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High photocurrent gain in NiO thin film/M-doped ZnO nanorods (M= Ag, Cd and Ni) heterojunction based ultraviolet photodiodes

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Abstract

The thermal evaporation method has been used to deposit p-type NiO thin film, which was combined with hydrothermally grown n-type pure and M-doped ZnO nanorods (M= Ag, Cd and Ni) to fabricate a high performance p-n heterojunction ultraviolet photodiodes. The fabricated photodiodes show high rectification ratio and relatively low leakage current. The p-NiO/n-Zn_{0.94}Ag_{0.06}O heterojunction photodiode displays the highest photocurrent gain ($\sim 1.52 \times 10^4$), a photoresponsivity of $\sim 4.48 \times 10^3 \text{ AW}^{-1}$ and a photosensitivity of ~ 13.56 compared with the other fabricated photodiodes. The predominated transport mechanisms of the p-n heterojunction ultraviolet photodiodes at low and high applied forward bias may be recombination-tunneling and space-charge limited current, respectively.

Keywords: ZnO nanorods, NiO thin film, Ultraviolet photodiode, Photocurrent gain.

1. Introduction

Wide bandgap semiconductor based ultraviolet (UV) photodiodes have attracted significant interest compared with UV photodiodes based on narrow bandgap semiconductors due to their high quantum efficiency, enhanced photocurrent gain and large UV/visible rejection ratio[1-5]. Wide bandgap semiconductor nanostructures such as nanorods are promising for the improvement of UV photodiodes since they have a large surface area to volume ratio which leads to high internal photoconductivity gain due to the surface enhanced electron-hole separation efficiency [6,7]. Among the different semiconductors, zinc oxide (ZnO) due to its wide band gap ($E_g=3.37$ eV), large exciton binding energy (60 meV) and strong cohesive energy (1.89 eV) has been widely used as an n-type semiconductor for the fabrication of UV photodiodes [8-11]. Recently, p-n heterojunctions have been highly adopted as appropriate structures for UV photodetectors. However, since the growth of reproducible p-type ZnO is still under development, hybrid structures of ZnO with other p-type semiconductors could increase the responsivity of UV photodiodes [12-14]. Nickel oxide (NiO) is another wide bandgap semiconductor ($E_g=3.7$ eV) which has shown potential applications in nanoscale optoelectronic devices such as photodiodes and field effect transistors [15,16]. These properties make NiO a suitable candidate for the fabrication of p-NiO/n-ZnO heterojunction based UV photodiodes [17-19]. Alternative inexpensive fabrication techniques are usually needed to realize large scale, mass production and low cost UV photodiodes with high performance. So far, there are only a few reports about using the thermal evaporation method for the synthesis of NiO thin film [20,21]. Moreover, there has been no detailed investigation about using the doping of ZnO nanorods with different elements to improve the photo-response characteristics of p-NiO thin film/n-ZnO nanorods heterojunction UV photodiodes.

In the present work, we have synthesized NiO thin film using the thermal evaporation method to fabricate p-n heterojunction with hydrothermally grown ZnO nanorods which results in low leakage current and high rectification ratio due to the improved properties of the heterojunction interface. Moreover, the effect of the doping using silver (Ag), cadmium (Cd) and nickel (Ni) elements in ZnO nanorods on the photo-

response characteristics of p-NiO/n-ZnO heterojunction UV photodiode has been studied. The materials were characterized by X-ray diffraction (XRD), field emission scanning electron microscope (FESEM) and photoluminescence (PL). The photo-response characteristics were investigated under UV illumination using semiconductor parameter analyzer. Furthermore, the obtained results were compared to those achieved from other UV photodetectors based on similar heterojunction grown by other methods.

