

Structural, Morphological, Optical, and NH₃ Gas Sensing Properties of Fe-Doped NiO Nanostructured Thin Films Prepared by Chemical Spray Pyrolysis

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Abstract— Pure and iron doped nickel oxide (NiO) thin films at 2% and 4% Fe levels were successfully deposited onto glass substrates by chemical spray pyrolysis (CSP) technique. That which relates to the effect of iron doping on the structural, morphological, optical, and gas-sensing properties of the films was studied in a systematic way out. X ray diffraction (XRD) analysis put forth a polycrystalline cubic structure with a preferential orientation along the (111) plane. Also, we saw an increase in the average crystallite size from 15.40 nm in pure NiO to 24.38 nm in 4% Fe doped films at the same time, we noted a drop in dislocation density from 24.45×10 to the power of 14 to 10.53×10 to the power of 14 lines/m squared. Atomic force microscopy showed that iron doping reduced surface roughness and also the average particle size. Also, we reported over 77.8% in the visible region for the optical transmittance which at the same time saw the absorption coefficient go up with iron concentration. Also, we saw the optical band gap narrow from 3.70 eV in pure NiO to 3.60 eV. At 180°C we saw that the sensitivity of ammonia (NH₃) gas to be 48.1% for pure NiO, which went down to 6.7% at 270 ppm for 4% Fe-doped films, which is a result of lower sensor response as we increased doping.

Keywords— Iron doped NiO, films, SPT, XRD, UV-VIS and AFM characterized, sensitivity, resistance

I. INTRODUCTION

Nickel oxide (NiO) is a very good p-type semiconductor oxide which reports wide bandgap of (3.6 - 4.0) eV, excellent chemical stability, and very variable functional properties. It reports great performance in terms of optical, electrical -- which in turn make it a right fit for advanced applications like electrochromic displays, optical switching glazing, lithium ion batteries, and gas sensor functional layers. Also, NiO films with a (111) orientation play very important role as buffer layers, which in turn facilitate the heteroepitaxial growth of perovskite type oxide films [6]. NiO has a rock salt (NaCl type) crystal structure in which Ni²⁺ and O²⁻ ions are arranged in an octahedral coordination environment [7]. That crystal structure plays a basic role in determining the

electronic, optical, and magnetic properties of the material. Also, NiO has a unique combination of antiferromagnetic ordering and at the same time high level of catalytic activity [10], great electrochromic behavior [11], and high chemical sensitivity [12]. What we see is a combination of these attributes that makes NiO a very attractive candidate for a wide array of tech applications, including in the field of advanced optoelectronics and dye-sensitized solar cells (DSSC) [13]. For these applications, NiO thin films have been successfully prepared by a large number of methods, which include chemical bath deposition [14], pulsed laser deposition [15], plasma-enhanced chemical vapor deposition, beam sputtering [16], RF sputtering [17], beam sputtering [18] and Chemical Spray Pyrolysis [19]. Out of all these, spray pyrolysis has gained much attention for its ease of use, low cost, scalability, and ability to put down uniform thin films over very large substrates [20]. In the present study we used a simple spray pyrolysis technique to deposit pure and iron doped NiO thin films on to glass substrates [21, 22]. We did a very detailed study to see the effect of iron doping on the structural, morphological, and optical properties of nanostructured NiO thin films.

II. MATERIALS AND METHODS

Thin films of Nickel oxide and Iron (Fe) doped NiO were grown onto a preheated glass substrate via SPT technique. An aqueous solution of 0.1M of Nickel chloride (supplied from Merck Chemicals) dissolved in deionized water as a matrix solution to form NiO thin films. The obtained solution of the doping agent was maintained by dissolving 0.1 M FeCl₃ (supplied from Merck Chemicals) in deionized water to obtain 0 %, 2% and 4% volumetric percentage, which was added to the matrix solution to obtain 0%, 2% and 4% doped. The preparation conditions were optimized at the following: The in thin films which we put onto glass substrates that we kept at a 450 °C during the whole depositing process. We used nitrogen as the carrier gas at a solution flow rate of 5 mL



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min⁻¹. We had a spray time of 10 sec which was followed by a 1.5 min break between each spray cycle to avoid excessive cooling of the substrate. We placed the spray nozzle a distance of 28 cm from the substrate. We measured the optical transmittance and absorbance spectra of the films with a double-beam UV-Vis spectrophotometer. For structural characterization, we used a Shimadzu XRD-6000 diffractometer (Japan) which has CuK α radiation (λ 0.15406 nm). We looked at the surface morphology of as deposited thin films with an AA3000 Scanning Probe Microscope, which was run in atomic force microscopy mode. We did gas sensing in a test chamber which had a radius of 7.5 cm and a height of 16 cm and we controlled the environmental conditions.

