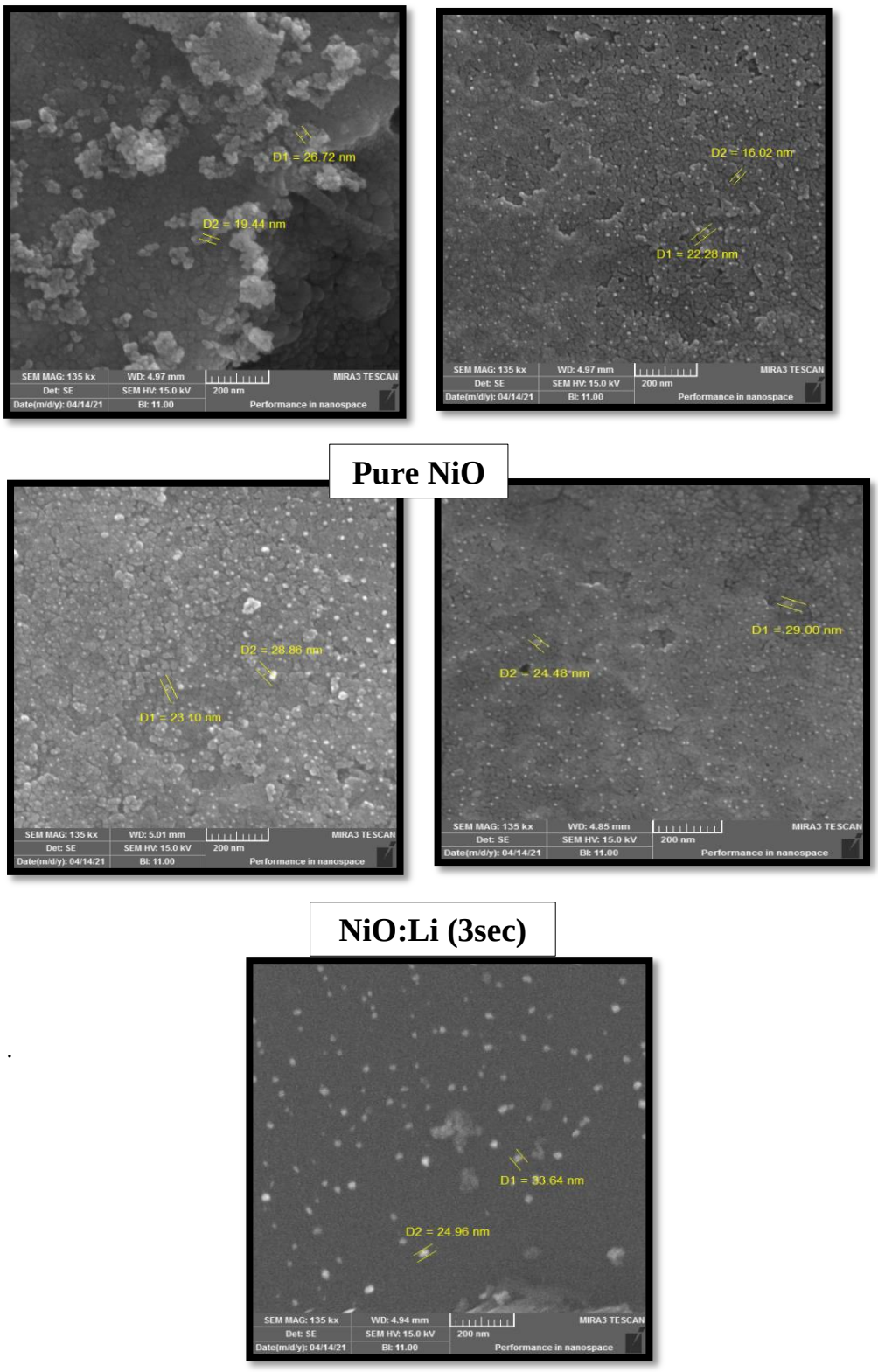

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Figure.3 SEM image for films (a) pure NiO ,(b) NiO:L ,
(c) NiO:Li after to non-thermal plasma at (3sec) exposure ,
(d) NiO:Li after to non-thermal plasma at (5sec) exposure,
(e) NiO:Li after to non-thermal plasma at (7sec) exposure.

