Supporting Information

Inverse opal anatase/rutile TiO₂ multi-heterojunctions enable efficient photoelectrochemical water splitting

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Experimental Section

Materials

Fluorine-doped tin oxide (FTO) was purchased from Hepta Chroma. Tetrabutyl titanate (TBT), hydrochloric acid, ethanol, titanium trichloride, acetone, and were purchased from Aladdin (Shanghai, China). All chemicals were used without further purification.

Synthesis of inverse opal TiO₂ (IO-TiO₂)

The FTO glass slide was placed vertically in a solution containing monodisperse polystyrene (PS: 230 nm, 0.2 wt % in water), as the water evaporated and the meniscus swept across the substrate, capillary forces caused the FTO glass slide. The PS spheres on the sheet surface are arranged in order, and the inverse opal TiO_2 structure is obtained by impregnation for 15 min and calcination at 450 °C. Among them, all the solutions of the dipping method are solutions prepared by volume percentage of titanium tetrabutoxide (21.5%), hydrochloric acid (8.6%), absolute ethanol (43.0%) and deionized water (26.9%).

Preparation of inverse opal TiO₂/nanorods (IO-TiO₂/NRs)

Using the as-synthesized IO-TiO₂ as a carrier, TiO₂ nanorods were directly grown on the surface of IO-TiO₂ by adding TBT precursor via a hydrothermal reaction. Briefly, a piece of IO-TiO₂-covered glass was placed at an angle into a Teflon autoclave. Then, tetra butyl titanate, concentrated hydrochloric acid, and water were mixed of the mixed solution was poured into the reactor, hydrothermally treated at 150 °C for 5 h, the FTO substrate was taken out, rinsed with deionized water, and air-dried to prepare IO-TiO₂/NRs. For comparison, FTO-NRs were prepared by directly hydrothermally growing TiO₂ nanorods on FTO substrates under the same conditions.

Synthesis of IO-TiO₂/NRs-TiO₂ multi-heterojunction photoanode

Using IO-TiO₂/NRs as carrier and TiCl₃ as titanium source, rutile TiO₂ were grown on TiO₂ nanorods by hydrothermal method, preparing IO-TiO₂/NRs-TiO₂ multi-heterojunction photoanode. Specifically, 20 mL deionized water and 0.25 mL HCl were mixed uniformly, and then TiCl₃ (0.25 mL) was slowly added dropwise to the solution, and then the solution containing TiCl₃ was poured into the already placed IO-TiO₂/NRs glass slides in a beaker, and then place the beaker in an oven (80 °C) for 120 min. Finally, the prepared samples were washed (ethanol), dried (60 °C) and calcined (450 °C), producing IO-TiO₂/NRs-TiO₂ catalyst. In the same way, small rutile grains were directly grown on FTO-NRs to prepare a comparative catalyst (NRs-TiO₂).

Characterization

Scanning electron microscope (SEM, JEOL JSM6330 F), Transmission electron microscope (TEM, Tecnai G2 F30 S-TWIN) and scanning transmission electron microscope (STEM) mapping were used to characterize the morphology and structure of the as-synthesized samples, while X-ray diffraction (XRD, D/MAX2500 V diffractometer) were employed to investigate their surface area, crystal structure, and binding energy, respectively. The chemical states of the samples were measured by using X-ray photoelectron spectroscopy (XPS, Thermo ESCALAB250Xi). In addition, photoluminescence (PL, FLS980), time-resolved photoluminescence (TRPL), transient-state surface photovoltage (TS-SPV), and ultraviolet visible spectrophotometry (Shimadzu UV-2600) were used to measure their photoelectronic properties.

