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Ag$_2$S/CdS/Ni ternary nanostructures: long-range electric field to enhanced photocatalytic hydrogen production activity

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Abstract

Silver sulfide, cadmium sulfide and nickel (Ag$_2$S/CdS/Ni) ternary nanostructures were fabricated by a two step process and characterized by x-ray powder diffraction, transmission electron microscopy, and UV–vis diffuse reflection spectroscopy. The photocatalytic hydrogen production activity of ternary nanostructures and reference samples were evaluated using triethanolamine (TEOA) as sacrificial reagent in water under visible-light illumination ($\lambda \geq 420$ nm). The result shows Ag$_2$S/CdS/Ni nanostructures exhibited a high visible light photocatalytic H$_2$ evolution rate of 1.54 mmolh$^{-1}$g$^{-1}$, which was 4.6 times and 1.4 times higher than that of 1.2% mol Ag$_2$S/CdS and Ni/CdS. The degree of photocorrosion of CdS were employed to study the photogenerated carriers transfer route by measuring and comparing the concentration of Cd$^{2+}$ in the solution of the photocorrosion experiments in a nonsacrificial system. A long-range electric field, which is similar to the p-i-n electric field structure, was proposed to be constructed by Ni nanoparticles and Ag$_2$S nanoparticles in CdS. Most of the photogenerated electrons and holes in CdS drift to the electron donor and electron acceptor respectively under the action of long-range electric field, which greatly improves the separation efficiency of photogenerated carriers and the photocatalytic H$_2$ production activity.

1. Introduction

As a potential clean and renewable energy resource that may be able to simultaneously solve the shortage of energy resources and environmental problems caused by rapid development of industrialization and urbanization, hydrogen energy is receiving more and more attention around the world [1, 2]. Mainstream industrial hydrogen production methods are electrolysis of water and steam reforming of natural gas [3, 4], which still consume non-renewable resources. Solar energy is the most abundant and clean source, and making semiconductor photocatalytic water splitting technology that can convert solar energy into hydrogen energy is quite possible, since the pioneering work of photoelectrochemical water splitting on titanium dioxide (TiO$_2$) electrodes reported by Fujishima and Honda in 1972 [5]. In addition to TiO$_2$, various semiconductor photocatalysts, such as oxides [6–8], sulfides [9–11], selenides [12, 13], oxynitrides [14, 15], and multi nanocomposites [16, 17] have been examined in searching for high-efficiency photocatalysts. In recent year, CdS has been considered to be a fascinating visible light photocatalyst because of moderate band gap, proper band structure, low cost and easy preparation. However, pristine CdS also suffers from several shortcomings, particularly rapid recombination of photoexcited charge carriers [18–20].

Hence, enormous efforts must be invested to reduce the recombination of photogenerated charge carriers of CdS before it can be practically used in commercial hydrogen production. So far, many methods have been used to enhance the separation of electron-hole pair, and improve the photocatalytic activity of CdS [21–24]. The most commonly used method is to introduce an interface built-in electric field by constructing a metal-
found that although the self-polarized electric diffusion, which reduces the separation of the photogenerated electrons and holes pairs. Fortunately, it has been shown that the built-in electric field can be extended. The photogenerated electrons and holes can be separated quickly by the built-in electric field. However, in either M-S heterojunction or S-S heterojunction, the built-in electric field is only confined within the depletion layer (as shown in scheme 1(a))), and the photogenerated electrons generated outside the depletion layer can only be transferred in the way of diffusion, which reduces the separation of the photogenerated electrons and holes pairs. Fortunately, it has been shown that although the self-polarized electric field in zinc oxide (ZnO) single crystal is very weak, it can still effectively separate the photogenerated electron-holes and then affect the photocatalytic activity of ZnO single crystal because of its long-range effect [34–36]. In view of this, it is possible to significantly increase the separation rate of photogenerated carriers if the range of action of the built-in electric field can be extended.

