



Ni_{0.9}Zn_{0.1}O/ZnO nanocomposite prepared by malonate coprecipitation route for gas sensing



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ABSTRACT

A nanocomposite material, Ni_{0.9}Zn_{0.1}O/ZnO, was synthesized by controlled thermal treatment of the corresponding nickel zinc malonate previously prepared by coprecipitation in aqueous solution. The co-existence, structure and morphology of the Zn-doped nickel oxide, Ni_{0.9}Zn_{0.1}O and zinc oxide, ZnO, nanoparticles was confirmed by a set of instrumental techniques such as XRD, SEM and ToF-SIMS. The gas-sensing properties of the as-synthesized nanocomposites have been evaluated for CO, NO₂, H₂ and the results show that the gas sensors based on the Ni_{0.9}Zn_{0.1}O/ZnO nanocomposites exhibit a good selectivity to NO₂ at low temperature (225 °C) and, comparing to ZnO and Ni_{0.9}Zn_{0.1}O individually, an increase in the response to CO gas at a high temperature (300 °C). The significant enhancement of the sensing performance of the Ni_{0.9}Zn_{0.1}O/ZnO nanocomposite is attributed to the formation of p-n junctions (metal oxide heterojunctions) between Ni_{0.9}Zn_{0.1}O (p-type semiconductor) and ZnO (n-type semiconductor) and to the catalytic activity of these metal oxides.

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1. Introduction

Semiconducting metal oxide based sensors have been widely used for the detection of toxic, hazardous and combustible gases for the safety of human beings and environmental control [1–7]. This is certainly due to their advantages such as simplicity of their use, high sensitivity to small concentration of gases, low fabrication cost and long term stability [7–9]. The gas detection principle of metal oxides semiconductors is based on the variation of electrical conductivity in the presence or absence of tested gas. In fact, when the metal oxide semiconductor is heated, it usually adsorbs the oxygen on its surface and, for an n-type semiconductor, this oxygen will extract electrons from the conduction band and form a depletion layer. In the presence of a reducing gas, for example, the negatively charged oxygen will oxidize the tested gas and re-inject

the electron back to the conduction band and in the case of n-type semiconductor, the electrical conductivity will increase [10,11]. For a p-type semiconductor, the oxygen adsorption increases the hole concentration. The presence of the target gas is then determined by the change of resistance resulting from the reaction between surface adsorbed oxygen species and the gas. The resistance change is directly related to the gas concentration, allowing for its measurement. Although, this type of sensors presents a good sensitivity, its selectivity remains a challenging issue as it gives a similar response to a wide range of gases. Many researchers have suggested various ways to overcome this problem of cross-sensitivity, which include the use of specific filters like zeolite thin films [12,13], the addition of a noble catalytic metal to promote the reaction to specific gases [14,15], and the use of composite metal oxides [16–19]. The latter is very important in that it can also be used to enhance the sensitivity of the gas sensor [7]. In fact, the interaction between the components of the metal oxide composite material can lead to a change in the electronic structure of the nanocrystals and, hence, affect their reactivity. For example, the contact between p and n-type semiconductors induces the build-up of an inner electric field at the p-n junction interface which could modify the properties of the resulting material. It is remarkable that the selectivity

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of the sensor response can be significantly improved by varying the composition [20,21], structure [22] and operating temperature [20] of such nanocomposite sensors. Indeed, various authors have reported selective and sensitive gas sensing characteristics of composite oxide materials prepared by different methods [23–26].

Zinc oxide (ZnO), an n-type semiconductor, has been considered as a viable material for gas sensors because of its high electrochemical stability, non-toxicity, suitability to doping and low cost [27]. It has been extendedly used as effective sensing material for gases such as H_2 , CO, NO_2 and acetone [28–31]. Various morphologies (nanorods [32], nanowires [33], nanoplates [34], etc.) of nanostructured ZnO have been synthesized by different methods and tested for gas sensor applications. It was found that the sensing performance strongly depends on the morphology, which, in turn, depends on the synthesis route. Nonetheless, ZnO has demonstrated some drawbacks, such as high working temperature (between 400 and 500 °C) and poor selectivity.

As a p-type wide-bandgap semiconductor, nickel oxide (NiO) has attracted increasing attention due to its excellent chemical stability and good electrical properties. In addition, NiO is a p-type semiconductor which is widely used for gas sensor applications [35] for such gases as NO_2 [36], H_2 [37], formaldehyde [38] and NH_3 [39]. Like all p-type semiconductors, the sensitivity of NiO is very low and, therefore, needs to be improved. Thus, recent years, a lot of effort has been geared towards the synthesis of nanostructured NiO with various morphologies in order to improve its performances [40–42].

As far as NiO–ZnO composites are concerned, several works have been reported in the literature on their use as gas sensors synthesized by various techniques [43–45]. In most cases; the formation of a p–n junction clearly affects the sensing properties. However, this p–n junction directly depends on the electrical properties of each component (NiO and ZnO for this specific case). To the best of our knowledge, we are reporting for the first time the investigation of the gas sensing properties of $Ni_{0.9}Zn_{0.1}O/ZnO$ nanocomposites forming intimate heterojunctions. This material was obtained at a relatively low temperature (500 °C) by the thermal decomposition of the corresponding nickel zinc malonate prepared by co-precipitation. In fact, the insertion of Zn in the NiO structure affects the NiO electrical properties thereby changing the resulting p–n junction effect on the gas sensing property of the target composite material.

