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Synthesis of Fibrous-Like Stripes ZnO: Ce Nano Structures for NO2 and NH3 Sensing

Raad S.Sabry[1], Mohammed J.Hassan[2], Tagrhid S. Muhsin[1], Baker A. Joda[3]

Department of Physics, College of Sciences, University of Al-Mustansiriya University, Baghdad, Iraq[1]

Department of Chemistry, College of Sciences, University of Al-Mustansiriya, Baghdad, Iraq[2]

Department of Chemistry, College of Science, University of Kerbala, Karbala, Iraq[3]

ABSTRACT:Zinc oxide and Ce doped nanostructures thin films with different ZnO:Ce ratios (3, 5 and 10) wt% have been prepared by Sol-gel spin coating method at optimum annealing temperature of 550 ˚C. The synthesized samples were characterized by X-ray diffraction (XRD), the spectra of the pure and ZnO: Ce nanostructures correspond to the various planes of a hexagonal ZnO phase, also the crystalline size decreased from about 37nm to 27nm by increasing Ce concentration, atomic force microscopy (AFM) analysis shows that the surface roughness of the samples increased from 1.14 for pure ZnO to 2.15 nm for 10% doping. FESEM measurements show that the ZnO is spherical shape particles which transfers into fibrous-like stripes embedded within a wrinkled shape with different size depending upon the Ce concentration. This change in the surface shape can improve the NO_2 and NH_3 sensing in the two cases of gas sensing measurements, namely static and dynamic case. It was found that the sensitivity was increased by using the same operating temperature. The dynamic results show that the response and recovery times are improved when the Ce concentration is increased. The optical measurements are shown that the transmittance was decreased with increasing of Ce concentration in zinc oxide.

KEYWORDS:NO_2, NH_3; Fibrous-like stripes, FESEM, ZnO: Ce nanostructures.

I. INTRODUCTION

Metal oxide semiconductors (MOSs) have been used as photo catalysts and other applications such as chemical sensors, detecting ultraviolet (UV) radiation, photoconductivity, gas sensory chemical sensors, and optoelectronic devices. Recently, ZnO has received the attention of many scientific researchers due to its remarkable combination of physical and optical properties. In addition, it has a wide band gap (3.37 eV at room temperature), high excitation binding energy (60 meV) [1, 2]. It was found that ZnO can use in various fields such as transparent electrode, gas sensors, room temperature ultraviolet lasers, solar cell windows, photovoltaic devices, catalysts, and piezoelectric cells [3–5]. Several deposition methods have been used to carry out undoped and doped ZnO films such as sputtering, pulsed laser deposition, vapor–liquid–solid (VLS) and hydrothermal, and sol–gel process [2,6].The last method, namelysol–gel process has several advantages when compared with other methods due to its simplicity, easy control of the film deposition, safety as well as low cost of the apparatus and raw materials[7]. A doping is an effective way to improve the properties of ZnO thin films. Therefore, adding selective elements to ZnO offers an important route to enhance and control its optical and electrical properties, which are crucial to its practical applications [8–10]. In recent years, many of rare earth elements such as Sc, Y, Ce, Eu, and Er have been added to ZnO, but the use of cerium doping has received more attention due to its important properties including: (a) the redox couple Ce^{+3}/Ce^{+4} that makes cerium oxide shift between CeO_2 and Ce_2O_3 under oxidizing and reducing conditions and (b) the easy formation of labile oxygen vacancies with the relatively high mobility of bulk oxygen species [11-13]. Therefore, the versatile properties of Ce-doped ZnO nanostructures have been explored for many applications such as photoluminescence, photo electro chemical properties under visible light, and as well as chemical and gas sensors [11]. The main aim of this study is to develop and validate the use of doping on structural and optical properties of the Ce (3,5 and 10 at.wt.%) - ZnO sol–gel deposited thin films by using various characterizationof the target gas sensor.