2. Experimental

2.1. Samples preparation

Commercially available fluorine doped tin oxide (FTO) glass substrate was used in this study and all the chemicals were of analytical grade and were purchased from Sigma Aldrich. The FTO glass substrate was cleaned by sonication in acetone, deionized water, and isopropanol, respectively. Part of the substrate at the edge was covered with scotch tape in order to have a metal contact area. Then the substrate preparation technique developed by Green et al. was used to improve the quality of the grown pure and doped ZnO nanorods [22]. To grow the ZnO, $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$, $\text{Zn}_{0.94}\text{Cd}_{0.06}\text{O}$ and $\text{Zn}_{0.94}\text{Ni}_{0.06}\text{O}$ nanorods, an equimolar concentration of hexamethylenetetramine (HMT) and zinc nitrate hexahydrate (94%) with silver nitrate hexahydrate (6%) or cadmium chloride (6%) or nickel nitrate hexahydrate (6%) (0.075 M) solutions were prepared and mixed together. Then the final solutions were poured into beakers and the pretreated substrates were immersed in the solutions with the growth side facing downward. The beakers were sealed and heated in a laboratory oven at 95 °C for 5 hours. Then they were allowed to cool down at room temperature. After the growth process, the samples were rinsed with deionized water to remove any residual salts and were then dried with blowing nitrogen. To fabricate p-n heterojunction UV photodiodes, Ni film with 50 nm thickness was deposited by thermal evaporation in a vacuum chamber having a base pressure of 2×10^{-6} mbar on the top of the pure and doped ZnO nanorods. Then to oxidize the Ni film, the samples were annealed in oxygen ambient at a temperature of 400 °C for 5 hours. Finally, Ag circular contacts with

80 nm thickness and 1 mm diameter were afterwards deposited on top of the NiO thin film using thermal evaporation. The schematic diagram of p-NiO thin film/n-ZnO nanorods heterojunction photodiode is shown in Figure 1.

2.2. Characterization

The structural and morphological properties of heterojunctions were examined by X-ray diffraction (Phillips PW 1729 powder diffractometer using CuK α radiation) and field emission scanning electron microscope (LEO 1550 Gemini). The light emission features of the samples were studied by a micro-photoluminescence (μ PL) setup at room temperature. The excitation was performed by a frequency doubled Nd:YVO laser as a continuous wave excitation source, giving a wavelength of $\lambda=266$ nm. The photo-response characteristics were evaluated by semiconductor parameter analyzer (Agilent 4155B) under UV lamp illumination at $\lambda_{\text{max}}=365$ nm with an incident intensity of 8 W/m².

3. Results and discussion

3.1. Structural and morphological properties

XRD analysis has been used to study the crystal structure of the materials forming the heterojunctions. Figure 2(a) shows the XRD patterns of the p-NiO thin film/n-pure and doped ZnO nanorods heterojunctions. It can be seen that the XRD patterns display diffraction peaks that corresponded to ZnO wurtzite structure (JCPDS No. 36-1451). The pure and doped ZnO nanorods have a preferential orientation along the c-axis since the (002) peak has the highest intensity. The observed (002) peak of the doped ZnO nanorods was found to be shifted a little bit towards a lower angle for Ag (34.403°) and Cd (34.401°) doped and to a higher angle for the Ni (34.441°) doped compared to the angular position of the (002) of the pure ZnO (34.419°). This can be attributed to the fact that substitution of larger Ag (144 pm) and Cd (151 pm) ions or smaller Ni (124 pm) ions with the Zn (134 pm) ions leads to expansion or contraction of

the ZnO lattice, respectively. Also, other factors like an electric force between Ag-Zn, Cd-Zn and Ni-Zn ions may have role in lattice expansion or contraction. Moreover, the XRD patterns show the characteristic peak of NiO corresponding to the (111) plane of cubic NiO structure consistent with the diffraction file (JCPDS No. 01-1239). Furthermore, no additional peaks related to impurities phases were observed. Figure 2(b, c) show the SEM images of the pure ZnO nanorods and the NiO thin film grown on the top of the ZnO nanorods. It is clearly seen that a relatively well aligned ZnO nanorods having hexagonal faces with an average diameter of approximately 50 nm were achieved. Since the doped ZnO nanorods have relatively the same size as the pure ZnO nanorods, their SEM images are not mentioned here. Also, as it can be observed the NiO thin film exhibits a relatively smooth surface morphology with uniformly distributed grains.

3.2. Optical properties

Photoluminescence (PL) study is an appropriate analysis to gain useful information about the optical properties of different semiconductor materials [23-26]. In order to determine the luminescence properties of the pure and the doped ZnO nanorods, the PL spectra of the samples were studied at room temperature (300 K) as shown in Figure 3(a). All the spectra were taken at the same excitation power and integration time and are therefore comparable. It can be seen that for ZnO nanorods the ultraviolet emission peak called near band edge emission (NBE) is centred approximately at 3.305 eV. The NBE emission is attributed to the recombination of electron-hole pairs between conduction and valence bands from which the optical bandgap can be extracted [27]. The optical bandgap of $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$, $\text{Zn}_{0.94}\text{Cd}_{0.06}\text{O}$ and $\text{Zn}_{0.94}\text{Ni}_{0.06}\text{O}$ were estimated to be 3.292 eV, 3.299 eV and 3.294 eV, respectively, as shown in the PL spectra. Also, two broad deep level emission (DLE) peaks were observed for the ZnO nanorods and were extended approximately from 1.80 eV to 2.08 eV (DLE1, red emission) and from 2.09 eV to 2.86 eV (DLE2, green emission). The green and red emissions are ascribed to the recombination of