Photoelectrochemical (PEC) Measurements

The PEC properties of samples were measured in a standard three-electrode cell. The reference wand counter electrodes were Ag/AgCl electrode and Pt wire, respectively. A 1.0 M KOH aqueous solution was used as the electrolyte. The photoelectrode film area was fixed at 1 cm² for all the PEC measurements. The measured potential versus Ag/AgCl reference electrode was converted to the reversible hydrogen electrode (RHE) scale according to the Nernst equation:

$$E_{RHE} = E_{Ag/AgCl} + 0.059 \times pH + 0.1976 \tag{1}$$

where E_{RHE} is the converted potential *versus* RHE, and $E_{Ag/AgCl}$ is the experimentally measured potential *versus* the Ag/AgCl reference electrode. The photoelectrodes with an exposure area of 1×1 cm² were illuminated under AM 1.5G light (100 mW cm⁻², 300 W Xe lamp). Linear sweep voltammetry (LSV) and Electrochemical impedance spectroscopy (EIS) were recorded using an electrochemical workstation (CHI instrument, CHI 660C).

The η_{sep} is the separation efficiency of the bulk charge in the photoelectrode, which can be derived from the following equation¹:

$$\eta_{sep} = J_{KOH} / J_{abs} \tag{2}$$

where, J_{KOH} is the photocurrent density obtained in 1.0 M KOH electrolyte solutions, J_{abs} represents the photocurrent density when the absorbed photon is fully converted into an electric current. According to the following equation, the overlapping region between the UV-Vis absorption spectrum and the AM 1.5G solar spectrum can be calculated²:

$$J_{abs} = \int \frac{\lambda}{1240} \cdot \varphi AM \ 1.5G(\lambda) \cdot LHE \ d\lambda \tag{3}$$

$$LHE = 1 - 10^{-A(\lambda)} \tag{4}$$

where, λ is the wavelength (nm), $\varphi AM 1.5G(\lambda)$ is the solar spectral irradiance (W·m⁻²·nm⁻¹) that provides the simulation, *LHE* is the light capture efficiency, and $A(\lambda)$ is the absorbance at the corresponding wavelength λ .

Applied bias photon-to-current efficiency (ABPE) is calculated by the following equation³:

$$ABPE(\%) = J_{pH} \times (1.23 - V_{app}) / P_{light} \times 100\%$$
(5)

where, J_{ph} is the photocurrent density, V_{app} is the application bias (vs. RHE), and P_{light} is the light intensity of AM 1.5G.

The incident photon to current conversion efficiency (IPCE) was calculated from amperometry measurements with three electrodes configuration at 1.23 V with respect to the RHE according to the follow equation:

$$IPCE = (1240 \times I) / (\lambda \times J_{light})$$
(6)

where *I* is the photocurrent density (mA cm⁻²) measured at the specific wavelength, λ is the wavelength of the incident light, and *J* is the irradiance (mW cm⁻²) measured at the specific wavelength. The EIS were recorded under AM 1.5G illumination (100 mW cm⁻²) at an AC potential frequency range from 10⁵ to 0.1 Hz with an amplitude of 10 mV. In the PEC hydrogen production process, a photoanode consisting of 5 mg IO-TiO₂/NRs-TiO₂ is used, with triethanolamine (10% concentration) as the sacrificial agent, and a 365 nm LED lamp serving as the light source.

Use the following equations to fit the time-resolved photoluminescence spectra and calculate average lifetimes (τ_{ave}):

$$\tau_{ave} = \left(A_1 \tau_1^2 + A_2 \tau_2^2\right) / \left(A_1 \tau_1 + A_2 \tau_2\right) \tag{7}$$

Photocatalytic Hydrogen Production

Photocatalytic hydrogen production was carried out in a 160 mL optical reaction vessel at room temperature with continuous stirring. 5 mg of the photocatalyst was scraped off the FTO and suspended in a solvent mixture consisting of triethanolamine (TEA) as sacrificial agents (10% concentration) and deionized water. In addition, the hydrogen evolution performance without the addition of sacrificial agents was also tested as a comparison. The mixture was thoroughly mixed to

achieve a homogeneous suspension, which was then stirred and purged with nitrogen for approximately 10 min to remove the air. Visible light irradiation was provided by an AM 1.5G light (100 mW cm⁻², 300 W Xe lamp). Circulating water was used throughout the reaction to maintain a steady temperature of approximately 25 °C. To measure hydrogen production, 200 μ L samples of the headspace were periodically withdrawn and analyzed using a gas chromatograph equipped. The hydrogen concentration was determined using a calibration curve based on an internal hydrogen standard.

