Electronic Supporting Information

Enhanced photocatalytic H_2 evolution over micro-SiC by coupling with CdS under visible light irradiation

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Experimental Details

1. Materials synthesis:

1.1 Chemicals: All chemicals are analytical grade and used as received without further purification. Green SiC particles (about 5 µm, mixed phases including 6H-SiC and 3C-SiC) were purchased from Shanghai ST-nano Science & Technology Co.Ltd., China. SiC powders were pre-treated in muffle furnace at 700 °C for 3 h to remove any organic residues and immersed in a HF (40%) solution to remove the oxidates on the surface. Then washed with deionized water thoroughly and dried at 60°C for 12 h in vacuum system.

1.2 Synthesis of SiC/CdS composites: In a typical procedure, 0.2284 g $CdCl_2{\cdot}2.5H_2O$ was well dissolved in 60 mL of ethanol, noted as solution A. 0.4 g NaOH was dissolved in 60 mL of ethanol, and added 0.144 g SiC and mechanically mixed for 30 min, which was named as solution B. The mixture of solution A and B was mechanically stirred for 1 h. Thereafter, another 10 mL of ethanol solution containing 0.24 g $Na_2S{\cdot}9H_2O$ was dropwise added into the mixture above and continued agitating for 1 h. Filtered and washed with de-ionized water until the pH value reached 7. The final precipitate was dried at 70 °C for 24 h in air, resulting in SiC/CdS(1:1) (mass ratio) composite. Other mass ratios of 1:0.2, 1:0.5 composites were fabricated by adjusting the amount of $CdCl_2{\cdot}2.5H_2O$ and $Na_2S{\cdot}9H_2O$. For comparison, CdS alone was prepared by a similar method but without SiC addition.

1.2 Synthesis of SiC/CdS-Pt composites: In a typical procedure, photodeposition method was employed to load Pt cocatalyst. 50 mg SiC/CdS composite was dispersed into 100 mL deionized water containing 0.1 M $Na_2S\cdot9H_2O$ and 0.1 M Na_2SO_3 as sacrificial reagents by ultrasonication for 5 min. Adding in 0.67 mL of hydrogen hexachloroplatinate hydrate ($H_2PtCl_6\cdot6H_2O$) aqueous solution (corresponding to 0.2 wt% Pt) into Pyrex cell. Under continuous stirring, the suspension was illuminated under the top irradiation of 300 W Xe arc lamp (PLS-SXE 300, Beijing Trusttech Co. Ltd.) with whole spectrum for 3 h. Different loading amount of Pt ranging from 0.2 to 5 wt % was controlled by regulating the volume of $H_2PtCl_6\cdot6H_2O$ aqueous solution.

2. Characterization:

X-ray diffraction (XRD) was used to identify the phase formation by a Philips X'PERT Pro X-ray diffractometer with Cu Kα radiation (λ=0.154 nm). The surface morphology of the products was observed by a scanning electron microscopy (SEM) system (S-4800, Hitachi, Japan). Transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM) images and Energy-dispersive X-ray spectroscopy (EDX) analysis were obtained with F20 S-TWIN electron microscope (Tecnai G^2, FEI Co.), using 200 kV accelerating voltage. The specific surface areas were measured by the multipoint Brunauer-Emmett-Teller (BET) method of nitrogen adsorption at liquid nitrogen temperature (77 K) using a Micromeritics TriStrar II in the relative pressure (P/P_0) range of 0.05-0.30. UV-Vis diffuse reflection spectra (DRS) for the dry-pressed disk samples was measured by UV-2550，SHIMADZU, in the wavelength range 200-800 nm. $BaSO_4$ was used as a reflectance standard in the diffuse reflectance experiments. Photoluminescence (PL) spectra were recorded on a Hitachi F-4500 fluorescence spectrophotometer, the excitation wavelength is 386 nm. Number of incident photons was calibrated with a Si photodiode (FZ-A, HANDY).

