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Electronic Supporting Information for

Enhancing Photoelectrochemical Hydrogen Production of n⁺p-Si Hetero-junction Photocathode with Amorphous Ti and Ni Layers

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I. Experimental Section

1. Materials and chemicals

Potassium hydroxide, hydrogen peroxide, hydrochloric acid, hydrofluoric acid, ethanol, acetone, phosphorus oxychloride and other chemical compounds were obtained from Sinopharm group Co., Ltd without further purification. Target materials of nickel and titanium (> 99.999%) were purchased from Zhongnuo New Material Co., Ltd. All the solutions were prepared using Millipore deionized water (18.2 $M\Omega$ ·cm). The gases with high purity were received from Hangzhou Jingong Co., Ltd.

2. Preparation of graphite disk supports for Ni layer deposition

The graphite rods are cut into thin disks with a diameter of 6 mm and a thickness of \sim 0.4 mm. Such graphite disks have a geometric area of 0.2826 cm². They are sonicated subsequently in deionized water for 20 min, and then were dried in an oven at 105 °C for 10 min before use. The prepared graphite disks were used as the working electrode for Ni layer deposition by thermal evaporation method, with the deposition rate of 0.2-0.3 Å/s.

3. Preparation of n⁺p-Si

Micropyramid arrays were fabricated on 200 μ m thick p-type (100) Si wafers with dopant concentration of 4.5 ×10¹⁵ cm⁻³ – 1.5 ×10¹⁶ cm⁻³ by chemical etching in 45 vol% potassium hydroxide solution, hydrogen peroxide and hydrochloric acid aqueous solution (H₂O₂:HCl:H₂O = 1:1:1.5 in volume), and then washed with hydrofluoric acid (HF, 40 vol%). The n⁺ emitter layer was formed by thermal diffusion of POCl₃ at 880 °C for 100 min.

4. Preparation of n⁺p-Si/Ti/Ni photocathodes

Thermal evaporation method was taken to deposit titanium and nickel layer. Before addition of functional layers, hydrofluoric acid (40 vol%) was used to remove the

silica, then washed with water and ethanol for several times, dried at nitrogen atmosphere finally. In the high vacuum degree, titanium metal and nickel metal were separated in two tungsten boats, typically 5 nm Ti and 5 nm Ni layer were evaporated with the deposition rate of 0.2-0.3 Å/s successively with the evaporative current of 142 A and 128 A respectively. Afterwards, the Ti and Ni coated silicon micropyramids arrays were cut into pieces (0.8 cm \times 0.8 cm) for photocathodes fabrication. Subsequently, the back side of the coated n⁺p-Si substrate was first scratched with a diamond scribe. A Cu wire was embedding in a Ga/In mixture to form an Ohmic contact instantly. Silver paint was then used to immobilize the Cu wire and the Ga/In liquid. After drying, the Cu wire was passed into a glass tube, and the Si electrode and Cu wire were encased in Hysol 9460 epoxy. The synthesis of n⁺p-Si/5nm Ti and n⁺p-Si/5nm Ni are the same process as noted above.

II. Structure characterization

Scanning electron microscopy (SEM) was performed on a SIRON-100(FEI America) field emission scanning electron microscopy. The samples were pretreated by spraying gold for 60 s. The transmission electron microscopy (TEM) and the energy dispersive spectroscopy (EDS) spectra were gained by field emission transmission electron microscope (Tecnai G2 F20 S-TWIN) coupled with energy-dispersive X-ray spectrometer at an accelerating voltage of 200 kV. It is really hard to observe the thin layer on the surface of n⁺p-Si by using the general method like scrape the surface of n^+p -Si then ultrasonic for a period of time. Therefore, we took Focused ion beam (FIB) as the effective method to prepare the TEM sample. In this experiment, gallium (Ga) was used as the ion source. When Ga flowed to the sharp Tungsten (W) needle, and field ionization and post-ionization process occurred with a high extraction field (> 10^8 V/cm). With an accelerating voltage of 30 keV, Ga⁺ ion is accelerated toward the target of n⁺p-Si/5nm Ti/5nm Ni, it enters the sample and creates a cascade of events which results in the ejection of a sputtered particle. This Ga⁺ cut n⁺p-Si/5nm Ti/5nm Ni into thin slices with a thickness of 80 nm to 100 nm, then glued onto copper grids for further TEM and elemental mapping characterization. X-ray photoelectron spectra (XPS) were obtained on Escalab 250Xi (Thermo Fisher Scientific) X-ray photoelectron spectrometer with an Mg K α X-ray resource. Calibration of binding energy was carried out by setting binding energy of C1s peak to 284.6 eV. UV-Vis spectra were recorded on a UV-3150 UV-Vis spectrophotometer and were collected over the range of 400-1200 nm. The crystal structures of samples were characterized using a X'Pert PRO X-ray diffractometer (XRD) (PNAlytical Netherlands) using Cu K α radiation ($\lambda = 1.54056$ Å), employing a sampling width of 0.033° in the 2 θ range of 10-80°. Wavelength-dependent external quantum efficiencies measurements were collected by using a QEX7 (PV Measurements, Inc.) calibrated by a NIST-certified Si photodiode.

