Research Article

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Effect of annealing temperature on the interface state density of n-ZnO nanorod/p-Si heterojunction diodes

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Abstract: The effect of post-growth annealing treatment of zinc oxide (ZnO) nanorods on the electrical properties of their heterojunction diodes (HJDs) is investigated. ZnO nanorods are synthesized by the low-temperature aqueous solution growth technique and annealed at temperatures of 400 and 600°C. The as-grown and annealed nanorods are studied by scanning electron microscopy (SEM) and photoluminescence (PL) spectroscopy. Electrical characterization of the ZnO/Si heterojunction diode is done by current–voltage (I – V) and capacitance–voltage (C – V) measurements at room temperature. The barrier height (ϕ_B), ideality factor (n), doping concentration and density of interface states (N_{SS}) are extracted. All HJDs exhibited a nonlinear behavior with rectification factors of 23, 1,596 and 309 at ± 5 V for the as-grown, 400 and 600°C-annealed nanorod HJDs, respectively. Barrier heights of 0.81 and 0.63 V are obtained for HJDs of 400 and 600°C-annealed nanorods, respectively. The energy distribution of the interface state density has been investigated and found to be in the range 0.70×10^{10} to 1.05×10^{12} eV/cm² below the conduction band from $E_C = 0.03$ to $E_C = 0.58$ eV. The highest density of interface states is observed in HJDs of 600°C-annealed nanorods. Overall improved behavior is observed

for the heterojunctions diodes of 400°C-annealed ZnO nanorods.

Keywords: ZnO nanorods, heterojunction, annealing, electrical characterization, interface states

1 Introduction

Zinc oxide (ZnO) is a direct and wide band-gap semiconductor (3.37 eV) studied widely for electronic and optoelectronic applications, such as light-emitting diodes, sensors, thin-film transistor (TFT), solar cells and UV photodetectors [1–3].

Zinc oxide shows n-type conductivity without any intentional doping and obtaining reproducible p-doped ZnO is very challenging. Therefore, its heterojunctions are realized on a variety of p-doped substrates such as Si, GaN, SiC, CuO₂, etc. [4–8]. Among these, ZnO/Si heterojunctions have attracted a great deal of interest due to low cost and fewer process steps. The heterojunctions of ZnO nanorods/Si are very flexible in fabrication but their electronic and optical properties are strongly influenced by the presence of point defects and strains contained in ZnO nanorods (NR) grown at low temperature. Since heterojunctions are widely employed as fundamental building blocks of several devices, therefore, growth of defect-free patterned and aligned one-dimensional nanorods with better optical quality is very important for the realization of efficient ZnO/Si heterojunction and other nanodevices [9–12].

Thermal treatment is a conventional and effective technique for reducing strains and defects and improving the crystalline quality [13,14]. The structural and optical properties of ZnO/Si heterojunctions can be modulated by controlling the growth temperature and thermal annealing atmosphere and conditions of ZnO nanorods (NR) [15,16]. Low defect concentration, high crystallinity and improved current–voltage (I – V) characteristics have been recently reported for ZnO/Si heterojunction diodes

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Figure 1: Step-by-step growth of ZnO nanorods.

(HJDs) of ZnO nanorods annealed at 450°C under different annealing conditions [15,17,18]. A significant improvement in ideality factor and reverse breakdown voltage is also observed in heterojunctions of nanorods annealed in a nitrogen atmosphere [19]. It is seen that the photosensitivity and photoresponsivity are also improved by annealing [20,21]. Improvements in current–voltage (I – V) characteristics and UV sensing are also observed for ZnO/Si HJDs annealed at 550°C [22].

In the present work, the effect of thermal annealing is studied for ZnO/Si HJDs. Well-aligned ZnO nanorods are grown on a p-Si substrate by the aqueous chemical growth (ACG) technique. Their post-growth annealing is done at 400 and 600°C. Nanorods are characterized by scanning electron microscopy (SEM) and photoluminescence (PL) measurements. n-ZnO nanorods/p-Si HJDs are fabricated and their electrical characteristics are studied by current–voltage (I – V) and capacitance–voltage (C – V) measurements. Current transport properties, rectification factor, reverse saturation current and ideality factor are studied for HJDs. The energy distribution of the density of interface states is also extracted and discussed. The results are compared with the heterojunctions fabricated from the as-grown nanorods.

2 Experimental

ZnO nanorods have been grown on the p-Si ($N_a = 10^{16}/\text{cm}^3$) substrate by aqueous chemical growth (ACG) method

[23–25]. The substrates were ultrasonically cleaned for 15 min with each acetone and methanol and then rinsed with deionized water to remove contamination and dust.