2. Experimental

2.1. Synthesis of the materials

The ZnO, $Ni_{0.9}Zn_{0.1}O$ and $Ni_{0.9}Zn_{0.1}O/ZnO$ nanoparticles were synthesized by a simple co-precipitation followed by thermal decomposition following the procedure reported elsewhere in our previous paper [46]. For instance, $Ni_{0.9}Zn_{0.1}O/ZnO$ was obtained via the thermal decomposition under air flow at 500 °C for 1 h, of corresponding mixed metal malonate co-precipitated in aqueous solution by mixing the malonate ligand with the nickel and zinc ions in the desired proportion (Zn/Ni = 1.5). For doped material, however, the nickel and zinc ratio was Zn/Ni = 0.11. As was clearly established in our previous paper [46], the Zn/Ni = 0.11 ratio corresponds to the solubility limit (~10%) of Zn in NiO.

For gas-sensing measurements, the as prepared $Ni_{0.9}Zn_{0.1}O$, ZnO and $Ni_{0.9}Zn_{0.1}O/ZnO$ powders were deposited as thick films on alumina substrates fitted with interdigitated gold electrodes and a platinum heating element, using a simple drop-coating of each corresponding ink. The ink was prepared by mixing the metal oxide powder with distilled water and the resulting solution (charged with 10% of metal oxide) was placed for 24 h in a TURBULA Shaker-

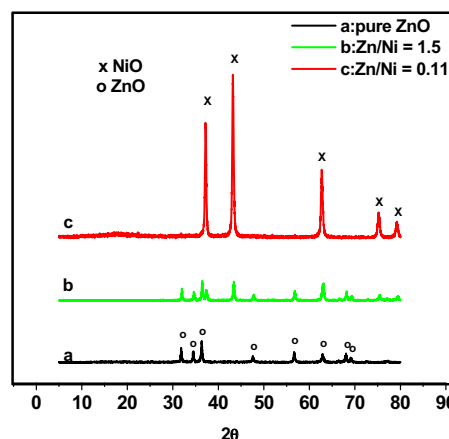


Fig. 1. Powder XRD of pure ZnO (a), Zn/Ni = 1.5 (b) and Zn/Ni = 0.11 (c).

Mixer to separate the agglomerated particles. Finally, the thick films were annealed at 400 °C for 1 h in open air. Before exposure to gases, the sensor was left for 3 h under air flow (50% of humidity) in the Teflon chamber at the highest temperature of the tests (400 °C) in order to stabilize the baseline resistance.

2.2. Characterization of the materials

The powder X-ray diffraction (PXRD) data for the decomposition products were collected at room temperature with a D5000 Siemens Kristalloflex θ -2 θ powder diffractometer which has a Bragg-Brentano geometry. It is also equipped with a graphite-monochromated Cu-K α ($\lambda = 1.54056$ Å) radiation, a standard scintillation counter detector and an automatic sample changer which can accommodate 30 samples. For the experiment, the decomposition product was spread out on a flat silicon plate in such a manner as to avoid preferred orientations. The patterns were recorded in the range of 5–80° with a scan step of 0.02 (2 θ) and a 2 s step⁻¹ acquisition interval. These patterns were compared to those of NiO and ZnO patterns of the ICDD using the HighScore Plus software for phase identification. The recorded patterns were also indexed and the unit cell refined by using the STOE WinXPOW software package (STOE WinXPOW Version 1.10; STOE & Cie GmbH, Darmstadt, Germany, 2002) [47]. The morphology of the particles deposited on the alumina substrate was determined by Scanning Electron Microscopy (SEM) using a high resolution FEG JEOL7600F with an accelerating voltage of 5 kV. The images were obtained with the semi-inlens secondary electron detector. SIMS depth profiles were made using a ToF-SIMS 5 (IONTOF GmbH, Münster, Germany) time-of-flight secondary ion mass spectrometer. The instrument is equipped with a Cs⁺ gun as sputtering source and a liquid metal ion gun (Bi) as analytical source both mounted at 45° with respect to the sample surface. The time-of-flight mass analyzer is perpendicular to the sample surface. Depth profiles were carried out in the 'non-interlaced' mode, consisting of period of 3.28 s of 30 keV Bi₅⁺ primary ions pulses for SIMS analysis followed by periods of 3.28 s of continuous Cs⁺ ion sputtering. Then, 25 s pauses whereas low energy electrons are sent to the sample in order to recover the initial surface potential. The Cs⁺ ion source was operated at 2 keV with a d.c. current of 160 nA. For depth profiling, the focused Cs beam of primary ions was rastered over an area of typically 450 × 450 μm^2 . A pulsed beam of 30 keV Bi₅⁺ (a.c. current of 0.12 pA) ions was employed to provide mass spectra from a 150 × 150 μm^2 area in the centre of the sputter crater. Charge compensation was done by electron flood gun ($E_k = 20$ eV). All data analyses were carried out using the software supplied by the instrument manufacturer, SurfaceLab (version 6.5).

