

Research Article

Special Effect of Urea as a Stabilizer in Thermal Immersion Method to Synthesis Porous Zinc Oxide Nanostructures

F. S. Husairi,^{1,2} Syahirah Mhd Ali,^{1,2} A. Azlinda,^{1,2} M. Rusop,^{1,3} and S. Abdullah^{1,2}

¹ NANO-SciTech Centre (NST), Institute of Science, Universiti Teknologi MARA (UiTM), 40450 Shah Alam, Selangor, Malaysia

² Faculty of Applied Sciences, Universiti Teknologi MARA (UiTM), 40450 Shah Alam, Selangor, Malaysia

³ NANO-ElecTronic Centre (NET), Faculty of Electrical Engineering, Universiti Teknologi MARA (UiTM), 40450 Shah Alam, Selangor, Malaysia

Correspondence should be addressed to F. S. Husairi; mhusairifadzilah@yahoo.com

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ZnO nanostructure was prepared by catalytic immersion method (90°C) with zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as a precursors and urea ($\text{CH}_4\text{N}_2\text{O}$) as a stabilizer. Different molarity concentration ratio of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ to $\text{CH}_4\text{N}_2\text{O}$, 2 : 1, 1 : 4, 1 : 6, and 1 : 8 is used in this work. The effect of urea concentration used during the synthesis process is discussed. The ZnO nanostructures were characterized by using field emission scanning electron microscope (FESEM), photoluminescence (PL), and *I-V* probe. Porous nanoflakes are successfully synthesized on p-type silicon substrate coated with gold layer with different size and dimension. High intensity photoluminescence (PL) at optimum concentration indicated that urea is good stabilizer to produce ZnO nanostructures with good crystallinity. Rectifying characteristics show dramatical change in turn-on voltage when the concentration of urea increases in aqueous solution. This is related to the theory about p-type doping of ZnO nanostructures by nitrogen from NH_3 .

1. Introduction

ZnO possesses unique characteristics because it is a semiconductor material with a direct band gap of 3.37 eV and large excitation binding energy (60 meV) which is important for near-ultraviolet emission and transparent conductivity. ZnO also is a piezoelectric material which is suitable for sensor and transducers application. Metal oxide semiconducting material becomes a selecting device comparing solid state sensor due to small dimension, lower cost, low power consumption, simple processing, and stable. The conductivity of metal oxide semiconducting material can be improved by introducing a doping material. The impurity used to reduce the band gap of TiO_2 and N^- and C^- dopant successfully narrows it [1]. Nitrogen is a good p-type dopant for II-VI semiconductors. Nitrogen dopant is used in metal oxide semiconductor to increase conductivity and produce p-type conductor material because it is more efficient than any other element since it contributes excess holes for conduction by forming an N-Zn(O)-N complex [2].

Differing from ZnO, it is difficult to introduce N^- in ZnO because Zn atoms always preferentially combine with O, rather than with N. Nitrogen has been known as a suitable impurity for p-type doping in ZnO. Based on theoretical study, incorporation of N in ZnO:N can be improved if we increase the chemical potential in the N source. From previous study, researchers used RF sputtering [3] technique, CVD method [4], spray pyrolysis [5], and so forth to dope ZnO with nitrogen. Most of them report that the conductivity of ZnO increases by increasing the concentration of the N source. As-grown ZnO typically has n-type conductivity with background concentrations between 10^{16} and 10^{17} cm^{-3} [4]. So, N^- is introduced in ZnO to increase the conductivity and change it into p-type conducting material.

In this paper, we report on the synthesis of ZnO:N by using the immersion method with varying molar concentration of urea in order to increase the source of N. When urea is dissolved in water, it will produce CO_2 and ammonia [6, 7]. This CO_2 will form a carbonate ion (CO_3^{2-}) which is used in ZnO formation, and ammonia molecules will supply the N

atom [8]. Due to the much lower N–H bonding energy in NH_3 , the decomposition of $\text{NH}_3\text{H}_2\text{O}$ will become the main source for nitrogen in ZnO:N formation. We found that when concentration of urea increases, the conductivity of ZnO film also increases and N_2 response becomes more effective.