III. RESULTS AND DISCUSSION

Figure (1) Reports we see that at 37.63° and 43.51° angles, there is a number of peaks which we identify as (111) and (200) planes, which we have fitted to the ICDD card No. (75-0197). The XRD patterns reveal that the diffraction peak corresponding to the (111) crystallographic plane exhibits the highest intensity, indicating that the NiO thin films possess a preferred orientation along the (111) direction. This preferred growth is commonly observed in NiO thin films and is attributed to the relatively low surface energy of the (111) plane compared with other low-index crystallographic planes [23].

The average crystallite size (D) of the deposited films was estimated using the Scherrer equation [24]:

$$D = \frac{0.94\lambda}{\beta \cos \theta} \quad (1)$$

Where λ is the X-ray wavelength (1.54056 Å), and β is the full width at half maximum. In Table 1 we present the

calculated values which show that the average crystallite size (D) went from 15.40 to 24.38 nm as the iron doping concentration increased. This growth in crystallite size indicates that the introduction of Fe ions promotes crystal growth and improves the crystallinity of the NiO thin films [25].

The dislocation density (δ) of the (111) preferred diffraction plane was calculated using Eq. (2) [26]:

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

The study reports that we see a large drop in dislocation density (δ) from 24.45×10^{14} lines/m² in pure NiO thin film to 10.53×10^{14} lines/m² in the film which also has 4% Fe [27]. Also we see that the reduction in dislocation density which in turn improves the crystalline quality of the films is due to better crystallite growth and also the that the number of crystallographic defects is reduced because of iron inclusion.

In the (111) diffraction plane, the microstrain (ϵ) was determined from Eq. (3) [28]:

$$\epsilon = \frac{\beta \cos \theta}{4} \quad (3)$$

The microstrain (ϵ) value for pure NiO thin film reported was 20.56×10^{-4} which we see drop to 11.19×10^{-4} for the film doped with 4% Fe as reported in Fig. 2. Also, we see that this reduction in microstrain is an indication of lattice distortion relaxation and a drop in internal stress of the crystal structure [29]. In Table 1, we present the calculated structural parameters which include crystallite size, lattice constant, dislocation density, and microstrain.

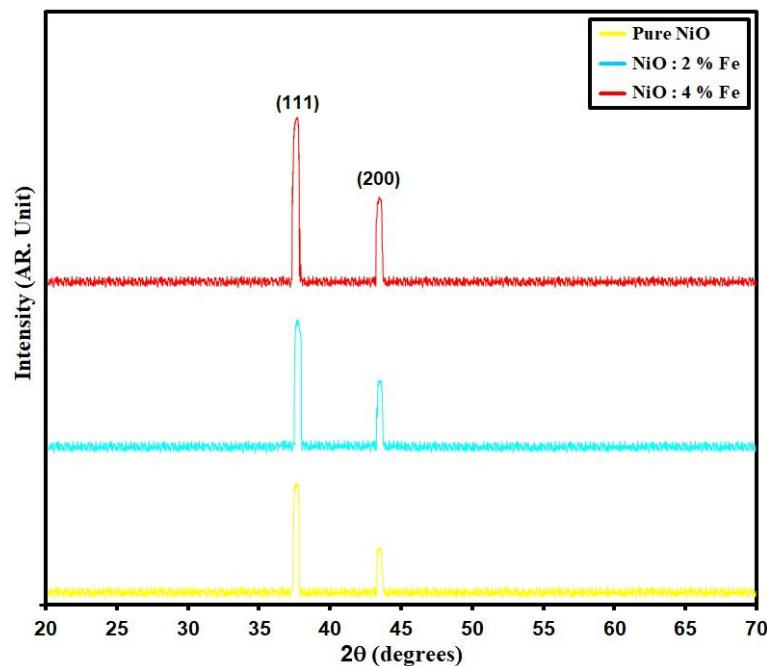
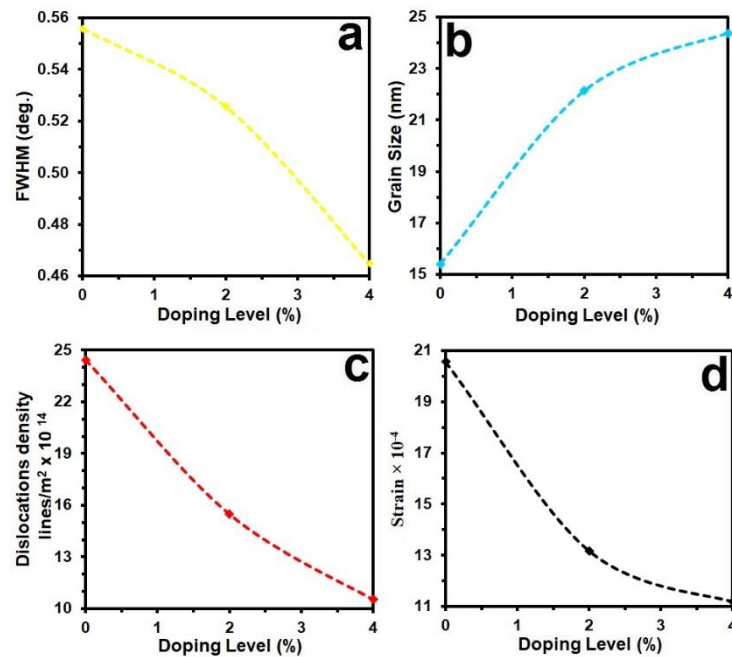


