





constructed solar-blind PEC PDs exhibited excellent responsivity of 48.8 mA W⁻¹, fast response speed (rise time of 83 ms and decay time of 19 ms) with large photocurrent of 55 µA cm⁻² at 254 nm illumination without external power supply (zero bias). density Such superior performance can be attributed to, firstly and foremost, the successful highly uniform and defect-free n-type AlGaN NWs which ensures efficient synthe of eration via effective light-harvesting, and secondly, the boosted carrier photoger separation and collection efficiency with the help of Ru decoration on nanowire's This novel architecture of AIGaN: Ru NW PEC PDs enables deep UV surfaces photodetection to work stably with low energy consumption, intriguingly, opening the possibility for the development of high-performance PEC-type PDs based on group IIInitride semiconductors (InN, GaN, AIN) covering the entire spectral range from infrared to deep Introduction This article is protected by copyright. All rights reserved.

Constructing self-powered devices that can work independently, wirelessly, and sustainably without external power supply emerges as a significant research topic for the next-generation electronic systems.^[1-3] Self-powered photodetectors (PDs), as one of the key family members in an energy-efficient optoelectronic sensing system, attract huge attention in past few years.^[4-6] Typical architectures of self-powered PDs are divided into p-n junction type, Schottky junction type, and photoelectrochemical (PEC) type.^[7-9] Among them PEC PDs exhibit a list of merits, including easy-to-fabricate process, low cost, accellent responsivity, and fast photoresponse,^[10-12] therefore being extensively investigated to construct highly efficient self-powered PDs. It should be noted that previous investigations of PEC PDs mainly focused on visible spectral range and ultraviolet (UV) spectral range (~360 nm), while there were limited

studies on solar-blind (~250 nm) detection,^[9, 10] which is particularly suitable for light

detection in harsh, remote, and aqueous environment, such as submarine oil leakage

and ozone monitoring, biological/chemical detection and ultraviolet spectroscopy

analysis, as well as invisible light communication system.[12-15] Therefore, searching for

semiconductors with larger bandgaps is the prerequisite to the realization of selfpowered solar-blind PEC PDs. By alloying binary gallium nitride (GaN) with aluminum AIN) and tuning their compositions, we can synthesis ternary aluminum gallium nitride nitride $(Al_xGa_{1-x}N: 0 \le x \le 1)$ as photoanodes which can cover a wide UV detection spectral 360 nm).^[16, 17] Moreover, the advantages of high carrier mobility and range 210chemical stability of Al_xGa_{1-x}N material system make AlGaN-based PEC PD perfectly suited to the applications of the sensitive solar-blind detection in harsh aqueous environment. for PEC cell, the electrolytes are crucial components for achieving the Additionally. electronic circuit, providing ionic conductivity between the working electrode and counter electrode.^[18, 19] Conventionally, the I^{-}/I_{3}^{-} redox couple (ionic liquid) was used as the electrolyte.^[8, 10] However, owing to the strong volatility, corrosivity, and latent interaction with metallic components, I-/I₃- ionic liquid gravely limits the long-term operation of PEC PDs, thus their short lifespan and poor stability become the biggest bottlenecks for practical applications.^[9] In recent years, aqueous solution has been explored as the

electrolyte to build PEC PDs, due to its safe, stable, and environmental-friendly characteristics. Unfortunately, previous aqueous PEC PDs have inferior responsivity and photores ponse compared with I^{-/I₃⁻-type,^[11, 20, 21] mainly due to the poor material quality} resulting in severe trapping and recombination of photoexcited carriers, as well as the activity of reaction sites which limits the electrochemical process and further low catalytic performance. Consequently, the surface decoration of high-quality affect semiconductors using well-designed co-catalysts is vitally significant for the performance improvement of PEC PDs. In this context, we demonstrate the self-powered solar-blind PEC-type PDs based on ntype AlGaN nanowires (NWs) grown by molecular beam epitaxy (MBE) on the conductive Si substrate and the growth conditions follow our previous recipe.^[22] These vertically-aligned, spontaneously formed NWs are particularly attractive for photodetection purposes owing to (1) their large surface-to-volume ratio for unambiguously enhanced light absorption and (2) their high crystalline quality that is free of dislocations and defects for sufficient photogeneration, effective separation and fast This article is protected by copyright. All rights reserved.