Computational methods

Electronic structure calculations were performed with the generalized gradient approximation (GGA) of the Heyd-Scuseria-Ernzerhof screened hybrid functional (HSE06) to the exchangecorrelation functional for calculating the band gap.^{4, 5} The kinetic cutoff energy for a plane-wave function was 35 Ry and the cut-off energy for the real space grid was 250 Ry. A 5 × 5 × 8 Monkhorst-Pack k-point sampling was used for the rutile unit cell and a 6 × 6 × 3 Monkhorst-Pack k-point sampling for the anatase unit cell. The anatase (101), rutile (110), rutile (111) and rutile (101) were modelled by eight layers of atoms with six TiO₂ building blocks and a 3 × 3 × 1 Monkhorst-Pack kpoint sampling was used.



Fig. S1 SEM images of (a) PS spheres, (b) IO-TiO₂, (c) IO-TiO₂/NRs, and (d) IO-TiO₂/NRs-TiO₂.

Fig. S2 SEM images of the IO-TiO₂/NRs prepared at a temperature of 80 °C with different concentration of TBT: (a) 0.1 mL, (b) 0.15 mL, and (c) 0.2 mL. SEM images of the IO-TiO₂/NRs-TiO₂ prepared at a temperature of 80 °C with different concentration of TiCl₃: (d) 0.15 mL, (e) 0.25 mL, and (f) 0.35 mL.

Fig. S3 TEM images of IO-TiO₂/NRs-TiO₂.

Fig. S4 Selective area electron diffraction of (a) anatase and (b) rutile in IO-TiO₂/NRs-TiO₂.

Fig. S5 EDS elements mapping of IO-TiO₂/NRs-TiO₂.

Fig. S6 (a, b) STEM images and (c) mapping of NRs-TiO₂ in IO-TiO₂/NRs-TiO₂.

Fig. S7 Cross-section SEM image of IO-TiO₂/NRs-TiO₂.

Fig. S8 (a) 2D height sensor image and (b) corresponding CAFM current images of IO-TiO₂/NRs-TiO₂, (c–f) line scan of surface morphology and current distribution of the dashed line 1 and 2, respectively.

Fig. S9 XPS full spectrum of IO-TiO₂/NRs-TiO₂.

Fig. S10 XPS spectra of (a) Ti 2p and (b) O 1s of IO-TiO₂, IO-TiO₂/NRs, and IO-TiO₂/NRs-TiO₂.

Fig. S11 UV-vis absorption spectra of $IO-TiO_2/NRs-TiO_2$ with different concentration of (a) TBT, (b) and TiCl₃.

Fig. S12 UV-vis absorption spectra of IO-TiO₂, IO-TiO₂/NRs, and IO-TiO₂/NRs-TiO₂.

Fig. S13 SEM images of (a) $IO-TiO_2/TiO_2$, (b) FTO-NRs, and (c) NRs-TiO₂.

Fig. S14 XPS spectra of (a) Ti 2p and (b) O 1s of FTO-NRs, IO-TiO₂/TiO₂ and NRs-TiO₂.

Fig. S15 LSV curves of NRs-TiO₂, FTO-NRs, and IO-TiO₂/TiO₂.

Fig. S16 (a) ABPE and (b) Charge separation efficiency of IO-TiO₂, IO-TiO₂/NRs, and IO-TiO₂/NRs-TiO₂.

Fig. S17 (a) SEM and (b) XRD patterns of $IO-TiO_2/NRs-TiO_2$ before and after long-term photocurrenttime test.

Fig. S18 EIS of IO-TiO₂, IO-TiO₂/NRs, and IO-TiO₂/NRs-TiO₂.

