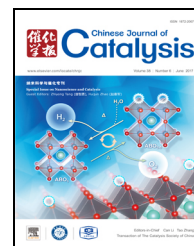


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Amorphous ferric oxide as a hole-extraction and transfer layer on nanoporous bismuth vanadate photoanode for water oxidation



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ABSTRACT

An amorphous ferric oxide layer was prepared on a bismuth vanadate photoanode. This resulted in improved charge carrier separation and surface catalytic performance compared with the photoanode without the oxide layer. The photocurrent of the oxide-layer-containing photoanode was 2.52 mA/cm² at 1.23 V versus the reversible hydrogen electrode, in potassium phosphate buffer (0.5 mol/L, pH = 7.0). The amorphous ferric oxide layer on the photoanode contained low-valence-state iron species (Fe^{II}), which enabled efficient hole extraction and transfer.

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1. Introduction

The direct capture and storage of solar energy via the formation of chemical bonds is a promising approach to realizing alternative energy sources [1,2]. Various approaches for solar energy storage have been reported. Photoelectrochemical (PEC) water splitting is one of the most promising approaches for hydrogen evolution [3–5]. n-Type bismuth vanadate (BiVO₄) is a promising photoanode in PEC systems, because of its narrow bandgap, appropriate conduction band energy level, and sufficient abundance [6]. However, typical incident photon-to-current conversion efficiencies (IPCE) of unmodified BiVO₄ have been unimpressive, because of the rapid recombination of photogenerated electron-hole pairs, poor electrical

conductivity, and slow hole transfer kinetics for water oxidation. Much effort has focused on enhancing the performance of BiVO₄-based water splitting, by coupling with dedicated electron collectors [7], oxygen-evolution catalysts (OEC) [8], and forming homo- or hetero-junctions [9]. A synergetic enhancement of surface reaction kinetics and bulk charge separation should be an ideal way to improve the PEC performance of BiVO₄. Chang et al. [10] prepared Co₃O₄ nanoislands on BiVO₄ to suppress the formation of recombination centers, but the coverage of Co₃O₄ on BiVO₄ was insufficient for long-term performance.

In the current study, we prepared an amorphous ferric oxide layer (FeO_x) on a BiVO₄ photoanode, to enhance the PEC performance of BiVO₄. The resulting photoanode (denoted Bi-

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VO_4/FeO_x) exhibited a photocurrent of 2.52 mA/cm² at 1.23 V versus the reversible hydrogen electrode (RHE) in 0.5 mol/L potassium phosphate buffer (0.5 mol/L, pH 7.0) under AM 1.5G simulated solar irradiation.

2. Experimental

2.1. Chemicals

Bismuth nitrate pentahydrate ($\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, 98%), dimethyl sulfoxide (DMSO), sodium hydroxide (NaOH, AR) and hexane were purchased from Sinopharm Chemical Reagent Co., Ltd. Vanadyl acetylacetonate ($\text{VO}(\text{acac})_2$, 99%) was purchased from J&K.

2.2. Preparation of nanoporous BiVO_4 photoanode

The nanoporous BiVO_4 photoanode was prepared in two steps: electrodeposition of a BiOI nanoarray, and subsequent conversion to nanoporous BiVO_4 by annealing and soaking treatment. Typically, 0.4 mol/L KI solution (0.4 mol/L) was adjusted to pH = 1.7 by adding HNO_3 . Then, 20 mmol of KI was added to give a transparent orange-red solution. This solution was mixed with 20 mL of absolute ethanol (99.99%) containing 0.23 mol/L p-benzoquinone, and the resulting solution was vigorously stirred for 15 min. Electrodeposition was carried out using a three-electrode system with a fluorine-doped tin oxide (FTO) substrate (2.5 cm × 1 cm) as the working electrode, platinum foil (2 cm × 2 cm) as the counter electrode, and a saturated Ag/AgCl electrode as the reference electrode. Cathodic deposition was performed potentiostatically at -0.1 V versus Ag/AgCl at room temperature for 5 min. BiVO_4 electrodes were obtained by depositing 0.1 mL of a DMSO solution containing 0.4 mol/L $\text{VO}(\text{acac})_2$ on the BiOI film, followed by annealing in a muffle furnace at 450 °C for 2 h. After soaking in 0.5 mol/L NaOH solution for 30 min to remove excess V_2O_5 , the resulting yellow BiVO_4 film was rinsed in pure water and allowed to dry.

