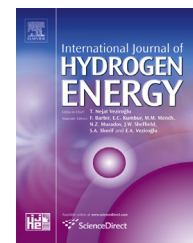


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Fabrication of nano-CdSe thin films from gas/liquid interface reactions and self-assembly for photoelectrochemical hydrogen production

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ABSTRACT

Thin films of CdSe nanoparticles (NPs) with good photoelectrochemical performances were rapidly fabricated by gas/liquid interface reactions and self-assembly. While dropping NaHSe ethanol solution onto the surface of CdCl₂ solution, CdSe NPs formed rapidly, spread over the solution's surface, and assembled into a dense thin film with the help of a surfactant (dodecyl sulfate). After annealing, the film loaded on conducting glass of fluorine-doped tin oxide (FTO) became flat, uniform and compact. Annealing also resulted in crystalline phase transformation, improved light absorption, and reduced charge transport resistance. Photoelectrochemical tests show that the prepared thin film of CdSe NPs (as photoanode) could generate a saturated photocurrent density as high as 4.4 mA cm⁻² at 0.69 V (versus RHE) and a solar-to-electricity conversion efficiency up to 4.31% under visible light illumination of 100 mW cm⁻² in a Na₂SO₃ solution for photoelectrochemical hydrogen production.

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Introduction

As a kind of abundant, clean and inexhaustible source of energy, solar energy can be converted into electrical energy, chemical energy, heat energy, biomass, mechanical energy, etc. Utilization of solar energy is one of the effective ways to solve the urgent problems in energy crisis and environment pollution [1]. Solar light-driven water splitting by photoelectrochemical (PEC) cells offers an ideal route for H₂ generation due to simple devices, abundant water resources and sustainable solar energy [2,3]. But the solar-to-hydrogen

conversion efficiency of PEC cells has always been the bottleneck.

For decades, nano-structured CdSe with a narrow band gap of ~1.7 eV has attracted worldwide researchers due to its broad applications in PEC solar cells [4,5], optoelectronic device [6], thin film transistors [7], photovoltaic devices [8], laser diodes [9], chem-/bio-sensors [10], and biomedical imaging [11]. CdSe films have been prepared by cathodic electrodeposition [4,12], chemical bath deposition [5,7], spray pyrolysis [13], sol-gel processes [14], chemical vapor deposition [15], and successive ionic layer adsorption and reaction (SILAR) [16]. To our knowledge, no CdSe semiconductor films were

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prepared from gas/liquid interface reactions and self-assembly up to date.

Herein, we propose a facile and rapid route to fabricate uniform dense thin films of CdSe nanoparticles (NPs) from gas/liquid interface reactions and self-assembly by successively dropping NaHSe ethanol solution onto CdCl₂ solution surface with the help of a surfactant (dodecyl sulfate). Reactions and assembly at gas/liquid interface offer a unique technique for preparing thin uniform films of semiconductor nanomaterials. It is a kind of simple and facile method without using expensive equipment and toxic organic solvents, and the prepared thin films can be easily transferred onto electrode substrates [17]. Especially, the as-prepared film of CdSe NPs from gas/liquid interface reactions and self-assembly exhibited better photoelectrochemical performance under visible light illumination for photoelectrochemical hydrogen production than the CdSe film prepared by drop-coating of CdSe suspension.

Materials and methods

Reagents

Sodium dodecyl sulfate (SDS), Na₂S·9H₂O, CdCl₂·2.5H₂O, anhydrous alcohol, Na₂SO₃, NaOH, methanol, selenium powders and NaBH₄ were in analytical grade and used without any further purification. All aqueous solutions were freshly prepared with Millipore ultrapure water.

Assembly of CdSe thin films from gas/liquid interface reaction

Firstly, NaHSe ethanol solution was prepared by using NaBH₄ to reduce Se powders in anhydrous alcohol according to the literature with an appropriate modification [18]. Optimally, to a solution of 0.08 g Se powders in 10 ml anhydrous alcohol, 0.04 g NaBH₄ was added, and the solution was sealed and stirred under ice-water bath for 15 min. Unless mentioned otherwise, this solution was used to prepare the CdSe film. In addition, other two NaHSe ethanol solutions were also prepared in a same way for comparison by adjusting the quantity of Se powders to 0.06 g and 0.12 g, respectively, maintaining the mass ratio as 2:1 between Se powders and NaBH₄.