2. Experimental

In this work, zinc oxide nanostructures were grown by the catalytic immersion method using zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) as a precursors and urea as a stabilizer. p-type silicon (100) as substrates was cut with the size that is $1.5 \text{ cm} \times 2.0 \text{ cm}$ and clean with acetone, methanol, and HF (48% HF: DI water (1:10)). The silicon substrate was sputter coated with 6 nm thickness of gold (Au) as a catalyst in argon plasma.

2.1. Synthesis of ZnO Nanostructures. For solution preparation, the molar ratio of the Zinc Nitrate Hexahydrate [$\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] to urea [$\text{CH}_4\text{N}_2\text{O}$] in 100 mL was varied with ratio 2:1, 1:4, 1:6, and 1:8. The aqueous solution stirred and heated at 60°C for 1 hour and follow by the ageing process for 24 hours to produce homogenous solution. After the ageing process, the silicon substrate inserted into test tube filled with 40 mL of the solutions of different molar ratio concentration. Then the test tubes were immersed in a water bath at temperature of 90°C . After 4 hours, samples were dried in 1 hour at temperature 150°C and were annealed at temperature 500°C for 1 hour. The morphology of the ZnO nanostructures was analyzed by Field Emission Scanning Electron Microscopy (JSM-7600F, FESEM). The optical properties are characterized by photoluminescence (HORIBA JOBIN YVON, PL) spectra at room temperature.

2.2. I-V Characteristics and N_2 Response Studies. ZnO nanostructures were characterized by I-V characteristic to measure electrical properties and N_2 gas response. Gold layer (as a metal) with thickness 6 nm was coated on ZnO nanostructures by using sputtering technique like in Figure 1. ZnO nanostructures were placed in tight box with inlet and outlet for N_2 gases. The current-voltage characteristics of ZnO nanostructures in vacuum and presence N_2 gas were measured by Keithely 2400 multimeter and voltage source.

3. Result and Discussion

3.1. Chemical Decomposition in Thermal Immersion Method. The particle nucleation involves two steps, nucleation and growth with the nucleation rate needs to be faster than growth rate. Growth rate that depends on the amount of reacting particles available, while nucleation will take place after supersaturation is achieved, and this is affected by the solubility of the reacting particles [9]. Urea is highly soluble in water, and when urea which is one of amine groups dissolved in water, it will slowly displacement by water molucules to produce ammonia and carbonate anion

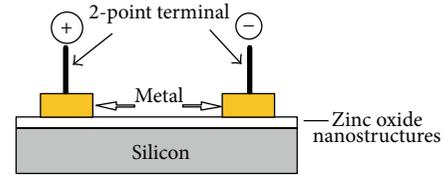
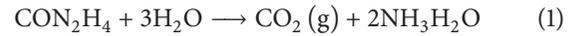


FIGURE 1: The schematic diagram of the electrodes plating on the surface of ZnO nanostructures.

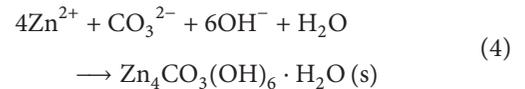
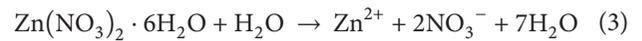
(CO_3^{2-}). The progression of chemical synthesis of ZnO in presence of urea is suggested as follows [6]:



When temperatures increase, urea decomposed and produced NH_3 . NH_4^+ ions generated from NH_3 ions will increase pH aqueous solution and support the ZnO crystal growth process:



Zinc nitrate hexahydrate will provide Zn^{2+} ions when dissolved in water as in



$\text{Zn}_4\text{CO}_3(\text{OH})_6 \cdot \text{H}_2\text{O}$ is formed during the reaction in aqueous solution and after heating at 500°C ; it decomposed ZnO [10]. The degree of supersaturation of $\text{Zn}(\text{OH})_2$ in the interfacial zone and the adsorption of organic/inorganic species on the surface of ZnO are the factors that can influence the nucleation and growth of ZnO nanostructures [11, 12].