Fig. 1. XRD analysis of Pure NiO and Iron doped.

Fig. 2. D , FWHM, strain and dislocation of Pure NiO and with doping of Iron films.Table 1. D , E_g and S_{para} of prepared films.

Specimen	2θ (°)	(hkl) Plane	FWHM (°)	E_g (eV)	D (nm)	δ ($\times 10^{16}$) (lines/m ²)	ϵ ($\times 10^{-4}$)
Pure NiO	43.51	200	0.56	3.70	15.40	24.45	20.56
NiO: 2% Fe	43.48	200	0.53	3.65	22.13	15.49	13.18
NiO: 4% Fe	43.42	2000	0.463.70	3.60	24.38	10.53	11.19

Figure 3 presents the 3D AFM surface topography of pure NiO and Fe doped NiO thin films which have 2% and 4% Fe concentration. We see that Fe introduction greatly changes the surface morphology of the films. The average grain diameter which is 79.10 nm for pure NiO goes down to 72.66 nm and 50.81 nm for the 1% and 3% Fe doped films respectively which means that Fe suppresses grain growth and promotes the formation of finer grains. Also we note a

change in surface roughness which reduces from 8.74 to 3.23 nm in terms of average roughness (R_a) and from 6.80 to 2.17 nm in terms of root mean square roughness (R_{rms}). As we see in Fig. 3(a_3 , b_3 , c_3) the smoother surface is a sign of improved film uniformity and compactness which is a result of modified nucleation and growth kinetics due to Fe incorporation [30]. Full AFM parameters are listed in Table 2.

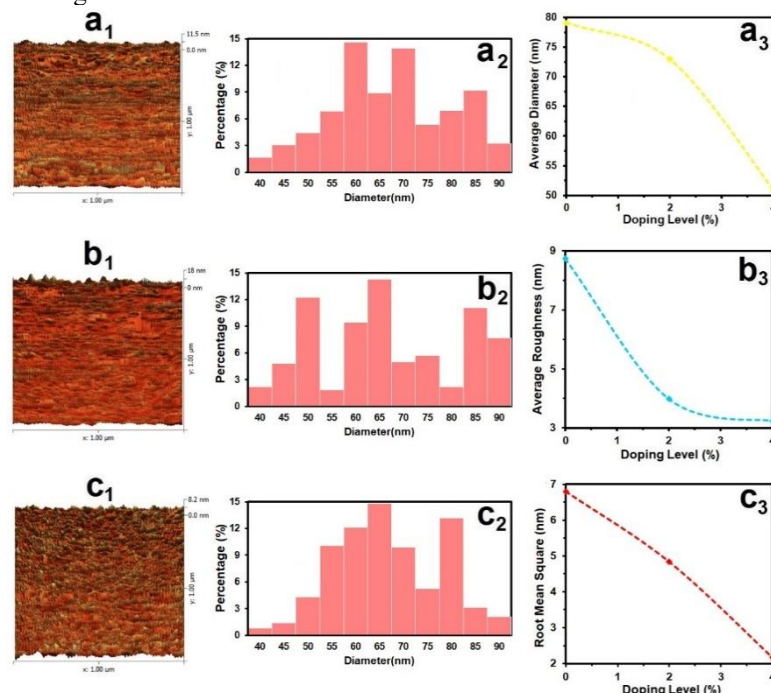


Fig. 3. In the case of Pure NiO and that of Iron films.