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II. MATERIALS AND METHODS

In this study, zinc acetate dihydrate (Zn $(CH_3COO)_2.2H_2O$, BDH, 98.5%) and cerium nitrate hexahydrate (Ce $(NO_3)_3 \cdot 6H_2O$, BDH, 99%) were used as starting material and doping sources, respectively. Moreover, ethanol (C_2H_5OH, Fluka, 99.9%) and monoethanolamine (MEA) (C_2H_7NO, Fluka, 98 %) were used as a solvent and stabilizer, respectively. In the preparation procedure for ZnO: Ce thin films, Zinc acetate dihydrate was dissolved in a mixture which was composed of ethanol and MEA at 70°C. The molar ratio of MEA to zinc acetate dihydrate maintained at 1 and the concentration of zinc acetate was 0.75 mol/L. The mixed solution was stirred at 70°C for one hour, then ethanolic solution of cerium nitrate hexahydrate with optimized Zn/Ce ratio at (3,5 and 10)% was added to mixture. The final solution was stirred for an additional hour. After stirring, the solution becomes clear and homogeneous. This solution was used as the coating solution after cooling at room temperature then allowed to age for 24h before initiating deposition. The films were obtained by a spin-coating procedure. The aged solution was dropped on glass substrates, which were rotated at 500 rpm for 10 sec, after that the speed was increased to 2000 rpm for 30 sec and 3000 rpm for 30 sec. After the coating step, the films were dried at 100 °C for 10 min. This step was repeated for ten times to obtain thickness about 325 nm, finally they were annealed at 550 °C in air for 1 h at rate of 10 min.

III. INSTRUMENTATION

The structure and lattice parameters of pure and ZnO:Ce films were analyzed by XRD instrument (MINIFLEX II RIGAKU, Japan), with the following specifications Cu (Kα) radiation with λ = 1.5405 Å (40 kV, 30 mA). The morphological properties of all films were investigated and observed by field emission scanning electron microscope (HITACHI S-4160) made in Japan. The optical transmittance and absorption were measured with a range of (300-900 nm.) by using (UV-1650PC Shimadzu software 1700 1650, UV-Visible recording Spectrophotometer, Japan). The surface morphological of ZnO thin films were analyzed using atomic force microscopy (SPM,AA-3000USA).

IV. RESULTS AND DISCUSSIONS

Figure.1 shows the typical XRD pattern of pure ZnO and ZnO:Ce thin film. The major peaks are identified as (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), and(1 0 3), plane of reflections for a single phase wurzite structure of ZnO and the peak located at 2Θ = 28.7˚ is attributed to (1 1 1) plane for the CeO_2 cubic structure (JCPDS standard card No. 75-0390) crystalline structure. Therefore, the diffraction peaks of pure ZnO and ZnO:Ce, without characteristic peaks of other impurities, are indexed to the ZnO hexagonal wurtzite structure according based on its JCPDS standard card (No. 75-0576).The average crystalline size (D) was also estimated by using scherrer's formula, $D = 0.9\lambda/(\beta\cos\Theta)$, based on the integral width of XRD peaks, where λ is the wavelength of the incident X-ray radiation, β is the full width at half-maximum (FWHM) of the desired peak (here (1 0 1) at 2Θ = 36.3˚) in radians and Θ is the diffraction angle.