3.2. Morphology of the Zinc Oxide Nanostructures. The presence of Au layer on Si substrate is a catalyst because it can provide an alternating energy pathway with lower activation energy. Heterogeneous nucleation promoted between Au particle and Zn and O ions is energetically favorable since the interfacial energy between the ions and adsorbed sites is lower. This is comparative to homogenous nucleation of two solid phases which has a higher activation energy barrier [13]. A large lattice match between ZnO nanostructures and Si is another factor of deposition of Au layer.

The morphology of ZnO nanostructures is observed by using Field Emission Scanning Electron Microscopy (JSM-7600F, FESEM). Different molarity of solution had been contributed to the different size and morphology ZnO nanoconflakes structures. Figure 2 shows that the FESEM images of ZnO nanostructures had grown on the silicon substrate coated with Au. As the urea concentration increases, the number of nanoflakes sheet also increases and their size reduced. At the lowest concentration of urea, the ZnO structures more to agglomerates structures and less sheet

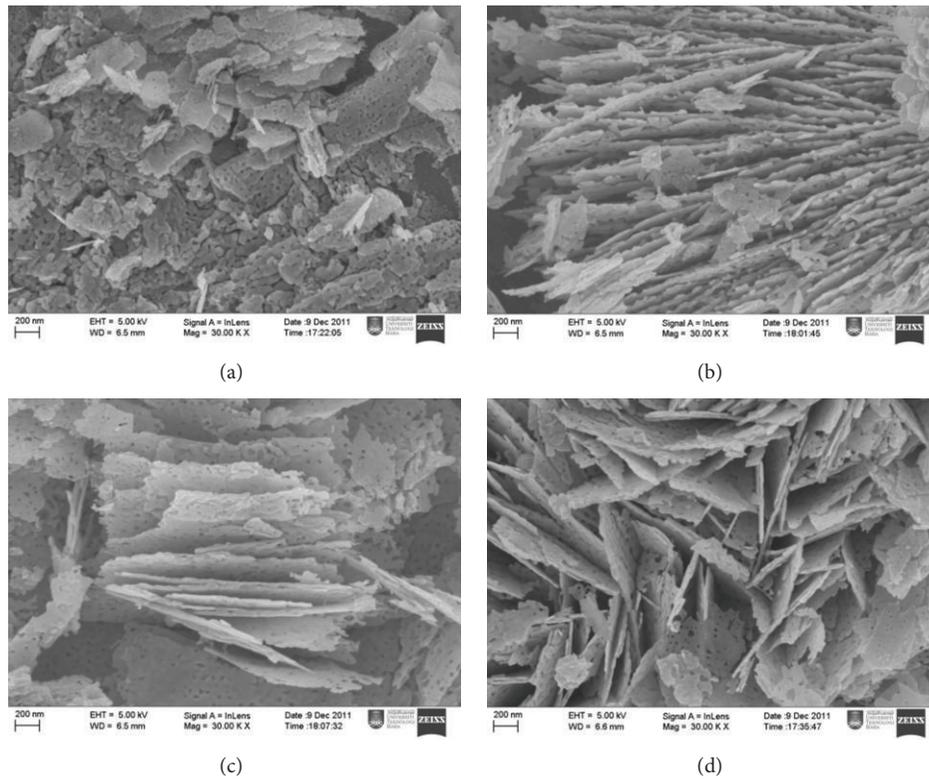


FIGURE 2: The FESEM micrographs of samples prepared at different concentration of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ to urea in aqueous solution: (a) 0.02 M : 0.01 M, (b) 0.01 M : 0.04 M, (c) 0.02 M : 0.06 M, and (d) 0.02 M : 0.08 M.

structures form. It may be caused by less of number carbonate ions (CO_3^{2-}) and OH^- combine with Zn^{2+} to form crystalline ZnO nanostructures. For the sample ZnO 0.01 M : Urea 0.04 M, the micrograph shows the early structures for ZnO before nanoflakes structures form. We can see that the primary nanoflakes structures with small size start to grow and no porous exists on it. From the observation on samples ZnO 0.01 M : Urea 0.06 M and ZnO 0.01 M : Urea 0.08 M as in Figures 2(c) and 2(d), the nanoflakes show a difference in the size and number as a concentration increase.

