

# Supporting Information

## **Construction of unique 2D/0D NiO/ZnCdS p-n heterojunction photocatalyst with highly improved photocatalytic H<sub>2</sub> generation**

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## **Characterization of the materials**

The X-ray diffraction (XRD) patterns were measured on a D/MAX 2500V diffractometer (Rigaku). UV-Visible (UV-Vis) absorption spectra were acquired using a Shimadzu 3600 UV-vis spectrophotometer. Scanning electron microscopy (SEM) images were obtained using a Zeiss Sigma 300. Transmission electron microscopy (TEM) images were carried out on a TECNAI F-30 at an accelerating voltage of 300 kV. XPS measurements were performed with an ESCALAB 250Xi. Under 370 nm excitation, photoluminescence (PL) and time-resolved photoluminescence (TRPL) were collected on the OmniFluo990LSP fluorescence spectrophotometer and HORIBA FL3C-111 fluorescence lifetime spectrophotometer, respectively. The photocurrent responses, electrochemical impedance spectroscopy (EIS), and Mott-Schottky plots were performed using a Bio-Logic VSP-300 multichannel potentiostat with a three-electrode system. The platinum sheet, Ag/AgCl electrode, and Na<sub>2</sub>SO<sub>4</sub> solution (0.1 M) serve as the counter electrode, reference electrode, and electrolyte, respectively. A working electrode was prepared by coating ITO glass (1.0 cm x 2.0 cm) with a catalyst slurry (5 mg of photocatalysts dispersed in 0.5 mL of ethanol and 10 μL of Nafion solution) and then dried in a vacuum drying oven.

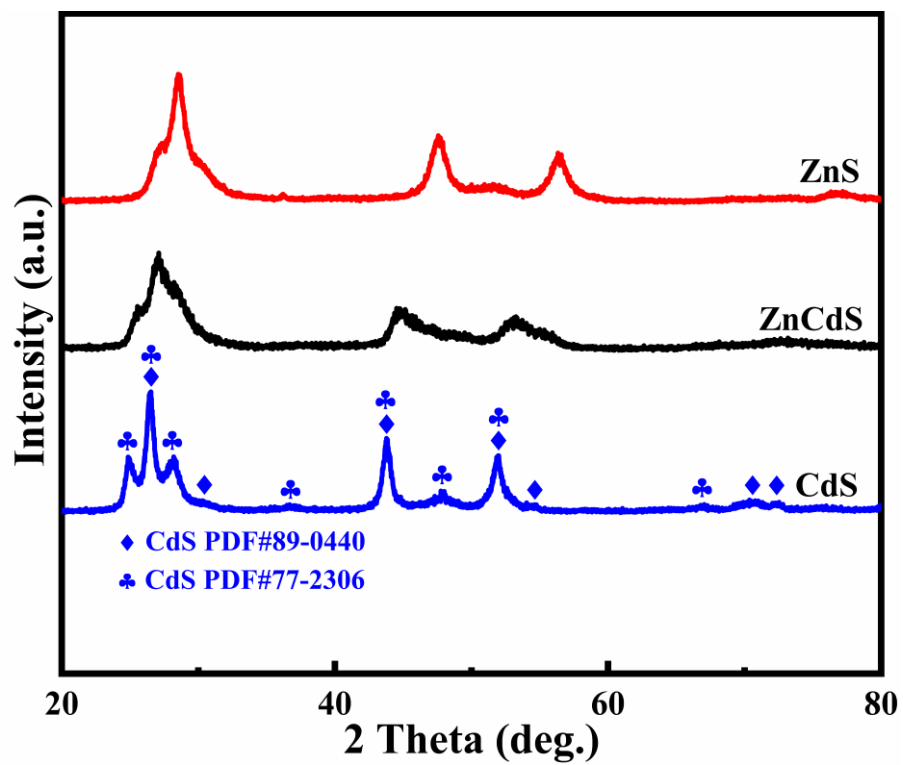


Fig. S1 XRD patterns of ZnS, CdS, and ZnCdS samples.

