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To cite this article before publication: Chen Huang *et al* 2021 *J. Phys. D: Appl. Phys.* in press <https://doi.org/10.1088/1361-6463/ac4185>

Manuscript version: Accepted Manuscript

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Boosted Ultraviolet Photodetection of AlGa_N Quantum-Disk Nanowires via Rational Surface Passivation

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E-mail: haiding@ustc.edu.cn
Received xxxxxx
Accepted for publication xxxxxx
Published xxxxxx

Abstract

Self-assembled AlGa_N nanowires (NWs) are regarded as promising structures in the pursuit of ultraviolet photodetectors (UV PDs). However, AlGa_N nanowire-based PDs currently suffer from degraded performance partially owing to the existence of outstanding surface-related defects/traps as a result of its large surface-to-volume-ratio feature. Here, we propose an effective passivation approach to suppress such surface states via tetramethyl ammonium hydroxide (TMAH) solution treatment. We successfully demonstrate a fabrication of UV PDs using TMAH-passivated AlGa_N quantum-disk NWs and investigate their optical and electrical properties. Particularly, the dark current can be significantly reduced by an order of magnitude after surface passivation, thus leading to the improvement of photoresponsivity and detectivity. The underlying mechanism for such boost can be ascribed to the effective elimination of oxygen-related surface states on the nanowire surface. Consequently, an AlGa_N nanowire UV PD with a low dark current of 6.22×10^{-9} A, a large responsivity of 0.95 A W⁻¹, and a high detectivity of 6.4×10^{11} Jones has been achieved.

Keywords: AlGa_N nanowires, ultraviolet photodetectors, dark current, passivation, tetramethyl ammonium hydroxide, surface states

1. Introduction

Searching for highly efficient, compact, and low-power-consumption ultraviolet photodetectors (UV PDs) (for example, photoelectrochemical-type PDs [1,2] and solid-state PDs [3]) has become significantly essential due to the increasing demands for advanced applications in military and civil fields, including space exploration, missile monitoring, fire alarm, and secure communication. The III-nitride

semiconductors, particularly, the Al_xGa_{1-x}N alloys possessing a wide bandgap ranging from 3.4 eV to 6.1 eV via tuning the Al composition, can cover the entire UV spectral range from 360 nm to 210 nm [4,5]. Additionally, the high carrier mobility, chemical robustness, and high thermal conductivity of AlGa_N alloys make them one of the top contenders for the realization of high-performance UV PDs [6]. However, conventional planar AlGa_N UV PDs still suffer from poor Al-rich AlGa_N epilayer quality and low light absorption efficiency, which results in performance degradation when

the detection wavelength moves toward shorter wavelength [7,8]. Fortunately, the AlGaIn nanowires (NWs), exhibiting many outstanding inherent properties relative to planar structures, have been considered as emerging structures to build AlGaIn UV PDs. First of all, during the nanowire growth, the lateral strain can be effectively released because of the nearly infinite critical thickness in one-dimensional structures, thus the defect-free crystalline quality of a single AlGaIn nanowire can be realized [9,10]. Secondly, the p-type doping efficiency in AlGaIn NWs can be remarkably improved due to more efficient Mg incorporation and lower activation energy, which are caused by sufficient surface dopants and reduced lattice strain in nanowire structures [11,12]. Last but not least, the large surface-to-volume ratios and multiple light scattering effects in AlGaIn NWs can enhance the light absorption efficiency [13]. Consequently, adopting AlGaIn NWs has been regarded as an promising approach to fabricate high-performance UV PDs.