Table 2. We present the Morphological values of Pure NiO and that of the Iron films.

Samples	T _p (nm)	R _a (nm)	R. M. S. (nm)
Pure NiO	79.10	8.74	6.80
NiO: 2% Fe	72.89	3.98	4.84
NiO: 4% Fe	50.89	3.23	2.17

Figure 4 shows that we have the optical transmittance spectra of pure and Fe doped NiO thin films in the range of (300-900) nm. In that range all films display high optical transparency. with transmittance values exceeding 77.8%, indicating their suitability for transparent optoelectronic applications [31]. The observed optical behavior suggests that Fe incorporation has only a limited effect on the overall transparency of the NiO thin films within the investigated spectral range. The optical absorption coefficient (α) was determined by the following relation [32]:

$$\alpha = 2.3026 \left(\frac{A}{t} \right) \quad (4)$$

In which A is the absorbance and t the film thickness. We present in Fig. 5 the absorption coefficient (α) as a function of the incident photon energy ($h\nu$). We report that at the fundamental absorption edge α values are of the order of 10^{-4} cm^{-1} which we see to increase as a function of Fe doping

concentration; this shows that iron incorporation into the structure enhances the light absorption of the films by modification of their electronic structure [33].

The values of optical bandgap were achieved by the Eq. (4) [34]:

$$\alpha h\nu = k(h\nu - E_g)^n \quad (5)$$

The optical band gap energy (E_g) in pure and Fe doped NiO thin films was determined by the use of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) plots as reported in Fig. 6. Band gap values were calculated by extending the linear part of the curves to the photon energy axis. It is found out that the band gap decreases stepwise from 3.70 eV for the pure NiO film to 3.60 eV as we increase the concentration of Fe doping, which in turn reports that upon Fe incorporation into the structure the electronic band configuration changes and the band gap widens [35].

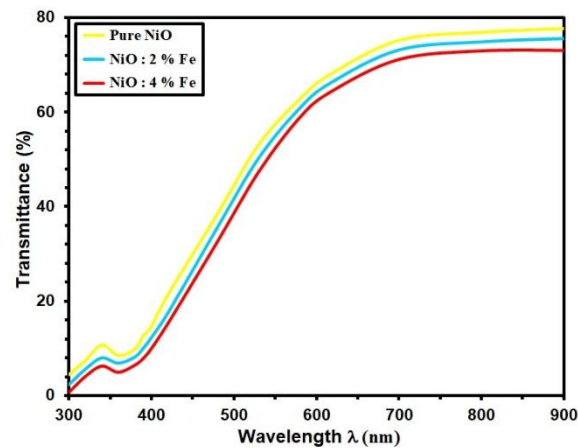
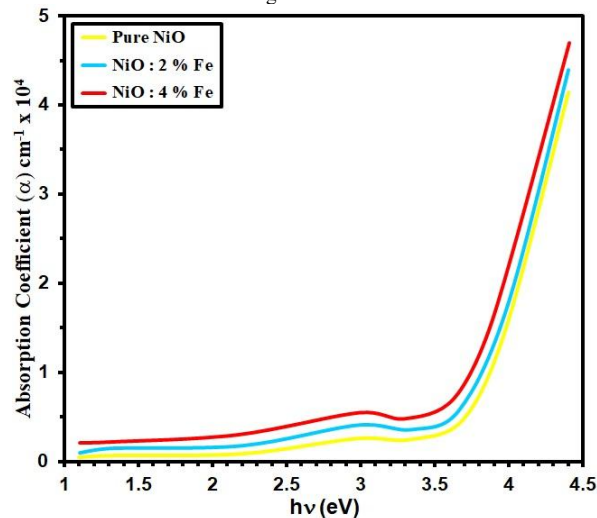


Fig. 4. Transmittance with wavelength of Pure & NiO: Fe with different dopant.

Fig. 5. α with photon energy (E_g) of Pure & NiO: Fe with different dopant.