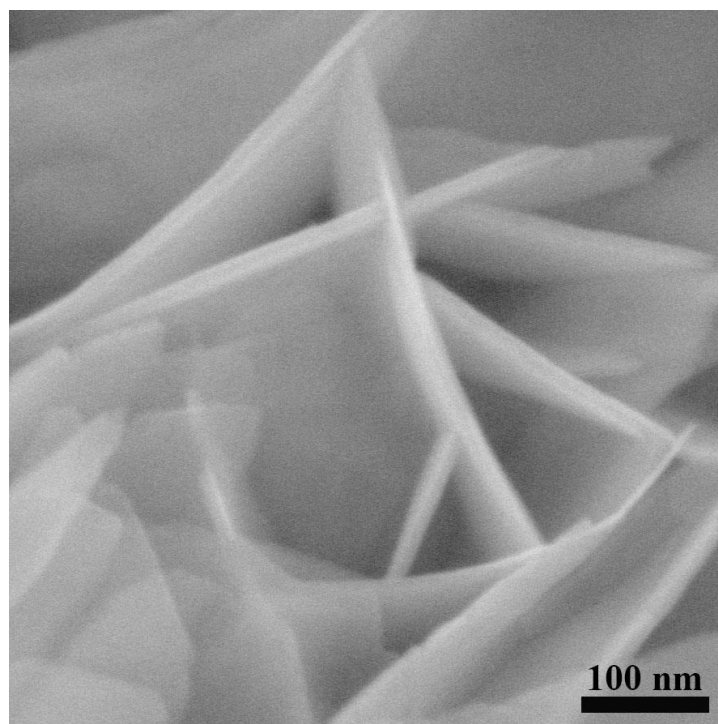


Fig. S2 SEM image of Ni(OH)<sub>2</sub> nanosheets.

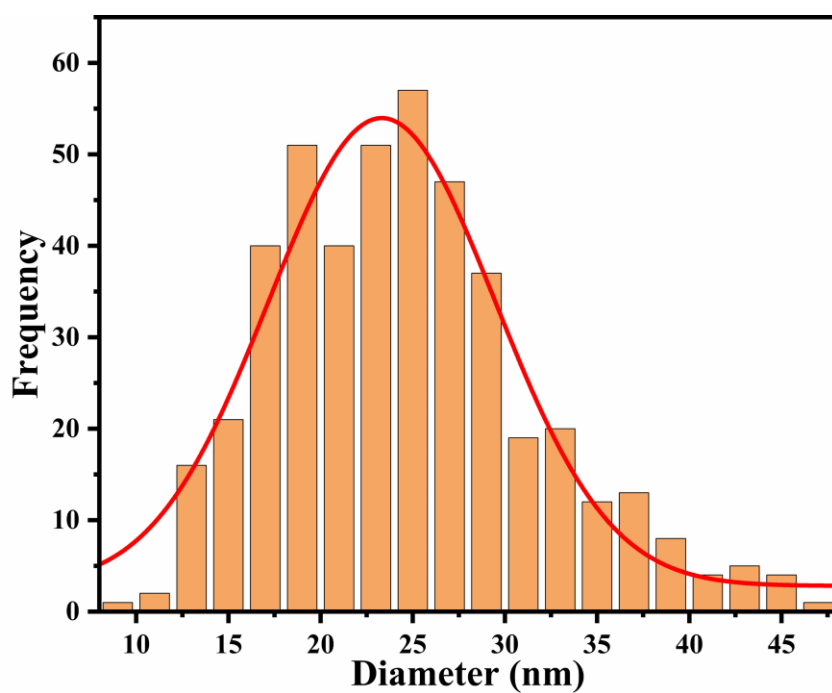


Fig. S3 Particle size distribution histogram of ZnCdS nanoparticles.

