Supporting information

NiS co-catalyst decorated $Zn_3In_2S_6$/g-C_3N_4 type-II ball-flower like nanosphere heterojunction for efficient photocatalytic hydrogen production

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1. Characterization

X-ray diffraction patterns (XRD) analysis was proceeded on a Bruker D8 Advance diffractometer to study the crystal phase of the photocatalysts. X-ray photoelectron spectroscopy (XPS) (ESCALAB 250 xi, USA) with Al Kα radiation sources was employed to verify the elemental chemical states. The surface morphology and microstructure of the photocatalysts were studied by scanning electron microscopy (SEM) (Phillips XL-30 FEG/NEW) and transmission electron microscopy (TEM) (Phillips Model CM200), respectively. N_2 adsorption-desorption measurement (Quantachrome Autosorb-iQ-AG instrument) was used to determine the specific surface area and pore structure of the samples. Ultraviolet visible diffuse reflection spectra (UV-vis DRS) and photoluminescence (PL) spectra were recorded to analyze the optical properties. The Fluorescence lifetime spectrophotometer (FLS980) was used to record the time-resolved fluorescence decay curves.

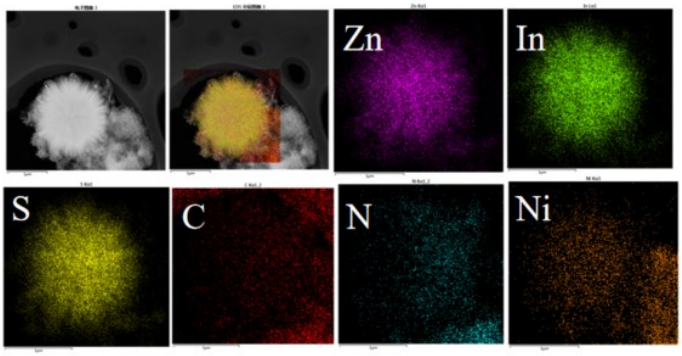

Fig. S1 Elemental mapping images of the 2.0% NiS/ZIS/CN composite.

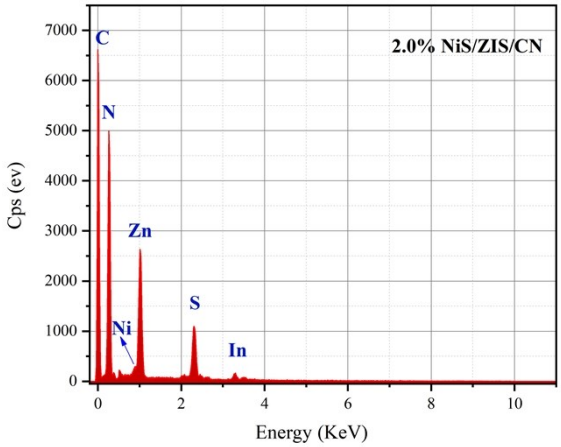

Fig. S2 The EDS spectra of 2.0%NiS/ZIS/CN) composite.

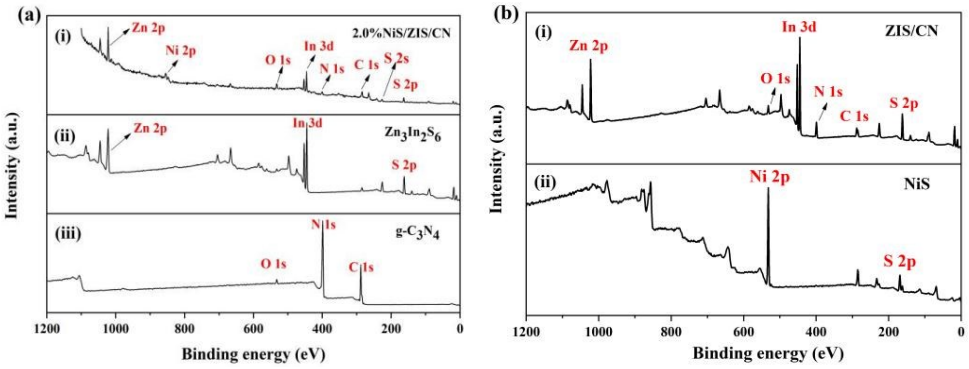

Fig. S3 Full survey scanned XPS spectra of (a) CN, ZIS, 2.0% NiS/ZIS/CN and NiS/CN and (b) NiS composites.