The procedure for preparing thin film of CdSe NPs at gas/liquid interface is shown in Scheme 1. Typically, 20 µl of freshly prepared NaHSe ethanol solution was successively dropped onto the surface of 40 ml of 0.1 M CdCl₂ solution along the wall of a Petri dish for 20 times. Red CdSe NPs were generated rapidly, which spread over the entire surface. They assembled into a compact thin film at the surface of CdCl₂ solution after dropping 8 µl of 2% SDS aqueous solution because SDS generated surface pressure. The floating CdSe film can be directly transferred onto a clean fluorine-doped tin oxide (FTO) substrate (NSG group, 16 Ω/square) by pulling up the inserted FTO plate, and the area of CdSe film was controlled as 1 cm × 1 cm. The collected CdSe film on FTO (CdSe/FTO) was dried under an infrared lamp and further annealed in a stove under air atmosphere at 300 °C for 30 min, which was then naturally cooled to room temperature. The film's color turned to taupe

brown after annealing. For comparison, CdSe films were also prepared by drop-coating of CdSe suspension onto FTO electrode, which were described in Supplementary data.

Characterization

Morphology images of the prepared thin films were obtained by transmission electron microscopy (TEM) on a JEOL-1223 microscope and by scanning electron microscopy (SEM) using a Quanta FEG 250 microscope. The crystalline phases were analyzed by X-ray diffraction (XRD) on a Bruker D8 Discover X-ray diffractometer using Cu K_α radiation (λ = 0.1542 nm). Raman spectra were measured by using a DXR Micro-Raman Spectroscopy System (Thermo Fisher Scientific) with a laser excitation wavelength of 780 nm. UV–Visible (UV–Vis) absorption spectra of the films on FTO substrates were characterized by a UV-2450 spectrophotometer (Shimadzu) with the FTO as reference. The conductance variation of CdSe films before and after annealing was measured by sandwiching the films in two pieces of conducting glass FTO/CdSe/FTO.

Photoelectrochemical (PEC) measurements

PEC measurements were conducted in 1 M Na₂SO₃ (pH = 8.92), 2 M NaOH + 2 M CH₃OH (pH = 14.3) and 0.35 M Na₂SO₃ + 0.24 M Na₂S (pH = 11.5) aqueous solution, respectively. A three-electrode system was used with the CdSe/FTO as the working electrode, a circular Pt wire as the auxiliary electrode and a saturated mercurous sulfate electrode (SMSE) Hg(l)|Hg₂SO₄(s)|K₂SO₄ (sat.) as the reference electrode. All potentials measured were calibrated to the reversible hydrogen electrode (RHE) potential according to the Nernst equation [19,20]: $E_{RHE} = E_{SMSE} + E_{SMSE}^{\ominus} + 0.059 \text{ pH}$, where E_{RHE} is the converted potential vs. RHE, $E_{SMSE}^{\ominus} = 0.615 \text{ V}$ at 25 °C, and E_{SMSE} is the experimentally measured potential against SMSE. A 500 W Xe lamp (CHF-XM-500W, Changtuo Beijing) coupled with a UVCUT400 filter (Changtuo, Beijing) served as the simulated visible solar light (>400 nm), which illuminated continuously on the photoanode through the electrolyte. The illumination power (P_{light}) was controlled at 100 mW cm^{−2}. Linear sweep voltammograms at a scan rate of 10 mV/s and chronoamperometric curves at constant potentials were carried out with a CHI 660C electrochemical station (Chenhua, Shanghai, China). Electrochemical impedance spectroscopy (EIS) measurements of the assembled CdSe thin films without and with annealing were carried out using above three-electrode system in 1 M Na₂SO₃ at −0.74 V vs. SMSE. The frequency range was 100 kHz–0.1 Hz, and the amplitude of the applied alternative voltage was 5 mV.

Results and discussion

Characterization of assembled CdSe thin films from gas/liquid interface reaction

As shown in Scheme 1, while the NaHSe ethanol solution was dropped onto the surface of CdCl₂ solution, reaction immediately occurred between Se^{2−} and Cd²⁺ at the gas/liquid

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