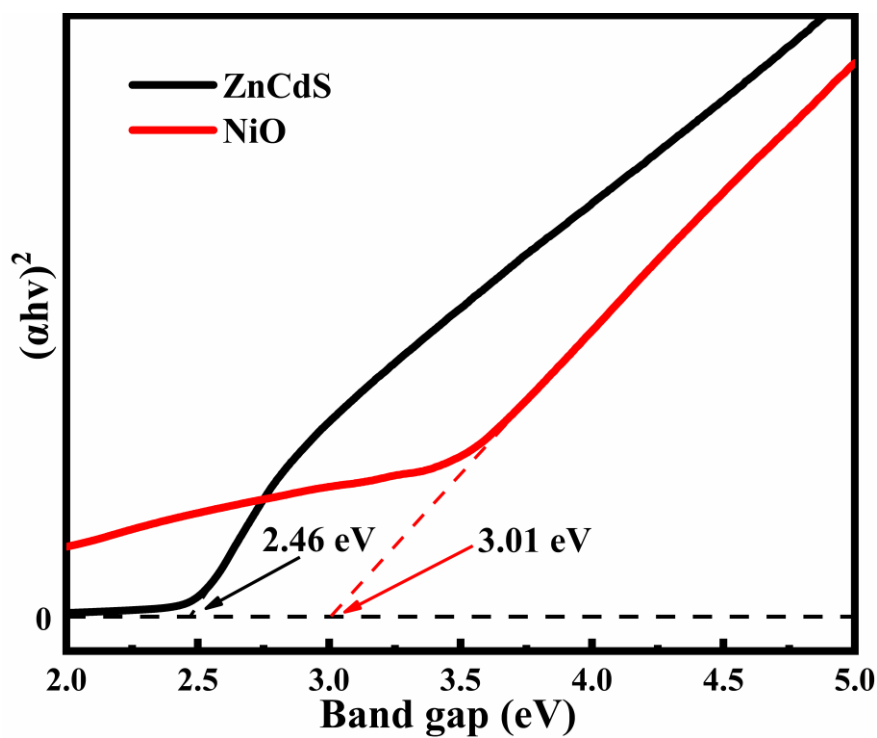
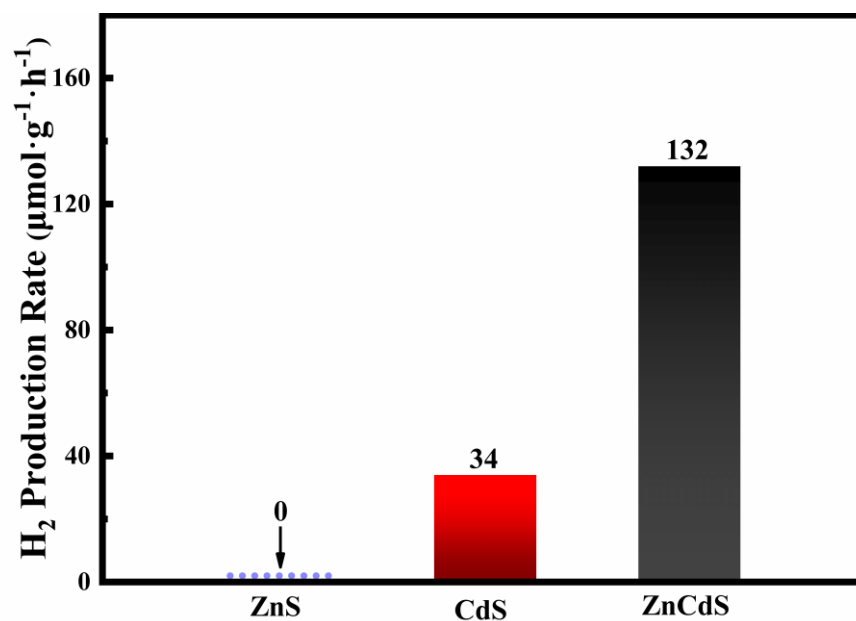
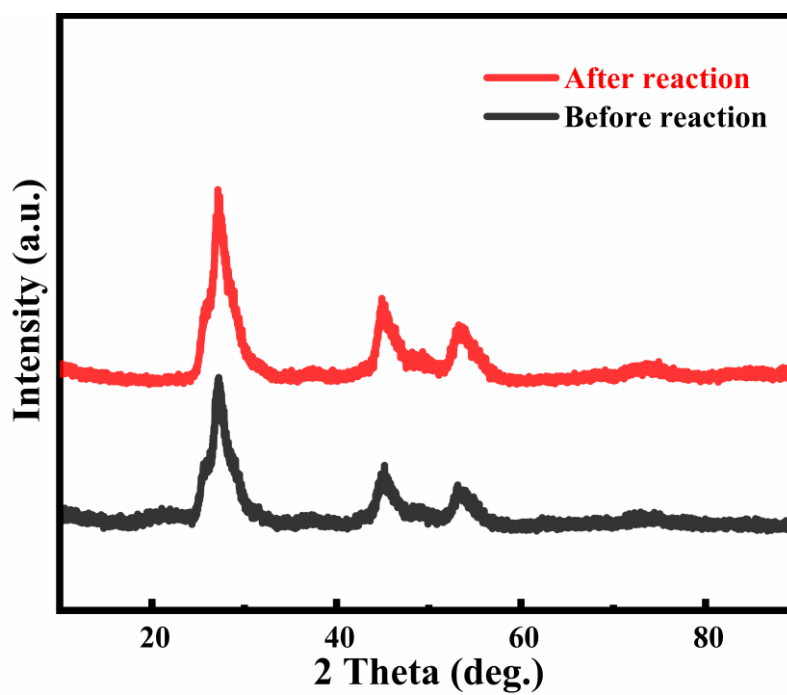