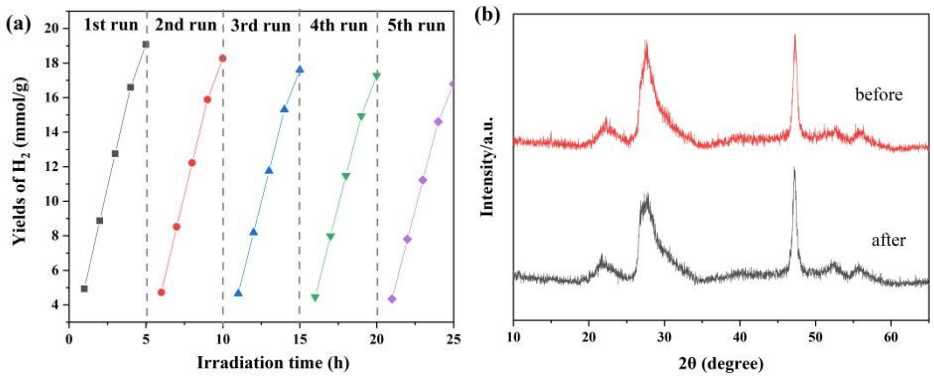

Fig. S4 (a) Photocatalytic H_2 production by the 2.0% NiS/ZIS/CN sample over 5 cycles; (b) XRD patterns of the 2.0% NiS/ZIS/CN composite before and after 5 cycles; (c) TEM of the 2.0% NiS/ZIS/CN composite before and after 5 cycles.

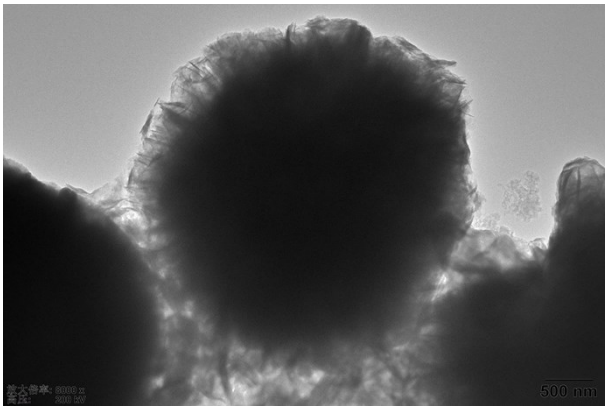

Fig. S5 TEM image of the 2.0% NiS/ZIS/CN sample over 5 cycles.