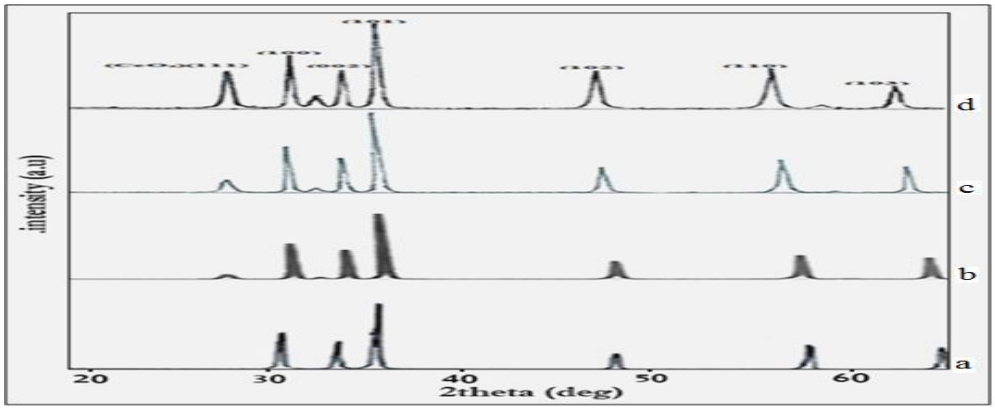

Figure 1: XRD patterns of ZnO thin film with different Ce concentrations: (a) pure (b) 3% Ce, (c) 5% Ce and (d) 10% Ce.

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The crystalline structure and averaged crystal size for different Ce doped ZnO system was summarized in Table.1. It was found that when increasing cerium concentration in the films, the average crystalline size decreased.

Table 1: Crystalline properties of pure ZnO and ZnO:Ce thin films.

Samples	Average grain size/ nm
Pure	37.1
3 %	33.9
5 %	32.4
10 %	27.0

This can be explained because as ionic radii of $Ce^{+3}$are 0.092 nm which are larger than that of Zn^{+2} ion of 0.074 nm, therefore, it is difficult for Ce^{+3} to enter the crystalline lattice of ZnO to substitute Zn^{+2}. The Ce^{+3}ions seem more likely to form complex with the surface oxygen of ZnO, which decreases the growth of ZnO crystallite [14, 15].
The results in Figure 2(a) show that the nanoparticals and a smooth grains to be formed. Interestingly, similar results were also reported in the literature by other authors [16].