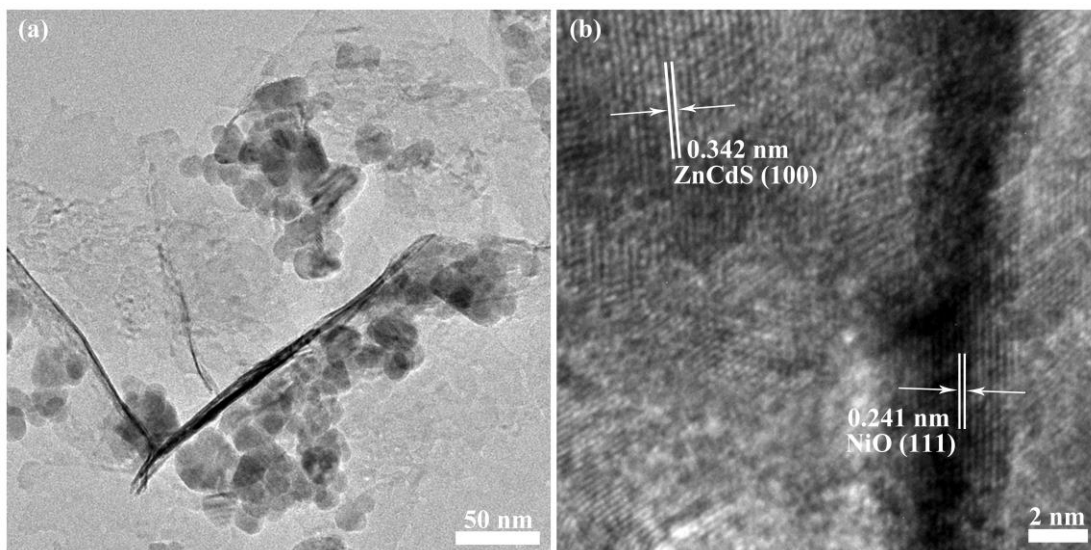
Fig. S4 Tauc plots of NiO nanosheets and ZnCdS nanoparticle.



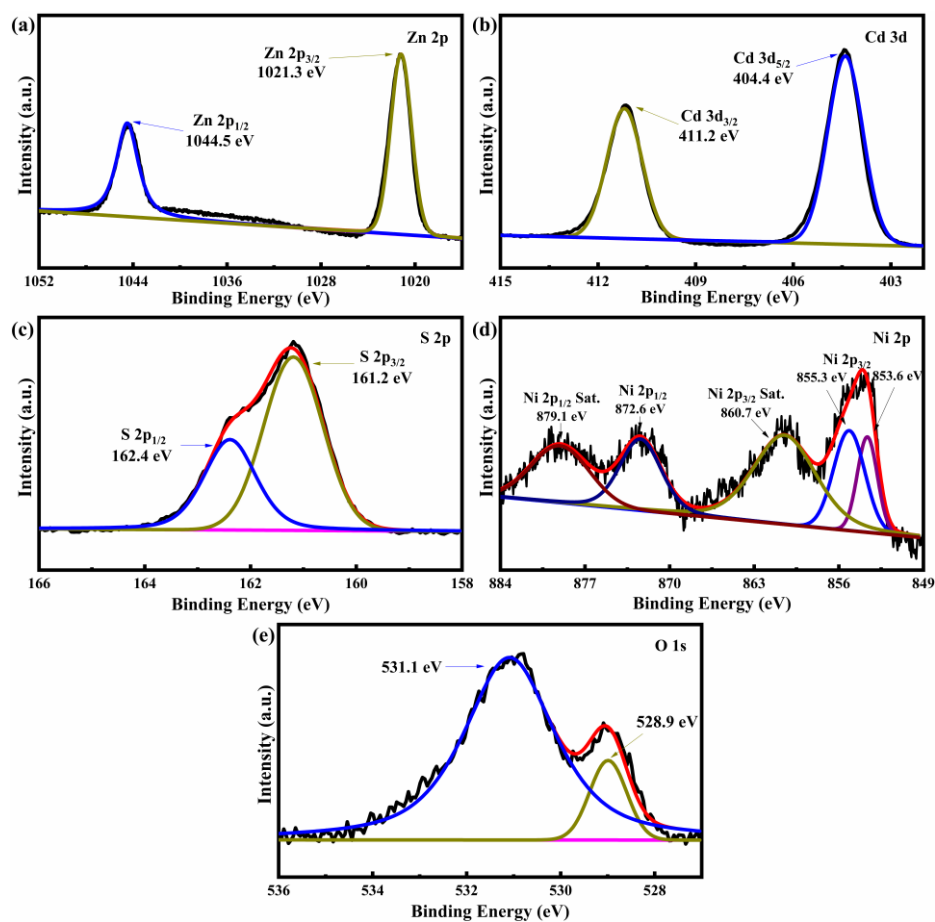
**Fig. S5** Average H<sub>2</sub> production rates of ZnS, CdS, and ZnCdS samples under visible-light irradiation ( $\lambda > 420$  nm).