The seed layer solution was prepared by diluting zinc acetate dehydrate in methanol. The seed solution was spin-coated on the substrate three times and then heated at 250°C in air for 20 min.

The growth solution was prepared with equimolar concentrations (0.022–0.075 mM) of analytic reagent grade hexamethylenetetramine (HMT) ($\text{C}_6\text{H}_{12}\text{N}_4$) and zinc nitrate ($\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) by dissolving in 200 mL of deionized water at room temperature. The solution was stirred at 60°C for 30 min by magnetic stirring for complete homogeneous mixing. The samples were placed in a holder and dipped in the solution with the seed layer facing downward at a certain angle. The container was sealed with foil and heated at 95°C for 4 h. After the growth process, the samples were washed with deionized water for removing organic salts and blow-dried with nitrogen. The samples were then annealed in air at 400 and 600°C for 30 min. For the fabrication of a heterojunction, an insulating layer of polymethyl methacrylate (PMMA) was spin-coated on the samples to prevent the carrier crosstalk among the nanorods by filling the gaps between them. Excess PMMA was removed from the top of the nanorods by oxygen plasma cleaning. Aluminum (Al) ohmic contacts of 150 nm thickness have been evaporated in a vacuum chamber on the p-Si substrate. Aluminum/platinum (Al/Pt) nonalloyed circular ohmic contacts of 50/60 nm thickness were then evaporated on ZnO nanorods. The diameter of contact is

0.58 mm with specific contact resistance of $1.2 \times 10^{-5} \Omega/\text{cm}^2$. A step-by-step growth of ZnO nanorods is shown in Figure 1.

The structural characteristics of ZnO nanorods were studied by using JEOLJSM – 6301F scanning electron microscope (SEM). Photoluminescence (PL) measurements were performed at room temperature using laser lines of wavelength 270 or 350 nm from an Ar⁺ laser as the excitation source. Keithley SCS-4200 semiconductor characterization System was used for current–voltage (*I*–*V*) and capacitance–voltage (*C*–*V*) measurements by placing the samples on a probe station.

3 Results and discussion

3.1 SEM

Typical morphology of hexagonal nanorods was revealed when examined by SEM. The nanorods were vertically aligned on the substrate with an almost uniform distribution as shown in Figure 2. The mean diameter of nanorods was 180–300 nm with an approximate height of 1.4 μm . No significant change was observed in the morphology of nanorods before and after thermal annealing [17,26,27].

3.2 Photoluminescence

Photoluminescence (PL) study was performed at room temperature to study the optical properties of nanorods. Typical PL spectra indicating the good optical quality of the synthesized nanorods were obtained. Two emission peaks were seen for all the samples. Sharp and strong peaks centered at wavelengths between 370 and 380 nm and weak broad peaks centered between 540 and 600 nm were observed as shown in Figure 3.

The sharp peaks observed below the wavelengths of 400 nm are due to the intrinsic band-gap emission, referred to as ultraviolet (UV) emission or near-band emission (NBE). This emission originated from the zinc interstitial and is due to the recombination of free-exciton across the band gap [18,28].

The broad peaks located between 540 and 600 nm are due to the deep levels, referred to as deep-level emission (DLE) or visible (VIS) emission. Weak DLE may be attributed to the low concentration of oxygen vacancies due to the absorption of oxygen atoms during the growth process. A significant increase in the NBE intensity was observed after

annealing at 400°C. The enhancement in the NBE peak depicts the improvement in the crystallinity. This also

Figure 2: SEM images of ZnO NRs: (a) as-grown, (b) annealed at 400°C and (c) annealed at 600°C.

Figure 3: (a) PL spectra of the as-grown and annealed ZnO nanorods, (b) DLE emission peaks, and (c) optical quality factor.

indicates that the number of recombination traps is reduced after annealing at 400°C. The NBE emission was found to dominate the DLE emission for all samples. This indicates that the annealing treatment did not effectively enhance the deep level emission, demonstrating a slight increase in the adsorbed oxygen. After annealing at 600°C, both NBE and DLE were suppressed by 49 and 172% respectively. This is often observed in nanorods annealed at high temperatures as reported by Huang et al. and Kurudirek et al. [29,30]. Generally, the crystallinity of ZnO nanorods continues to improve with temperature; however, there is also the formation of point defects (oxygen and zinc vacancies) with an increase in the annealing temperature [31]. Sometimes the increased defects, caused by the breaking of the ZnO–O bonds, degrade the structural quality [32].