When concentration of urea (stabilizer) increases, the chance for ZnO nuclei to grow in their orientation is high. The probability of ZnO nuclei to agglomerate decreases and ZnO will form a large sheet number of nanoflakes. It can be deduced that smaller amount of Zn^{2+} ions leads to slower nucleation rate and induces smaller sheets growth. Too small a concentration may impede nuclei growth due to lack of starting material. The porous structures form on sheet flakes will increase the surface area that is good for sensing application.

3.3. Photoluminescence (PL) Spectra. Optical properties of ZnO nanostructures are being intensively studied for implementing photonic devices ZnO-based material [14]. Figure 3 shows the PL spectrum of ZnO nanoflakes structures at varying stabilizer concentrations. Two emitting bands, including strong UV emission at 385–400 nm and weak orange band

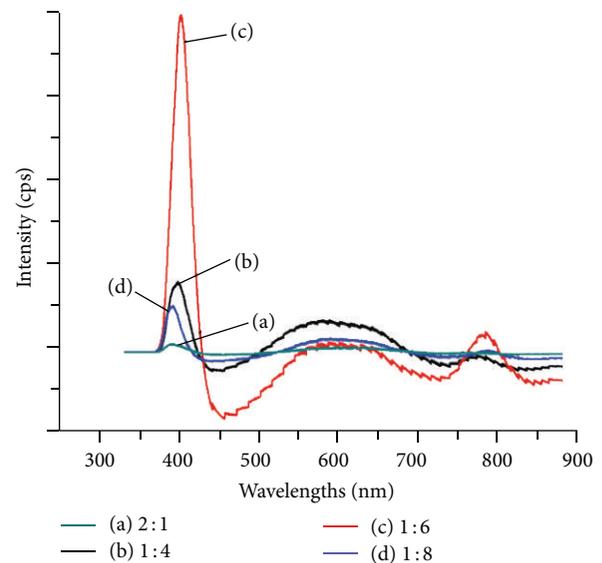


FIGURE 3: PL spectra of ZnO nanostructures at varying Zn^{2+} : Urea concentration ratio: (a) 0.02 M : 0.01 M, (b) 0.01 M : 0.04 M, (c) 0.01 M : 0.06 M, and (d) 0.01 M : 0.08 M.

(585–620 nm), were observed. Peaks centered at ultra violet band-edge attributed to near band gap emission (NBE) and emission on visible range are due to the recombination of photogenerated holes with singly ionized charge states

TABLE 1: Energy band gap ZnO nanostructures calculated based on photoluminescence (PL) spectra at UV emission.

$(\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}) : (\text{CH}_4\text{N}_2\text{O})$	Wavelength (nm)	Energy band gap (eV)
(a) 0.02 M : 0.01 M	387	3.21
(b) 0.01 M : 0.04 M	395	3.20
(c) 0.01 M : 0.06 M	399	3.11
(d) 0.01 M : 0.08 M	390	3.19

in intrinsic defects like oxygen vacancies (V_{O}) and zinc interstitial (Zn_i) [15, 16].

The excitonic emission in the UV range (from 385 to 400 nm) is an intrinsic property of the wurtzite ZnO nanostructures. It originates due to excitonic recombination where electrons come back after being excited to this energy level in band gap and associate with a hole to form a pair of exciton. It can be seen that the emission at 387 nm (3.21 eV) shifts to 399 nm (3.11 eV) for ZnO 0.01 M : Urea 0.06 M before shifted back to short wavelength (Table 1). Due to quantum confinement effect theory, the energy emission shifts to higher energy when the size of the nanostructures decreases [17]. Emission energy of this band edge emission (NBE) obeys an inverse dependence on the size of nanostructures because it tale with FESEM result which nanoflake increase until optimum ratio (1 to 6) before reduced back. Besides, the shift of the exciton band to lower energy may be attributed to increase the carrier concentration closed to the valence band in the band gap [18] and the reducing of band gap [19]. When concentration of stabilizer increases (precursor concentration constant), the intensity of UV emission also increases until lowest energy, 3.11 eV. That means that the crystalline properties of the films improved and the intrinsic donor defects, such as V_{O} and Zn_i , decreased [16]. At low urea concentration (less number of N atom), the formation of ZnO:N is lower. This will produce the structural defect because of deficiency of oxygen to form the correct ZnO structures in the sample. The optimum stabilizer ratio was found at 0.01 M of precursor to 0.06 M because the intensity UV emission produced is higher compared to others.