2.3. Preparation of $\text{BiVO}_4/\text{FeO}_x$ photoanode

Iron (III) 2-ethylhexanoate (50% (w/w) in mineral spirits, J&K) was dissolved in hexane to give a 10% (w/w) solution. 5 μL of this solution was deposited onto the BiVO_4 photoanode using a pipette. The electrode was irradiated by an ultraviolet light-emitting diode for 50 h to achieve decomposition of precursor complex, and was then annealed in an oven at 100 °C for 1 h. A similar procedure was used to prepare the FTO/ FeO_x samples.

2.4. Characterization

The crystal structures of the samples were determined using X-ray diffraction (XRD, Bruker D8 Advanced diffractometer, $\text{Cu K}\alpha$ radiation, 40 kV). The morphologies and microstructures of the samples were characterized by field-emission scanning electron microscopy (FESEM, Hitachi S4800, Japan) and high-resolution transmission electron microscopy (HRTEM,

JEOL JEM-2010F, F20, 200 kV).

2.5. Photoelectrochemical measurements

PEC experiments were conducted by back-side illumination using a three-electrode configuration. The sample electrode, platinum foil, and Ag/AgCl in saturated KCl solution were used as the working, counter, and reference electrodes, respectively.

Simulated solar radiation was provided by a solar simulator (Oriel, 91160, AM 1.5 globe), the power of which was calibrated to be 100 mW/cm² using a Newport Oriel PV reference cell system (model 91150V). A potassium phosphate (KPi) buffer solution (0.5 mol/L, pH = 7.0) with and without Na_2SO_3 (1 mol/L) as a hole scavenger was used as the electrolyte. The IPCE was measured by a solar simulator (Newport 70525) coupled with a filter (Newport 70625) and an aligned monochromator (Newport 7400). All experiments were performed using an electrochemical workstation (Parstat 2237, Princeton, UK).

Electrochemical impedance spectroscopy (EIS) measurements were carried out by applying an AC amplitude of 50 mV with a frequency range from 10⁵ to 0.01 Hz, in KPi buffer (0.5 mol/L) under the open-circuit potential. Mott-Scotky plots were obtained under the same solution and scanned from 0 to 1 V versus RHE. The AC potential frequency was 5000 Hz under dark conditions, with an amplitude of 3 mV.

The potential versus Ag/AgCl was converted to the RHE scale using the Nernst equation:

$$E_{\text{RHE}} = E_{\text{Ag/AgCl}} + 0.059 \times \text{pH} + 0.1976$$

3. Results and discussion

Fig. 1(a) shows that the gossamer-like amorphous FeO_x was uniformly deposited on the surface of the BiVO_4 nanoparticles, without obvious aggregation. Energy dispersive X-ray spectroscopy was used to confirm the presence of the ferric oxide layer (Fig. 1(b)). The mapping images show the uniform distri-

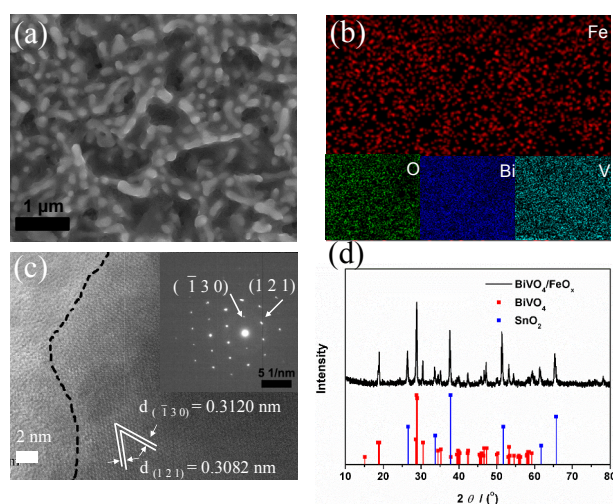


Fig. 1. (a) Top-view SEM image of the $\text{BiVO}_4/\text{FeO}_x$ photoanode; (b) Elemental mapping images of the $\text{BiVO}_4/\text{FeO}_x$ photoanode; (c) HRTEM image and corresponding SAED pattern of the $\text{BiVO}_4/\text{FeO}_x$ film; (d) XRD pattern of the $\text{BiVO}_4/\text{FeO}_x$ photoanode.

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