In our recent work, based on the synergistic effect of the dual heterojunctions, two heterojunctions were constructed on the graphite phase carbon nitride (g-C3N4) nanoparticles to form a long-range electric field, in which photogenerated electrons were transferred from the semiconductor to one metal while holes to the other metal [37]. In this article, we present an Ag2S/CdS/Ni ternary nanostructures photocatalyst fabricated by a solution method and solubility product difference-guided synthesis method [38, 39]. As shown schemes 1(b) and (c), after charge balance, the Ag2S in the Ag2S/CdS composite heterojunction is enriched with negative charges (formed a built-in electric field towards the Ag2S) [40, 41], while the Ni in the Ni/CdS composite material is enriched with positive charges (formed a built-in electric field towards the CdS) [42, 43], which helps to form a long-range electric field in the CdS nanoparticles [37]. The morphology, physicochemical properties as well as the photocatalytic H2-production rate from aqueous solutions containing TEOA of these Ag2S/CdS/Ni composites and contrast samples were characterized and investigated. To further study the photo-generated carrier transfer route, photocorrosion behavior of the Ag2S/CdS/Ni composites and contrast samples in a nonsacrificial system were also investigated. It can be found that photo-generated carrier transfer route is not what it be thought was. The role of Ag2S and Ni nanoparticles in the photocatalytic hydrogen-production process was discussed. This work may provide a stepping stone towards searching for new high efficiency CdS-based photocatalysts in photocatalytic hydrogen production.

2. Experimental procedure

2.1. Materials synthesis

CdS powder was prepared by chemical precipitation, and the Ni nanoparticles loaded CdS samples were synthesized by a solution method. Three samples were prepared, 1.2% mol/mol Ni/CdS, 1.2% mol/mol Ag2S/CdS and 0.6% mol/mol Ag2S/CdS/Ni, in which the molar ratio of Ag/Cd and Ni/Cd are both 0.6%. Ag2S nanoparticles were loaded on CdS and CdS/Ni composite photocatalyst fabricated by a solution method and solubility product (Ksp) difference-guided synthesis method based on the Ksp of CdS 1.4 × 10−29 and Ag2S 6.7 × 10−50 [44, 45]. Briefly, 0.1 M sodium sulfide (Na2S) solution was dripped slowly to 0.1 M cadmium nitrate (Cd(NO3)2) solution with stirring continuously. After the reaction is completed, the precipitate is repeatedly washed and centrifuged to remove the residual precursor solution. Finally, the cleaned precipitation was freeze-dried for 24 h. 0.5 g CdS powder was dispersed in 175 ml water and sonicated and stirred for 10 min.
respectively. To obtain metal 0.6% mol/mol Ni loaded CdS samples (Ni/CdS), an aqueous solution of nickel nitrate hexahydrate (Ni(NO₃)₂·6H₂O) were added to the above mixed solution and stirred for 18 h at room temperature. After 18 h, the mixed solution was bubbled with N₂ for 20 min to remove the O₂ inside. And then 1 g of sodium borohydride was added into solution with constant stirring, and the reaction was stirred for another 16 h at 50 °C. For 0.6% mol/mol Ag₂S nanoparticles loaded CdS and CdS/Ni, 0.5 g CdS or CdS/Ni powder was dispersed in 175 ml water and sonicated and stirred for 10 min, respectively, then an aqueous solution of silver nitrate (AgNO₃) was added to the above mixed solution and stirred for 18 h at room temperature. All the samples were collected by centrifugation, washed with deionized water for three times, and dried by freeze-drying.

All chemicals were of analytical grade and used without further purification. Cd(NO₃)₂·4H₂O, Na₂S·9H₂O, Ni(NO₃)₂·6H₂O, AgNO₃, and TEOA were received from Aladdin (Shanghai, China), distilled water was made by Milli-Q Direct16 (Millipore, Billerica, MA, USA).

2.2. Characterization
The photocatalytic hydrogen production experiments were performed in a 100 ml commercial glass reactor with optical quartz window at ambient temperature and atmospheric pressure. A 300 W xenon lamp PLS-XE300 (Perfectlight, Beijing, China), which was positioned 16.5 cm away from the reactor, was used as a light source to trigger the photocatalytic reaction. In a typical photocatalytic experiment, 50 mg of catalyst was dispersed by a constant stirring in mixed aqueous solution containing 40 ml deionized water and 10 ml TEOA, and then irradiated by xenon lamp (300 W, λ ≥ 420 nm) under 25 °C circulating water cooling. The focused intensity on the reactor was about 510 mW cm⁻². Before irradiation, the reactor was bubbled with N₂ for 30 min to remove the dissolved oxygen and ensured that the reaction system was under an anaerobic condition. A 1 ml portion of gas at the center of reactor was sampled through the septum, and hydrogen was analyzed by gas chromatograph GC2060 (Ramiin, Shanghai, China) (nitrogen as a carrier gas and 5 Å molecular-sieve column).

