



Cobalt/molybdenum ternary hybrid with hierarchical architecture used as high efficient counter electrode for dye-sensitized solar cells

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Abstract

Cobalt selenide/molybdenum selenide/molybdenum oxide ($\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$) ternary hybrid is synthesized by a facile hydrothermal method and used as efficient Pt-free counter electrode (CE) for dye-sensitized solar cells (DSSCs). Field emission scanning electron microscopy observes that $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ possesses hierarchical architecture with one-dimensional (1D) nanorods, two-dimensional (2D) nanosheet and three-dimensional (3D) nanoparticle. Cyclic voltammogram indicates that $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ hybrid have larger current density than sputtered Pt electrode. Electrochemical impedance spectroscopy shows that cooperation among $\text{Co}_{0.85}\text{Se}$, MoSe_2 and MoO_3 reduces series resistance and charge transfer resistance. The DSSC based on $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ CE achieves a power conversion efficiency of 7.10%, which is higher than that of the DSSC based on sputtered Pt CE (6.03%).

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1. Introduction

Dye-sensitized solar cells (DSSCs) have attracted growing interests around the world due to their low cost, simple preparation procedures and relatively high power conversion efficiency (PCE) (O'regan and Gratzel, 1991; Hagfeldt et al., 2010; Wu et al., 2015). So far, a breakthrough was made in Co-based electrolyte DSSCs, giving a PCE of 13% (Mathew et al., 2014). Basically, a DSSC consists of a dye-sensitized photoanode, an electrolyte with I^-/I_3^- redox couple, and a counter electrode (CE). As an important component in a DSSC, the mission of a CE is to collect electrons from the external circuit and catalyze

the reduction of I_3^- to I^- at the counter electrode/electrolyte interface. Thus, an ideal CE should have high electrocatalytic activity, high electrical conductivity, and good stability (Arbab et al., 2015; Ke et al., 2014). At present, platinum (Pt) is the most-common CE material for I^-/I_3^- based DSSCs owing to its excellent electrocatalytic activity, superior conductivity and stability. However, as a noble metal, Pt is expensive and rare on the earth, causing difficulties in commercialization of DSSCs. Therefore, searching for competent substitutes for Pt is of great importance.

To date, the most common-used substitutes for Pt include carbonaceous materials (Chang et al., 2015; Dao et al., 2015; Ma et al., 2015; Yeh et al., 2014), conductive polymers (Duong et al., 2014; Park et al., 2015; Wu et al., 2014; Yue et al., 2012) and transition metal compounds such as nitride (He et al., 2014; Wu et al., 2014),

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carbide (He et al., 2014; Wu et al., 2014; Xu et al., 2014) and chalcogenide (Huang et al., 2015; Ke et al., 2014; Kung et al., 2012; Lee et al., 2014; Shahpari et al., 2015). Among them, transition metal chalcogenide has been considered as one of the most prospective CE material in DSSCs due to their low cost, abundant resources, excellent catalytic activity and high conductivity. As for CE materials in DSSCs, transition metal selenide is less reported compared to transition metal oxide and transition metal sulfide even though they show good electrochemical properties. Recently, Gong et al. prepared binary metal selenide $\text{Co}_{0.85}\text{Se}$ CE in situ and achieved a PCE of 9.40% versus 8.64% for the Pt CE (Gong et al., 2012). Chen et al. proposed a facile MoSe_2/Mo counter electrode and obtained a power conversion efficiency of 8.13% which was comparable to the PCE of Pt (8.06%) (Chen et al., 2014). Lee et al. prepared few-layer MoSe_2 and yielded a PCE of 9.00% versus 8.68% for the Pt CE (Lee et al., 2014). Here, cobalt selenide/molybdenum selenide/molybdenum oxide ($\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$) hybrid counter electrode is synthesized by a facile hydrothermal reduction method without any scarce elements, and the molar ratio of Co/Mo/Se in the hybrid is optimized. When the molar ratio of Co/Mo/Se is 2/2/4, the DSSC based on $2\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ CE achieves PCE of 7.10% while the PCE of the DSSC based on Pt CE is only 6.03% under the same condition.

