

Supporting Information

Heterojunction Design between WSe₂ Nanosheets and TiO₂ for Efficient Photocatalytic Hydrogen Generation

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Figure S1. Scheme illustration of synthesis procedure of TW-x catalysts

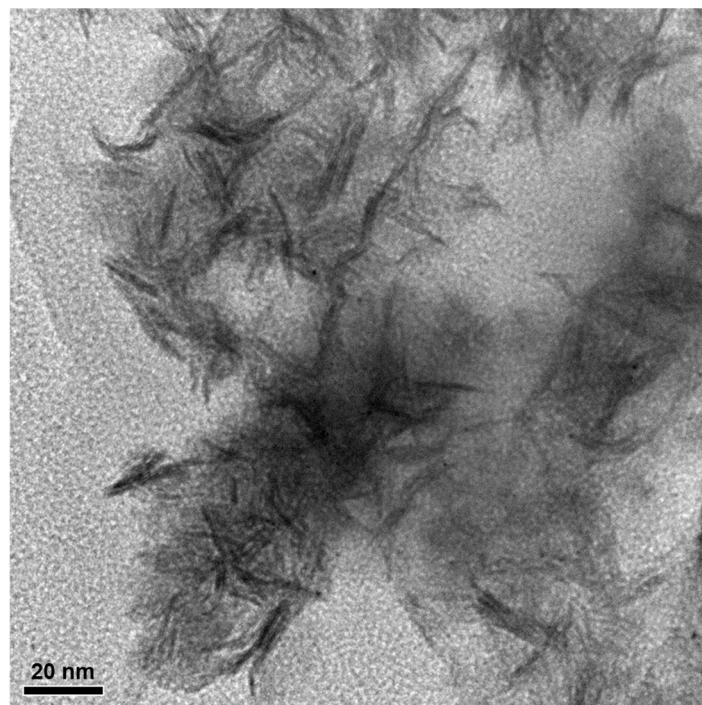


Figure S2. TEM image of WSe₂ nanosheets with low-magnification. The nanosheet was about ~20 nm.

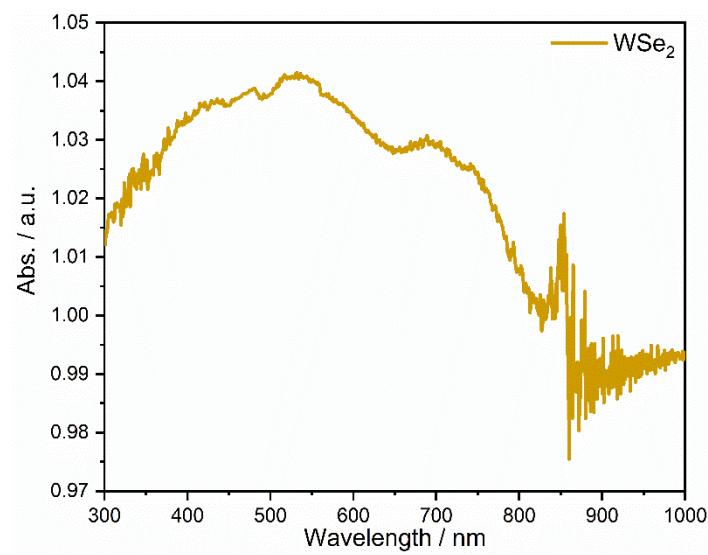


Figure S3. UV-Vis absorption spectra of WSe₂ nanosheets from 300 nm to 1000 nm.

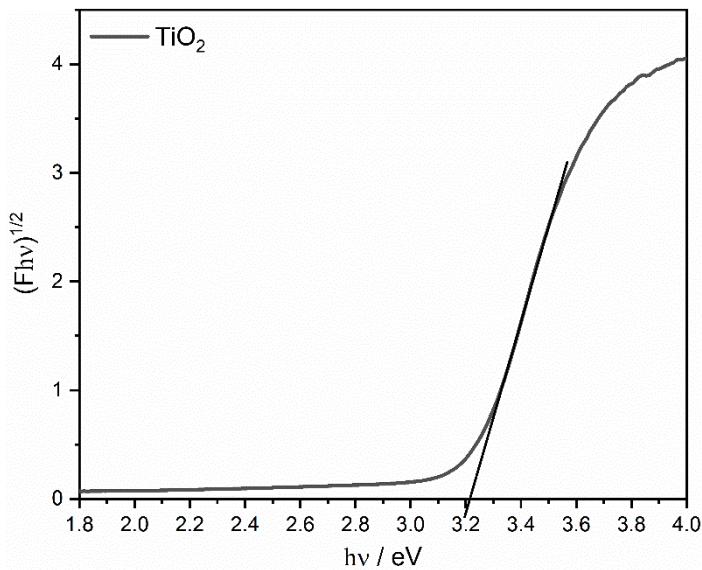


Figure S4. Band-gap evaluation of TiO_2 from Tauc plot.