transport of carriers. Thereafter the nanowire growth, in analogous to visible-light PEC artificial photosynthesis by loading co-catalyst on the surface of semiconductor,^[23, 24] we decorated the AIGaN NWs using novel metal ruthenium (Ru) which could drive the active redox reactions during PEC process and subsequently fabricated the entire AlGaN: Ru photoanode for solar-blind photodetection test. Intriguingly, the solar-blind NWs a the exhibits an extremely large photocurrent density of 55 µA cm⁻² with a record-PEC P high responsivity of 48.8 mA W⁻¹ under 254 nm illumination at zero bias. Furthermore, the device features a record fast response/recovery time of 83/19 ms, which has never previously achieved in other self-powered solar-blind PEC PDs. These superior been performance reveals that the AlGaN: Ru nanostructure is a promising candidate for selfpowered, high-sensitivity solar-blind photodetection application with low cost, large scalability, and excellent stability. **Results and Discussion** This article is protected by copyright. All rights reserved.

The detailed morphology of the AlGaN: Ru NWs were discerned using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The SEM images of the AlGaN NWs captured at a tilt angle of 30° are shown in Figure 1a and Figure S1. The asgrown NWs are almost vertically aligned on the planar (001) Si substrate with relative uniform diameters of ~55 nm and lengths varying from 180 to 200 nm. The TEM characterization in Figure 1b and Figure S2 demonstrate the intact appearance of a single nanowire. As the high-resolution transmission electron microscopy (HRTEM) image shows in Figure 1b inner, the fringe lattice parameter of the AlGaN nanowire was determined as 5.167 Å, which can be assigned to the lattice spacing between the two adjacent (0001) plane,^[25] suggests the growth direction along the c-axis.

Through the above morphology characterizations, however, no obvious Ru-related nanoparticles were observed. To further investigate the interaction between Ru species and AlGaN NWs after photodeposition, the photoluminescence (PL) measurements were performed (Figure 1e). Under an excitation wavelength of 266 nm at room temperature,

the PL peaks of two samples are centered at 320 nm which indicates the AIGaN NWs

possess about 25% Al composition calculated by the following formula:

$$E_g(Al_xGa_{1-x}N) = xE_g(AlN) + (1-x)E_g(GaN) - bx(1-x)$$

where b is the bowing parameter given 0.98 eV.[26]

In principle, the recombination of electrons and holes in direct-bandgap semiconductors, have the same crystal momentum, excluding the involvement of phonons in the recombination process.^[27] The intensity of PL peaks is indicative of the amounts of photons generated from radiative recombination of photo-generated carriers. The steady-state PL peak of AlGaN: Ru NWs was significantly suppressed with respect to those of pristine AIGaN NWs. It could be explained by the fact that the metal Ru could allow smooth charge migration from the photoexcited semiconductor, according to the built-in electric field at the interface that results from the alignment of the potentials of the Ru and AIGaN nanowires. [28-30] In other words, the decoration of Ru species reduces the possibility of radiative electron-hole recombination, promoting the photocarrier separation the pristine AIGaN nanowires. with respect to

To further determine the distribution of the Ru species, scanning transmission electron microscopy (STEM) characterization was carried out. Energy dispersive spectroscopy napping analysis of the STEM images revealed the homogeneous dispersion of (EDS) Ru species in AlGaN: Ru NWs (Figure 1c,d). Therefore, it is concluded that the Ru loaded adequately on the surface of NWs. In order to examine the valence specie are pecies loaded on the nanowire surface, the X-ray photoelectron spectra state (XPS) survey spectra of the AlGaN: Ru samples are exhibited in Figure 1f and Figure S3. All binding energies were calibrated by the C 1s peak (284.6 eV) arising from carbon. Due to the loading quantity at a minute magnitude, the Ru 3d adventitious signals were covered by C 1s peak. However, in the captured Ru 3p spectrum (Figure S3), the peak at 463.0 eV is different from the typical peaks at 461.2 eV for Ru metal, which has been likely attributed to the presence of an oxidation state close to Ru(III) or N 1s (Figure 1f) spectra of AlGaN: Ru NWs and bare AlGaN NWs, the Ru(IV) 1 and 398.0 eV can be consistent with the value of Al–N and Ga–N bonds neak respectively [22] and the 398.6 eV, which does not appear in N 1s spectrum of bare This article is protected by copyright. All rights reserved.