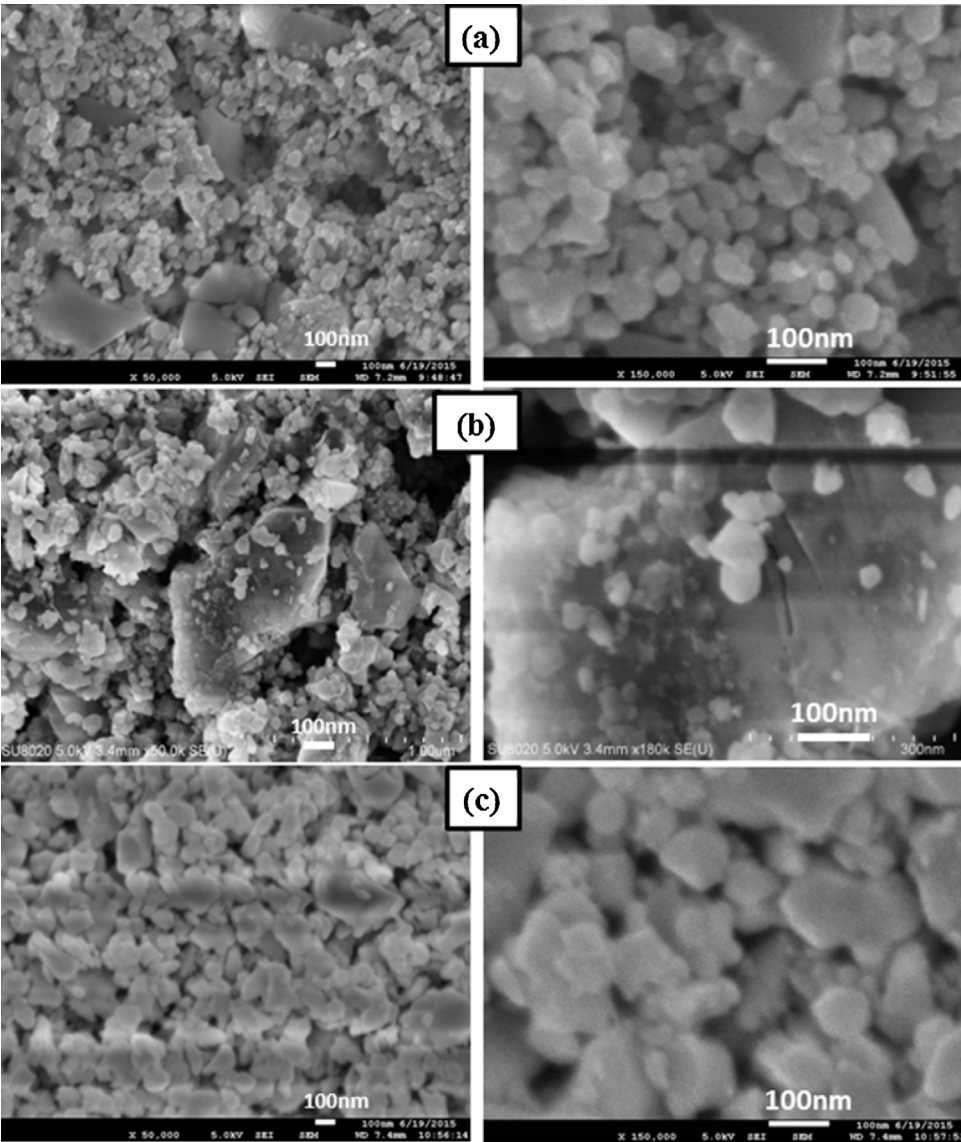


Fig. 2. SEM images of ZnO (a), Ni_{0.9}Zn_{0.1}O/ZnO (b) and Ni_{0.9}Zn_{0.1}O (c).

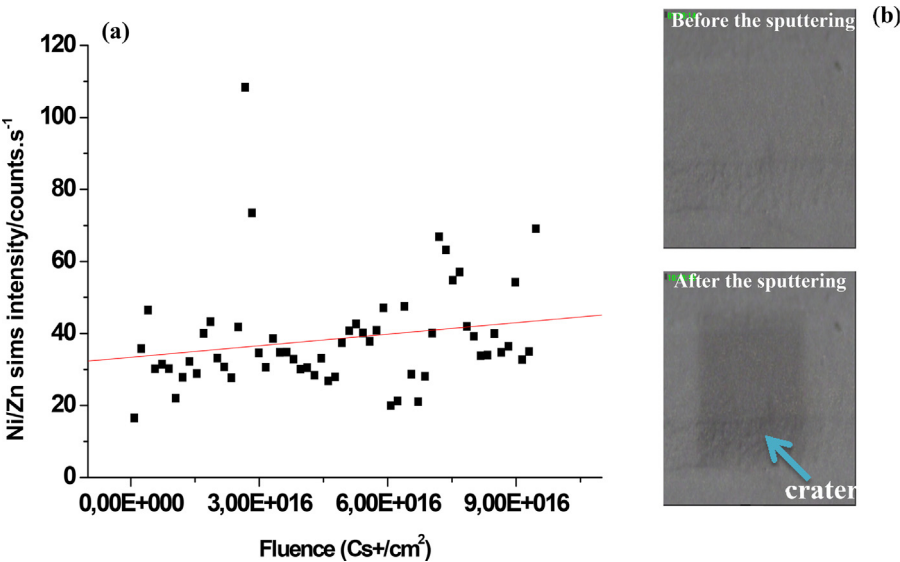


Fig. 3. Ratio of the Ni⁺ intensity to the Zn⁺ intensity with increasing Cs⁺ ion fluence for Ni_{0.9}Zn_{0.1}O (a) and image before and after the sputtering (b).