Furthermore, as a result of high-temperature annealing, small crystallites merge together to form larger crystallites, and some void spaces are introduced on the surface effecting the smoothness and uniformity. The optical quality ratio (I_{NBE}/I_{DLE}) has been investigated, and improvement in the ratio has been observed from 9.40 (as-grown) to 39.12 for nanorods annealed at 400°C, as shown in Figure 3(c) and

listed in Table 1. This increase in the NBE and DLE ratio indicates the suppression of structural defects and improvement in the optical properties [18,26,33]. The decrease in the intensity of NBE after annealing at 600°C may also be attributed to the dissociation of hydrogen donors.

No other obvious peaks were observed in the PL spectra. The parameters obtained from the PL emission spectra of the as-grown, 400 and 600°C-annealed ZnO nanorods are listed in Table 1.

3.3 Electrical characterization

3.3.1 Current–voltage (I – V) characteristics

The current–voltage (I – V) characteristics of ZnO/Si HJDs fabricated from the as-grown and annealed nanorods were measured at bias voltages between -10 and $+10$ V. The linear I – V characteristics and diode schematic (inset) are shown in Figure 4(a). All diodes exhibited nonlinear rectifying behavior with a turn-on voltage of ~ 1 V. The

Table 1: Parameters obtained from the PL emission spectra of the as-grown, 400 and 600°C-annealed ZnO nanorods

	As-grown	Annealed at 400°C	Annealed at 600°C
NBE peak λ_{UV} (nm)	379	374	377
NBE peak energy E_{UV} (eV)	3.27	3.31	3.28
DLE peak λ_{GL} (nm)	545	576	599
DLE energy E_{GL} (eV)	2.27	2.15	2.07
NBE intensity ratio ($I_{Annealed}/I_{As-grown}$)	1	3.49	0.503
DLE intensity ratio ($I_{Annealed}/I_{As-grown}$)	1	0.73	1.71
NBE/DLE intensity ratio I_{NBE}/I_{DLE}	9.40	39.12	2.76

Figure 4: (a) I - V characteristics and device schematic (inset) and (b) $\log I$ - $\log V$ characteristics.

current increased in the forward direction and decreased in reverse directions after annealing as shown in Figure 4(b).

The electrical behavior of the diodes improved with annealing. The rectification (I_F/I_R) factor increased from 23 (as-grown) to 1,596 and 309 for diodes of NRs annealed at 400 and 600°C measured at ± 5 V, respectively. The ratios are approximately 69 and 13 times the as-grown. The highest rectification ratio of 1,596 indicates that annealing at 400°C improves the crystallinity and interface between Si and ZnO nanorods and decreases the

internal defect concentration. Conversely, more defects are produced after annealing at 600°C due to the thermal diffusion process leading to reduced rectification factor [16,34]. Furthermore, high-temperature annealing may give rise to the leakage current and low conductivity caused by the partial melting between the nanorods. The barrier heights (ϕ_B) were calculated by using equation (1)

$$I_0 = AA^*T^2e^{-q\phi_B/kT}, \quad (1)$$

where I_0 is the saturation current, A is the area, k is Boltzmann's constant, and A^* is the Richardson constant having a value of 32 A/cm² K² for n-ZnO [25]. The barrier heights were found to be 0.75 eV (as-grown), 0.81 (400°C) and 0.63 eV (600°C). The highest barrier height of 400°C-annealed nanorod HJDs corresponds to its highest rectification factor demonstrating that the diode performance is improved by annealing the ZnO nanorods at 400°C.

The ideality factor (n) was obtained from the slope of the linear portions of the semi-log forward bias I - V plot using the Shockley equation given by equation (2):

$$I = I_0(e^{(qV/nkT)} - 1). \quad (2)$$

The ideality factors were found to be 3, 3.09, and 2.95 for the as-grown, 400 and 600°C-annealed nanorod diodes, respectively. Usually, a decrease in the ideality factor is observed after annealing [35]. The values of the barrier height and ideality factor are listed in Table 2.

The $\log I$ - $\log V$ characteristics were studied during the forward sweep voltage in order to compare the charge transport behavior of HJDs. Three slopes corresponding to different conduction mechanisms were observed for each diode as shown in Figure 5.