AlGaN NWs, is assigned to Ru-N bond.^[33] The XPS analysis unambiguously prove the strong interaction between Ru and AlGaN NWs. Together with above PL, STEM-EDS mapping results, we can confirm that the Ru co-catalysts were successfully anchored to

the AIGaN nanowire surface.

shows the schematic illustration for measuring the photodetection Figure characteristics of the AlGaN: Ru NW PEC PDs by using the three-electrode system. First, the n-AlGaN: Ru NW samples were fabricated as the photoanodes and then loaded in PEC cell for photodetection measurement. In theory, when an n-type semiconductor is in contact with electrolyte, it would exhibit an upward band bending at the AlGaN/electrolyte interface due to the transport of electrons from the semiconductor to the electrolyte to achieve electrochemical equilibrium (as shown in Figure 2 inner).^[18] In this case, when external UV illumination is applied, the photoexcited holes are more likely to transport to the AlGaN/electrolyte interface. Driven by Ru co-catalysts at the interface, holes are captured by hydroxide ions to undergo redox reactions to form redox molecules (h+ + OH*). Meanwhile, electrons drift into AlGaN NWs and flow through the external OH- -

circuit to Pt counter electrode. Thereafter, the redox molecules, which diffuse through the H₂SO₄ electrolyte to the surface of the Pt counter electrode, are reduced to hydroxide ions with the help of electrons re-entered from the external circuit ($e^- + OH^* \rightarrow OH^-$), completing the current loop.^[34-36] The Ag/AgCl electrode serves as the reference electrode which has a stable and well-known electrode potential. Its only role is to act as measuring and controlling the working electrode's potential and at no point a refei pass any current and undergo chemical reactions. The electron flow throughout does it photodetection circuit can be clearly observed in Figure 2. It is worth mentioning the PE that, during the PEC operation process, the upward surface band bending can lead to of photoexcited holes to gather at the surface of semiconductors, possibly majority resulting in oxidation metamorphism,^[37, 38] namely the photo-corrosion effect. In this regard, the Ru co-catalysts can help drive the photoexcited holes into reaction sites to participate in redox reactions instead of etching the semiconductor material itself,[39] alleviating such unedifying photo-corrosion.

To validate the conductivity type of the AlGaN NWs, we first conducted the open circuit potential (OCP) measurement under periodically switching illumination with different = 254, 365, 254+365 nm) in 0.01 M H_2SO_4 , as presented in the top of wavelength. Figure 3a. The observed negative shift of the OCP behavior illustrates the n-type of our AIGaN NWs. This can be explained as: under UV illumination, the nconduc Ws would absorb photons and generate free minority carriers which shift type A the electron quasi-Fermi level upward and flatten the band at the AlGaN/electrolyte interface.^[40] Therefore, the OCP measurements would exhibit a negative shift, which n-type conductivity of the grown samples. Furthermore, ΔOCP (the confirms he between OCP in the dark/light conditions) reflects the steady-state difference photogenerated carrier concentration.[40] The lower ΔOCP indicates negligible light absorption at 365 nm wavelengths, compared to direct bandgap transition under 254 nm irradiation, suggesting the high potential of n-AIGaN NWs for solar-blind detection. Thus, the corresponding solar-blind detection performance of the AlGaN: Ru to evaluate nanostructures, we measured time-dependent photocurrent (Iphoto) of AIGaN: Ru NWs at This article is protected by copyright. All rights reserved.

fixed 0.93V bias under same irradiation condition, shown in the bottom of Figure 3a.

Here we define I_{photo} using the following equations:



As shown in Figure 3b, the photocurrent density of as-prepared AlGaN: Ru NW photoanodes demonstrates a steady increase when the applied bias is raised. The photocurrent density is 149 µA cm⁻² for 0.8 V, which is 2.7 times higher than 55 µA cm⁻² This could be attributed to the fact that the applied bias voltage across the for 0 photoanode would construct the increasing potential gradient within AIGaN: Ru NWs and separation of photoexcited holes and electrons, suggesting that optimization promo of photodetection performance can be legitimately modulated by bias voltage in our device.^{41]} Impressively, the PEC-type PD performs an obvious switching behavior under indicating its potential in self-powered device applications. To further study the 0 bias electrolyte concentration on the device performance, the photoresponse impact of switching behaviors at different H₂SO₄ concentration are compared in Figure 3c. With 1.15 mW cm⁻² illumination, the photocurrent of the device increases in 0.05 and 0.1 M H_2SO_4 electrolyte, but sharply reduces in 0.5 M H_2SO_4 electrolyte. Such phenomenon can be rationally explained by considering that the increase in the concentration of protons enhances the conductivity of the electrolyte, but it also inhibits the oxidation half This article is protected by copyright. All rights reserved.