electron-hole pairs between the conduction band or zinc interstitial (Zn_i) energy level to oxygen vacancy (V_o) and oxygen interstitial (O_i) energy level, respectively [28,29]. To investigate the optical quality of the pure and doped ZnO nanorods, the intensity ratio I_{NBE}/I_{DLE} was measured, and is demonstrated in Figure 3(b), where I_{NBE} and I_{DLE} are the integrated intensity of the NBE and the DLE peaks, respectively. As it can be observed the doping of Ag and Cd leads to increase the optical quality of the ZnO nanorods, although the doping with Ni results in decrease of the optical quality. In general, the $Zn_{0.94}Ag_{0.06}O$ nanorods have the highest value of the I_{NBE}/I_{DLE} ratio which means that they have the best optical quality in comparison to the other samples. It seems that the presence of Ag and Cd leads to produce more energy levels close to the conduction band of the ZnO nanorods instead of deep energy levels. Therefore, most of the electron-hole recombination processes could occur between the conduction and valence bands which results in an increase of the integrated intensity of the NBE peak compared to the DLE peaks. However, doping with Ni gives rise to enhance the deep level defects in ZnO nanorods which leads to increase the integrated intensity of the DLE peaks compared to the NBE peak.

3.3. *I-V characteristics*

Figure 4 (a-d) shows the current-voltage (I-V) characteristics of the p-NiO/n-ZnO, p-NiO/n- $Zn_{0.94}Ag_{0.06}O$, p-NiO/n- $Zn_{0.94}Cd_{0.06}O$ and p-NiO/n- $Zn_{0.94}Ni_{0.06}O$ heterojunction photodiodes, respectively. The rectifying behavior of the p-n heterojunctions is obviously observed which confirms the formation of a junction barrier at the interface of the NiO thin film with the pure and doped ZnO nanorods. The threshold voltage (V_{th}), forward (I_F) and reverse (I_R) bias currents, rectification ratio (I_F/I_R) and series resistance (R_s) of the fabricated p-n heterojunctions were measured in the dark for an applied bias varying from -8.0 to $+8.0$ at room temperature and are listed in Table 1. The relatively high values of the V_{th} can be related to the high series resistance of p-n heterojunctions. The low leakage current and high rectification ratio observed in the fabricated p-n heterojunctions may be related to the formation of a proper junction interface between the NiO thin film and pure

and doped ZnO nanorods which results in low imperfections of the heterojunction interface. The thermionic emission current-voltage relationship of a p-n heterojunctions is usually written as a function of the applied voltage as [30]:

$$I = I_o (\exp (qV/nK_B T) - 1) \quad (1)$$

where I_o is the saturation current, q is the electronic charge, V is the applied voltage, n is the ideality factor, K_B is the Boltzmann's constant and T is the temperature in Kelvin. The ideality factor (n) of the heterojunctions which can be calculated from the slope of the straight line region of the forward bias in $\ln(I)$ - V plot shown in the inset of Figure 4 (a-d) is listed in Table 1. The high values of n indicate that the characteristics of the fabricated heterojunctions have deviated from the ideal diode ($n=1$) which may be attributed to the fact that the transport mechanism is not dominated by the thermionic emission, but consists of other mechanisms like defect assisted tunnelling with conventional electron-hole recombination [31].