The emission near the yellow region most probably is caused by two factors. Firstly, it can be caused by an excess of oxygen, and the presence of hydroxyl (OH) group which found in ZnO was synthesized by using thermal immersion method [20, 21]. The formation of interstitial oxygen ions is given by aqueous chemical growth because this is an oxygen-rich growth method for ZnO [22]. Secondly, it may be due to presence of deep-level defect in ZnO nanostructure layer [23], where it can be reduced substantially by thermal treatment like annealing [24]. According to the PL spectra, ZnO grew at lowest stabilizer concentration ($\text{CH}_4\text{N}_2\text{O}$) solution (0.01 M with 0.02 M of $\text{Zn}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$), and a weak violet emission was observed.

3.4. I-V Characteristics of Zinc Oxide Nanostructures. The I-V characteristic of ZnO nanostructures is measured by using 2-point probe in vacuum condition. Figure 4 shows

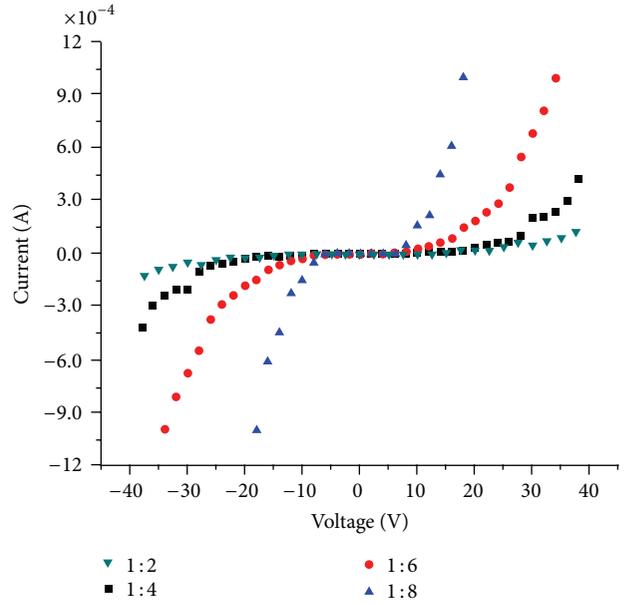


FIGURE 4: I-V characteristics of ZnO nanostructures with Au contact in air ambient.

the results of the I-V measurements of ZnO deposited on p-Si substrate. Rectifying behavior is clearly illustrated for all samples produced but different in turn-on voltage value. Rectifying behavior for all samples existence because by the junction form at interface of ZnO film and p-Si substrate [25]. ZnO exhibits n-type semiconductors due to their dominant donor defects, such as oxygen vacancies and Zn interstitials [2]. The turn-on voltage decreases as the molar ratio of urea to zinc precursor increases. The observed values of turn-on voltage are 3.27 V, 2.79 V, 1.58 V, and 1.42 V for 2 : 1, 1 : 4, 1 : 6, and 1 : 8, respectively. This negative trend is possible when the film is getting doped with “N.” The decreasing of resistance can be related to two aspects: the first one reduced net compensation between holes and electrons due to reduction of oxygen vacancy, and the second one is doping of N into the lattice [26, 27].

Based on the theoretical study of the chemical trends in the defect energy levels by Kobayashi et al. [28], N would produce a shallow acceptor level in ZnO to form ZnO:N form. This prediction made is based on the theoretical study of the chemical trends in the defect energy levels where the substitutional impurities are considered in a number of wurtzite-structured semiconductors. According to that finding, when the number of nitrogen in ZnO structures increases, the numbers of free carriers also increase. Beside, the introduction of “nitrogen” as a p-type dopant is more efficient than any other element due to formation of an N-Zn(O)-N complex and contributes excess “holes” for conduction [2].