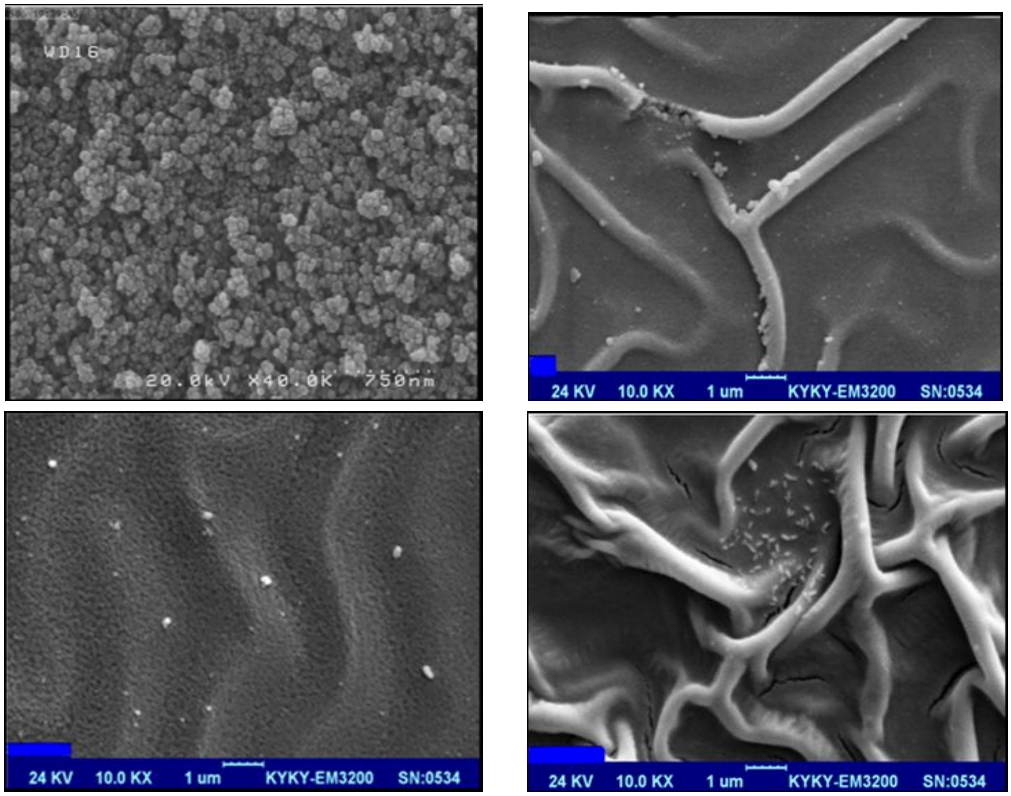

Figure 2: FE-SEM images of (a) pure ZnO (b) 3% Ce, (c) 5% Ce and (d) 10% Ce.

In general the film is homogeneous, continuous and uniform distribution and Spherical shape. An image of 3% Ce concentration shows that the films consist of small regions of fibrous-like stripes (ZnO:Ce) embedded within a wrinkled, as shown inFigure 2 (b). A previous study has suggested the two mechanisms can be used to explain wrinkle formation. The main reason may be related to the increase in volumetric strain [17]. Another reason is that a substantial loss of the hydroxyl/alkoxy groups during annealing induced stress formation [18]. Figure 2 - (c) shows that the SEM micrographs of a typical ZnO:Ce nanostructure hybrid the ZnO:5% Ce has a wrinkled/perturbed morphology with

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small patches scattered on its surface. It was found that the increase of Ce (5%) lead to decrease the volume and height of ganglia-like, as shown in Figure 2(c). On the other hands, the results in Figure 2 (d) show that increase of Ce (10%) lead to increase the volume and height of the ganglia-like content and become more complex. Therefore, it looking more distorted and branched at their ends, induced pronounced wrinkling, Bulk shrink age is most likely caused by rapid solvent evaporation. The derived films evolve from desorption of the hydroxyl groups with subsequent ZnO: Ce crystallization. It was found that a wrinkle formation is beneficial and prestressing phase effectively reduces the intrinsic stress within the film during operation making the hybrid nanostructure.

Figure 3 (a,b,c,d) Shows the pure ZnO and ZnO:Ce thin films at different concentration (3, 5 and 10) % ,annealed in 550˚Cfor 2h. The root mean square (RMS) surface roughness is observed to be 0.693, 1.14, 1.47 and 2.15 nm for a, b, c, and d samples, respectively when compared with the pure ZnO. The roughness is increased with increasing the cerium concentration in the films. This increase is attributed to the difference in ion size between Zn (rZn^2 = 0.074 nm) and Ce (rCe^3 = 0.092 nm).

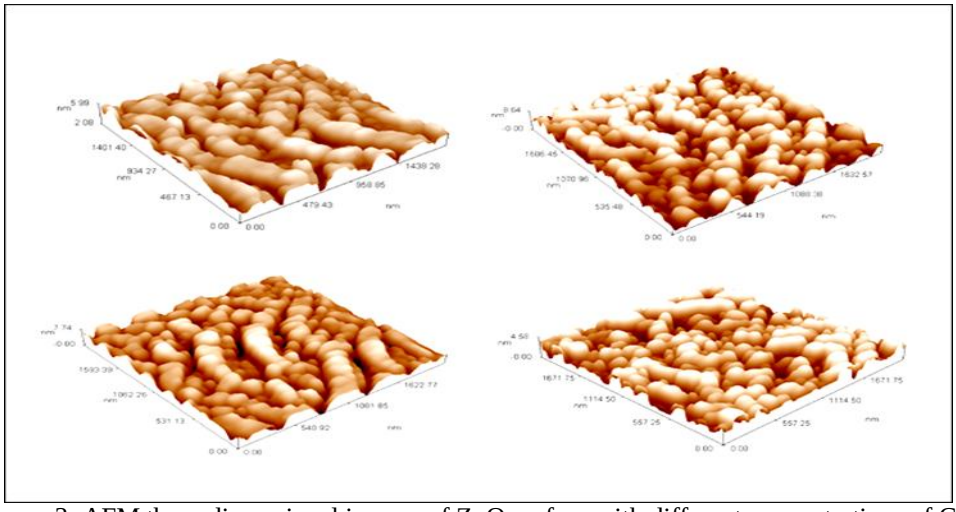

Figure 3: AFM three-dimensional images of ZnO surface with different concentrations of Ce.