2. Experimental

2.1. Synthesis of $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ hybrid CEs

The $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ hybrid was prepared through a one-step hydrothermal reaction similar to Wang et al. (2005). 1 g NaOH and 8 ml oleic acid were added to 32 ml absolute ethyl alcohol to form solution A. A transparent solution B comprises predetermined amount of MoCl_5 and $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ and 14 ml deionized water. 0.2 g Se powder was dissolved in 10 ml $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (80 wt %) to form a transparent solution C. Then solution B was added to solution C under stirring. After stirred for 10 min, the above solution was added to solution A. Finally, the mixture was added to a 100 ml autoclave and reacted at 160 °C for 12 h. After cooled down to room temperature, the black precipitate at the bottom of the autoclave was collected, centrifuged and washed with absolute ethyl alcohol for several times. Then, the product was re-dispersed in hexamethylene with concentration of 0.08 g ml^{-1} to form $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ ink. $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ hybrid CE was fabricated by directly drop-casting the ink on the cleaned FTO glass and dried at room temperature. In this experiment, we synthesized a series of $\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$ hybrids by controlling the molar ratio of Co/Mo/Se in the reactant at 4/0/4, 3/1/4, 2/2/4, 1/3/4, and 0/4/4. The corresponding products are simply denoted as A, B, C, D, and E, respectively. Which stand for the hybrid $\text{Co}_{0.85}\text{Se}$, $3\text{Co}_{0.85}\text{Se}/0.5\text{MoSe}_2/0.5\text{MoO}_3$,

$2\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$, $2\text{Co}_{0.85}\text{Se}/\text{MoSe}_2/\text{MoO}_3$, and $\text{Co}_{0.85}\text{Se}/1.5\text{MoSe}_2/1.5\text{MoO}_3$, respectively.

2.2. Fabrication of DSSCs

A layer of mesoporous TiO_2 was prepared according to our previous paper (Wu et al., 2007). After that, the TiO_2 photoanode was sensitized by immersing into an ethanol solution of N719 dye (2.5×10^{-4} mol ml^{-1}) for 24 h. A DSSC device was fabricated by sandwiching redox electrolyte between a dye-adsorbed TiO_2 photoanode and various CEs. The active area of DSSC devices was 0.12 cm^2 . The redox electrolyte was composed of 0.10 M tetramethyl ammonium iodide, 0.1 M tetraethyl ammonium iodide, 0.1 M tetrabutyl ammonium iodide, 0.1 M sodium iodide, 0.1 M potassium iodide, 0.1 M lithium iodide, 0.05 M iodine and 0.50 M 4-tert-butyl-pyridine in acetonitrile.

2.3. Measurements

The as-prepared sample was characterized by X-ray photoelectron spectroscopy (XPS, ESCALAB 250XI, Thermo). The morphologies of as-synthesized samples were examined by a field emission scanning electron microscopy (FESEM) (SU8000, HITACHI) at an acceleration voltage of 5 kV. The photocurrent–voltage (J – V) curves of the assembled DSSCs were recorded on a Keithley 2400 source meter under the irradiation of AM 1.5G simulated solar light supplied by an AAA solar simulator (Newport-94043A) equipped with a Xe lamp (450 W) and an AM 1.5G filter. The incident light was calibrated with a reference Si solar cell (Oriel-91150). The incident photon-to-current conversion efficiency (IPCE) curves were measured using the Newport IPCE system (Newport, USA). The cyclic voltammetry (CV) curves were carried out using CHI660E setup in a three-electrode system with a Pt sheet as counter electrode, an Ag/AgCl electrode as reference electrode and various CEs as working electrode. The electrolyte is acetonitrile solution containing 10 mM LiI, 1 mM I_2 , and 0.1 M LiClO_4 . The scan rate is 50 mV s^{-1} . Electrochemical impedance spectroscopy (EIS) measurements were implemented with a Zennium electrochemical workstation (IM6) in a frequency range of 100 mHz–100 kHz at an AC amplitude of 5 mV. The impedance spectra were analyzed with Zview software. Tafel polarization curves were implemented on the same Workstation by assembling symmetric cell consisting of FTO/CE/redox electrolyte/CE.

3. Results and discussion

3.1. Morphology observation

The field emission scanning electron microscopy (FESEM) images of the five samples (A, B, C, D, and E) on FTO are shown in Fig. 1. From the FESEM images shown in Fig. 1(a), (e), and (f), it can be seen that sample

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