III. Electrochemical and photoelectrochemical tests

Electrochemical measurements were performed in three-electrode a electrochemical cell using a Bio-Logic VSP potentiostat. All measurements were performed in 70 mL of 1 M KOH (aq) electrolyte and constantly purged with H₂ gas to maintain a constant Nernst potential for H^+/H_2 redox couple by using a platinum plate as the counter electrode, and mercuric oxide electrode (Hg/HgO) as the reference electrode. To allow potentials to be referenced against the reversible hydrogen electrode (RHE), the Hg/HgO was calibrated against the reversible hydrogen potential, using platinum plate as both the working and counter electrodes. Cyclic voltammograms was measured from -0.8 V to - 1.1 V vs. RHE at a scan rate of 3 mV/s. Electrochemical impedance spectroscopy (EIS) was performed in potentiostatic mode at - 0.2 V (vs. RHE) from 200 kHz to 60 mHz.

The photoelectrochemical characterizations of photocathodes were also measured in a three-electrode configuration using a Bio-Logic VSP potentiostat. The prepared n^+p -Si/Ti/Ni served as the working electrode, a platinum plate as a counter electrode, and Hg/HgO as reference electrode. The simulated 1.0 Sun illumination was supported by 500 W Xe short arc lamp solar simulator (Perfect Light FX 3000) with an AM 1.5 G filter. The light intensity was calibrated to 100 mW·cm⁻² with an AM 1.5 G filter by an irradiatometer (Aulight, Beijing, CEL-VIS 400). The "1 sun" calibration was carried out by the irradiatometer inside the cell. We calibrated the solar simulator with the Oriel PV Reference Cell (Newport Model 91150V). All measurements were performed in 70 mL of 1 M KOH (aq) electrolyte and constantly purged with H₂. The potentials were also referenced against the reversible hydrogen electrode (RHE). Electrochemical impedance spectroscopy (EIS) was performed was performed when the working electrode was biased at a constant potential of 0.1 (vs. RHE) in 100 mW·cm⁻² (AM 1.5 G filter), while sweeping the frequency from 200 kHz to 60 mHz. The capacitance measurement on the electrode/electrolyte was employed to determine the flat band potential (E_{fb}) and carrier density (N_A), which can be obtained in a Mott-Schottky (M-S) plot with $1/C^2$ vs. potential at a fixed frequency of 100 kHz in dark as the following equation below:

$$1/C^2 = 2(V - V_{fb} - k_B T/e)/\varepsilon_0 \varepsilon e N_A A^2$$

Where C is the space-charge capacitance of the semiconductor, V is the applied potential, V_{fb} is the flat-band potential, k_B is the Boltzmann constant, T is the absolute temperature, ε_0 is the electric permittivity of a vacuum, ε is the dielectric constant of semiconductor (taken as 11.8), N_A is the carrier density that can be calculated from the slope, and the A is the area of electrode. The ABPE was estimated using the following expression:

 $ABPE=|J_{ph}(mA \cdot cm^{-2}) \times (0 - V_b)(V)|/P_{in}(mW \cdot cm^{-2})$

Where J_{ph} is the photocurrent, P_{in} is the irradiance of the AM 1.5G (100 mW·cm⁻²), V_b is the applied potential (vs. RHE). The Faradaic efficiency of the photoelectrochemical cell was determined by measured the amount of generated H_2 and O_2 using a gas chromatography (GC, 9790II, Hangzhou Gatai Scientific Instruments), and N_2 severed as carrier gas. A chronoamperometry test was conducted on the n⁺p-Si/5nm Ti/5nm Ni photocathode at 0 V (vs. RHE) under simulated 1 Sun irradiation. A syringe was used to transfer 200 µL gas from the sealed polytetrafluoroethylene cell to the GC instrument every 30 minutes. The Faradaic efficiency was then calculated by comparing the theoretical amount with the GC-detected amount of generated H₂ and O₂. The spectra were measured by a thermal conductivity detector (TCD), at a column temperature of 20 °C, injection temperature

of 80 °C, and thermal conductivity cell of 100 °C.