Figure 4 shows the optical transmittance spectra for ZnO and ZnO doped Ce thin films using the UV–visible wavelength (350–800 nm).

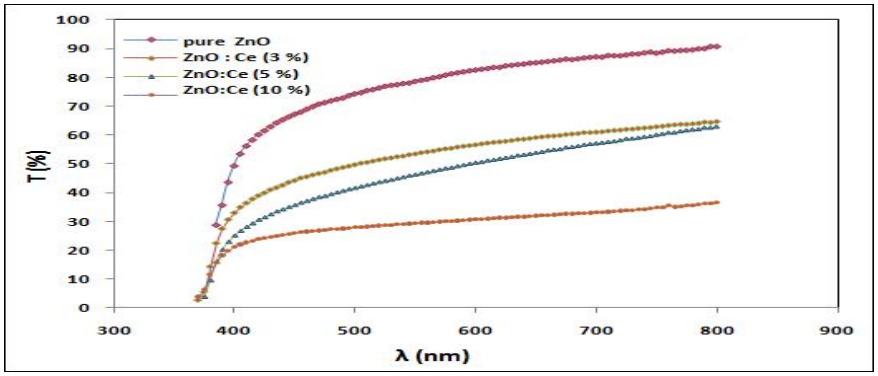

Figure 4: Transmittance spectra of ZnO thin film with different concentrations of Ce.

In the light of these results, it can be seen that the transmittance of pure ZnO thin films were measured at approximately 95% in the visible light ranged from 370 to 800 nm. It was found that, the percentage of transmittance is decreased

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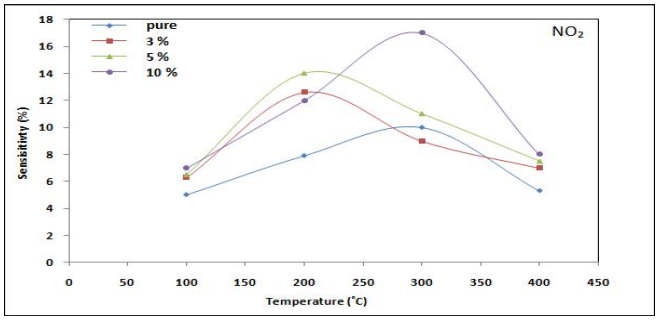
until 10% ZnO when the concentration values of ZnO:Ce thin film increase. This reduction in the transmittances of the ZnO doped Ce thin films may be due to the observed morphology for the corresponding sample. According to AFM analysis, there is a difference in the measured root mean square (rms) surface roughness values between samples. The rms values, namely 0.693, 1.14, 1.47, and 2.15 nm were determined for pure, 3, 5, and 10 % ZnO doped Ce samples, respectively. The findings show that the sample with the highest Ce doping Ce (10 %) has the largest surface roughness. As a result it has the highest scattering and the lowest transmittance. Moreover, the RMS surface roughness of the 3% ZnO doped Ce sample is lower than the 5 % ZnO doped sample. Thus, the higher measured transmittance is due to lower scattering of this sample. According to these results, the values of band gap energy for ZnO and 3, 5, and 10% of Ce were found to be 3.219, 3.222 and 3.232 eV, respectively.