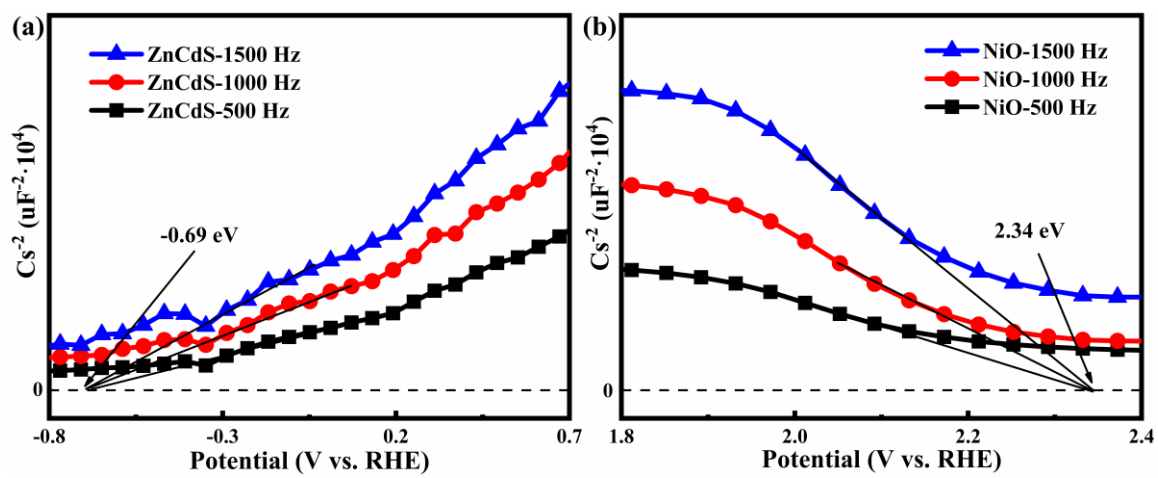
**Fig. S6** XRD patterns of the 5% NiO/ZnCdS sample before and after cycling photocatalytic reactions.