3.4. Photo-response characteristics

Figure 5 (a-d) shows the semi-log I - V characteristics of the fabricated p-n heterojunction photodiodes in the dark and under UV illumination at room temperature. It can be seen that both reverse and forward bias currents for all the photodiodes are increased under UV light. The photosensitivity ($= I_{pc}/I_d$) of the p-NiO/n-ZnO photodiode was found to be about 3.54 and 24.67 at forward and reverse applied bias of ± 8.0 V, respectively, where I_{pc} and I_d are the current under UV illumination and in the dark, respectively. The photosensitivity of the doped heterojunction photodiodes were measured and listed in Table 2. It can be noted that p-NiO/n-doped ZnO photodiodes show a slightly higher value for the photosensitivity under forward applied bias, although the observed enhancement in the photosensitivity under reverse applied bias was much more than that obtained for the p-NiO/n-ZnO heterojunction photodiode. The photoresponsivity (R) is the current value per unit optical power and is expressed as $R = \Delta I / P_{opt}$, where $\Delta I = I_{pc} - I_d$ and P_{opt} is the

absorbed optical power. The photocurrent gain (G) is the ratio of the number of electrons collected per unit time to the number of absorbed photon per unit time. If the absorption quantum efficiency of the photon is assumed to be unity, G can be expressed as [32-34]:

$$G = R / (q/h\nu) \quad (2)$$

where q is the elementary charge and ν is the frequency of the absorbed photon. The calculated values of R and G under applied forward and reverse bias are listed in Table 2, respectively. Also, the curve of the photocurrent gain of the photodiodes as a function of forward bias voltage is shown in Figure 5. The obtained values of R and G at +8.0 V applied forward bias for the p-NiO/n-ZnO photodiode were $3.20 \times 10^3 \text{ AW}^{-1}$ and 1.09×10^4 , respectively. As it can be noted the photoresponsivity and photocurrent gain of the p-NiO/n- $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$ photodiode is increased at forward bias voltage compared to the p-NiO/n-ZnO photodiode, but these values were slightly decreased for the p-NiO/n- $\text{Zn}_{0.94}\text{Cd}_{0.06}\text{O}$ and the p-NiO/n- $\text{Zn}_{0.94}\text{Ni}_{0.06}\text{O}$ photodiodes. Also, the photoresponsivity and photocurrent gain of the p-NiO/n-doped ZnO photodiodes under reverse bias voltage is increased compared with p-NiO/n-ZnO photodiode; however, these values were much less than those obtained at applied forward bias. The photo-response characteristics obtained at applied forward bias of +8.0 V in the present work especially for the p-NiO/n- $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$ heterojunction photodiode is much higher than those reported for other p-n heterojunction photodiodes [19,34,35].

To investigate the origin of the high photocurrent gain of the fabricated heterojunction ultraviolet photodiodes at applied forward bias of +8.0 V, the log-log plot of the I-V characteristics in the dark and under UV illumination has been studied. The log-log plot of I-V characteristics of all the photodiodes under both dark and UV illumination can be divided into three separate regions as shown in Figure 6 (a-d). At low forward bias voltage, i.e. <0.3 V (region i), the current was increased linearly with the applied forward bias which is related to the ohmic conduction. By further increase in the applied forward bias to about 4.0 V (region ii), the current was enhanced exponentially which is usually observed in p-n junction diodes based on wide bandgap semiconductors due to

recombination-tunneling mechanisms [32,33]. Under UV illumination the absorbed photons in the vicinity of the heterojunctions give rise to the generation of electron-hole pairs. The enhancement in the carrier densities results in the redistribution of space charge in the depletion region of the heterojunctions, leading to shrink the barrier width [36,37]. In this case, more charge carriers are allowed to tunnel across the narrowed depletion region of the heterojunctions, increasing the current exponentially as can be seen in Figure 6. At higher applied forward bias, $>4.0\text{V}$ (region iii), the current behaves as $I \sim V^m$ relation, where m is found to be more than 4 for all the heterojunction photodiodes under UV illumination. The observed trend of power law is related to trap-assisted space charge limited current (SCLC) conduction mechanism [33,36]. Since the NiO thin film is prepared by annealing the Ni in oxygen ambient, oxygen molecules are absorbed onto the pure and doped ZnO nanorods surface by capturing free electrons [$\text{O}_2(\text{g}) + \text{e}^- \rightarrow \text{O}_2^-$], leaving behind a depletion region near the surface. Once electron-hole pairs are generated under UV illumination, the photo-generated holes sweep to the surface due to the built-in electric field to discharge the negatively charged absorbed oxygen ions [$\text{O}_2^- + \text{h}^+ \rightarrow \text{O}_2(\text{g})$], leaving the photo-induced electrons inside. The separation of electrons and holes leads to the reduction of electron-hole recombination rate. So the charge carrier lifetime increases and the photocurrent gain is enhanced [32,37]. The double effect of the recombination-tunneling and SCLC transport mechanisms results in high photocurrent gain for the fabricated p-n heterojunction ultraviolet photodiodes. Since there is more radiative electron-hole recombination between the conduction and valence band edges than the non-radiative recombination in the samples with high optical quality which can lead to increase the carrier charge lifetime [38], the p-NiO/n- $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$ heterojunction photodiode shows the highest photocurrent gain compared with other heterojunctions because $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$ nanorods has the highest optical quality.