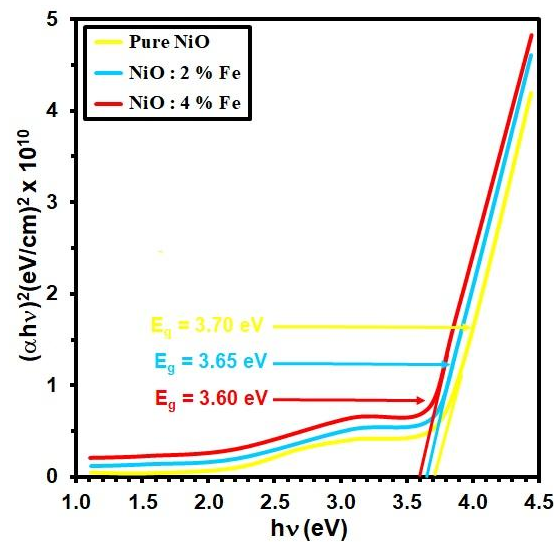


Fig. 6. Eg of Pure & NiO: Fe with different dopants.

The extinction coefficient (k) values were determined by use of Eq. 6 [30]:

$$K(\lambda) = \frac{\alpha(\lambda)\lambda}{4\pi} \quad (6)$$

Figure 7 shows that the extinction coefficient (k) varies with wavelength (λ). We see that the extinction coefficient goes down as we increase the Fe doping concentration, which in turn reports a decrease in optical losses within the NiO thin films [36].

The refractive index (n) was determined from that in Eq. (7) [31]:

$$n = \left(\frac{1+R}{1-R} \right) + \sqrt{\frac{4R}{(1-R)^2} - k^2} \quad (7)$$

Figure 8 also presents the variation of the refractive index (n) with wavelength (λ) for pure and Fe doped NiO thin films. We see a trend of decreasing refractive index with increased Fe doping concentration, which in turn indicates that iron's introduction into the structure modifies the films' optical response. This trend is put forward to be due to changes in crystal structure, packing density, and electronic polarizability brought about by the Fe introduction [37].

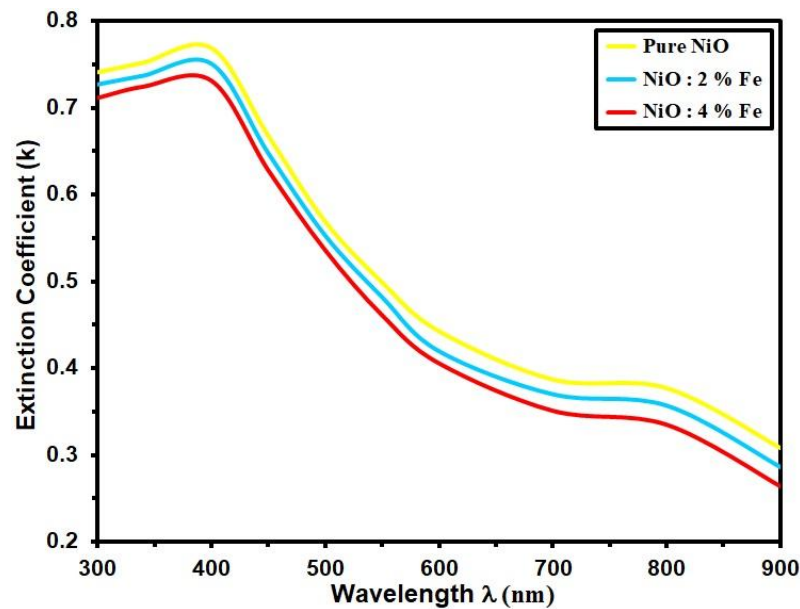


Fig. 7. k of pure & NiO: Fe with different dopants.