IV. SEM image and EDS of n⁺p-Si/5nm Ti/5 nm Ni



Figure S1. (a) Top-view SEM image of n⁺p-Si/5nm Ti/5nm Ni. Elemental mapping images of Ni (b), Si (c), and Ti (d), respectively.



V. XRD patterns of n⁺p-Si, n⁺p-Si/5nm Ti and n⁺p-Si/5nm Ti/5nm Ni

Figure S2. XRD patterns of n⁺p-Si, n⁺p-Si/5nm Ti and n⁺p-Si/5nm Ti/5nm Ni.

VI. External quantum efficiencies of n^+p -Si, n^+p -Si/5nm Ti and





Figure S3. External quantum efficiencies of n^+p -Si, n^+p -Si/5nm Ti and n^+p -Si/5nm Ti/5nm Ni.

VII. J-V curves of low applied potential for different photoelectrodes



Figure S4. *J-V* curves at lower applied potentials for bare n^+p -Si, n^+p -Si/ 5 nm Ti, n^+p -Si/ 5 nm Ti/ 5 nm Ni.

VIII. *J-V* curves of n⁺p-Si, n⁺p-Si/5nm Ti and n⁺p-Si/5nm Ni



Figure S5. *J-V* curves of n⁺p-Si, n⁺p-Si/5nm Ti and n⁺p-Si/5nm Ni.

IX. Band diagrams of n⁺p-Si and n⁺p-Si/5 nm Ti/5 nm Ni in electrolyte under irradiation



Figure S6. Band diagrams of n^+p -Si and n^+p -Si/5 nm Ti/5 nm Ni in electrolyte under irradiation. Figure S5 shows the energy band diagrams obtained at open-circuit conditions under illumination. As can be seen, open circuit voltage (V_{oc}) of n^+p -Si can be determined from the quasi-Fermi level of electrons in n^+p -Si (E_{f,n}) and E(H⁺/H₂). When the metal and n^+p -Si are in contact, the free electrons will transfer between metals and n^+p -Si until the Fermi levels of metals and n^+p -Si (E_{f,n}) are aligned, due to the work function difference.^[1] The low work function of Ti affords a high Schottky barrier to the n^+p -Si valence band.^[2] This high Schottky barrier results in a high built-in potential (V_{bi}), thus reduces the majority carrier (hole) density at the band edge of n^+p -Si, affording a high photovoltage finally.^[3] In addition, the large work function of Ni results in a high V_{oc}. By inserting the Ti layer underneath Ni, a larger V_{oc} and a higher photovoltage are obtained compared with bare n^+p -Si.^[4]

X. SEM image of n⁺p-Si/5nm Ti/Pt NPs



Figure S7. SEM image of Pt nanoparticles (NPs) electrodeposited on n^+p -Si/5nm Ti. The preparation of n^+p -Si/5nm Ti/Pt was conducted by photo-assistant electrodeposition at -0.7 V (vs. SCE) in the electrolyte of 1.0 mmol H₂PtCl₆ solution (a deposition time of 60 s).

XI. *J-V* curves of n⁺p-Si/5nm Ti/Pt NPs photocathode



Figure S8. A typical *J-V* curves of n^+p -Si/5nm Ti/Pt NPs photocathode under 100 mW·cm⁻² with an AM 1.5 G filter in 1 M KOH. Inset image is the *J-V* curves with the lower applied potentials.

XII. Reflectance spectrum of n⁺p-Si/5 nm Ti/Pt NPs



Figure S9. Reflectance spectra of n⁺p-Si /5 nm Ti, n⁺p-Si /5 nm Ti/5 nm Ni, n⁺p-Si /5 nm Ti/Pt NPs.

XIII. J-V curves of n⁺p-Si with different thickness of Ti and Ni layers



Figure S10. (a) *J-V* curves of n^+p -Si/Ti/Ni with 5 nm Ti and Ni with different thicknesses. (b) *J-V* curves of n^+p -Si/Ti/Ni with 5 nm Ni and Ti with different thicknesses, it was clearly seen that n^+p -Si/5 nm Ti/5 nm Ni exhibited the optimal performance in photocurrent (0 V vs. RHE) and onset potential. All tests were irradiated under AM 1.5 G illumination (100 mW·cm⁻²).

XIV. ABPE of n⁺p-Si with different thickness of Ti and Ni layers



Figure S11. ABPE curves of n^+p -Si/Ti/Ni photocathodes measured under AM 1.5 G illumination (100 mW·cm⁻²) with different thicknesses, (a) Ti layer and (b) Ni layer.

XIV. The stability test plots of n⁺p-Si/5nm Ni



Figure S12. The stability test plots of n^+p -Si/5nm Ti and n^+p -Si/5nm Ni at constant current density of -10 mA·cm⁻² under 100 mW·cm⁻² irradiation in 1M KOH.