2- Optical properties

Optical band gap

The energy required to shift an electron from the valance band to the conduction band is known as the energy gap. The energy gap is affected by ambient conditions such as substrate temperature and the method used to prepare thin films [23]. Optical measurements revealed that the energy gap (E_g) of pure NiO and NiO:Li films has decreased (from 3.65eV to 3.60 eV). This is consistent with researches [24]. Because the doping by (Li) leads to the production of new local energy levels bottom conduction band ready to receive electrons and generate tails in the optical energy gap and these tails work in the direction of reducing the optical energy gap, which is one of the crystal defects [25]. The energy band gap was reduced after exposure to non-thermal plasma for 3,5,7 seconds. For the permitted direct electronic transitions, the optical energy gap of undoped and doped films formed on glass substrate was computed using the relationship [26]:

$$\alpha h\upsilon = B_o (h\upsilon - E_g^{opt})^r \quad \textbf{.... (3)}$$

Where α: is the absorption coefficient, hυ: the photon energy units (eV), B_o: is constant, E_g^{opt} : the optical energy gap units (eV), r: exponential coefficient determines the type of transmission .

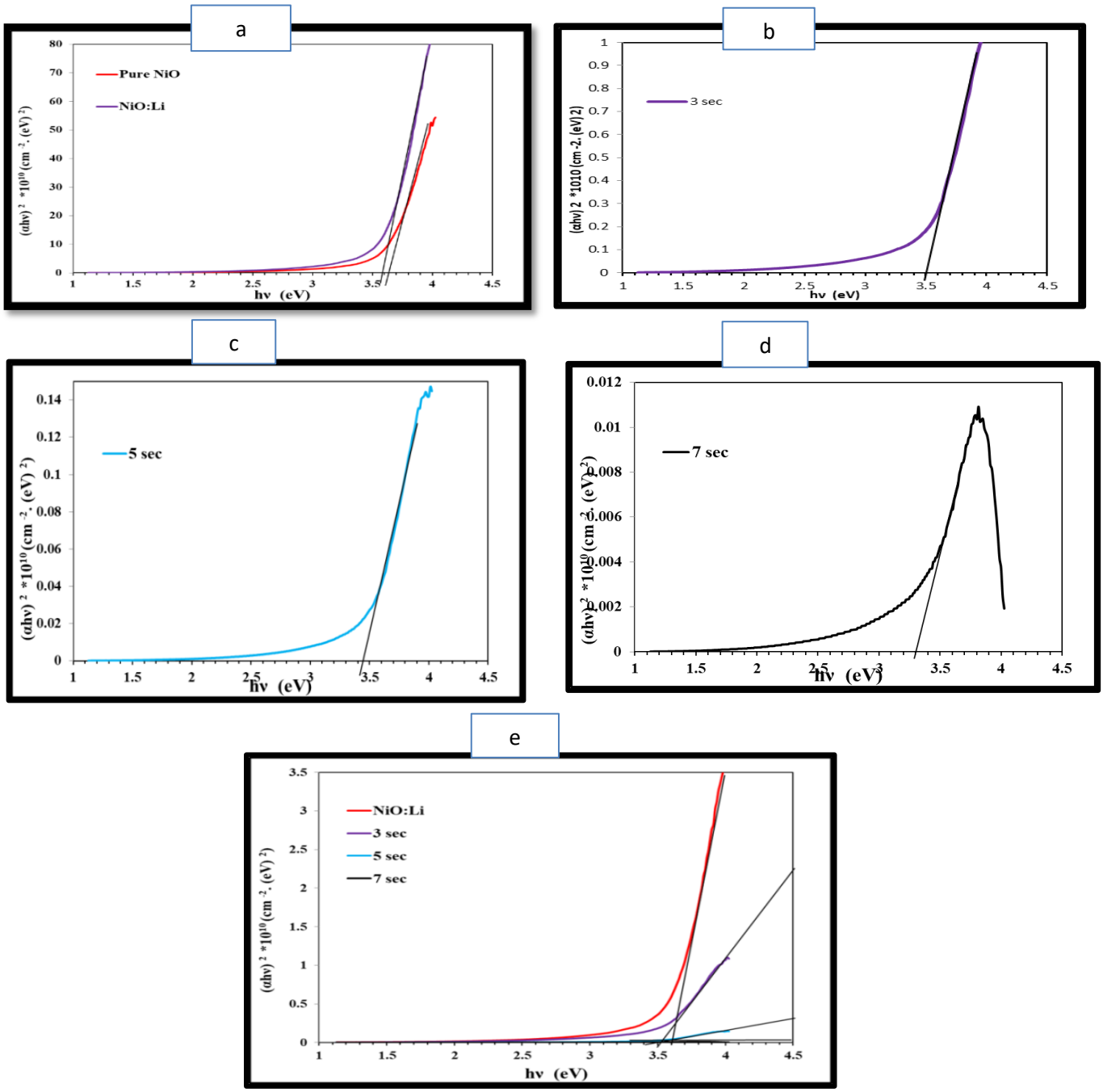

Figure 4. Energy gap for films a) pure NiO , NiO:Li, (b) NiO:Li after exposure to non-thermal plasm 3sec, (c) NiO:Li after exposure to non-thermal plasma at 5sec , (d) NiO:Li after exposure to non-thermal plasma at 7sec , e) NiO:Li before and after exposure to non -thermal plasma.

Table .5 Values of energy gap for NiO:Li before and after exposure to non- thermal plasma