However, the development of AlGaIn nanowire UV PDs is still in its infancy by two main challenges: the difficulty of nanowire-based device fabrication and the extensive surface states [9]. It is noted that a low filling factor of nanowire array will cause the metal penetration into the nanowire gaps so that the metal deposition during the electrode formation has to be carefully designed and optimized during the fabrication process [10]. Researchers have tried the planarization step using polyimide or parylene to fill the gaps before depositing metal pads. Unfortunately, the introduction of such polymer results in an undesirable increase in fabrication cost and time. It may also block the incidence of UV light and aggravate the heat accumulation, which may cause serious damage to the device operation [14,15]. To avoid this obstacle, we intentionally grew the nearly coalesced NWs, on which we can directly deposit metal pads without using any filling materials, simplifying the fabrication process [16]. On the other side, the presence of a large number of surface states (such as dangling bonds and surface defects) on the nanowire surface can introduce the Shockley-Read-Hall (SRH) nonradiative recombination centers, which will capture the photoexcited carriers and lower the quantum efficiency of photodetection devices [17,18]. Many efforts have been dedicated to removing such surface states to suppress surface recombination for performance improvement. In particular, surface treatment using various wet chemicals has been proved to be an effective method to passivate defect-related surface states. Zhao et al. reported the successful mitigation of SRH recombination by octadecyl thiol (ODT) and inorganic sulfide passivation in InGaIn/GaN disk-in-nanowire light-emitting diodes (LEDs) [17]. Moreover, Sun et al. introduced the potassium hydroxide (KOH) solution treatment as an additional passivation process to achieve significantly enhanced luminescence performance of AlGaIn nanowire

LEDs [19]. Bui et al. used ammonium sulfide $((\text{NH}_4)_2\text{S}_x)$ to passivate the p-i-n AlGaIn nanowire LEDs and found that the electrical and optical properties of LED devices improved obviously after sufficient passivation [20]. However, limited studies and exploration on the effect of surface passivation on the performance of AlGaIn nanowire-based UV PDs have been carried out so far.

In this work, we report on the fabrication of UV PDs using as-grown and passivated p-i-n AlGaIn quantum-disk NWs grown by molecular beam epitaxy (MBE) on Si substrates. The surface passivation was performed by using the tetramethyl ammonium hydroxide (TMAH) solution, which is commonly used as a follow-up step after dry etching of GaN-based thin films to improve the sidewall roughness for light-emitting devices or to suppress the leakage current in the fabrication of power devices [21,22]. Here, we investigate the underlying TMAH passivation mechanism and its effects on the electrical and optical characteristics of AlGaIn nanowire PDs. Surprisingly, passivated AlGaIn nanowire PDs show much lower dark current and higher responsivity compared to the passivation-free devices. The further spectroscopic characterization provides strong evidence that surface defects and oxides can be effectively eliminated through simple TMAH wet passivation, thus enhancing the UV photodetection.

2. Experimental section

The AlGaIn quantum-disk NWs studied in this work were grown on n-Si (001) substrates using Veeco plasma-assisted MBE under nitrogen-rich conditions. Prior to the AlGaIn nanowire growth, ~ 50 nm Si-doped n-GaN seeds were initiated at a substrate temperature of 550 °C. The Si effusion cell was kept at 1180 °C, the Ga beam equivalent pressure (BEP) was 6.0×10^{-8} Torr, and the high-brightness nitrogen plasma was sustained using 350 W RF power and 1 sccm flow rate. The followed p-i-n AlGaIn structures were then grown at a higher temperature of 630 °C. We estimated the nominal Al composition based on the ratio of the Al BEP to the total metal BEP as measured by the beam flux monitor. Around 150 nm Si-doped n-Al_{0.4}Ga_{0.6}N layers were firstly grown, followed by the active region consisting of 10 pairs Al_{0.25}Ga_{0.75}N/Al_{0.4}Ga_{0.6}N quantum disk/quantum barrier with a thickness of ~ 3 and ~ 6 nm, respectively. Then ~100 nm Mg-doped p-Al_{0.4}Ga_{0.6}N layers were grown under the Mg cell temperature of 360 °C with the same nitrogen plasma condition. Finally, the additional highly Mg-doped p-GaN contact layers (~ 5 nm) were deposited by increasing the Mg cell temperature to 380 °C at a reduced growth temperature of 580 °C. The morphology of the AlGaIn NWs was observed using field emission scanning electron microscopy (SEM) (SU8220, Hitachi) at an accelerating voltage of 3 kV. A schematic of the grown AlGaIn nanowire structure and the

corresponding SEM image of the vertically aligned nanowire arrays on Si are shown in figure 1(a) and figure 1(b), respectively. We found that the coalesced NWs with relatively uniform height (~ 400 nm) and diameter (~ 80 nm) distribution exhibit a high filling factor ($>95\%$), indicating that we can directly deposit metal contacts on the top of the NWs. After the growth, samples were dipped into 25 % TMAH at 50 °C for 30s, 1 min, 2 min, and 3 min, respectively.