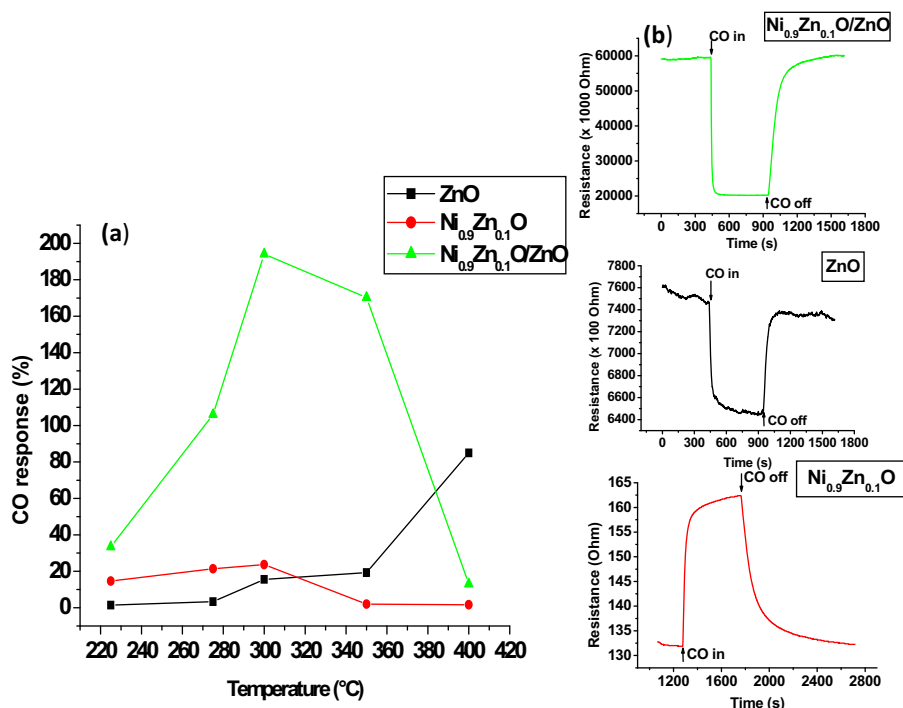


Fig. 4. Response of ZnO, $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ and $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ to 300 ppm of CO at different operating temperatures (a) and their dynamic response at 300 °C (b).

2.3. Gas-sensing tests

The measurements of the electrical resistances of the sensors in the presence of the target gases were made in a Teflon chamber. The sensors are connected to a tailor-made system to measure their electrical resistance. Moist air (50% relative humidity at 20 °C) was used as reference gas in the tests. After the electrical resistance of the sensors was stable in reference air (R_{air}) and then waiting for another 20 min, NO_2 , H_2 or CO in air diluted with the reference moist air to get the desired target gas concentrations, were introduced into the chamber. R_{gas} is obtained when the sensor resistance is stable in the presence of target gas. The sensor response (S) for ZnO and $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$, which are n-type semiconductors is defined by $S = (R_{\text{gas}}/R_{\text{air}} - 1) \times 100$ for oxidizing gases (the resistance increases after the reduction of the target gas) and $S = (R_{\text{air}}/R_{\text{gas}} - 1) \times 100$ for reducing gases (the resistance decreases after the oxidation of the target gas). For $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$, which is p-type semiconductor, $S = (R_{\text{gas}}/R_{\text{air}} - 1) \times 100$ for reducing gases (the resistance increases after the oxidation of the target gas) and $S = (R_{\text{air}}/R_{\text{gas}} - 1) \times 100$ for oxidizing gases (the resistance decreases after the reduction of the target gas). These measurements were carried out at different operating temperatures ranging from 225 to 350 °C. Response time is the time it takes sensor to reach 90% of its steady-state gain value after introduction of target gas. The recovery time is the time that it takes the sensor to be within 10% of the value it had before exposure to the target gas.

3. Results and discussion

3.1. Structure, morphology and composition

The decomposition products obtained after pyrolysis of the precursors in air at 500 °C for 1 h were analyzed by X-Ray diffraction (XRD). As evident from Fig. 1, the diffractograms of (a) and (c) show one single phase each, identified as hexagonal ZnO (P63mc, $a = 3.2490(1)$ Å and $c = 5.2052(6)$ Å) and cubic NiO ($Fm-3m$, $a = 4.187$ Å) respectively. The absence of any of zinc phase (Zn metal

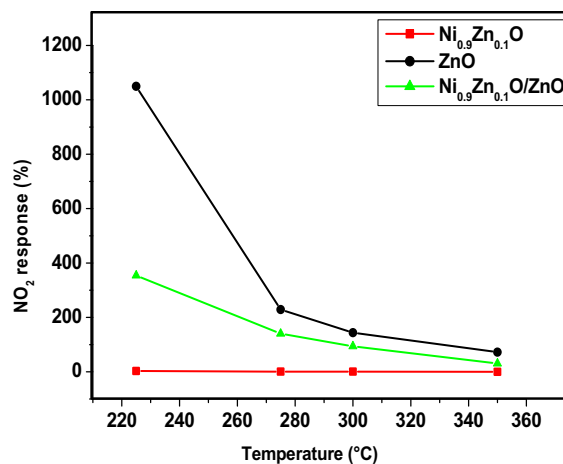


Fig. 5. Response of ZnO, $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ and $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ to 5 ppm of NO_2 at different operating temperatures.

and ZnO) in the pattern (c) can be explained by the fact that during pyrolysis, the Zn was incorporated in the NiO crystal structure by substituting Ni. Moreover, the lattice parameter of NiO obtained here (4.187 Å) is greater than that of pure NiO (4177 Å) [46]. The diffractogram of sample (b) shows two different phases identified as cubic NiO and hexagonal ZnO which indicate the formation of the composite material. The lattice parameter of NiO is 4.190 Å which is different from the pure NiO (4177 Å). This difference again suggests the partial insertion of Zn into the NiO structure since the radius of Zn^{2+} (74 pm) is slightly greater than that of Ni^{2+} (69 pm) [48]. This result undoubtedly excludes the formation of a simple NiO/ZnO composite material, rather favoring the formation of a $\text{Ni}_{1-x}\text{Zn}_x/\text{ZnO}$ composite ($x \sim 0.1$) which will be tested for the first time as gas sensor.