Plasma exposure	$E_{g\ (eV)}$
0	3.60
3	3.50
5	3.45
7	3.30

3- Electrical properties

Hall Effect

The electrical mobility, carrier concentration, and conductivity of the produced films are determined by measuring the Hall Effect at room temperature, which provides information about the sort of predominant charge carriers. The results of the Hall effect showed that the electrical conductivity of the pure NiO and NiO:Li films were p-type semiconductors. That is, the lithium atoms behaved as acceptable impurities. Based on the study of the relationship between Hall voltage and electric current these results have a good agreement with [27]. The conductivity results were consistent with prior research, which indicated that electrical conductivity is in the range of $(10^{-6}\text{-}10^{-1})$ $(.cm)^{-1}$ [20]. There was also an increase in the mobility of all films. The electrical mobility mainly expresses the effect of charge carriers on the electric field in the crystal lattice of the material. Improving the electrical characteristics of transparent conductive oxides [28] necessitates enhancing this feature. This can be explained by minimizing the dispersion of surface charge carriers as well as eliminating imperfections in thin films. as a result of an increase in crystal size, which reduces the number of grains These results are consistent with the research [29].The Improvement of crystal quality reduces the carrier scattering from structural defects ,leading to increases mobility [30].

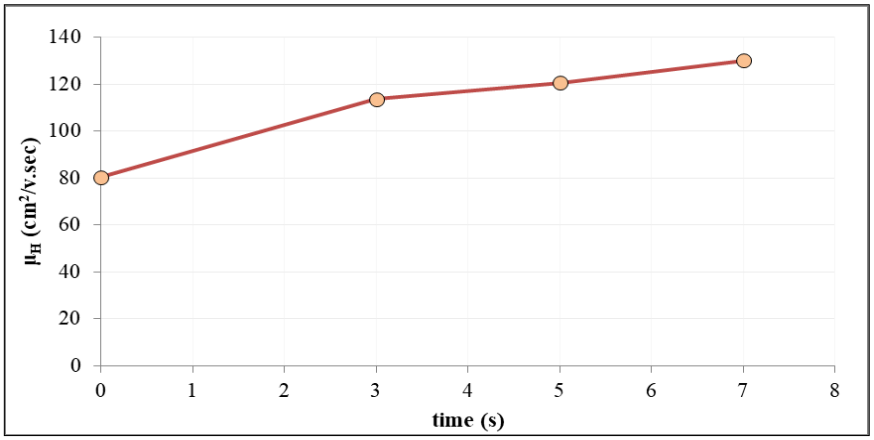

Figure.5 Change of electrical mobility of the prepared films of NiO: Li thin film before and after exposed to non-thermal plasma at (3,5,7 sec)