reaction: (h⁺ + OH⁻ \rightarrow OH^{*}) at the photoanode/electrolyte interface. From the above analysis we can conclude that this system is more suitable for a weak acid aqueous environment. As critical indicators to judge how fast it responses to external illumination, we investigated the response and recovery characteristics of the device, as shown in he rise time (t_r) refers to the time required for the photocurrent to increase Figure 90% of the maximum value and the decay time (t_d) refers to the time from required for the photocurrent to recover from 90% to 10% of the maximum value. The corresponding tr of 83 ms and td of 19 ms indicate the high photoresponse speed of this reports have shown that the solution concentration in the electrolyte has a PD. E great influence on the photoresponse speed of PEC type photodetector^[20-21]. Thus the rising and decay time could highly depends on the H₂SO₄ concentration in this study which requires further optimization. To analyze the quantitative dependence of the photocurrent on the illumination intensity, the time-dependent photoresponse under different illumination power is presented in The measured photocurrent density at 0 bias as a function of illumination Figure 4 a. This article is protected by copyright. All rights reserved.

power intensity is plotted in Figure 4 b (red triangles), in which the photocurrent density is 5.86 µA cm⁻² at 0.2 mW cm⁻² and linearly increases to 71.5 µA cm⁻² at 1.5 mW cm⁻², indicating that the photoexcited electron-hole pairs are effectively separated and can hardly be captured by trap states.^[42] Here, we define responsivity (R) as the photocurrent generated by unit power of the incident light on the effective area, namely where P_m is the incident light power density. The calculated maximum R (blue circles in Figure 4b) of the self-powered AIGaN: Ru NW PEC PD is 48.8 mA W⁻¹ under the 254 nm light irradiation with the incident light intensity of 1.5 mW cm⁻². Such value is superior to most PEC-type UV PDs made by oxide-based nanostructures (see Table S1 for details),^[43-49] The extraordinary performance should be attributed to (1) the highly uniform AIGaN nanowire structure which possesses large light absorption capability, ensuring the high irradiation utilization; (2) the MBE-grown AIGaN materials with high crystalline quality enabling sufficient carrier photogeneration and trap-free carrier transport; (3) the high catalytic activity of Ru decoration which helps drive the photoexcited carriers to This article is protected by copyright. All rights reserved.

redox reaction sites, thus promoting the carrier separation and alleviating the photocorrosion of AlGaN nanowire surface. Furthermore, to testify the robustness of the experimental conditions, we execute an additional measurement of photocurrents under 0.4 V for 600 s. The device exhibits excellent stability in terms of on/off switching during shown in Figure 4c, indicating the reliability and accuracy of all the performed the tes measure For a better comparison of the solar-blind photodetection ability, Figure 4d plots the device performance of the newly designed PEC PDs in this work and recently reported self-powered solar-blind PEC PDs with nanostructure photoelectrodes based on wide bandgap oxides and sulfides.^[12,34-36] Obviously, our proposed AIGaN: Ru NW PEC PD shows ever-reported fastest photoresponse speed and highest responsivity, revealing the great potential of such device architectures in the field of the high-efficiency solarblind PEC photodetection. Moreover, the device performance of AIGaN: Ru NW PEC PDs not only on the excitation and transport of carriers within the depends semiconductor itself, but also on the diffusion of ions in the aqueous environment, which

provide us a new method to modulate the photodetection performance to meet desired

requirements.

Conclusion

In summary, we have explored the first self-powered solar-blind PEC PD based on highly-uniform n-AlGaN NWs grown by the MBE technique. Ru co-catalysts were used to decorate the surface of NWs to ensure that device performance would not be hampered by insufficient chemical activity during PEC photodetection. The PEC PD performance can be readily modulated by varying external experimental conditions, for instance, bias voltage, light source wavelength, light power density and electrolyte properties. The photoresponse characteristics of the fabricated self-powered PEC UV PD were investigated systematically. A large photocurrent density of 55 µA cm⁻² and excellent responsivity of 48.8 mA W⁻¹ at 254 nm illumination, together with extraordinary fast response speed (rise time of 83 ms and decay time of 19 ms) were measured, which are superior to ever-reported self-powered solar blind PEC PDs made of oxide and sulfidebased nanostructures. This work confirms the feasibility of III-nitride NWs as excellent

photoanodes to achieve highly sensitive and stable photodetection and demonstrates great prospects to realize highly efficient self-powered PDs for compact energy harvesting nanosystems in the near future.