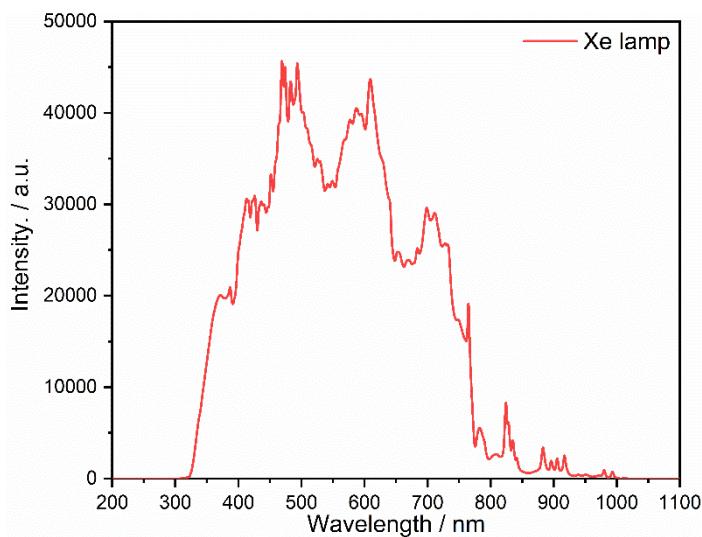


Figure S5. The spectrum of Xe lamp used for light irradiation.

Table S1. Fitting data for photoluminescence emission decay curves using a biexponential function.

Studied Samples	a_1	t_1 (ns)	a_2	t_2 (ns)	Average lifetime
TiO_2	0.82	0.28	0.21	2.10	1.48
TW-2	1.00	0.21	0.22	1.83	1.27

Table S2. Binding energies of studied samples

Binding Energy / eV	TiO ₂	TW-2	WSe ₂
Ti	458.34	458.36	/
Ti	464.03	464.04	/
O	529.56	529.58	/
O	531.41	531.46	/
W	/	30.65	31.52
W	/	32.87	33.63
W	/	34.66	35.32
W	/	36.59	37.50
Se	/	53.38	54.10

Table S3. Comparison of hydrogen evolution for cocatalysts-TiO₂ photocatalysts.

Photocatalyst	Light source	Sacrificial reagent	Activity (mmol/g·h)	AQY (365 nm)
PtAu/TiO ₂	Xe lamp	Methanol	61.5	72.9%[1]
Pt/TiO ₂	Xe lamp	Methanol	29.63	45.6%[2]
Pd/TiO ₂	365 LED	Ethanol	86.7	14.9%[3]
NiCuS _x /TiO ₂	365 nm LED	Methanol	8.56	34.67%[4]
Cu ₂ (OH) ₂ CO ₃ /TiO ₂	Xe lamp	Ethylene glycol	0.50	31.9%[5]
Cu-NW/TiO ₂	365 nm LED	Methanol	5.104	17.2%[6]
MoS ₂ /TiO ₂	Xe lamp	Methanol	1.84	13.6%[7]
Ni-NiS _x /TiO ₂	365 nm LED	Ethanol	4.47	12.78%[8]
MoS ₂ /GO/TiO ₂	Xe lamp	Ethanol	2.07	9.7%[9]
WSe ₂ /TiO ₂	Xe lamp	Methanol	2.28	43.8%

- Wei, T.; Ding, P.; Wang, T.; Liu, L.-M.; An, X.; Yu, X., Facet-regulating local coordination of dual-atom cocatalyzed TiO₂ for photocatalytic water splitting. *ACS Catal.* **2021**, *11*, 14669-14676.
- Ruan, X.; Cui, X.; Cui, Y.; Fan, X.; Li, Z.; Xie, T.; Ba, K.; Jia, G.; Zhang, H.; Zhang, L.; Zhang, W.; Zhao, X.; Leng, J.; Jin, S.; Singh, D.; Zheng, W.; Favorable energy band alignment of TiO₂ anatase/rutile heterophase homojunctions yields photocatalytic hydrogen evolution with quantum efficiency exceeding 45.6%. *Adv. Energy Mater.* **2022**, *12*, 2200298.
- Chen, Y.; Soler, L.; Armengol-Profitós, M.; Xie, C.; Crespo, D.; Llorca, J., Enhanced photoproduction of hydrogen on Pd/TiO₂ prepared by mechanochemistry. *Appl. Catal. B-Environ.* **2022**, *309*, 121275.
- Zhong, W.; Gao, D.; Yu, H.; Fan, J.; Yu, J., Novel amorphous nicus H₂-evolution cocatalyst: optimizing surface hydrogen desorption for efficient photocatalytic activity. *Chem. Eng. J.* **2021**, *419*, 129652.
- Xu, L.; Li, L.; Ao, Y.; Long, F.; Guan, J., Engineering highly efficient photocatalysts for hydrogen production by simply regulating the solubility of insoluble compound cocatalysts. *Int. J. Hydrogen Energy* **2014**, *39*, 11486-11493.
- Xiao, S.; Liu, P.; Zhu, W.; Li, G.; Zhang, D.; Li, H., Copper nanowires: a substitute for noble metals to enhance photocatalytic H₂ generation. *Nano Lett* **2015**, *15*, 4853-8.
- Du, F.; Lu, H.; Lu, S.; Wang, J.; Xiao, Y.; Xue, W.; Cao, S., Photodeposition of amorphous MoS_x cocatalyst on TiO₂ nanosheets with {001} facets exposed for highly efficient photocatalytic hydrogen evolution. *Int. J. Hydrogen Energy* **2018**, *43*, 3223-3234.
- Wang, P.; Xu, S.; Chen, F.; Yu, H., Ni nanoparticles as electron-transfer mediators and NiS_x as interfacial active sites for coordinative enhancement of H₂-evolution performance of TiO₂. *Chinese J. Catal.* **2019**, *40*, 343-351.
- Xiang, Q.; Yu, J.; Jaroniec, M., Synergetic effect of MoS₂ and graphene as cocatalysts for enhanced photocatalytic H₂ production activity of TiO₂ nanoparticles. *J. Am. Chem. Soc.* **2012**, *134*, 6575-8.