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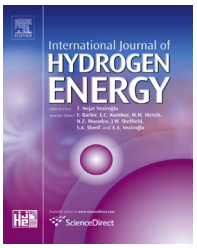

CdS-sensitized 1-D single-crystalline anatase TiO_2 nanowire arrays for photoelectrochemical hydrogen production

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ABSTRACT

We report photoelectrochemical (PEC) properties of single crystalline anatase TiO_2 nanowire (NW) arrays which are sensitized with CdS for visible light activity. Photocurrent varies largely with increasing the growth time (from 2 to 24 h) of the TiO_2 NWs and also increasing the deposition cycles (from 7 to 13 cycles) of CdS, which are attributed to changes in morphology of the TiO_2 NWs and CdS layer. TiO_2 NW array grown for 3 h with 9-cycles of CdS deposition exhibits the maximum photocurrent of 3.6 mA/cm^2 at 1.23 V vs. reversible hydrogen electrode (RHE) under the 1 SUN solar light. Morphological analysis combined with photoresponse analysis show that the 3 h-grown TiO_2 NW array has the most suitable structure for PEC electrode because of large surface area for light absorption and chemical reaction, and that the 9-cycles deposited CdS layer provides the optimum light absorption and electron lifetime.

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Introduction

The procurement of clean and renewable energy resources is one of the recent important issues, due to the eventual exhaustion of fossil fuels, the environmental pollution, and the world's constantly increasing energy consumption [1,2]. Photoelectrochemical (PEC) hydrogen production is currently being considered as a potential clean and renewable route for energy generation [3]. After the discovery of PEC hydrogen

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production reaction using n-type TiO_2 by Fujishima and Honda in 1972, PEC hydrogen production using TiO_2 as a photoanode have been widely studied [4–6]. However, the large band gap (3.2 eV) of TiO_2 restricts the absorption of solar energy in visible range.

Over the past several decades, many attempts have been made to improve the water splitting performance of TiO_2 photoanodes [7–13], largely in the manner of modifying the band gap by doping with transition-metal cations [7] or anions [8,9], or forming the heterostructure with a narrow band-gap inorganic sensitizer such as CdS [10,11], CdSe [12], and CdTe [13]. For successful development of highly-efficient, sensitized TiO_2 photoanodes, it should possess a large surface area to load a sufficient amount of sensitizers, a short distance for minority carrier to diffuse to the surface, a fast majority carrier collection to the substrate electrode, and a long optical path length [14–16]. One dimensional (1-D) TiO_2 nanostructures, such as nanotube, nanorod and nanowire (NW) arrays have been reported to satisfy the requirements, exhibiting superior PEC properties to planar TiO_2 photoelectrodes [16]. Because of difficulties in synthesis of single-crystalline anatase TiO_2 1-D arrays, most of the PEC property of 1-D TiO_2 have been studied using single-crystalline rutile NW arrays [16–18] or polycrystalline anatase nanotubes [19]. There has been lack of study on single crystalline anatase TiO_2 1-D structures in PEC systems [20], although anatase is the most suitable phase for a highly efficient PEC system among the TiO_2 polymorphs, owing to its highly-active hydroxylated surface, low charge carrier recombination rate [21], fast electron transport properties [22], and electrochemically appropriate conduction band edge position [23–25].

Here in, we report single crystalline anatase TiO_2 NW based photoanodes which are functionalized to be active in visible-light by CdS sensitization. By varying the growth time of the TiO_2 NW and deposition cycles of the CdS layer, we systematically investigated the morphology, light absorption, and charge collection property of the CdS-sensitized TiO_2 NW-based photoanodes. Based on the analysis, TiO_2 NW growth time and CdS deposition cycles were optimized for PEC performance under the 1 SUN condition.