Figure 1. (a) The schematic structure of the *p-i-n* AlGaIn NWs on Si substrate. (b) A 20°-tilted SEM image of the AlGaIn nanowire arrays.

To study the optical properties of the samples after TMAH treatment, room-temperature photoluminescence (PL) measurements were performed (using a 266 nm laser as the excitation source). The results are shown in figure 2, it is noticed that the PL peak wavelength of the TMAH treated device exhibits a slight red-shift from 307.6 nm to 309.4 nm. This phenomenon is consistent with our previous results and can be explained by the TMAH etching of Al-rich AlGaIn shell coating on the nanowire surface [19]. More importantly, the peak PL intensity increases by around 80% (suggesting a significantly enhanced radiative recombination) using TMAH passivation method. We did not extend the passivation time further, as the PL enhancement after 2 min was not significant (as shown in figure 2).

Figure 2. Room-temperature PL spectra of the samples with different TMAH passivation time.

We then fabricated the devices using standard UV contact lithography techniques. Every device size was defined as $300 \times 300 \mu\text{m}^2$. A Ni (7.5 nm)/Au (7.5 nm) stacking metal layer was first deposited by electron-beam evaporation followed by rapid thermal annealing in a 4:1 $\text{N}_2:\text{O}_2$ ambient at 500 °C for 1 min. A stack of Ni (20 nm)/Au (50 nm) was then deposited as the top pad for probing. Lastly, a thickness of ~ 200 nm of Si was etched away from the Si backside using reactive-ion etching (RIE) and a Ti (20 nm)/Au (50 nm) metal layer was deposited as the n-pad, upon annealing under N_2 ambient at 250 °C for 1 min. The electrical performance of the PDs was tested by a semiconductor device analyzer (B1500A, Keysight). A 265 nm UV LED was applied as the illumination source and the light intensity was calibrated by an optical power meter (2936-R, Newport). TRPL spectra were measured by a time-correlated single-photon counting (TCSPC) system containing a femtosecond pulsed diode laser (PHAROS, Light Conversion, ≈ 221 fs, 1030 nm, 50 kHz, 10 W), an optical parametric amplifier (ORPHEUS, Light Conversion, 190-2000 nm), a stand-alone TCSPC module and a single photon counting photomultiplier. The average excitation power of the 235 nm laser on the sample surface was maintained to be 1 mW at a 50 kHz repetition rate. XPS measurements were executed using an Al $K\alpha$ X-ray source of energy 1253.6 eV (K-Alpha, Thermo Fisher Scientific).

3. Results and discussion

The current-voltage (I-V) characteristics of the reference and TMAH-treated samples under dark and 265 nm light illumination with a power density of 3.75 mW cm^{-2} are measured, as presented in figure 3(a). It is found that the dark current reduces by nearly an order of magnitude after TMAH passivation, from 7.23×10^{-8} A to 6.22×10^{-9} A at a bias of -4 V. We have measured more than four devices on the TMAH-treated AlGaIn nanowire PDs, and found that our devices exhibited nearly consistent dark currents in the range from 5.4×10^{-9} to 9.5×10^{-9} A. This corroborates with previous observation during the optimization of PDs using other passivation methods [18,23]. Interestingly, the influence of such passivation on photocurrent enhancement is negligible. The photocurrents of the two devices increase to 2.86×10^{-6} A and 3.21×10^{-6} A, respectively at -4 V bias voltage. The value of responsivity (R) is a critical feature indicator of PDs, which is given by $R = (I_{\text{photo}} - I_{\text{dark}}) / (PS)$, where I_{photo} is the photocurrent, I_{dark} is the dark current, P denotes the light power density, and S is the irradiated area of the device. After calculation, we find that after TMAH passivation, the R

of the AlGaIn nanowire PD increases from 0.83 A W^{-1} to 0.95 A W^{-1} under -4 V bias with the incident power density of 3.75 mW cm^{-2} at 265 nm light illumination.