3. Photocatalytic activity measurements:

The photocatalytic hydrogen evolution reactions were performed in Labsolar II system (Beijing Perfectlight Technology Co.Ltd.). 300 mL Pyrex reaction vessel is connected to a closed gas circulation and an evacuation system at ambient temperature. The evolved gases were measured by an online gas chromatograph (GC 7890II, Techcomp) equipped with a molecular sieve (5 Å pore size) and N_2 carrier gas. Under continuous stirring, 50 mg photocatalysts were dispersed into

100 mL deionized water containing 0.1 M $Na_2S{\cdot}9H_2O$ and 0.1 M Na_2SO_3 as sacrificial reagents by ultrasonication for 5 min. Prior to irradiation, the system was vacuumized for 30 min to remove the dissolved gases in water. A continuous magnetic stirrer was applied at the bottom of the reactor in order to keep the photocatalyst particles in suspension status during the whole experiment. A 300 W Xe lamp was used as a light source and 1 cm away from the reactor. A UV cut-off filter ($\lambda \geq 420$ nm) was used to be the visible light source for the photocatalytic hydrogen evolution reaction.

The apparent quantum efficiency (AQE) was measured under the same conditions except that the 420 nm cut-off filter was changed to 420 nm band-pass filter. In the experiment, the irradiation intensity after the 420 nm band-pass filter was determined to be 10.1 mW/cm^2. The reaction solution was irradiated for 3 h to calculate AQE according to the following equation:

$$\mathrm{AQE} = \frac{\text{number of reacted electrons}}{\text{number of incident photons}} \times 100\%$$

$$= \frac{\text{number of evolved } \mathrm{H_2} \text{ molecules} \times 2}{\text{number of incident photons}} \times 100\%$$

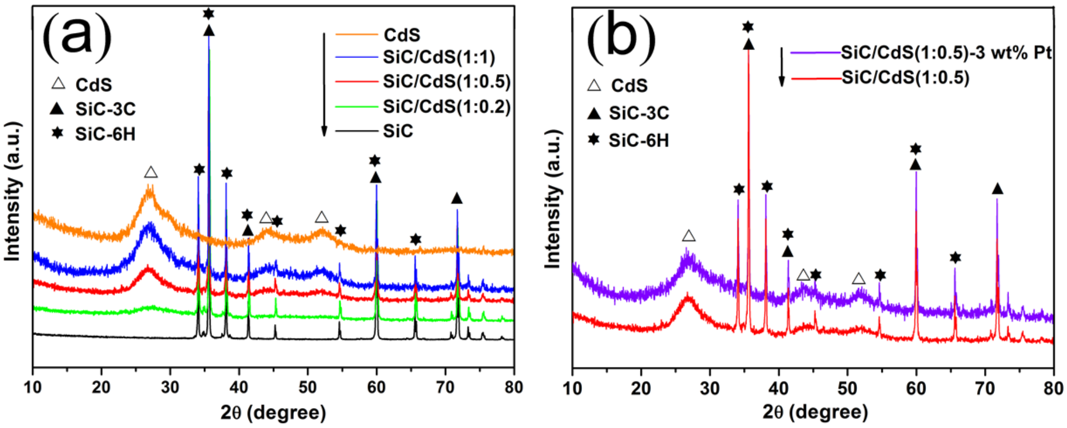

Fig. S1 XRD patterns of CdS, SiC and SiC/CdS composites with different mass ratios (a) and SiC/CdS(1:0.5)-Pt photocatalyst (b).