For a low-voltage region (<0.5 V), the current increased linearly ($I \propto V$) with slopes of 1.2, 1.4 and 1 for the as-grown, 400 and 600°C-annealed NR diodes, respectively. This confirms that ohmic transport is the dominant conduction mechanism in Region-I. For a moderate voltage range ($0.5 < V < 2$ V), the current increased exponentially ($I \propto e^{mV}$) in the as-grown and 400°C-annealed diodes but followed the power law ($I \propto V^m$) in 600°C-annealed diode. The exponent values higher than 2 indicate that the charge

Table 2: Electrical parameters calculated from I - V and C - V characteristics for the ZnO/Si heterojunction diodes

Annealing temperature (°C)	I_F/I_R @ ± 5 V	Barrier height (ϕ_B), V	Ideality factor (n)	Built in potential (V_{bi}), V	Donor density (N_d), (/cm ³)
As-grown	23	0.75	3	1	7.8×10^{12}
Air-400	1,596	0.81	3.09	1.3	5.9×10^{15}
Air-600	309	0.63	2.95	0.7	7.04×10^{14}

Figure 5: log I -log V characteristics of the HJDs: (a) as-grown, (b) 400°C annealed, and (c) 600°C annealed.

transport is subjected to the space-charge effect. In fact, the current increased due to increased injection of access carriers after filling the deep traps in the band. The exponential and power-law dependence of current with

applied voltage shows that the diode behavior is dominated by Region-II in all diodes. In Region-III of higher voltages ($V > 2$ V), log I -log V of all HJDs showed a power law ($I \propto V^m$) dependence with a decrease in slope ($m \sim 1.7$). At higher voltages, most traps are occupied by injected carriers and the accumulation of space charge near the electrode results in the creation of a field hindering further injection. Hence, the carrier transport approaches the trap-filled limit. This behavior may be attributed to the presence of oxygen vacancies [36,37].

3.3.2 Capacitance-voltage (C - V) characteristics

The capacitance-voltage (C - V) characteristics of HJs are measured at a frequency of 1 MHz at room temperature, as shown in Figure 6(a). An increase in the junction capacitance has been observed. This increase in capacitance may be attributed to the change in the depletion region width followed by the change in the carrier concentration after annealing [14].

Figure 6: (a) Capacitance-voltage (C - V) characteristics and (b) $1/C^2$ vs V plot at a frequency of 1 MHz.

The concentration of electrons (N_d) is obtained by plotting the inverse squared junction capacitance ($1/C^2$) against the applied reverse voltage (V). The concentrations were extracted from the slope of the linear segment of the curve as shown in Figure 6(b) by using equation (3):

$$N_d = \frac{2}{q\epsilon_s\epsilon_0} \times \frac{dV}{d(A^2/C^2)}. \quad (3)$$

Here, ϵ_s is the relative permittivity of ZnO having a value of 8.2. The extracted concentrations are 7.8×10^{12} , 5.9×10^{15} and 7.04×10^{14} for the as-grown, 400 and 600°C-annealed nanorods diodes, respectively. An increase in the carrier concentration is observed after annealing and a higher carrier concentration is achieved after annealing at 400°C. This increase in carrier concentrations might be due to the creation of oxygen vacancies and Zn-interstitial-related donor-type defects [38]. An increase in the concentration of hydrogen after annealing might also be one of the factors for the increase in the carrier concentration [39]. The lowest concentration obtained for the as-grown might be due to the presence of additional oxygen in the form of OH groups due to incomplete reactions at a low growth temperature. These OH groups may act as acceptors giving a low carrier concentration [14,40]. The barrier height has been calculated using equation (4):

$$\phi_{B(C-V)} = V_{bi} + \frac{kT}{q} \ln\left(\frac{N_C}{N_d}\right). \quad (4)$$

Here, N_C is the effective density of states in the conduction band (CB). Its value is $3.5 \times 10^{18}/\text{cm}^3$ for ZnO, obtained from equation (5):

$$N_C = 2 \left(\frac{2\pi m_n^* kT}{h^2} \right)^{3/2}, \quad (5)$$

where m_n^* is the effective mass of electrons given by $m_n^* = 0.27m_0$. The obtained barrier heights are 1.3 V (as-grown), 1.4 V (400°C) and 0.9 V (600°C) for HJDs. The barrier heights obtained from the $C-V$ are comparatively larger than the barrier heights obtained from the $I-V$ measurements. These discrepancies may be attributed to barrier inhomogeneity, surface defects and image force [25,41]. Second, the interface traps do not respond to high-frequency ac signals and hence do not contribute to capacitance [42,43].

3.3.3 Interface states density (N_{SS})

During the growth of ZnO, an unavoidable SiO_x interfacial layer is formed at the ZnO/Si interface. This thin interfacial layer is formed due to incomplete covalent

bonds and chemical reactions resulting in a new dielectric phase. The electric charge in the thin insulating interfacial layer introduces a high density of interface states. They affect the diode properties and recombination in these states is sometimes the main loss mechanism in heterojunctions, especially heterojunction solar cells [14].