XV. The SEM image of n⁺p-Si/5nm Ti/5nm Ni after PEC test



Figure S13. The SEM image of n⁺p-Si/5nm Ti/5nm Ni (a) before, and (b) after PEC test.

XVI. SEM image and EDS spectra of amorphous Ni/graphite electrode



Figure S14. (a) SEM image of bare graphite; (b) SEM image and EDS spectrum of amorphous Ni/graphite electrode. SEM image of amorphous Ni/graphite electrode illustrate no morphological differences with bare graphite, and the EDS spectrum confirms the Ni layer is successfully integrated with graphite electrode.

XVII. Electrocatalytic activity of amorphous Ni layer on graphite disk



Figure S15. (a) *J-V* curves of amorphous Ni layer (5 nm) on the graphite disk, the amorphous Ni layer shows an overpotential of -198 mV to drive the current density of -10 mA·cm⁻² (b) Stability test of amorphous Ni layer (5 nm) on the graphite disk at constant current density of -10 mA·cm⁻². The amorphous Ni layer also exhibits the favourable long-term stability in EC system in alkali that only a small decrease of operation potential is observed at the constant current density of -10 mA·cm⁻² after 70h test.





Figure S16. (a) XPS spectra of Ni 2p before and after PEC-HER of n^+p -Si/Ti/Ni. (b) XPS spectra of O 1s before and after PEC-HER of n^+p -Si/Ti/Ni. In addition to the changes in the main peaks area, some of the spectral peaks were founded to shift to higher binding energies, thus indicating that the transition is accompanied by a significant change in the energy level of the material. (c) XPS spectra of Ti 2p before and after PEC-HER of n^+p -Si/Ti/Ni. (d) The XPS element survey of n^+p -Si/Ti/Ni illustrates the existence of Ti, Ni, and O elements before and after test.

Table S1. Comparison of the PEC performance of n⁺p-Si/5nm

Photocathode	Onset potential	J at 0 V vs. RHE (mA·cm ⁻²)	Electrolyte	Light power	Ref
Our work	0.61	-38.7	1M KOH	1 Sun	
p-Si/Ti/NiFe LDH	0.3	-7	1М КОН	1 Sun	S ^[5]
p-Si/Ti/Ni	0.3	-5	1М КОН	1 Sun	S ^[6]
Si/SiO _x /Ni-rich	0.5	-20	1М КОН	1 Sun	S ^[7]
n ⁺ p-Si/TiO ₂ /Pt	0.5	-15	1М КОН	0.39 Sun	S ^[8]
MoS _x Cl _y /Si MPs	0.41	-43	$0.5M H_2 SO_4$	1 Sun	S ^[9]
p-Si/SiO _x /TiO ₂	0.5	-33	1M HClO ₄	1 Sun	S ^[10]
p-Si/NiCoSe _x	0.25	-37.5	$1 M H_2 SO_4$	1 Sun	S ^[11]
p-Si/Ti/Pt/SrTiO ₃	0.42	-26.3	1M HClO ₄	1 Sun	S ^[4a]
n ⁺ p-Si/Ni-Mo	0.44	-10.2	KHP buffer	1 Sun	S ^[12]
Si MWs/CoS ₂	0.248	-3.22	$0.5M H_2 SO_4$	1 Sun	S ^[13]
n^+p -Si/MoS ₂	0.31	-14	$0.5M H_2 SO_4$	1 Sun	$S^{[14]}$
p-Si/TiO ₂ /MoS ₂	0.35	-28	$0.5M H_2 SO_4$	1 Sun	S ^[15]
p-Si MWs/Co-P	0.47	-25	$0.5M H_2 SO_4$	1 Sun	S ^[16]
n ⁺ p-Si/CoP	0.46	-19.8	$0.5M H_2 SO_4$	1 Sun	S ^[17]

Ti/5nm Ni photocathode with other Silicon based photocathodes

n ⁺ p-Si/Ti/TiO ₂ /Pt	0.52	-22	1M HClO ₄	1 Sun	$\mathbf{S}^{[18]}$
Si/SiO ₂ /NiO _x	0.5	-30	1M KOH	1 Sun	S ^[19]

Table S2. The parameters of equivalent circuit model for differentphotoelectrodes

Photoelectodes	$R_1(\Omega)$	Q ₁ (F)	$R_{2}\left(\Omega ight)$	Q ₂ (F)
n ⁺ p-Si/5nm Ti	67	9.25×10^{-4}	41	1.32×10^{-6}
n ⁺ p-Si/5nm Ti/5nm Ni	21	1.186×10^{-3}	4	2.082×10^{-6}

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