The time-dependent photoresponse characteristics were measured under the same test conditions. As shown in figure 3(b), both devices exhibit reproducible and stable on/off switching behavior. By extracting the photo-to-dark-current ratio ($PDCR = (I_{\text{photo}} - I_{\text{dark}}) / I_{\text{dark}}$), the TMAH-treated device shows a $PDCR$ of ~ 500 , which is 10 times higher than that of the reference sample. This is attributed to the obviously reduced dark current in the passivated PDs. To evaluate the ability to detect the lowest detectable signal, the detectivity

(D^*) defined as $D^* = RS^{1/2} / (2qI_{\text{dark}})^{1/2}$ were calculated to be 1.6×10^{11} Jones for the reference device and 6.4×10^{11} Jones for the passivated device, respectively. The rise and decay time (t_{rise} , defined as the time for the current increasing from 10 % to 90% of the peak value; t_{decay} , defined as the time for the current dropping from 90% to 10% of the peak value) of the two devices were also studied to judge the response speed of PDs to external photo source. Both devices exhibit fast response speed. Particularly, figure 3(c) gives the t_{rise} of $\sim 33 \text{ ms}$ and t_{decay} of $\sim 27 \text{ ms}$ in the passivated device (operated in -4 V bias).

Figure 3. (a) The I–V curves of the AlGaIn nanowire PDs with and without TMAH passivation under dark and 265 nm UV light illumination (the power density is 3.75 mW cm^{-2}). The inset shows the device under test. (b) Time-dependent photoresponse characteristics of the reference and TMAH-treated AlGaIn PDs under -4 V bias with the light power density of 3.75 mW cm^{-2} . (c) Enlarged curves of a single cycle of the normalized photoresponse curves.

To understand the effect of surface passivation on the fundamental carrier dynamics after photoexcitation, we carried out the room temperature TRPL measurement on the reference and passivated AlGaIn NWs, as shown in figure 4.

Figure 4. TRPL carrier lifetimes of the untreated and TMAH-passivated AlGaIn NWs. The fast and slow decay time constants (τ_1 and τ_2) are indicated in the figure.

Through biexponential decay fitting process, we observe that the fast and slow decay time constants (τ_1 and τ_2) of

reference NWs are 104 and 1067 ps, while those of TMAH-treated NWs were 152 and 1262 ps, respectively. The fast decay component can be attributed to the trapping of surface states, and the slow decay component comes from the carrier relaxation of deep defect states. Therefore, the obtained larger τ_1 in the treated devices suggests that the TMAH treatment reduced nonradiative recombination centers at nanowire surface and renders more time to collect and transport photogenerated carriers in the PDs [11,24].

We further implemented the XPS measurements to explore the element variations of the AlGaIn NWs after surface passivation. All binding energies were calibrated based on the C 1s peak position (284.8 eV). Presented in figure 5(a) are the captured Al 2p spectra for reference and TMAH-passivated AlGaIn NWs. Based on the combination of Gaussian and Lorentzian fitting, the Al 2p peak can be deconvoluted into two contributions of Al–N bond at 73.0 eV and Al–O bond at 74.2 eV, respectively. Noticeably, after TMAH passivation, the ratio of the normalized concentration of Al–O bond to that of Al–N bond (ratio of Al–O/Al–N) has reduced from 40.4% to 20.7%. A similar decreasing trend of the oxide-related peak was observed in the captured Ga 3d spectra as well. As shown in figure 5(b), the Ga 3d peak can be deconvoluted into three contributions of Ga–N bond at

19.4 eV, Ga-O bond at 20.4 eV, and N 2s at 15.8 eV. The calculated ratio of Ga-O/Ga-N of 24.5% in the passivated sample is relatively lower than that of 41.9% in the reference sample, indicating the effective removal of surface oxides via TMAH passivation [25-27]. Our XPS observations in regards to the reduction of oxygen-related complex on nanowire's surface are consistent with a report where they confirmed the disappearance of oxygen atoms on the nanowire's surface by using a detailed chemical mapping on nitrides NWs after surface passivation treatment [28].