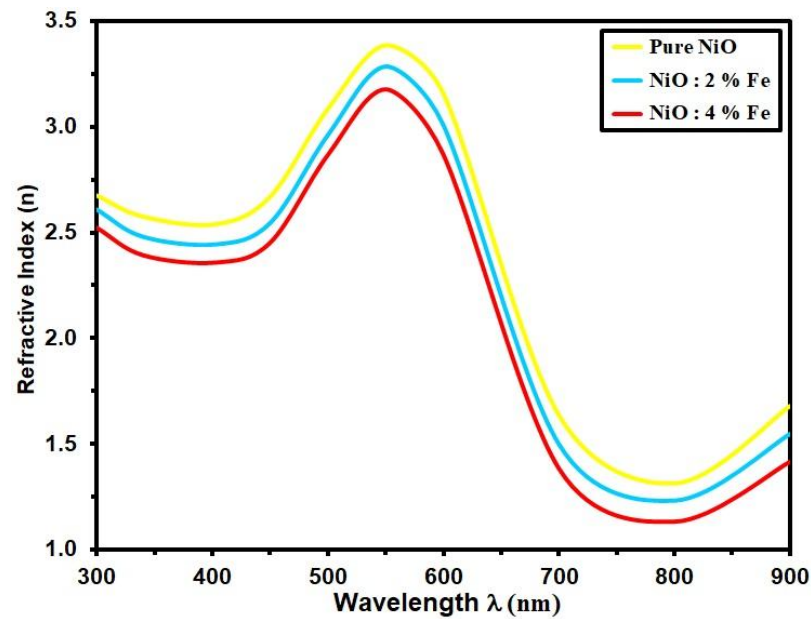


Fig. 8. n of Pure & NiO: Fe with different dopants.

The sensor performance was evaluated via the standard sensitivity metric [38]:

$$Sensitivity = \frac{\Delta R}{R_g} = \left| \frac{R_g - R_a}{R_g} \right| \times 100 \% \quad (8)$$

Figure 9 displays the resistance change over time of the various films at 270 ppm is presented. The sensor was operated at a temperature of 180°C. We see that upon exposure to ammonia (NH₃) what we have is surface oxidation of the NiO surface which in turn causes adsorbed O²⁺ ions to give up trapped electrons back into the conduction

band thus raising resistance because of a higher potential barrier at the grain boundaries. Also within our set of samples the NiO film which had 4% Fe doping had the highest resistance which we put forth as a sign of greater sensitivity [39].

Figure 10 also shows the effect of Iron doping on the ammonia (NH₃) gas response of NiO thin films. As Fe concentration goes up sensitivity clearly goes down which is mainly due to charge carrier recombination. We see a drop in sensitivity from 42.9% to 2.2% at 90 ppm, from 45.4% to 4.6% at 180 ppm, and from 48.1% to 6.7% at 270 ppm for pure, 2% Fe-doped, and 4% Fe-doped materials respectively [40].

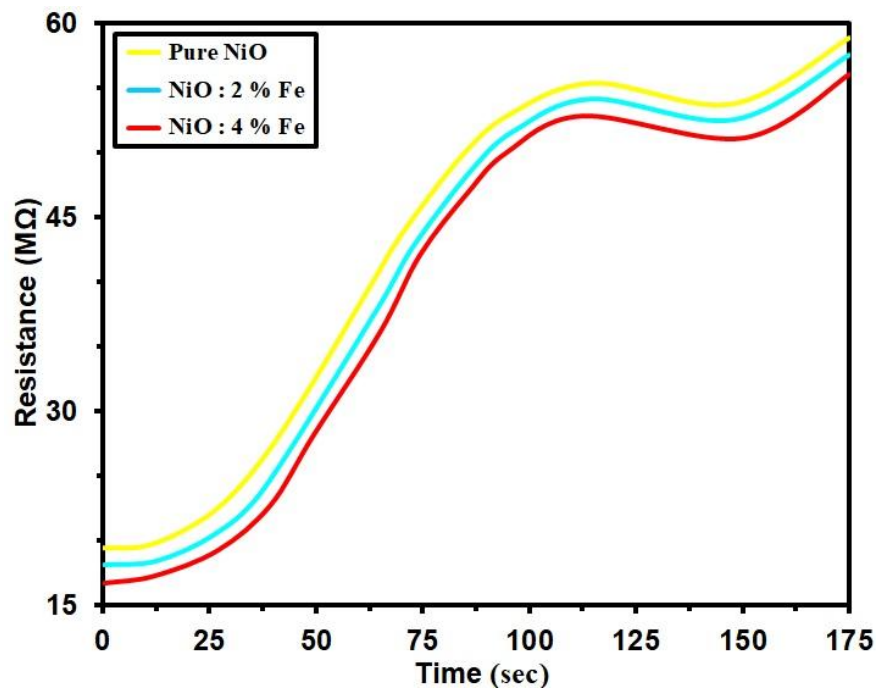


Fig. 9. Resistance via time of Pure & NiO: Fe with different dopants.