4. Conclusion

In summary, p-NiO thin film/n- pure and doped ZnO nanorods heterojunction ultraviolet photodiodes were fabricated using a simple two-step fabrication process. Structural investigation indicated that well aligned pure and doped ZnO nanorods with

hexagonal face having a preferential orientation along the c-axis (002) have been achieved and the NiO thin film covered the nanorods with a relatively smooth surface morphology. The $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$ nanorods show the highest optical quality with the lowest optical bandgap compared to the other samples. The p-NiO/n-ZnO heterojunction photodiode shows a threshold voltage of 5.39 V with an excellent rectifying ratio (3.05×10^3). The doping of Ag element with ZnO nanorods leads to increase the photo-response characteristics of p-NiO/n-ZnO heterojunction photodiode. It was also observed that the twin effect of the recombination-tunneling and SCLC transport mechanisms results in high photocurrent gain for the fabricated p-n heterojunction ultraviolet photodiodes at applied forward bias.

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Figure 1. Schematic diagram of p-NiO thin film/n-ZnO nanorods heterojunction photodiode.

Figure 2. XRD patterns of p-NiO thin film/n-pure and doped ZnO nanorods heterojunctions (a), SEM images of pure ZnO nanorods (b) and NiO thin film (c).

Figure 3. (a) PL spectra and (b) $I_{\text{NBE}}/I_{\text{DLE}}$ ratio of ZnO, $\text{Zn}_{0.94}\text{Ag}_{0.06}\text{O}$, $\text{Zn}_{0.94}\text{Cd}_{0.06}\text{O}$ and $\text{Zn}_{0.94}\text{Ni}_{0.06}\text{O}$ nanorods.

Figure 4. The I-V characteristics of the heterojunction photodiodes in the dark. The inset shows the $\ln I$ vs. V plot for extraction of ideality factor (n) of the heterojunctions.

Figure 5. The semi-log I-V characteristics in the dark and under UV illumination and the photocurrent gain of heterojunction photodiodes as a function of forward bias voltage.

Figure 6. The log-log plot of I-V characteristics of heterojunction photodiodes in the dark and under UV illumination.

Table 1. I-V characteristics in the dark of the p-n heterojunction photodiodes.

Heterojunction	V_{th} (V)	I_F (A)	I_R (A)	I_F/I_R	n	R_s(KΩ)
p-NiO/n-ZnO	5.39	5.03×10 ⁻⁴	1.65×10 ⁻⁷	3.05×10 ³	8.10	3.32
p-NiO/n- Zn _{0.94} Ag _{0.06} O	6.28	1.43×10 ⁻⁴	4.06×10 ⁻⁷	3.52×10 ²	9.83	3.55
p-NiO/n- Zn _{0.94} Cd _{0.06} O	6.12	9.28×10 ⁻⁵	2.61×10 ⁻⁸	3.55×10 ³	8.35	3.89
p-NiO/n- Zn _{0.94} Ni _{0.06} O	5.01	3.01×10 ⁻⁴	2.76×10 ⁻⁸	1.09×10 ⁴	8.25	5.97

Table 2. Photo-response characteristics of the p-n heterojunction photodiodes at applied forward and reverse bias.

Heterojunction	Forward Bias			Reverse Bias		
	I_{pc}/I_d	R (AW⁻¹)	G	I_{pc}/I_d	R (AW⁻¹)	G
p-NiO/n-ZnO	3.54	3.20×10 ³	1.09×10 ⁴	24.67	9.76	33.18
p-NiO/n-Zn _{0.94} Ag _{0.06} O	13.56	4.48×10 ³	1.52×10 ⁴	233.01	235.52	800.16
p-NiO/n-Zn _{0.94} Cd _{0.06} O	5.35	1.01×10 ³	3.42×10 ³	181.23	11.75	39.92
p-NiO/n-Zn _{0.94} Ni _{0.06} O	4.35	2.52×10 ³	8.56×10 ³	1028.98	70.87	240.80