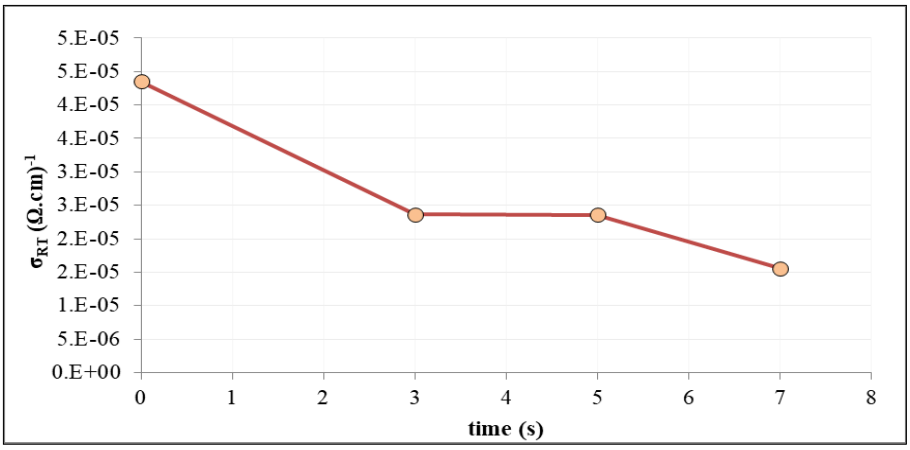

Figure .6 Change of electrical conductivity of the prepared films of NiO: Li thin film before and after exposed to non-thermal plasma at (3,5,7sec)

Table . 6 Hall Effect measurements

Sample	$n\times10^{12}(cm)^{-3}$	μ_H (cm^2/v.sec)	$\sigma_{RT}(\Omega^{-1}.cm^{-1})$
NiO pure	5.95	56.70	5.40E-05
NiO:Li	3.38	80.50	4.35E-05
3s	2.30	113.80	2.36E-05
5s	1.92	120.60	2.35E-05
7s	1.20	130.20	1.56E-05

Gas sensing measurements

Invasive sensitivity can be defined as the ratio between air resistance air (R_{air}) to resistance after gas exposure gas (R_{gas}) [31]:

$S = R_{air}/R_{gas}$ -------- for n-type **..... (4)**

$S = R_{gas}/R_{air}$ -------- for p-type **..... (5)**

where S is allergy, The percentage is also used as follows

$S\ (\%) = (R_{gas} - R_{air})/(R_{gas}) \times 100$ ---------- for p-type **..... (6)**

Sensor measurements

In this project, by exposing NiO pure and NiO:Li films to nitrogen dioxide NO_2 gas at operational temperatures, the gas sensing performance was examined (RT, 100 °C, 200 °C, and 300 °C). The gas sensing performance of the prepared NiO:Li films was also investigated after exposed to cold plasma at (3,5,7)sec. Equation (6) was used to get the sensitivity (S) and response time (S percent). When a P-type semiconductor interacts with an oxidizing gas such as (NO2), the majority of charge carriers are holes, resulting in an increase in conductivity and a decrease in resistivity (With an oxidizing atmosphere, a resistance decrease is noticed, which is attributed to the oxygen ion extracting an electron from metal oxide).