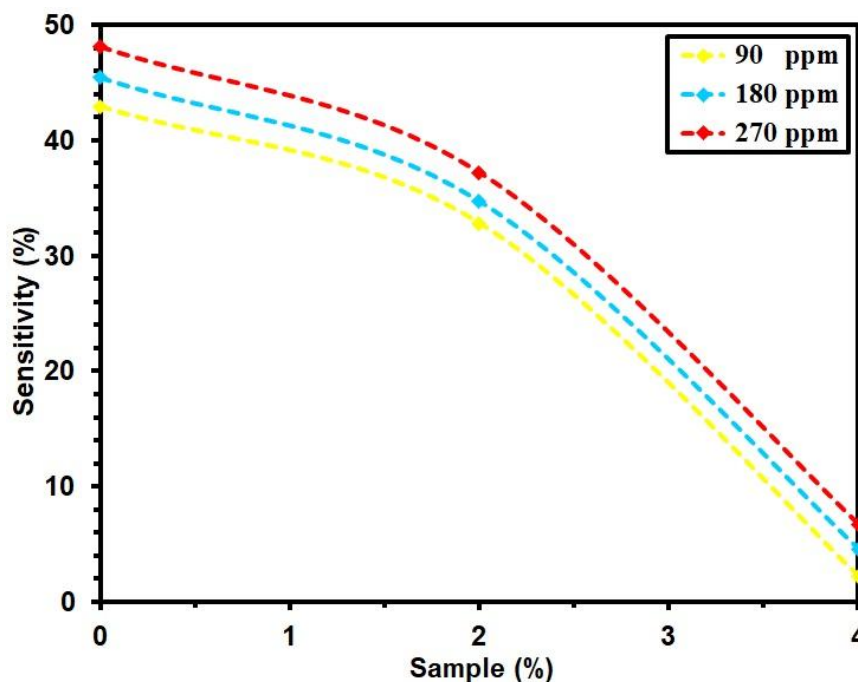


Fig. 10. sensitivity (S) of Pure & NiO: Fe with different dopants.

Aluminum thin films were at 175 °C during operation. In Figure 9 we see that the resistance of the films which resulted from exposure to 225 ppm of NO₂ gas. Pure CdO films had the lowest resistance [18]. Also we see that the CdO films doped with 3% Aluminum had a great deal more resistance which is a result of what we attribute to changes in the electronic structure brought about by the Aluminum dopant [36].

The gas sensor's sensitivity is ascertained, based on the relationship expressed in Equation (8), as described in reference [19].

$$Sen. = \frac{\Delta R}{R_g} = \left| \frac{R_g - R_a}{R_g} \right| \times 100 \% \quad (9)$$

Where R_a is the resistance in the presence of NO₂ gas and R_g is the baseline resistance in air. As shown in Figure 10, sensitivity measurements were conducted for pure CdO, CdO doped with 1% Aluminum, and CdO doped with 3% Aluminum under varying NO₂ concentrations. A clear trend is observed: Increasing the Aluminum content results in a large drop in sensitivity. For example, sensitivity values decrease from 54.1% to 7.8 % at 75 ppm, from 43.6% to 10.3% at 150 ppm and from 54.1% to 14.2% at 225 ppm as Aluminum doping increases from 0% to 3%. This drop in sensitivity we see is mostly due to increased charge carrier recombination at higher voltages which in turn reduces the effective separation of charge carriers and Reduces the total sensor response to NH₃ gas. [37].

IV. CONCLUSION

In this work, we report the synthesis of undoped and Fe doped NiO nanostructured thin films via chemical spray pyrolysis at 450°C. XRD analysis showed that all films are polycrystalline with a cubic structure, which displays a preference for orientation along the (200) plane. Iron doping, which we noted, brought in an increase in crystallite growth from 15.40 nm to 24.38 nm; we also saw a great drop in

dislocation density which went from 24.45×10^{14} to 10.53×10^{14} lines/m². Also we saw that iron incorporation reduced surface roughness and average particle size as reported by AFM studies. Optically we noted high transmittance which was greater than 77.8% in the visible region and also we saw that the absorption coefficient increased with increasing Fe concentration. Also we noted that the optical bandgap which for undoped NiO is at 3.70 eV narrowed to 3.60 eV at 4% Fe. Also we noted that the refractive index and extinction coefficient went down with higher Fe content. At the gas sensing tests for NH₃ at 180°C we reported that as we increased Fe doping sensitivity went down going from 48.1% at 270 ppm to 6.7% at 270 ppm

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