Early, the sample may exhibit an n-type. As the N atom present in ZnO film increased, it will be activated as electron acceptors of the ZnO:N thin film and turn to p-type ZnO behavior. Minimum turn-on voltage was observed at high concentration of urea, ZnO 0.01 M : Urea 0.08 M, 1.42 V, so

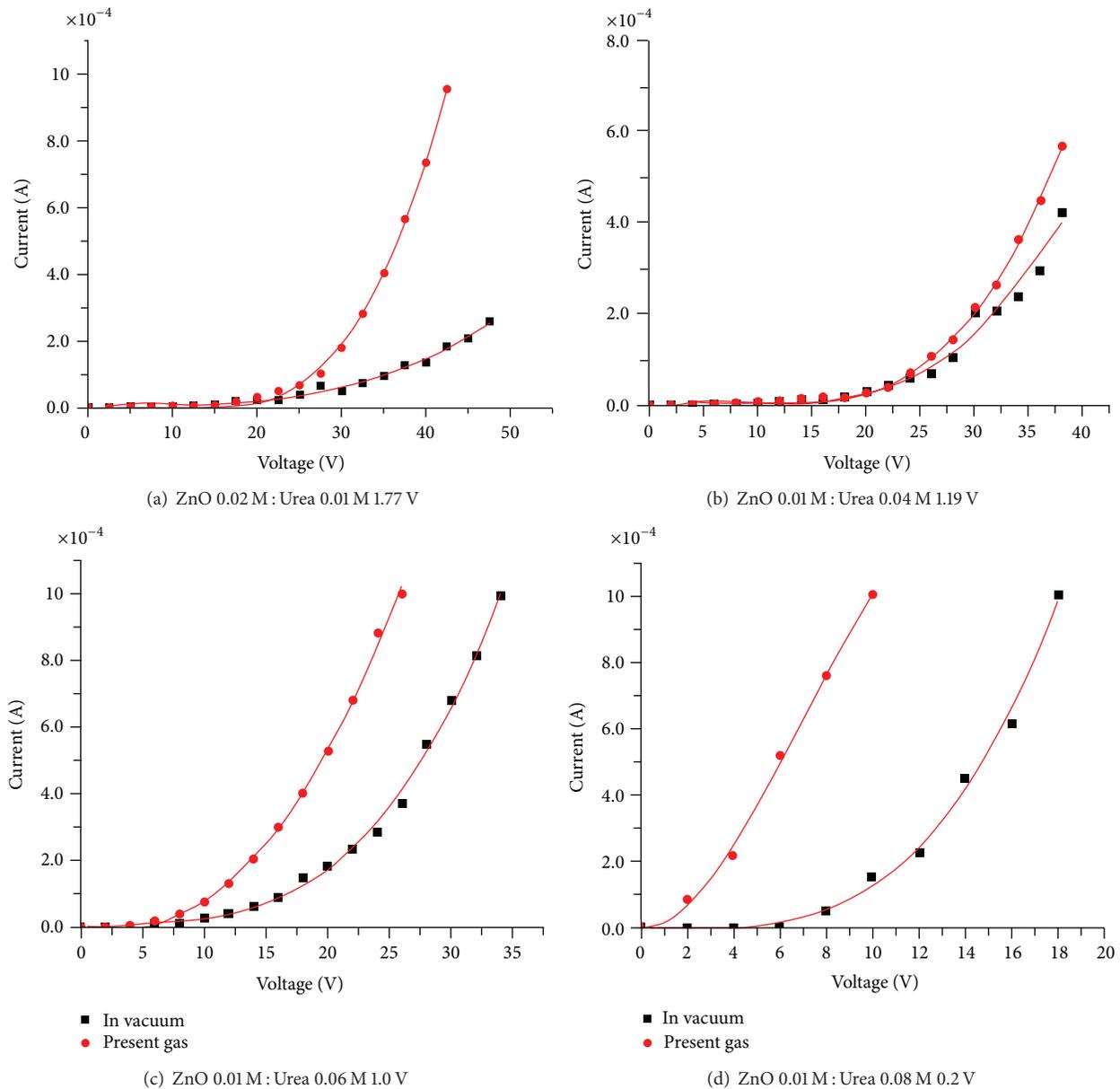


FIGURE 5: I - V characteristics of ZnO nanostructures when exposed to N_2 gas ambient.