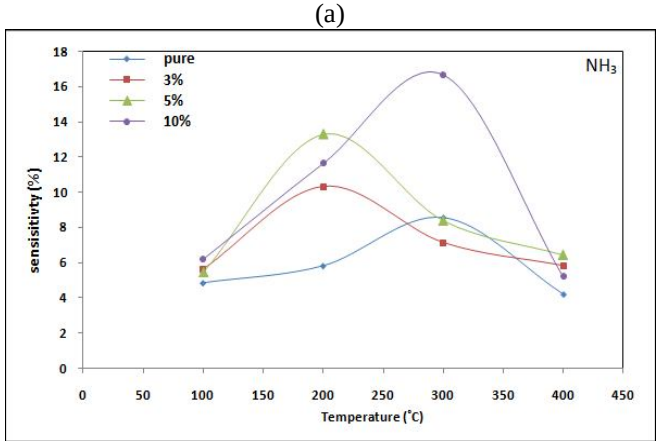
The gas sensing behavior of pure ZnO and ZnO: Ce thin films annealed at 550˚C were studied by using static and dynamic measurements system. The thin films resistors were measured in air and 100 ppm of NH3, and NO2 as well. The operating temperature was range at the interval of 100 to 400 ˚C. The sensitivity value of gas was determined at particular operating temperature by using the following equation.

$$S = \left| (Rg - Ra) \right| / Ra \times 100$$

WhereRa is electrical resistance of the sensor in air, Rg is electrical resistance of the sensor in gas. Figure 5 shows the variation of the sensitivity with temperature for pure and ZnO: Ce (3%, 5 %, and 10 %) sensor samples toward 100 ppm NH_3 and NO_2 gases.



(a)



(b)

Figure 5: Sensitivity of ZnO thin film with different Ce concentrations as a function of operating temperature for NO2 (a) and NH3 gases (b).

It can be seen that the sensitivity for all the samples gradually increases with increasing the temperature values to reach the maximum values, and then it is dropping at high temperature. The maximum values corresponding to an optimum operating temperature for NH_3 and NO_2 were appeared at 300°C for pure and 10 % samples and 200 °C for 3 % and 5 %

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samples, as shown Table 2. The change in the sensitivity with temperature depends on the interaction between the adsorbed oxygen species and gases [19, 20]. When the pure and ZnO:Ce sensor samples are heated, the oxygen molecules begin to adsorb and dissociate ($O_2 \rightarrow 2O^- \rightarrow O^{2-}$) on their surfaces and its concentration rise gradually with increasing the temperature until certain temperature which accompanied with the increase in the electrical resistance. While at higher temperatures, desorption of oxygen species from the sensor surfaces leads to decrease the concentration of oxygen species. So, the high sensitivity values of gases at temperature of 300˚C can be to the high concentration of adsorbed oxygen species and high oxidation activity of gases [21-23]. The results in Table 2 show that the sensitivity is increased when the concentration of Ce increase. A possible explanation is that the addition of Ce may be caused to decrease the particle size, as shown from the XRD analysis. As a result, the change in the surface shape from smooth to Wrinkle is also found, as shown from FE-SEM measurements. This means that an increasing in the surface to volume ratio leads to enhance the interactions between the cases and surface. The results of AFM analysis show that the roughness is increased when the concentration of Ce increase, and lead to produce a largest effective surface area in order to react with the gas.

Table 2: Sensitivity of ZnO and ZnO:Ce using the operating temperatures ranged from 200 to 300 °C forNO_2 and NH_3 gases.

Doping	Sensitivity%			
	NO_2		NH_3	
	200°C	300°C	200°C	300°C
Pure ZnO	5.80	8.54	7.9	10
3%	10.31	7.12	12.6	9
5%	13.28	8.83	14.0	11
10 %	11.64	16.66	12.0	17

Figure 6 show the measured resistances against the values of time for for pure, 3, 5, and 10 % ZnO doped Ce samples by using various concentrations of gases, namely 100, 150, and 200 ppm of NH_3 and NO_3 at room temperature. It can be seen that the resistance increased with increasing the concentrations of gases during the exposure to gases (NO_2 and NH_3). In addition, the minimum resistances are observed at concentration of 10% Ce for two gases, whereas the maximum values observed at 3% concentration of Ce and pure ZnO for NH_3 and NO_2 gas, respectively.