Experimental

Preparation of CdS-sensitized TiO_2 nanowire-based photoanode

Anatase TiO_2 NW arrays were grown on 0.127 mm-thick titanium foil (Sigma Aldrich) via three steps, following a previously reported method [26]. The first step is as in the following. A piece of titanium foil (1.5 × 1.6 cm^2) was ultrasonically cleaned in a solution of distilled water, acetone and 2-propanol (volume ratio 1:1:1) for 10 min and then placed against the wall of a 110 ml Teflon-lined stainless steel autoclave filled with 36 ml of a 1 M NaOH aqueous solution. Then, as the first step, the autoclave was put in an electric oven at 220 °C for 2–24 h. From here, the growth time of TiO_2 NW designates the reaction time of the first step. After the completion of this alkali hydrothermal growth process, the second and third steps followed Boercker et al. [27]. For the second step, the titanium foil covered with the $Na_2Ti_2O_5 \cdot H_2O$ NW array was immersed in a 0.6 M HCl solution for 1 h to exchange the Na^+ ions with the H^+ ions. The titanium foil was rinsed with pure ethanol and dried under ambient conditions. In the third step, the dried titanium foil covered with NWs was calcined in a furnace at 450 °C for 4 h. Then CdS inorganic sensitizer was coated on anatase TiO_2 NW using a modified successive ionic layer adsorption and reaction (SILAR) route reported by Baker et al. [11]. The prepared TiO_2 NW array electrodes were immersed in a solution containing 50 mM cadmium acetate dihydrate ($Cd(CH_3COO)_2 \cdot 2H_2O$, Sigma Aldrich, 98%) in ethanol for 1 min in order to adsorb the Cd^{2+} ions onto the TiO_2. The electrodes were then rinsed with pure ethanol and blown with N_2. The dried electrodes were immersed in a solution containing 50 mM sodium sulfide (Na_2S, Sigma Aldrich, 98%) in methanol for 1 min, where the pre-adsorbed Cd^{2+} ions reacted with the S^{2-} ions to form the desired CdS. The electrodes were then rinsed in distilled water to remove the excess ions and dried again with N_2. This SILAR cycle was repeated 7–13 times on different samples to investigate the effects of the absorbed amount of CdS. The sensitized samples were then annealed at 300 °C for 30 min in an ambient atmosphere to further increase the crystallinity of CdS [28].

Characterization

The crystal structures of the anatase TiO_2 NW arrays were characterized by an X-ray diffractometer (New D8-Advance, Bruker Miller Co.). The morphologies of the prepared TiO_2 NW and CdS/TiO_2 NW were investigated using a field-emission scanning electron microscopy (FESEM) (JSM-6330F, JEOL). High-resolution transmission electron microscopy (HRTEM) (SEM-3000F, JEOL) was used for analyzing the morphology and crystal structure of an individual TiO_2 NW and CdS/TiO_2 NW. The optical absorbance of the CdS/TiO_2 NW photoelectrodes was measured between 400 nm and 700 nm using ultraviolet–visible light (UV–vis) spectroscopy (Cary 5000, Agilent).

Measurements of photoelectrochemical properties

The photocurrent density, electrochemical impedance spectroscopy (EIS), and photoresponse of the anatase CdS/TiO_2 NW electrodes were measured by aids of potentiostat (CHI 608C, CH Instruments) and solar simulator (HAL-320, Asahi spectra), in an electrolyte (0.35 M Na_2SO_3 and 0.25 M Na_2S 1:1 volume ratio mixed solution, pH = 13) with a Ag/AgCl reference electrode and a Pt plate counter electrode. The measured potentials vs. the Ag/AgCl were converted to the reversible hydrogen electrode (RHE) scale via the Nernst equation, as follows:

$$E_{RHE} = E_{\frac{Ag}{AgCl}} + E^0_{\frac{Ag}{AgCl}} + 0.059 \times pH$$

The simulated solar light was calibrated using a reference cell (PV Measurements) to be 1 SUN (1.5 AM, 100 mW/cm^2). The applied bias photon-to-current efficiency (ABPE) values were calculated from the 2-electrode J–V curves with an assumption of 100% faradic efficiency.

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