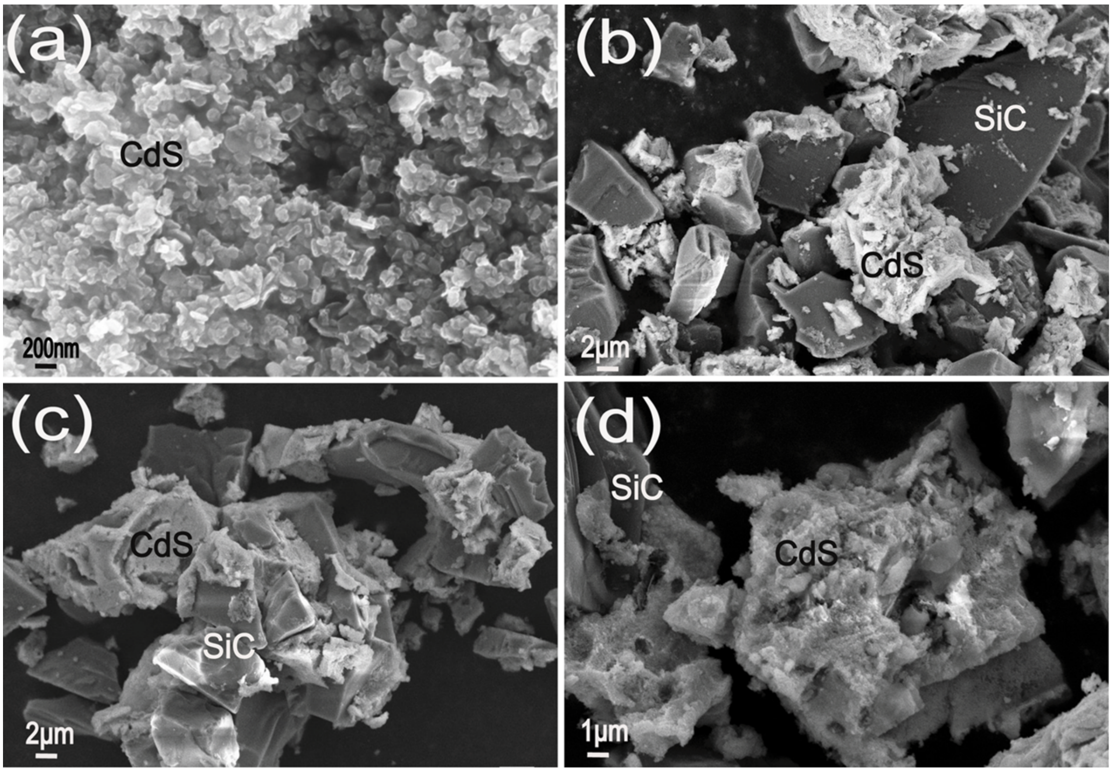

Fig. S2 SEM images of prepared CdS (a) and SiC/CdS composites with different mass ratios of 1:0.2 (b), 1:0.5 (c) and 1:1 (d).

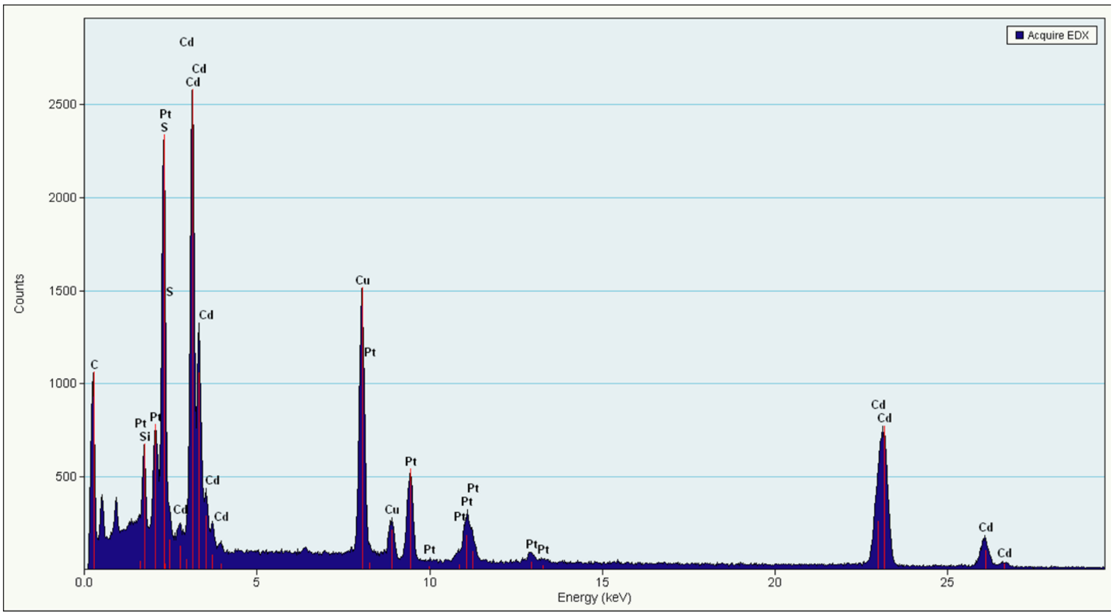

Fig. S3 Acquired EDX image of SiC/CdS(1:0.5)-Pt photocatalyst.

Fig. S4 Digital pictures of SiC, SiC/CdS composites and CdS.