NO_2 as a kind of oxidizing gas that will react with the oxygen ion and keep the electrons at the surface therefor will increase the concentration of holes inside). As a result, after being exposed to NO_2, the conductance of p-type metal oxide semiconductors would increase. It should be noticed that the resistance of all samples lowers, demonstrating the p-

type nature of the sensor as a result of NiO semiconducting nature. Moreover, sensitivity, response and recovery time vary by operating temperature .Figure.7 showed the variation of sensitivity for prepared films towards NO_2 gas at operating temperature RT . The study showed that all films deposited can detect gas NO_2 at room temperature. The sensitivity of pure NiO and NiO:Li porous structures was 26.1 percent and 36.8%, respectively. For RT operating devices, quick response and recovery times are essential [33]. Exposing NiO:Li samples to cold plasma increased sensitivity, with the best sensitivity of 49.9% for samples exposed to cold plasma at time 7 sec. At an operating temperature of 200 ^{o}C. The sample itself revealed a high allergic reaction of 80.2 percent. This is owing to the porous nature of the material, which increases the number of active sites and the surface area available for gas diffusion [7]. Figure.8 showed the variation of sensitivity for NiO:Li films towards NO_2 gas at all operating temperature. Sensors with an operating temperature of less than 100 degrees Celsius might be beneficial in a variety of applications[34]. The NiO:Li sensor characteristics improved after being exposed to non-thermal plasma.. Because cold plasma increased the porosity of the surface through plasma etching, the microscopic structure of the surface became highly effective for interacting with donor gases parked on the sample's surface.

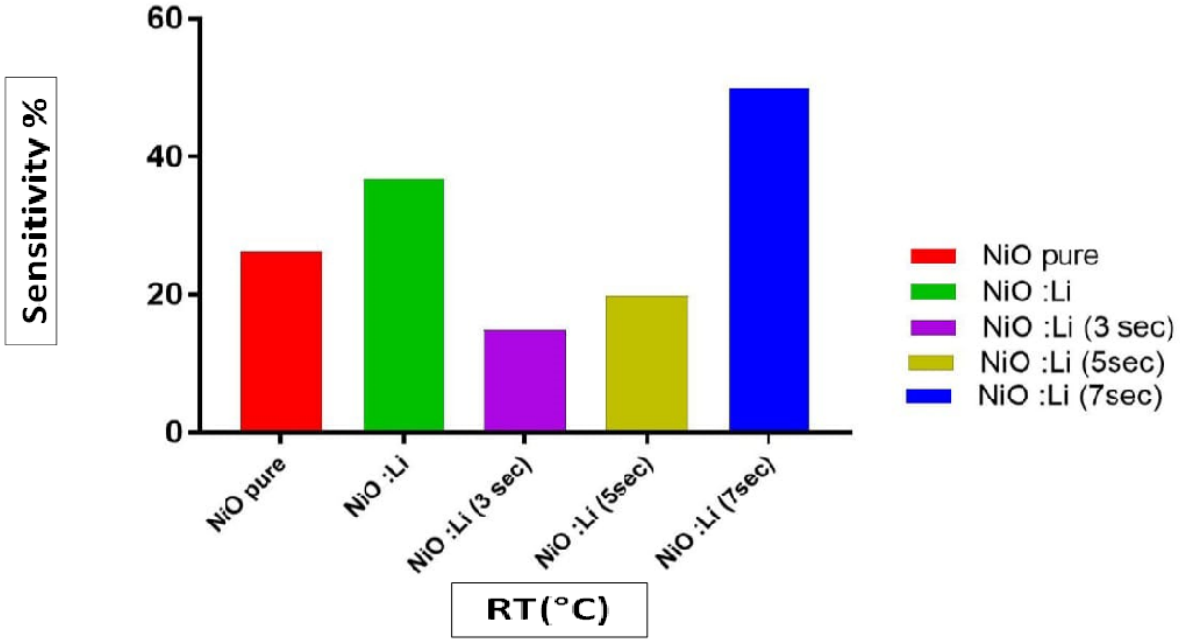

Figure7. Sensitivity variation with operating temperature at RT of NO_2 gas for all samples prepared.