The response and recovery times for sensor were reduced for all samples when the concentrations ofCe increase, as shown in Table 3. Moreover, the minimum response and recovery times are observed at the concentration of 10% Ce. It was found that the values of resistance are increased for NH_3 exposure when compared to NO_2, as shown in Figure 6. A relative change in the electrical conductance due to an oxygen ion acts as a trap to electrons from the thin films. The electrons are taken from ionized donors through conduction band. The density of majority charge carriers at the gas–solid interface was reduced. This leads to form a potential barrier for electrons with increasing the density of oxygen ions on the surface. The adsorption of further oxygen is inhibited. Thus at the junctions between ZnO grains, the depletion layer and potential barrier leads to the increasing of the electrical resistivity value. This value is strongly dependent on the concentration of adsorbed oxygen ions on the surface. Introducing the n-ZnO films in an NH_3 ambient will change the concentration of these ions and increase the resistance. These results are in agreement with those reported in the literatures [23-25]. The detection of an oxidizing gas such as NO_2 can be associated with reaction in which conduction electrons are consumed and the subsequent detection reaction lead to increase the barrier height and increase the surface resistance. The NO_2 response can be explained below:

$$NO_2 + e^- \rightarrow NO + O_{ads.}$$

The electrons consumed in these reactions are extracted from the conduction band, thus raising the resistivity of Ce-doped zinc oxide film. It is necessary to mention that in the last case, the reaction products emerging from the primary detection reactions escape further detection within the sensing layer. The improvement of sensing properties of doped zinc oxide sensor structure towards hazardous gas is determined by the use of a particular dopant for the detection of specific gas species. The presence of chemical adsorbed molecules (NO_2) may becaused electron depletion in the thin

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film surface and the building up of a Schottky surface barrier; consequently, the electrical resistivity of the thin film increases to a maximum value [26, 27].