The morphology of all the metal oxide thick films was investigated by SEM analyses. As is evident from Fig. 2, the samples, in general, show undefined morphology. However, ZnO (a) and

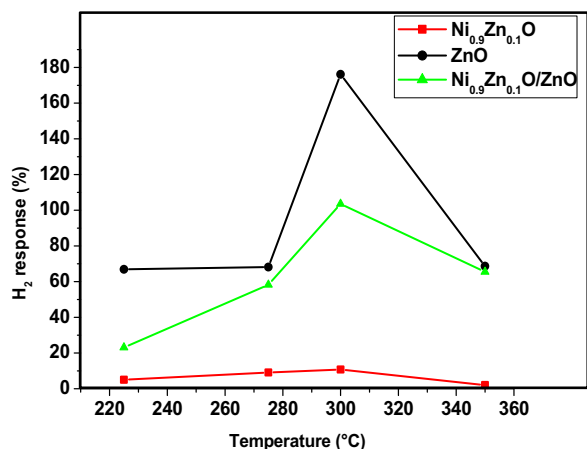


Fig. 6. Response of ZnO, Ni_{0.9}Zn_{0.1}O and Ni_{0.9}Zn_{0.1}O/ZnO to 300 ppm of H₂ at different operating temperatures.

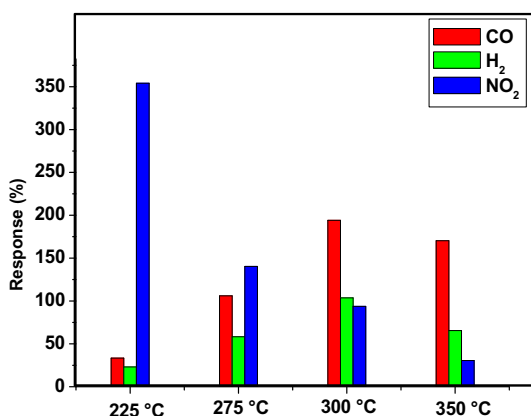


Fig. 7. Response of Ni_{0.9}Zn_{0.1}O/ZnO to 300 ppm of CO, 300 ppm of H₂ and 5 ppm of NO₂ at different operating temperatures.

Ni_{0.9}Zn_{0.1}O/ZnO (b) are almost spherical while Ni_{0.9}Zn_{0.1}O (c) is more similar to small pellets. It is also evident that the ZnO and Ni_{0.9}Zn_{0.1}O/ZnO particles are smaller (average size was estimated to 30 nm) than those of Ni_{0.9}Zn_{0.1}O. It is, also, important to mention that the very large particles observed in Fig. 2(a) and (b) were identified as constituting the substrate (alumina) by the EDX mapping.

A deeper insight into the composition of the system was obtained by ToF-SIMS, and a representative in-depth profile is shown in Fig. 3. A rough estimate of the depth of the crater (picture on Fig. 3(b)) based on the sputter yield of the oxides gives around 10 nm. As can be seen, the Ni_{0.9}Zn_{0.1}O shows an approximately homogeneous composition in the depth. As observed from the Ni⁺/Zn⁺ ion intensity ratio in Fig. 3(a), despite the statistical variation, the best linear regression of the data is almost a constant. However the signal of Ni⁺ is higher than that of Zn⁺ and this can be explained by the difference of ionization probability of the considered elements. This composition uniformity was also observed in the composite material (not shown).

3.2. Gas-sensing tests

The first test carried out was addressed to the study of the response to 300 ppm of carbon monoxide (CO) gas for the as prepared three sensors at different working temperatures. In fact, it is well-known that the operating temperature is an important parameter for a metal oxide semiconductor and the gas response is highly dependent on operating temperature. The resistance of ZnO and

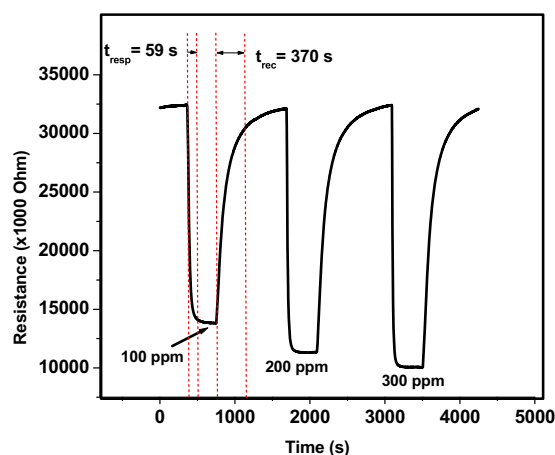


Fig. 8. Resistance variation of Ni_{0.9}Zn_{0.1}O/ZnO in the presence of CO at 300 °C (100, 200 and 300 ppm, RH = 50%).