The photocorrosion experiments of the selected samples in a nonsacrificial system were investigated in a gas-closed reactor with water cooling. An assembling quartz window (60 mm in diameter) on the top of the reactor was used for light entrance. 50 mg of the sample was dispersed in 100 ml of distilled water, and the system was bubbled with nitrogen for 30 min to create an anaerobic condition. The suspension was irradiated by the above xenon lamp with continuous stirring. At certain time intervals, 2 ml aliquots were sampled and centrifuged at 10 000 rpm for 5 min to separate the solutions and catalysts. The concentration of released Cd²⁺ in the reaction solution was determined by inductively coupled plasma (ICP, Vario El Cube, Elemental, Hanau, German). No Pt cocatalysts such as Pt were used in the photocorrosion tests.

X-Ray Diffraction (XRD) test was carried by Miniflex300 (Rigaku, Japan) and transmission electron microscope (TEM/HRTEM) and High Angle circular dark field scanning transmission electron microscope (HAADF-STEM) images were performed by Tecnai F20 (FEI, Hillsboro, OR, USA). X-ray photoelectron spectroscopy (XPS) tests were carried out on Escalab 250Xi (Thermo Fisher, Waltham, MA, USA). Diffuse reflectance UV–vis spectra of the samples were collected using a Shimadzu UV-2550 UV–vis spectrophotometer equipped with an integrating sphere and barium sulfate (BaSO₄) was used as the reference sample.

3. Results and discussion
The crystal structure and phase composition of the samples were characterized by power XRD. As shown in figure 1, the diffraction peaks of all the samples can be easily indexed to hexagonal CdS (JCPDS NO. 41-1049) [39], and there were no other impurities peaks in the patterns. Furthermore, there were no peaks of Ag₂S and Ni metals, which should be because the content of these two components is too low in these samples. According to Scherrer formula [46], the particle size of CdS/Ag₂S, Ni/CdS and Ni/CdS/Ag₂S nanocomposites were about 5 nm, 7 nm and 5 nm respectively.

The morphological and structural features of the Ag₂S/CdS/Ni sample were further examined by TEM/HRTEM, as shown in figure 2. Figure 2(a)) show the relative positions of the CdS base, Ag₂S and Ni three nanoparticles. HRTEM observations indicate that the distances between the adjacent lattices fringes in figure 2(b)) were approximately 0.20 nm, and those in figures 2(c) and (d) were approximately 0.31 nm and 0.34 nm, in agreement with the (111) d spacing of Ni [47], the (111) d spacing of Ag₂S [41] and the (002) d spacing of CdS [41]. This result shows that both Ag₂S and Ni nanoparticles are loaded on CdS base successfully. Ag₂S were prepared by a solubility product difference-guided synthesis method, which means that the ion exchange between Cd²⁺ and Ag⁺ only happen on the bare surface of CdS. If a part of the CdS surface is covered by Ni nanoparticles, then ion exchange will not occur. Therefore, Ag₂S and Ni nanoparticles must be grown in different positions on the surface of CdS substrate, which provides a basis for the formation of long-range electric fields.
The HAADF-STEM and elemental mapping images in figures 3(a) and (b)–(d) also show that elements Al and Ni homogeneously dispersed on the surface of CdS. This shows that ion exchange between Cd$^{2+}$ and Ag$^{+}$ occurs uniformly on the surface of CdS nanoparticles, and so that the synergistic effect between Ag$_2$S/CdS and Ni/CdS heterojunctions is also more likely to form.