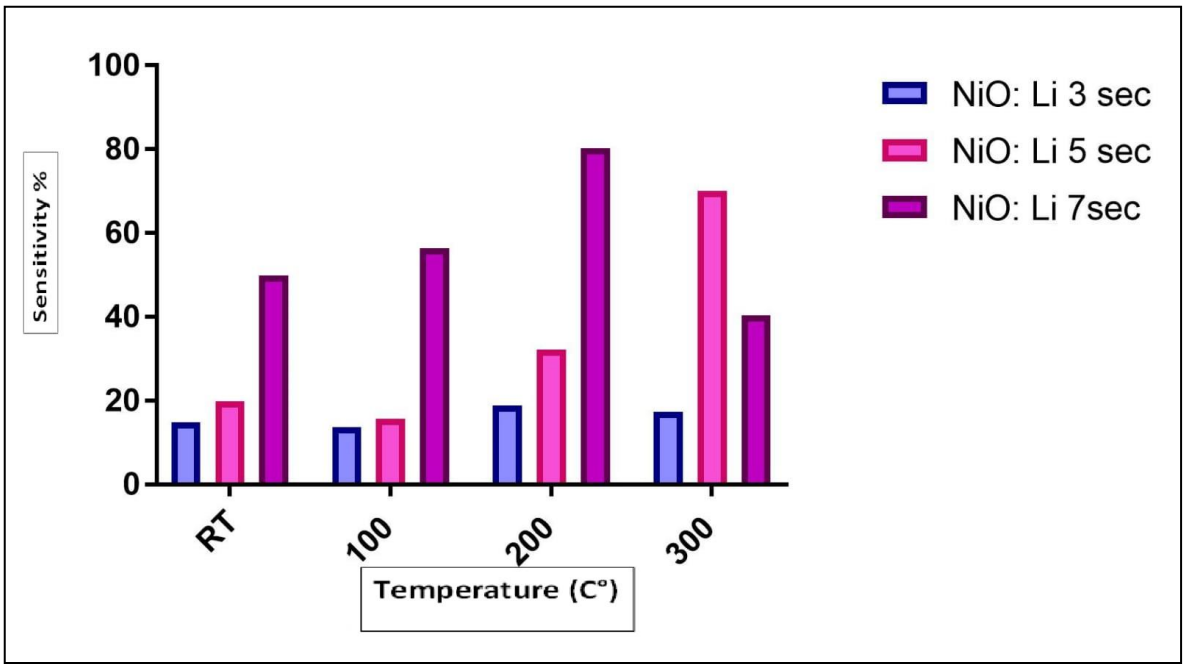

Figure 8. Sensitivity of the NiO:Li films towards NO_2 gas at different operating temperature after exposure to non-thermal plasma .

Table (5): Sensitivity , response time and recovery time for pure NiO, NiO:Li films before and after exposure to non-thermal plasma at (3,5,7 sec).

			NO2	
Samples	Temp	Sensitivity%	Res. Time (s)	Rec. Time (s)
Pure	30	26.1	11.3	67.7
	100	15.6	23.1	75.9
	200	53.5	18.7	78.6
	300	21.8	37.8	70.9
Doped	30	36.8	27.2	88.9
	100	80.8	20.4	87.9
	200	67.6	16.6	88.4
	300	49.3	11.8	46.5
3 sec	30	14.8	33.2	93.9
	100	13.7	23.0	87.0
	200	18.9	22.6	68.4
	300	17.4	12.6	27.8
5 sec	30	19.9	16.3	54.2
	100	15.7	24.5	79.3
	200	32.1	18.9	60.3
	300	70.0	13.6	50.4
7 sec	30	49.9	26.3	84.1
	100	56.4	26.4	92.1
	200	80.2	19.9	84.0
	300	40.4	22.8	27.3

Conclusions

The NiO:Li films were successfully prepared using the spray pyrolysis process (SPT). A pure NiO and NiO:Li films phase was found having a polycrystalline cubic structure, with growth preferentially along the (111) plane, according to XRD studies. SEM images yielded useful properties of the analyzed sensors. The optical energy gap for NiO:Li films decreased after exposure to non-thermal plasma at (3,5,7sec). Hall effect measurements showed that the all films were of p -type. Sensor behavior measurements showed a good sensitivity of pure NiO and NiO:Li towards NO_2 at RT. Treatment by non thermal plasma led to increasing sensitivity and improve the properties of the NiO:Li sensor. The combination of doping and non-thermal plasma of NiO thin films would be a promising approach for improving the sensing performance a type of semiconductor-based devices.

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