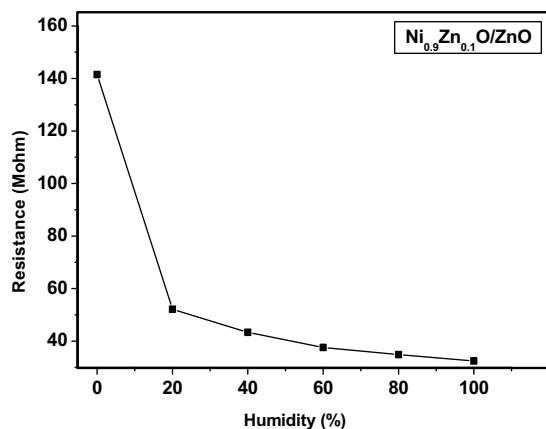


Fig. 9. Variation of baseline resistance of Ni_{0.9}Zn_{0.1}O/ZnO at different relative humidity levels.

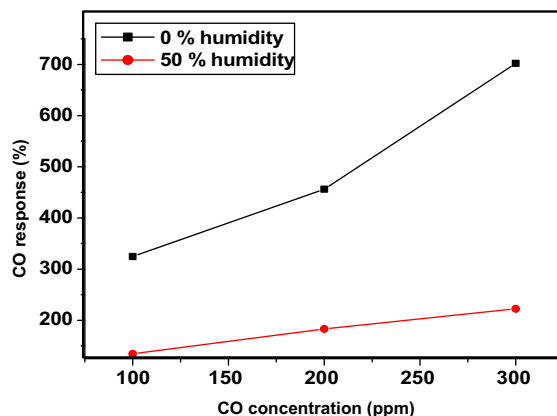


Fig. 10. Response of Ni_{0.9}Zn_{0.1}O/ZnO to 300 ppm of CO at 300 °C in the presence and in the absence of humidity.

Ni_{0.9}Zn_{0.1}O/ZnO, both which are n-type semiconductors decreases in the presence of CO but increases for Ni_{0.9}Zn_{0.1}O, a p-type semiconductor (Fig. 4(b)). As Fig. 4(a) indicates, the response of the sensor based on Ni_{0.9}Zn_{0.1}O/ZnO to 300 ppm of CO increases rapidly with an increase in the operating temperature up to 300 °C and then decreases with a further increase in the operating temperature. This implies that this sensor reaches its maximum response of 194% at 300 °C (the optimal operating temperature). A similar

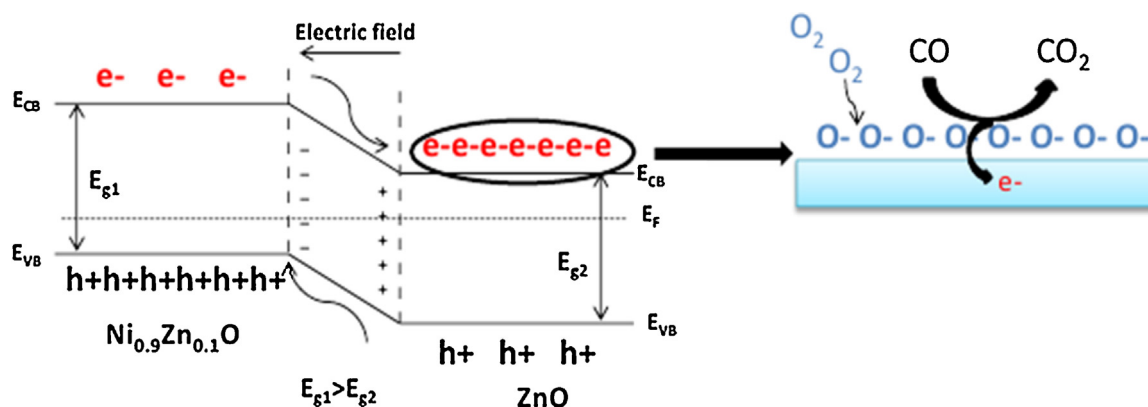


Fig. 11. Sensing mechanism.

behavior could be observed in the case of the sensor based on $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ but its maximum response at optimal operating temperature (300°C) is 24% which is 8 times lower than that of sensor based on $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$. In the case of pure ZnO, however, we observe an increase of response with increasing operating temperature in the range of the temperature chosen for the test. At 400°C , the response is 85% which is 2.2 times less than that of the sensor based on $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ at 300°C . From these results it can be observed that the sensor based on $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ compared to $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ and ZnO, shows a strong increase of response to 300 ppm of CO and a drop in the maximum operating temperature. This suggests an influence of the formation of heterojunction on the gas sensing response and on the optimal operating temperature. The existing of the heterojunction can be proved by the dynamic response to CO at 300°C for the three sensors (Fig. 4(b)), where it can be seen that the baseline resistance in air of the composite material ($R_{\text{air}} = 60\text{ M}\Omega$) is far higher than that of the single components (ZnO: $R_{\text{air}} = 750\text{ k}\Omega$, $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$: $R_{\text{air}} = 131\text{ }\Omega$). This kind of heterojunction effect has also been reported for other heterojunction systems like CuO–NiO [49] and Fe_2O_3 –NiO [50].

The same response studies were carried out on the three different sensors with 5 ppm of NO_2 . As Fig. 5 indicates, the response of the $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ based sensor and the two others ($\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ and ZnO) decreases with increasing operating temperature. The responses are 1049% for ZnO, 354% for $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ and 3% for $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ at 225°C , and 72% for ZnO, 30% for $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ and 0.1% for $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ at 350°C . For the range of temperatures covered in this work, the optimum temperature is 225°C and at this temperature the ZnO showed higher response than the other two.