the minimum operating potential for ZnO nanostructures as a based material because of increasing of oxidation and N dilution into ZnO lattice as a dopand. When the nitrogen atom in solution increased, the donor defects insufficient to compensate for the N substitutional acceptor and leads to an acceptor density increases. N atom that is present in ZnO film is activated as electrons acceptor of ZnO:N thin film. More N atoms is present will lead to the further pronounced p-type ZnO behavior [25].

3.5. N_2 Response of ZnO Nanoflakes Synthesized at Different Molar Concentration of Urea. Most of research in gas sensor were done more to gases which contain oxygen molecule and explained the response based of free electron on oxygen

ions [29]. In this project, we test the N_2 gas response by ZnO nanostructures which had a modification in term of conductivity (doping). Figure 5 shows the change of I - V characteristics of ZnO nanostructures with variety of molarity of urea when exposed to N_2 gas. From that figure, we can see that all the samples give a response to N_2 gas in different sensitivity with good response detected at sample ZnO 0.01 M : Urea 0.08 M.

Table 2 shows the changes of turn-on voltage and sensitivity of the samples when exposed to N_2 gas. The detecting of chemical species usually determined by surface defect which may act as an active site to absorb the testing gas, and the ratio of area to volume [30]. The basis of the sensing mechanism for metal oxide is chemoresistivity, which changes in term of conductance or resistance when surface chemical reacts

TABLE 2: Sensitivity of ZnO nanostructures prepared at different ratio concentration of N₂ gas by I-V characteristics of testing.

Samples	Turn-on voltage, V _O		Response, S S = ((V _b - V _a)/V _b) × 100%
	Before exposed, V _b	After exposed, V _a	
ZnO 0.02 M : Urea 0.01 M	3.27 V	1.77 V	45.87
ZnO 0.01 M : Urea 0.04 M	2.79 V	1.19 V	57.35
ZnO 0.01 M : Urea 0.06 M	1.58 V	0.53 V	66.46
ZnO 0.01 M : Urea 0.08 M	1.42 V	0.21 V	85.21

with testing gases. When ZnO is exposed to N₂ gas, the N molecules will be adsorbed on the ZnO surface and cause the change in chemical bonding and carrier concentration of ZnO:N. Molecules N arrived intact at the surface, so high concentration of (N₂)_O centers will be observed at Zn-rich conditions [3]. Generally, nitrogen exists in two forms, (N)_O which acts as an acceptor and (N₂)_O which acts as donor in ZnO:N [31]. (N)_O formed when N substitutes at O site, and (N₂)_O formed when N₂ substitute at O site also. Based on theoretical calculations, as-grown ZnO:N films contain higher electron concentration than undoped ZnO because (N₂)_O has a smaller formation energy than (N)_O. That makes the resistance of film decreased when ZnO:N, exposed to N₂ gas especially for ZnO 0.01 M : Urea 0.08 M. The responses were increased by the conductivity of ZnO film. The result of ZnO film responses was detailed in Table 2.

4. Conclusion

Porous ZnO nanoflakes were successfully synthesized at various molar concentrations of urea by using thermal immersion method. The analysis of FESEM images illustrates nanoflake formation on silicon substrate. The PL spectra show that the ZnO nanoflake has two emission bands: one is relatively strong UV emission centered about 390–400 nm, and the other is weaker emission observed in the green-yellow range of the visible spectrum with an emission peak about 600 nm. The study of PL and I-V measurements has demonstrated that an n-type ZnO doped with N (ZnO:N) thin film is successfully fabricated by using simple immersion method. It is shown that, by controlling concentration of urea, we can obtain a good conductivity and N₂ gas response of ZnO nanostructures. The minimum value of rectifying characteristic is observed at high concentration of urea in solution. It can be concluded that when high concentration of nitrogen atoms is introduced in the film, the p-type ZnO nanostructure behavior is observed.

Acknowledgments

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