Materials	Electrolyte	Photocurrent	Lamp	Ref.
BCN/CsTaWO ₆₋ _x N _x array	1.0 M KOH	1.90 mA cm ⁻² at 100 mW cm ⁻² 1.23 V vs. RHE 300 W Xe lamp		6
$SrTaO_2N$	1.0 M KOH	1.10 mA cm ⁻² at 100 mW cm ⁻² 1.23 V vs. RHE AM 1.5G sunlight		7
$LaTaON_2$	1.0 M KOH	0.80 mA cm ⁻² at 1.6 V <i>vs.</i> RHE	30 mW cm ^{−2} 500 W Xe lamp	8
TiO ₂ array	1.0 M KOH	1.24 mA cm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² AM 1.5G	9
BiVO ₄	1.0 M KOH	0.66 mA cm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² Xe lamp	10
Branched TiO ₂ - NRs	1.0 M KOH	1.83 mA cm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² AM 1.5G solar light	11
2D Porous TiO ₂	1.0 M KOH	1.0 mA cm ⁻² 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² 150 W Xe lamp	12
$ZnFe_2O_4/TiO_2$	1.0 M KOH	0.78 mA cm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² AM 1.5G	13
$g-C_3N_4/TiO_2$	1.0 M KOH	0.80 mA cm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² 300 W Xe lamp	14
TiO ₂ nanoflower	1.0 M KOH	0.75 mAcm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² 300 W Xe lamp	15
IO-TiO ₂ /NRs- TiO ₂	1.0 M KOH	1.01 mAcm ⁻² at 1.23 V <i>vs.</i> RHE	100 mW cm ⁻² 300 W Xe lamp	This work

Table S1 PEC water splitting performance of $IO-TiO_2/NRs-TiO_2$ photoelectrode in comparison of various photoelectrodes.

Photocatal yst	Morphology	Co- catalyst	Sacrificial reagent	Lamp	H ₂ evolution rate (μmol g ⁻ ¹ h ⁻¹)	Ref.	
Anatase TiO ₂	Mesoporous structure	Pt (1 wt%)	Methanol (20 vol%)	100 mW cm ⁻² 300 W Xe lamp	1362	16	
Rutile TiO ₂	Nanoparticle	Pt (1 wt%)	Methanol (10 vol%)	100 mW cm ⁻² 300 W Xe lamp	34	17	
S doped rutile TiO ₂	Nanoparticle	Pt (0.5 wt%)	Methanol (25 vol%)	100 mW cm ⁻² AM 1.5G	258	18	
Anatase TiO₂	Nanoparticle	None	Glycerol (5 vol%)	7.5 W m ⁻² Philips PL-S 9W lamps	115	19	
Anatase TiO ₂	Nanoparticle	Pt (2.5 wt%)	Ethanol (20 vol%)	100 mW cm ⁻² 300 W Xe lamp	1100	20	
Anatase TiO ₂	Nanoparticle	Pt (1 wt%)	Methanol	12.5 W m ⁻² Philips PL-S 9W lamps	900	21	
poly- pyrrole TiO ₂	Nanoparticle	Pt (0.1wt%)	Methanol (20 vol%)	100 mW cm ⁻² 300 W Xe lamp	850	22	
TiO ₂	Inverse opal	Pt (0.2wt%)	Formic acid- sodium formate 0.1 M	365 nm Philips PLS-10	1300	23	
B-TiO ₂ /g- C ₃ N ₄	Hollow spheres	None	Triethanolam ine (10 vol%)	100 mW cm ⁻² 300 W Xe lamp	808.97	24	
In ₂ S ₃ /Pt- TiO ₂	Nanoparticle	Pt (1 wt%)	Na ₂ S, Na ₂ SO ₃ (0.25 M)	100 mW cm ⁻² 300 W Xe lamp	90	25	
Anatase/ Rutile TiO ₂	IO-NRs- nanosheets	Pt (1 wt%)	Methanol (10 vol%)	100 mW cm ⁻² 300 W Xe	1308.4	This work	
Anatase/ Rutile TiO ₂	IO-NRs- nanosheets	none	TEA (10 vol%)	100 mW cm ⁻² 300 W Xe	555	This work	

Table S2 Photocatalytic water splitting performance of IO-TiO₂/NRs-TiO₂ in comparison of various reported photocatalysts.

Sample	A ₁	τ ₁	A ₂	τ2	R ²
IO-TiO ₂	1754.3	7.0	633257.2	1.1	0.990
IO-TiO ₂ /NRs	911.2	11.1	164784.4	1.4	0.997
10-TiO ₂ /NRs-TiO ₂	67085.4	1.9	1038.1	22.7	0.998

 Table S3 The fitted parameters of time-resolved photoluminescence.

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