To further analyze the valance state of Ag and Ni, XPS was employed to investigate both the elemental composition and the valence bond structure of Ag$_2$S/CdS/Ni, Ni/CdS and Ag$_2$S/CdS three sample. XPS survey spectrum in figure S1 (available online at stacks.iop.org/MRX/8/045508/mmedia) shows that Ag$_2$S/CdS/Ni is composed of Cd, S, O Ag and Ni elements. As presented in figure 4(a), the high-resolution XPS spectrum for the
Ni region of Ni/CdS shows a signal at 852.5 eV, corresponding to Ni 2p3/2, a peak at 870.0 eV belonging to Ni2p1/2, and a peak at 859.5 eV belonging to a Ni 2p3/2 satellite [48]. Since the content of Ni element in Ag2S/CdS/Ni sample is only 0.6%, the signal of XPS is relatively weak, only the peak at 852.5 eV can be found. According to previous reports [49–51], the Ni2p3/2 peak of Ni/CdS and Ag2S/CdS/Ni was shifted towards lower binding energy values by 0.3–0.4 eV, indicating that Ni nanoparticles loaded on CdS maybe act as electron acceptors under our experimental conditions [48, 52]. The spectrum for Ag region of Ni/CdS and Ag2S/CdS/Ni in figure 4(b)) shows that the peaks located at 368.1 and 374.1 eV can be assigned to AgI3d5/2 and AgI3d3/2, respectively, which indicates the formation of a monovalent compound on the surface of metallic Ag [53, 54]. This confirms that Ag+ ions replace the Cd2+ ions through ion exchange, and the Ag2S nanoparticles were successfully loaded on the CdS and CdS/Ni surfaces. The two main peaks observed at 405.1 and 411.8 eV in figure 4(c)) were attributed to the Cd 3d3/2 and Cd 3d1/2 photoelectrons of the Cd2+ species in CdS, respectively, which corresponded to the S2− species of CdS [15, 56].

Figure 5 depicts the UV–vis diffuse absorption spectra for samples pristine CdS, Ag2S/CdS/Ni, Ni/CdS and Ag2S/CdS composites. There is a slight light absorption in the 550–700 nm wavelengths in the sample Ag2S/CdS because of the loading of Ag2S, so do the sample Ag2S/CdS/Ni. The Tauc plot inserted in the inset of figures 5 and S2 were used to determine the band gaps of the samples by fitting the optical transition at the absorption edges. The fitting results indicate that the band gap of the four samples are about 2.39 ~ 2.41 eV, which is consistent with the existing results [19, 39]. The result shows that the loading of Ag2S and Ni nanoparticles do not significantly change the optical absorption feature of CdS nanoparticles.

The results of photocatalytic H2 production activities of the Ag2S/CdS, Ni/CdS and Ag2S/CdS/Ni are shown in figure 6(a)). After 3 h of irradiation, the Ag2S/CdS exhibited a H2 production activity of 0.33 mmolh−1 g−1; the Ni/CdS exhibited a H2 production activity up to 1.13 mmolh−1 g−1, which is more than three
times as high as the activity of Ag₂S/CdS. Generally speaking, if metal Ni nanoparticles on the surface of Ni/CdS are replaced by Ag₂S, its photocatalytic activity should be reduced, because the photocatalytic activity of Ag₂S/CdS is lower than that of Ni/CdS. However, it could be seen in figure 6(a), after Ni was replaced by Ag₂S, the Ag₂S/CdS/Ni exhibited the highest H₂ production activity approaching 1.54 mmol h⁻¹ g⁻¹, which is nearly five times the activity of Ag₂S/CdS and about 36% higher compared with that of Ni/CdS. To evaluate the stability of the most active sample, recycling tests were performed on the H₂ production activity of Ag₂S/CdS/Ni. As shown in figure 6(b), after three recycles, no significant decrease was observed in the H₂ production rate, indicating the good stability of the catalysts during the photocatalytic reaction.

In order to confirm whether the valence states of Ag and Ni in Ag₂S/CdS/Ni change during the photocatalytic process, the sample was centrifuged, dried and recycled and then used to test the XPS again, which is showed in figures 7 and S3. By comparing the XPS data before and after the photocatalytic hydrogen production, it can be determined whether the valence states of Ag and Ni change.
production test in figures 4 and 7, we can see that the valence states of Ni and Ag have not changed, indicating that the sample is very stable.