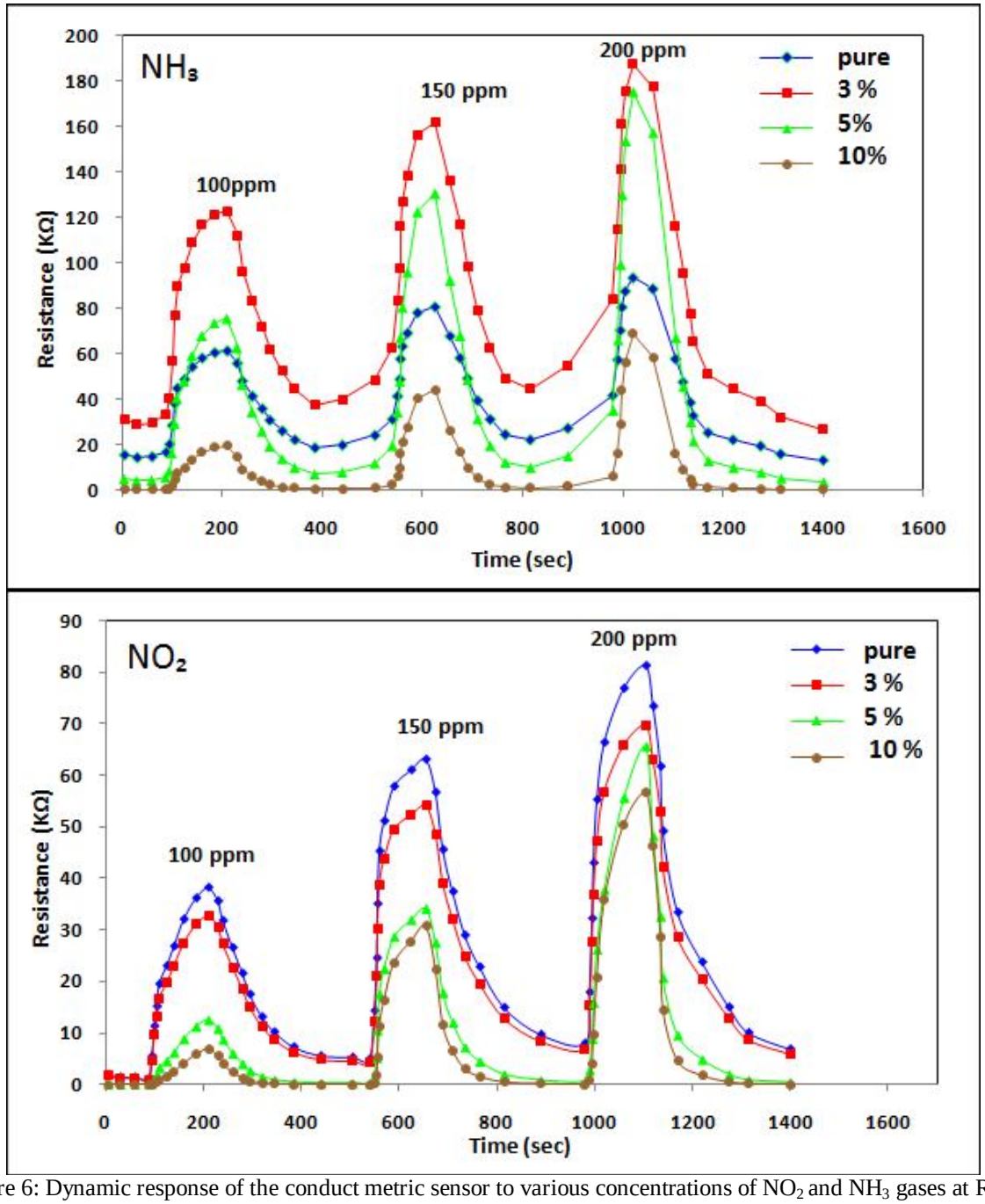

Figure 6: Dynamic response of the conduct metric sensor to various concentrations of NO_2 and NH_3 gases at Room temperature.

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Table 3: Response and Recovery time values of ZnO and variusZnO:Ce.

Sample	Response time (s)			Recovery time (s)		
NH_3 gas	100ppm	150 ppm	200ppm	100 ppm	150 ppm	200 ppm
pure	85	97	130	112	115	190
3 % Ce	120	122	125	130	135	155
5 % Ce	80	99	121	110	119	135
10 % Ce	70	90	115	105	111	120
Sample	Response time (s)			Recovery time (s)		
NO_2 gas	100ppm	150ppm	200ppm	100ppm	150ppm	200ppm
pure	121.1133	105.566	120.352	130.759	135.68	200.5
3 % Ce	98.9263	114.566	124.383	120.206	140.393	150.22
5 % Ce	94.255	110.566	111.837	115.293	120.293	117.3
10 % Ce	65.1063	78.343	85.29	93.92	95.56	98.47

V. CONCLUSION

In conclusion, the results of FE-SEM are shown that the structures for pure ZnO thin films are spherical, nanoparticles distributed a regular, basis and homogeneous, while doped thin films were shapes like fiber and a wrinkle network. On the other hands, the findings of AFM technique are indicated that the roughness increased with increasing the cerium concentration in the films from (1.14) to (2.15). The optical properties of undoped and Ce doped ZnO films were shown thatthe higher transmittance (95%) wasfound for undopedZnO films when compared with(65-40%) for doped Ce films within wavelength ranged (350-900nm).The energy gap is slowly increased when the doping increase. The energy gap is slowly increased when the doping increase. Furthermore, the sensitivity of all the samples is increased when the operating temperature increase. It was found that the maximum value was reported corresponding to an optimum operating temperature, which is 300°C for sample (10 %) and 200 °C for samples (3 % ,5 %). The results of dynamic sensitivity at room temperature have concluded that the 3% doping have a maximum resistance for NH_3 while pure ZnO have a maximum value for NO_2 gas.

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