The n-type AlGaN NWs were grown on Si (100) substrates using a Veeco plasma-

assisted molecular beam epitaxy (PAMBE). Before the Si wafer was loaded into the MBE chamber, the surface oxide on the wafer was removed using the HF-H₂O solution. After cleaning, the Si wafer was then outgassed in the load-lock chamber at 200 °C for 1 h followed by outgassing in the buffer chamber at 650 °C to remove any water components and organic-based contaminants. For the nanowire growth, the Si-doped GaN seeds were first nucleated on top of the Si wafer at ~500 °C for 1 min to reduce Ga adatom desorption and increase the nucleation probability. The nitrogen plasma source was operated with a flow rate of 1.0 standard cubic centimeter per minute (sccm) and RF-power of 300 W and metal source are supplied by standard Knudsen cell with beam

equivalent pressure (BEP) values of (Ga) 6.0 × 10⁻⁸ Torr. The k-cell temperature of Si dopant is 1180 °C. After the nucleating layer of less than 3 nm n-GaN was grown, the temperature was then increased to 630 °C to grown the n-AlGaN layer for 2 hours with Al and GaN BEPs of 5 × 10⁻⁸ and 9 × 10⁻⁸ Torr, respectively, while the Si cell temperature at 1180 °C with the same nitrogen plasma condition. was kept PL measurements were measured by an OceanOptics QEPro spectrometer, performed to investigate the optical properties of the nanowire samples (using 266 nm laser as the excitation source). The morphology of the AlGaN NWs was characterized using field scanning electron microscopy (FESEM) on Hitachi, SU8220 systems at an emission accelerating voltage of 3 kV. TEM, EDS, and high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) measurements were performed on a JEM-ARM200F instrument (Anhui University) with probe corrector operating at an acceleration voltage of 300 kV. The TEM specimens were prepared by focused ion beam (FIB). XPS was acquired on the Photoemission End station at the BL10B beamline in the National Synchrotron Radiation Laboratory (NSRL) in Hefei, China.

Before the PEC measurement, Ru co-catalysts were in-situ photodeposited from an aqueous RuCl₃ (10 mg/mL) solution for introducing active sites on the nanowire surface to accelerate the redox reaction (see Supporting Information S1 for details). The PEC experiments were conducted in a typical three-electrode cell. Pt mesh and Ag/AgCl were the counter electrode and reference electrode, respectively. The PEC cell was utilized tz with good transmittance for both visible and UV light. For the working made electrode, Ga-In eutectic alloy (Alfa Aesar) was first deposited on the backside of the Si substrate to form an ohmic contact, which was subsequently connected with a copper silver paste. The entire photoanode device, except the nanowire surface sheet usina (5×5 mm was then covered with an insulating epoxy to avoid any leakage current. The photoanodes were dried at room temperature in air for at least 24 h before measurement. M H₂SO₄ was used as the electrolyte instead of an alkaline solution since the A 0.01 alkali would readily corrode III-nitride materials.^[50] A Tanon UV-100 lamp was used to G65 nm monochromatic deep UV light and the light intensity was calibrated genera by an optical power meter (S401C and PM100D).



Supporting Information

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Supporting Information is available from the Wiley Online Library or from the author.

Author contributions

H.D.S. developed the idea and H.D.S., B.S.O., Z.T.M., and J.H.H. discussed the experiments. D.H.W., C.H., X.L., H.C.Z., H.B.Y. and S.F. fabricated the photocathode and performed the characterizations and photoelectrochemical experiments, collected, and analyzed the data. C.H., D.H.W., and X.L. performed photodeposition synthesis. D.H.W. and H.D.S. performed the STEM characterization. D.H.W., C. H. and H.D.S. wrote the initial draft of the paper and the manuscript was revised by other co-authors. All authors discussed the results and commented on the manuscript.



The authors declare no conflict of interest.



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Figure 2. Typical three-electrode PEC system built for evaluating the photodetection



Figure 4. a) Photocurrent density of the PD under the incident light intensity of 0.2, 0.65, 1.15, and 1.5 mW cm⁻². b) Fitting curve and corresponding calculated photoresponsivity of the PD under different light intensities in 0.01 M H₂SO₄. c) Long-time photocurrent measurements of the PD under 0.4 V bias potential with 1.15 mW cm⁻² illumination. d) The comparison of responsivity and recovery speed in previous self-powered solar-blind this work. PEC F uniform, self-assembled AIGaN nanowires fabricated as self-powered solar-blind Highly with fast-response speed and high responsivity is demonstrated, in the photodetector configuration of photoelectrochemical cell. Owing to the defect-free wide bandgap AlGaN-based nanowires with appropriate surface decoration, the high-performance solar-blind photodetection is realized. Coupled with the self-powered characteristics of photoelectrochemical photodetectors, the proposed novel device architecture unveils an opportunity for designing future energy-efficient and sustainable unprecedented optoelectronic system. This article is protected by copyright. All rights reserved.