The photocorrosion of CdS-based photocatalysts is inevitable if there is no corresponding anti-photocorrosion strategy [57, 58]. The surface sulphide ions of CdS are easily oxidized by photogenerated holes to form solid sulphur, as shown in equation (1):

\[
\text{CdS} + 2h^+ \rightarrow \text{Cd}^{2+} + S
\]  

In the CdS-based heterojunction, the photogenerated electrons and holes can be separated by the built-in electric field. If the photogenerated holes in CdS are transferred to semiconductor in contact, the degree of photocorrosion of CdS would be reduced, and the concentration of Cd\(^{2+}\) in the solution would be declined.
Conversely, if the photogenerated holes in CdS stay on itself, the degree of photocorrosion of CdS would be increased, and the concentration of Cd$^{2+}$ in the solution would be raised. Consequently, by measuring and comparing the concentration of Cd$^{2+}$ in the solution after the photocorrosion experiments, the photogenerated carriers transfer route could be qualitatively judged. As mentioned above, the photo-generated holes in Ag$_2$S/CdS should be transferred to Ag$_2$S, while the holes in Ni/CdS remain in CdS. Therefore, under the same conditions, the Cd$^{2+}$ ion concentration in Ag$_2$S/CdS solution should be lower than that of Ni/CdS.

The concentration of Cd$^{2+}$ as a function of photocorrosion time of samples CdS, Ag$_2$S/CdS, Ni/CdS and Ag$_2$S/CdS/Ni was shown in figure 8. After 8 h of photocorrosion experiment, the concentration of Cd$^{2+}$ of Ag$_2$S/CdS solution is 232.14 mg L$^{-1}$, while that of CdS and Ni/CdS are 225.62 mg L$^{-1}$ and 142.58 mg L$^{-1}$, respectively. Obviously, in the Ag$_2$S/CdS heterojunction, the photogenerated holes should remain in the CdS to make its photocorrosion degree higher than that of CdS, while in the Ni/CdS heterojunction, the holes are transferred to the Ni nanoparticles, so that the photocorrosion degree is greatly reduced. This result is consistent with the conclusion of Ni as electron acceptor in figure 4. This is not an exception. Ag$_2$S act as electron donor in Ag$_2$S/CdS heterostructure [39] and Ni nanoparticles act as an electron acceptor in Ni/g-C$_3$N$_4$ heterostructure [37] had also been reported. The photocorrosion degree of Ag$_2$S/CdS/Ni sample is between Ag$_2$S/CdS and Ni/CdS, which is mainly because the loading amount of Ni is less than that of Ni/CdS.

Figure 9 presents the schematic of Ag$_2$S/CdS/Ni nanocomposites for photocatalytic H$_2$-production. Given the above results and discussions, we propose a possible photocatalytic H$_2$ production mechanism in Ag$_2$S/CdS/Ni under irradiation. Ni and Ag$_2$S nanoparticles are loaded on CdS respectively. After re-equilibrium of the charge migration caused by the potential difference, Ni nanoparticles were negatively charged while Ag$_2$S...
nanoparticles were positively charged, forming a long-range electric field similar to the p-i-n electric field structure inside CdS. When the Ag$_2$S/CdS/Ni is under illumination, most of the photogenerated electrons and holes in CdS drift to the electron donor and electron acceptor respectively under the action of long-range electric field, which greatly improves the separation efficiency of photogenerated carriers and the photocatalytic activity.

4. Conclusion

In summary, this article has successfully prepared Ag$_2$S/CdS/Ni ternary nanostructures through a solution method and solubility product difference-guided synthesis method. As revealed by the degree of photocorrosion of CdS, the photogenerated electrons of CdS are prone to transfer to Ag$_2$S while the photogenerated holes are prone to transfer to Ni nanoparticles. This Ag$_2$S/CdS/Ni ternary nanocomposite exhibits superior and durable photocatalytic H$_2$ production activity. The enhanced photocatalytic performance can be attributed to the formation of long-range electric field caused by the synergistic effect between two heterojunctions Ag$_2$S/CdS and CdS/Ni.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Conflicts of interest

The authors declare no conflict of interest.

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