**Fig. S7** TEM images of the 5% NiO/ZnCdS sample after cycling photocatalytic reactions.



**Fig. S8** XPS spectra of the 5% NiO/ZnCdS sample after cycling photocatalytic reactions.



**Fig. S9** Mott-Schottky plots of (a) ZnCdS nanoparticles and (b) NiO nanosheets.

**Table S1.** Photocatalytic H<sub>2</sub> evolution activity of ZnCdS-based photocatalysts.

	<b>Photocatalyst</b>	<b>Light source</b>	<b>Scavenger</b>	<b>Activity (<math>\mu\text{mol h}^{-1} \text{g}^{-1}</math>)</b>	<b>Enhancement factor* (Reference)</b>	<b>Refs</b>
1	MoS <sub>2</sub> /Zn <sub>0.5</sub> Cd <sub>0.5</sub> S /g-C <sub>3</sub> N <sub>4</sub>	300 W Xe lamp	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	4914	/	1
2	Zn <sub>0.5</sub> Cd <sub>0.5</sub> S @MoS <sub>2</sub> /RGO	300 W Xe lamp	Lactic acid	2310	9 (Zn <sub>0.5</sub> Cd <sub>0.5</sub> S)	2
3	Zn <sub>0.5</sub> Cd <sub>0.5</sub> S @PAN	350 W Xe lamp	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	475	27.4 (Zn <sub>0.5</sub> Cd <sub>0.5</sub> S)	3
4	Zn <sub>0.5</sub> Cd <sub>0.5</sub> S @Ni-Glycerate	CEL-LED 100	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	2800	/	4
5	Zn <sub>0.7</sub> Cd <sub>0.3</sub> S /PDI	/	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	5166	6.5 (Zn <sub>0.7</sub> Cd <sub>0.3</sub> S)	5
6	CdS @Cd <sub>x</sub> Zn <sub>1-x</sub> S	300 W Xe lamp ( $\lambda \geq 420 \text{ nm}$ )	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	5170	12.3 (Cd <sub>x</sub> Zn <sub>1-x</sub> S)	6
7	Zn <sub>0.7</sub> Cd <sub>0.3</sub> S /NiWO <sub>4</sub>	5 W LED ( $\lambda \geq 420 \text{ nm}$ )	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	15,950	3.16 (Zn <sub>0.7</sub> Cd <sub>0.3</sub> S)	7
8	Zn <sub>0.5</sub> Cd <sub>0.5</sub> S /C	300 W Xe lamp ( $\lambda \geq 420 \text{ nm}$ )	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	2018	/	8
9	Ni <sub>2</sub> P /ZnCdS	300 W Xe lamp ( $\lambda \geq 420 \text{ nm}$ )	Na <sub>2</sub> S /Na <sub>2</sub> SO <sub>3</sub>	1040	5 (ZnCdS)	9
10	<b>NiO /ZnCdS</b>	<b>300 W Xe lamp (<math>\lambda &gt;</math> 420 nm)</b>	<b>Na<sub>2</sub>S /Na<sub>2</sub>SO<sub>3</sub></b>	<b>5042</b>	<b>38.2 (ZnCdS)</b>	<b>This work</b>



**Table S2.** Fitting results of the Nyquist plots for ZnCdS, 5% NiO/ZnCdS, and NiO photocatalysts.

Photocatalyst	$R_s$ ( $\Omega$ )	$R_{ct}$ ( $k\Omega$ )	CPE ( $F \cdot cm^{-2} \cdot S^{n-1}$ )	n
ZnCdS	9.859	612	1.02E-4	0.89
5% NiO/ZnCdS	9.832	67.1	2.37E-4	0.84
NiO	9.804	8.03	2.73E-4	0.62

**Table S3.** Decay parameters of pure ZnCdS and 5% NiO/ZnCdS.

Samples	Lifetime, T (ns)	Rel (%)	$T_{av}$ (ns)
ZnCdS	$T_1= 2.04$	$B_1= 64.82$	3.06
	$T_2= 41.24$	$B_2= 35.18$	
5% NiO/ZnCdS	$T_1= 2.79$	$B_1= 45.69$	5.92
	$T_2= 110.21$	$B_2= 54.31$	

## References

1. Y. Tang, X. Li, D. Zhang, X. Pu, B. Ge and Y. Huang, *Mater. Res. Bull.*, 2019, **110**, 214-222.
2. S. N. Guo, Y. L. Min, J. C. Fan and Q. J. Xu, *ACS Appl. Mater. Interfaces*, 2016, **8**, 2928-2934.
3. J. Fu, B. Zhu, W. You, M. Jaroniec and J. Yu, *Appl. Catal. B*, 2018, **220**, 148-160.
4. J. Huang, X. Ma, Y. Sun, L. Wang, H. She, J. Li and Q. Wang, *Mater. Sci. Semicond. Process.*, 2020, **105**, 104703.

5. J. Song, Y. Chen, D. Sun and X. Li, *Inorg. Chem. Commun.*, 2018, **92**, 27-34.
6. S. Kai, B. Xi, Y. Wang and S. Xiong, *Chem. Eur. J.*, 2017, **23**, 16653-16659.
7. Y. Liu, G. Wang, Y. Li and Z. Jin, *J. Colloid Interface Sci.*, 2019, **554**, 113-124.
8. X. Zhou, X. Wang, X. Feng, K. Zhang, X. Peng, H. Wang, C. Liu, Y. Han, H. Wang and Q. Li, *ACS Appl. Mater. Interfaces*, 2017, **9**, 22560-22567.
9. S. Peng, Y. Yang, J. Tan, C. Gan and Y. Li, *Appl. Surf. Sci.*, 2018, **447**, 822-828.