These unintentionally introduced interface states are distributed continuously within the forbidden energy gap states and a leakage current is caused by the high density of these states. The origin of these states is exactly not known; however, they are introduced due to oxidation of semiconductor surfaces from extended air exposure, surface dipoles, incomplete covalent bonds, hydrogen, chemical reactions during fabrication processes and sharp discontinuity between semiconductor crystals [44,45]. The large surface area and small diameter of ZnO nanorods are also the reason for these states [46].

The density of interface states (N_{SS}) can be extracted using $I-V$ and $C-V$ measured values using equation (6) [25]:

$$N_{SS} = \frac{1}{q} \left[\frac{\epsilon_i}{t_i} \{n(V) - 1\} - \frac{\epsilon_s}{W_D} \right]. \quad (6)$$

Here, W_D is the width of the space-charge region, and ϵ_i and t_i are permittivity and thickness of the interfacial layer. The capacitance of interfacial layer (C_i) is given by equations (7) and (8);

$$C_i = C_{ma} \left[1 + \left(\frac{G_{ma}}{\omega C_{ma}} \right)^2 \right], \quad (7)$$

$$\frac{C_i}{A} = \frac{\epsilon_i \epsilon_0}{t_i}. \quad (8)$$

Here, ω is angular frequency given by $2\pi f$, and C_{ma} and G_{ma} are capacitance and conductance measured at a frequency of 1 MHz. The voltage-dependent ideality factor $n(V)$ is extracted from equation (9):

$$n(V) = \frac{qV}{kT \ln\left(\frac{I}{I_0}\right)}. \quad (9)$$

Taking into account the voltage-dependent ideality factor and series resistance, the bias-dependent effective barrier height (ϕ_e) and the energy distribution of interface states with respect to the conduction band ($E_C - E_{SS}$) are obtained by using equations (10) and (11), respectively:

$$\phi_e = \phi_{bo} + \left(1 - \frac{1}{n(V)} \right) (V - IR_s), \quad (10)$$

$$E_C - E_{SS} = q(\phi_e - V). \quad (11)$$

The electrical behavior of the diode is affected when the electrons excited to the conduction band are trapped by these states. The influence of these interface states is more pronounced for the compound semiconductor nanostructure as their surfaces have more defects [47].

The distribution of the interface state density below the conduction band is shown in Figure 7 for the as-grown, 400 and 600°C-annealed NR diodes. The study of the effect of annealing temperature on the interface state density of ZnO nanorod/Si HTJs has not been extensively carried out earlier. The density is maximum near the conduction band edge (E_C) and decreased monotonically with energy from the band edge toward the mid of the band gap. The change in the density and energy distribution of the interface states varies with the bias due to the potential drop across the interfacial layer. It affects the diffusion potential and depletion capacitance of HJDs [48]. An increase in N_{SS} is seen with the increase in the annealing temperature, indicating that the influence of annealing on the interface state density is higher at higher temperatures. This increase in N_{SS} is often observed with annealing at high temperatures [48,49]. For ZnO nanorods, the oxygen vacancies tend to decrease thermodynamically with an increase in the annealing temperature up to 400°C [21,50]. Therefore, improved crystallinity/optical quality, enhanced electrical properties and the low interface state density is observed for the 400°C-annealed NR diodes.

For nanorods annealed at higher temperatures, the surface defects come into play due to desorption of

oxygen ions and thermal diffusion of the oxygen vacancies on the surface owing to the evaporation of oxygen atoms [16,17]. Therefore, decreased crystallinity/optical quality, inferior electrical properties and a high density of interface states are observed for the 600°C-annealed NR diodes [32,51,52].

4 Conclusion

The effect of annealing temperatures on the performances of ZnO nanorod/Si HJDs is studied. The as-grown ZnO nanorods were annealed at 400 and 600°C and their characterizations have been performed. The photoluminescence results exhibited an improvement in the optical quality of the ZnO nanorods after annealing treatment at 400°C. Likewise, the heterojunctions of 400°C-annealed nanorods exhibited an enhancement in the electrical properties with higher rectification factor and lesser density of interface states than the 600°C-annealed ZnO/Si HJDs. The reported study suggests that ZnO nanorods annealed at 400°C will be promising for the realization of more efficient electronic devices such as heterojunctions, solar cells and UV sensors.

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Figure 7: Energy distribution of the interface states density.

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