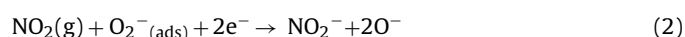
The response of the three sensors to 300 ppm of H_2 has been also investigated at different operating temperatures and the results are shown in Fig. 6. The response of the 3 kinds of sensors follows the same trend, increasing with temperature up to 300°C and then decreasing. Among the three sensors investigated at 300°C , ZnO shows a higher response (176%) than for $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ (104%) and $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ (11%).

The low gas response of sensor based on $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$ (p-type semiconductor) compared to the others (n-type semiconductors), can be explained by the theory supported by the Hübner et al.'s calculation which states that the gas response of p-type oxide semiconductors is the square root of that of n-type oxide semiconductors with the same morphology [51]. Moreover, owing to its high conductivity ($R_{\text{air}} = 131\text{ }\Omega$) compared to others ($R_{\text{air}} = 750\text{ k}\Omega$ for ZnO and $R_{\text{air}} = 60\text{ M}\Omega$ for $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$), the variation of the resistance at the surface of the particle is negligible because the Debye length is very short and the relative charge carrier change is small when adsorbed gas exchanges electrons. The high gas response of the sensor based on composite material

($\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$) for CO specifically can be explained by the formation of heterojunction in one hand but moreover by the catalytic activity of both component (ZnO and $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}$) on the oxidation of CO in other hand [52–54]. This cumulative CO oxidation catalytic activity should favor more adsorption of CO on the surface via the active sites thus enhancing the sensor response. This will not be the case for H_2 and NO_2 .

Based on the fact that the heterojunction and catalytic activity have interesting effects on the detection of CO among the others testing gases (H_2 and NO_2), further investigations on the gas sensor properties of the nanocomposite material ($\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$) were carried out by studying its selectivity, dynamic response and the influence of humidity.

To this end and bearing in mind that selectivity is one of the most problematic properties of metal oxide gas sensors, the selectivity of $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ nanocomposite with respect to CO, NO_2 and H_2 at different operating temperatures was investigated. Fig. 7 shows the different responses of $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ to the three gases at different temperatures. These results show that at 225°C the response to 5 ppm of NO_2 (354%) is higher than that of 300 ppm of CO (33%) and 300 ppm of H_2 (23%). This implies that this sensor is at least 10.7 times and 15.4 times more sensitive to NO_2 than CO and H_2 , respectively, at 225°C which is indicative of its good selectivity to NO_2 . This good selectivity to NO_2 can be ascribed to the difference in the optimum operating temperature for each gas. At low temperature (225°C) NO_2 reacts more with the material than CO. In fact NO_2 reacts with the sensing material in two ways: directly with free electron (Eq. (1)) and with adsorbed oxygen on the surface (Eq. (2)). However the CO reacts only with the adsorbed oxygen on the surface as shown in Eq. (3). At low temperature owing to the fact that the kinetic of reaction of CO with adsorbed oxygen species is low leading to a low oxygen desorption rate and electron release, a decrease of the response to CO is expected while this phenomenon does not happen for NO_2 .



At 300°C and 350°C , the responses to 300 ppm of CO (194% and 170%, respectively) are higher than those of 300 ppm of H_2 (103% and 65%, respectively). This implies that at 300°C this sensor is 1.9 times more sensitive to CO than H_2 while at 350°C it is 2.6 times. Thus, even though at 300°C the $\text{Ni}_{0.9}\text{Zn}_{0.1}\text{O}/\text{ZnO}$ shows the highest response to CO (194%), its best selectivity to this gas relative to H_2 is, however, obtained at 350°C (2.6 times).

The response of this sensor to 5 ppm of NO_2 at 300 and 350°C are 93% and 30%, respectively. Even though the responses to 300 ppm

of CO at these temperatures are 2.1 and 5.7 times higher than the response to 5 ppm of NO₂, the selectivity against NO₂ is not evident because of the difference of the test gas concentration. However, the composite material is more sensitive to CO than ZnO and Ni_{0.9}Zn_{0.1}O at this temperature as shown in Fig. 4. This increase of response to CO at 300 °C is ascribed to the formation of a heterojunction in the composite material and to the combined catalytic activity of the components.

From these results, we can conclude that the gas sensor material based on Ni_{0.9}Zn_{0.1}O/ZnO shows a good selectivity at 225 °C for NO₂ and an increased response to CO at 300 °C.

Fig. 8 shows the dynamic and reproducible response of the Ni_{0.9}Zn_{0.1}O/ZnO gas sensor exposed to different concentrations of CO at 300 °C. As can be seen, the sensor resistance reaches an equilibrium value upon gas exposure and settles back to the original value when the test gas is vented and this indicates a good reproducibility of the sensor. The dependence of the sensor response on the concentration of CO was also noticed showing that the response time is limited by the reaction rate of CO with adsorbed oxygen species. The response of the sensor is 135, 183 and 222 for 100, 200 and 300 ppm of CO respectively. The results indicate that the gas response increases with increasing concentration of CO from 100 to 300 ppm. The response and recovery times are calculated to be 59 s and 370 s respectively.