Figure 5. (a) Al 2p and (b) Ga 3d core levels of the AlGaIn nanowire surfaces before and after TMAH passivation.

In order to confirm that the passivated devices are quantitatively dependent on the external illumination intensity, the time-dependent photoresponse under different illumination power density is presented in figure 6(a). The measured photocurrent as a function of light power density is plotted in figure 6(b) (red squares), in which the photocurrent is 0.19 μA at 0.12 mW cm^{-2} and later linearly increases to 3.21 μA at 3.75 mW cm^{-2} . Such linear behavior can be explained by the fact that photoexcited electron-hole pairs are effectively separated and can scarcely be captured by trap states [29]. Additionally, we observed the value of R varying sensitively to different incident light power densities, as in figure 6(b) (orange circles), indicating its excellent

photodetection ability. We further compare our device performance with other early reported AlGaIn nanowire/nanorod based UV PDs, as presented in Table 1, and find that our proposed TMAH-treated AlGaIn NWs (not including GaN NWs) shows ever-reported highest R , revealing the great potential of such effective and environmentally-friendly passivation method in the pursuit of high-efficiency nanowire-based UV photodetection.

Figure 6. (a) Photocurrent of the TMAH-passivated device under -4 V bias with the incident light power density of 0.12, 0.56, 1.18, 2.56, and 3.75 mW cm^{-2} , respectively. (b) Fitting curve and corresponding calculated responsivity of the TMAH-passivated device under different light power density.

Table 1. Detailed comparison between previously reported AlGaIn nanowire-based PDs with our current AlGaIn quantum-disk nanowire PDs with or without TMAH passivation.

AlGaIn structure	R (A W ⁻¹)	Dark current (A)	Rise/decay time (ms)	Detectivity (Jones)	Reference
AlN <i>p-i-n</i> quantum-disk nanowires	~0.001 (-6V)	~10 ⁻¹⁰	-	~ 0.2×10 ¹¹	7
AlGaIn nanorods	0.12 (3V)	-	-	-	8
AlGaIn nanoflowers	0.72 (2V)	-	-	4.76×10 ⁷	30
AlGaIn nanowires with graphene electrode	0.18 (-2V)	5.4×10 ⁻⁸	4000/4700	~ 0.9×10 ¹¹	31
AlGaIn/AlGaON nanowires	0.73 (-5V)	10 ⁻⁴	-	-	32
AlGaIn <i>p-i-n</i> quantum-disk nanowires (as-grown)	0.83 (-4V)	7.23×10 ⁻⁸	35/29	1.6×10 ¹¹	This work
AlGaIn <i>p-i-n</i> quantum-disk nanowires (TMAH-treated)	0.95 (-4V)	6.22×10 ⁻⁹	33/27	6.4×10 ¹¹	This work

4. Summary and conclusions

In conclusion, we have demonstrated that the TMAH wet chemical treatment is a promising method to boost the electrical and optical performance of AlGaIn nanowire UV PDs. By introducing the TMAH surface passivation, the dark current was reduced from 7.23×10⁻⁸ A to 6.22×10⁻⁹ A in the AlGaIn nanowire PDs, leading to the enhancement in *PDCR*, *R*, and *D**. We attribute the reasons to the reduction of nonradiative recombination centers originated from surface oxides and states. This work provides an effective and convenient method to realize low-dark-current and high-responsivity AlGaIn nanowire UV PDs.

Acknowledgements

This work was funded by National Natural Science Foundation of China (Grant No. 61905236, 51961145110), Fundamental Research Funds for the Central Universities

(Grant No. WK2100230020), USTC Research Funds of the Double First-Class Initiative (Grant No. YD3480002002), and was partially carried out at the USTC Center for Micro and Nanoscale Research and Fabrication.

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