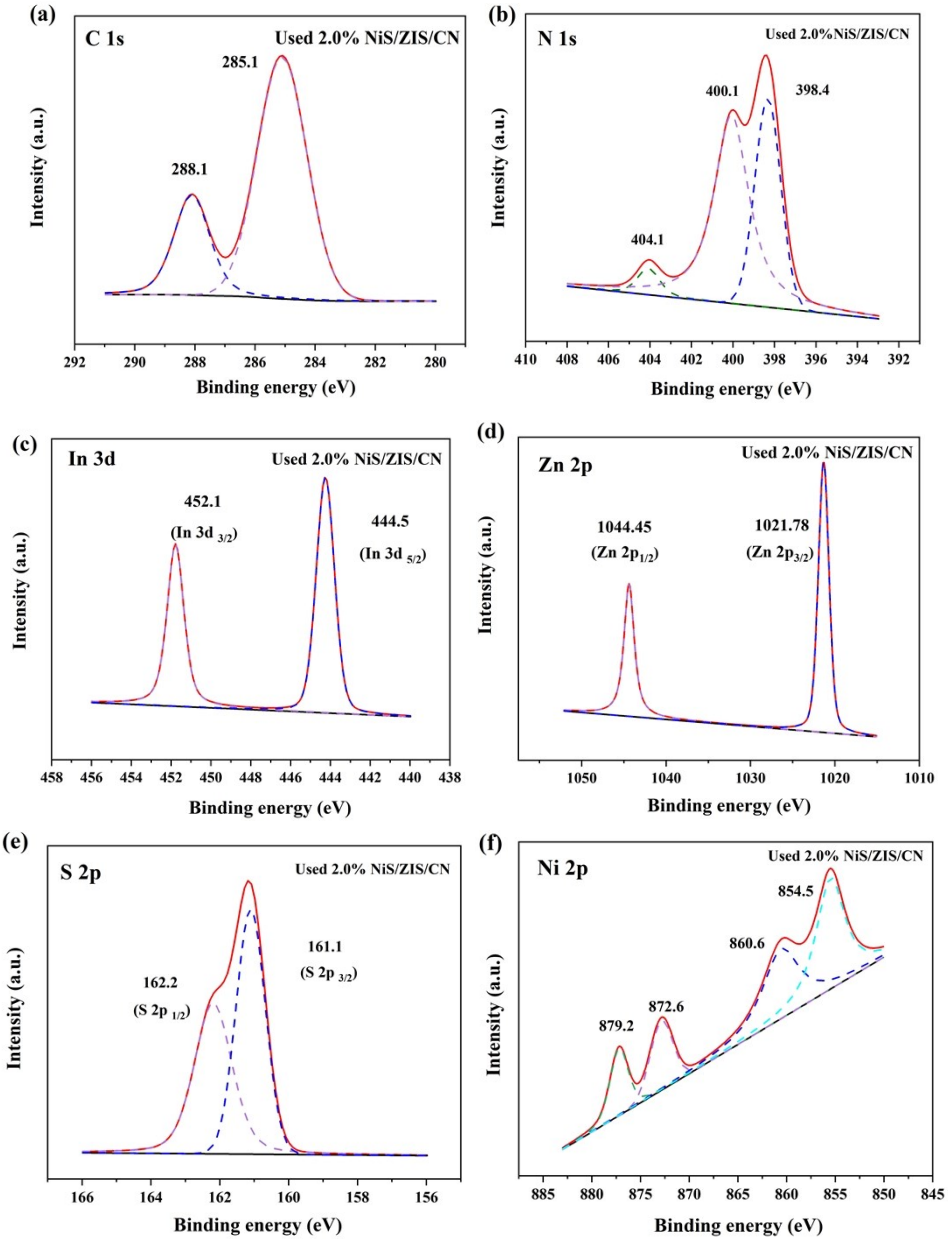

Fig. S6 (a) High-resolution of C 1s and (b) N 1s for CN and 2.0% NiS/ZIS/CN. (c) In 3d spectra and (d) Zn 2p spectra of 2.0% NiS/ZIS/CN composite. (e) S 2p spectra of ZIS and 2.0% NiS/ZIS/CN. (f) Ni 2p spectra of 2.0% NiS/ZIS/CN composite over 5 cycles.

Table S1. The surface properties of samples.

Samples	S_{BET} (m^2/g)	Pore diameter (nm)
CN	21.49	4.151
ZIS	87.23	4.153
NiS	10.51	1.938
NiS/CN	48.09	3.821
ZIS/CN	86.35	3.833
1.0% NiS/ZIS/CN	91.08	4.152
1.5% NiS/ZIS/CN	81.71	4.157
2.0% NiS/ZIS/CN	108.10	3.830
2.5% NiS/ZIS/CN	84.06	3.838

Table S2. The activity corresponding to the type and dosage of sacrificial agent.

Sacrificial agent	Amount（mL）	Corresponding activity(mmol·g^{-1}·h^{-1})
Glycerine	20	2.976
Lactic acid	20	3.025
Methyl alcohol	20	3.436
TEOA	10	3.497
TEOA	20	4.135
TEOA	30	3.682