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# Enhanced cyclability of CdS/TiO<sub>2</sub> photocatalyst by stable interface structure

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#### ABSTRACT

CdS nanoparticles were *in situ* deposited on TiO<sub>2</sub> nanosheets and nanorods under hydrothermal conditions, respectively. The effect of CdS–TiO<sub>2</sub> interface structure on hydrogen production activity was mainly investigated under visible light irradiation. The results showed that the TiO<sub>2</sub> nanosheet-based CdS/TiO<sub>2</sub> showed a higher activity and a higher cyclability than the nanorod-based sample due to the stronger interaction of CdS with the (001) facets of TiO<sub>2</sub> than with the (101) facets. It was proposed that the strong interaction between CdS nanoparticles and TiO<sub>2</sub> nanosheets effectively refrains the recombination of electrons and holes.

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# 1. Introduction

Hydrogen is an attractive clean and efficient energy alternative to non-renewable fossil resource. Since the first report on photoelectrochemical water splitting over  $TiO_2$  electrode [1],  $TiO_2$  has been extensively studied due to its nontoxicity, low cost, high activity and stability. However, the photocatalytic hydrogen production efficiency of  $TiO_2$  is fairly low due to the fast recombination rate of photogenerated carriers [2–30]. Among various modification methods, the coupling of  $TiO_2$  with semiconductors (e.g., CdS) is a promising strategy to overcome the disadvantage, since they can compensate for the disadvantages of individual component and induce a synergistic effect [3–11]. Nevertheless, the photocatalytic activities of the as-reported coupled photocatalysts are not high, since the used

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0749-6036/\$ - see front matter @ 2012 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.spmi.2012.03.026 TiO<sub>2</sub> in these composites mainly consists of the thermally stable (101) facets. Recently, TiO<sub>2</sub> nanosheets with the highly reactive (001) facets have been reported and the hydrogen production activity of TiO<sub>2</sub> can be significantly improved by the exposed (001) facets [12–19]. It has been reported that water molecules can chemically dissociate on the (001) facets, whilst only physically adsorption can take place on the stable (101) facets [20,21]. For example, Amano et al. [25] reported that the decahedral anatase with the exposed (001) facets had a higher hydrogen production rate than Degussa P25. Liu et al. [26–28] also reported that the nitrogen-doped TiO<sub>2</sub> nanosheets with the dominant (001) facets showed an improved hydrogen production activity under visible light. Furthermore, Yu and Xiang et al. [32,33] have reported the enhanced hydrogen production activity of the TiO<sub>2</sub> nanosheet-based CdS/TiO<sub>2</sub> photocatalysts. However, most of the reports are mainly paid attention to the activities of these photocatalysts. It is well known that the stabilities of the surface coupled composites are very important for the practical applications. To the best of our knowledge, little work has been performed to study the stability of the surface coupled photocatalysts.

Herein, CdS nanoparticles have been *in situ* deposited on the TiO<sub>2</sub> nanosheets and nanorods by a simple hydrothermal method, respectively. The samples were characterized by X-ray diffraction, scanning electron microscopy, transmission electron microscopy, X-ray photoelectron spectroscopy, UV–visible diffuse reflectance spectroscopy, photoluminescence (PL) spectroscopy and nitrogen adsorption. The effect of interface structure on the cyclability of the CdS/TiO<sub>2</sub> photocatalysts was mainly researched.

# 2. Experimental section

# 2.1. Sample preparation

All reagents were of analytical grade (purchased from Beijing Chemical Reagents Industrial Company, China) and were used without further purification.

#### 2.1.1. Preparation of TiO2 nanosheets

The sample was prepared by the hydrothermal method similar to the report by Yu et al. [17] Typically, 25 mL of  $Ti(OC_4H_9)_4$  was mixed with 3 mL of 40 wt.% HF solution in a 120-mL Teflon-lined autoclave. The nominal molar ratio of F to Ti was kept at 1. Then the mixture system was heated at 180 °C for 24 h. After reaction, the white precipitate was separated, washed and dried at 80 °C overnight. It has been reported that the adsorbed fluorine ions on the surface of TiO<sub>2</sub> can be easily removed by calcination at 600 °C [18] or alkaline washing in a NaOH solution without altering the crystal structure and morphology<sup>22</sup>. The surface fluorine species on TiO<sub>2</sub> nanosheets were calcined at 600 °C for 2 h to obtain pure TiO<sub>2</sub>, which was designated as S1.

# 2.1.2. Preparation of TiO<sub>2</sub> nanorods

The TiO<sub>2</sub> nanorods were prepared following the reference [31]. Typically, 2 g of Degussa P25 was dispersed 80 mL of 10 mol/L KOH solution under intensive stirring. The mixture was loaded into 120 ml of Teflon-lined autoclave and heated at 200 °C for 60 h. The white powders were separated by centrifuging, washing with 0.1 mol/L HNO<sub>3</sub> solution, and washed with deionized water until pH reach 7, respectively. The sample was dried at 110 °C overnight. The powders were also calcined at 600 °C for 2 h to obtain the product, which was designated as S2.

# 2.1.3. Preparation of CdS/TiO<sub>2</sub> hybrids

In the synthesis of CdS/TiO<sub>2</sub>, the *in situ* hydrothermal method is adopted under the same conditions except for with different TiO<sub>2</sub> (i.e., S1 and S2). Typically, 0.2 g of TiO<sub>2</sub> powders was dispersed in 100-mL aqueous solution of 0.0014 M Cd(NO<sub>3</sub>)<sub>2</sub> and 0.0014 M (NH<sub>2</sub>)<sub>2</sub>C=S under ultrasonic stirring. The suspension was hydrothermally treated at 140 °C for 24 h. The precipitates were separated by centrifuging, washed, and finally dried in an oven at 80 °C overnight. In our studies, it was found that the optimal molar ratio of CdS to TiO<sub>2</sub> was 1:4 (Fig. 1S of supporting information). Therefore, the molar ratios of CdS to TiO<sub>2</sub> were kept at 1:4. The as-obtained CdS/TiO<sub>2</sub> samples with S1 and S2 were designated as S3 and S4, respectively.

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