The effect of humidity on the performance of a sensor is very important for practical applications. To investigate this effect, we have measured the baseline resistance of Ni_{0.9}Zn_{0.1}O/ZnO at 300 °C at different humidity levels. Fig. 9 shows that in the absence of humidity the baseline resistance has a highest value whereas with 20% of relative humidity, this baseline resistance drops drastically to lower values. Beyond 20% of relative humidity, the baseline resistance slightly decreases with the introduction of 40, 60, 80 and 100% of relative humidity. This implies that beyond 20% of relative humidity the response of the sensor is no longer affected by humidity which is interesting from the point of view of practical applications. To further elucidate the effect of humidity on the performance of the sensor, the response of the sensor based on Ni_{0.9}Zn_{0.1}O/ZnO in the absence of humidity (0% of relative humidity) and 50% of relative humidity was investigated for different concentrations of CO gas at 300 °C. The results are shown in Fig. 10. In the former (0% of relative humidity) the responses are 325%, 456% and 702% for 100, 200 and 300 ppm of CO, respectively. These values are, at least 2.4 times higher than those obtained in 50% of humidity (135%, 183% and 222%) for the same gas concentrations (Fig. 10). This observation can be explained as follows: firstly, in the absence of humidity all the active sites on the surface of the sensor are occupied by only adsorbed oxygen whereas in the presence of humidity some active sites are occupied by water molecules. In other words, since CO reacts with adsorbed oxygen, more oxygen adsorbed onto the surface implies that more CO will react, thus, enhancing the sensor response. Secondly, the drastic increase in the value of the baseline resistance in the absence of humidity (from 32.4 MΩ in 50% of humidity to 150 MΩ in dry air) (Fig. 9) implies that the decrease in the resistance in the presence of CO gas, due to the reinjection of electrons in the conduction band, could be more visible, thus, resulting in an increase in the sensor response in the absence of humidity.

3.3. Gas sensing mechanism

The sensing mechanism is based on the change in resistance of the sensor by the adsorption and desorption processes of oxygen molecules on the surface of oxides. In general, when an n-type metal oxide semiconductor gas sensor is exposed to air, oxygen molecules will be adsorbed onto the surface of the sensing materials and ionize into species such as O₂⁻, O⁻, and O²⁻ by reducing

electrons at the valence band of the n-type semiconductor and this will form a depletion layer on the surface. In this process, oxygen molecules act as electron acceptor to decrease the electron concentration and, thus, increase the resistance of the sensor. Ni_{0.9}Zn_{0.1}O/ZnO behaves as an n-type semiconductor with electron carriers. Upon exposure to a CO gas atmosphere, for example, at an appropriate temperature, these gas molecules will react with the adsorbed oxygen ions on its surface and release electrons, thus, decreasing the resistance of the sensor. The reactions involved are shown in Eqs. (3) and (4).



The enhancement of the sensor response for CO gas in the nanocomposite material can be ascribed to the p-n heterojunction formation. Similar results of sensor performance improvement using metal oxide hetero-structure are reported by various authors [45,55,56]. In fact, the creation of inner electric fields at the semiconductor p-n interface will allow the electrons to flow from Ni_{0.9}Zn_{0.1}O (with higher band gap) to ZnO while holes will flow in the opposite direction until the fermi level equalizes. This creates an electron depleted layer at the interface of ZnO and Ni_{0.9}Zn_{0.1}O which bends the energy band. The formation of two depleted layer, one at the surface of individual grain by the adsorption of oxygen species and other at the hetero-interface of ZnO and Ni_{0.9}Zn_{0.1}O, promotes higher oxygen adsorption on the sensor surface which might provide higher reaction sites. Therefore, by reacting with the CO gas, more electrons will be released back to the conduction band, leading eventually to an enhanced response (Fig. 11). The influence of p-n junction can also be explained as follows: the normal ambient resistance of the nanocomposite material in air (R_{air}) will be higher than that of each component (Ni_{0.9}Zn_{0.1}O: $R_{\text{air}} = 131 \Omega$, ZnO: $R_{\text{air}} = 750 \text{ k}\Omega$, Ni_{0.9}Zn_{0.1}O/ZnO: $R_{\text{air}} = 60 \text{ M}\Omega$ at 300 °C as observed in Fig. 4(b)), due to the fact that at the contact between both components, there is a depletion layer increasing the contact resistance between the grains. The overall resistance of the layer is dominated by this contact resistance which explains the drastic increase in the resistance of the composite. When a reducing gas (CO) is introduced, the electron releases from the CO oxidation will decrease the holes concentration in p-type Ni_{0.9}Zn_{0.1}O by combining with them and this will result in an increase of the electron concentration according to the law of mass action for semiconductor ($n_0 \times p_0 = n_i^2$). As a result, the concentration gradient of the same charge carrier on both sides of p-n junction is reduced. This leads to decrease of the potential barrier height of depletion layer at the interface. This result in a large decrease in the resistance, thus, increasing the sensor response compared to the situation without heterojunction.

4. Conclusion

A novel nanocomposite material, Ni_{0.9}Zn_{0.1}O/ZnO, has been successfully obtained via the thermal decomposition of mixed precursor compound synthesized using a facile coprecipitation route and its gas sensing properties investigated for many gases (CO, H₂ and NO₂). According to the XRD results the nanocomposite materials were a homogeneous mixture of Ni_{0.9}Zn_{0.1}O and ZnO, excluding the possibility of the simple NiO/ZnO nanocomposite. Enhanced sensing properties with respect to CO were observed by comparison with those of pure Ni_{0.9}Zn_{0.1}O and ZnO structures and the optimal operating temperature was found to be 300 °C. It was also found that the as synthesized composite material can be used at lower operating temperature (225 °C) as selective NO₂ gas sensor. The humidity has no significant influence on the response of sensor beyond 20%. The heterojunction between Ni_{0.9}Zn_{0.1}O and ZnO and their catalytic activities are probably the principal factor

responsible for the enhanced gas sensing performance of the composite material.

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