



# Transparent nickel selenide used as counter electrode in high efficient dye-sensitized solar cells



Jinbiao Jia, Jihuai Wu\*, Yongguang Tu, Jinghao Huo, Min Zheng, Jianming Lin

Eng. Res. Center of Environment-Friendly Functional Materials, Ministry of Education, Institute of Materials Physical Chemistry, Huaqiao University, Quanzhou 362021, China

## ARTICLE INFO

### Article history:

Received 3 December 2014  
Received in revised form 27 March 2015  
Accepted 28 March 2015  
Available online 9 April 2015

### Keywords:

Dye-sensitized solar cell  
Counter electrode  
Nickel selenide

## ABSTRACT

A transparent nickel selenide ( $\text{Ni}_{0.85}\text{Se}$ ) is prepared by a facile solvothermal reaction and used as an efficient Pt-free counter electrode (CE) for dye-sensitized solar cells (DSSCs). Field emission scanning electron microscopy observes that the as-prepared  $\text{Ni}_{0.85}\text{Se}$  possesses porous structure. Cyclic voltammogram measurement indicates that  $\text{Ni}_{0.85}\text{Se}$  electrode has larger current density than Pt electrode. Electrochemical impedance spectroscopy shows that the  $\text{Ni}_{0.85}\text{Se}$  electrode has lower charge-transfer resistance than Pt electrode. Under simulated solar light irradiation with intensity of  $100 \text{ mW cm}^{-2}$  (AM 1.5), the DSSC based on the  $\text{Ni}_{0.85}\text{Se}$  CE achieves a power conversion efficiency (PCE) of 8.88%, which is higher than the solar cell based on Pt CE (8.13%). Based on the transparency of  $\text{Ni}_{0.85}\text{Se}$ , the DSSC with  $\text{Ni}_{0.85}\text{Se}/\text{mirror}$  achieves a PCE of 10.19%.

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## 1. Introduction

Nanocrystalline dye-sensitized solar cell (DSSC) has attracted considerable interest since it was reported by O'Regan and Gratzel in 1991 [1]. As a new kind of solar cell, DSSC displays many virtues such as low cost, easy preparation, good photovoltaic performance and environmental benignity compared with traditional photovoltaic devices [2]. Recently, Simon Mathew et al. reported a DSSC with power conversion efficiency (PCE) of 13% through the molecular engineering of porphyrin sensitizers and a Co(II/III) tris(bipyridyl)-based redox electrolyte [3]. The conventional DSSC consists of a dye-sensitized photoanode, a counter electrode (CE) and an electrolyte containing a redox couple such as an iodide/triiodide couple [2]. As a key component, the role of CE is to collect electrons from the external circuit and catalyze the reduction of triiodide ( $\text{I}_3^-$ ) to iodide ( $\text{I}^-$ ) at CE/electrolyte interface. Hence, an efficient CE should have excellent electrocatalytic activity, high electrical conductivity, and good stability [2,4]. So far, Pt CE is widely used in high-efficiency DSSCs due to its excellent conductivity and high catalytic activity toward  $\text{I}^-/\text{I}_3^-$  couple [5,6]. However, Pt CE used in DSSCs has some disadvantages. Pt is expensive and can be decomposed to  $\text{PtI}_4$  or  $\text{H}_2\text{PtI}_6$  by  $\text{I}^-/\text{I}_3^-$  redox couple [4] which influences large-scale manufacturing of the DSSCs. Besides,

Pt CE is prepared by sputtering process or high-temperature hydrolysis, which also increases the device cost. In order to overcome these disadvantages, numerous attempts have been done to explore competent substitutes for Pt [4]. The substitutes reported so far include carbon materials [7–11], conducting polymers [12–16] and inorganic compounds such as nitride [17–19] and carbide [20,21].

Metal chalcogenide is a prospective CE material in DSSCs owing to their high conductivity and excellent catalytic activity for the reduction of  $\text{I}_3^-$  [4]. Among them, the metal sulfides are the most researched CE materials [22–31]. Recently, Hsu et al. synthesized CoS nanoparticles by CTAB-assisted preparation of a metal organic framework, ZIF-67, and subsequent oxidation and sulfide conversion to CoS [25]. The generated CoS nanoparticles was used as CE for DSSCs, leading to an improved efficiency of 8.1%. Metal oxides also are used as CE materials in DSSCs [32,33], using  $\text{WO}_2$  as a CE, the DSSC obtained power conversion efficiency of 7.25% [32], which can match the performance of the DSSC based on a Pt CE. Metal selenides are fewer reported as CE materials [34,35], although they possess good electrochemical properties.

Here, we synthesize transparent nickel selenide ( $\text{Ni}_{0.85}\text{Se}$ ) by a facile solvothermal reaction and used  $\text{Ni}_{0.85}\text{Se}$  as Pt-free CE in DSSCs. Under simulated solar light irradiation with intensity of  $100 \text{ mW cm}^{-2}$  (AM 1.5), the DSSC based on the  $\text{Ni}_{0.85}\text{Se}$  counter electrode achieves a power conversion efficiency (PCE) of 8.88%, which is higher than the solar cell based on the Pt CE (8.13%).

\* Corresponding author. Tel.: +86 595 22693899; fax: +86 595 22692229.  
E-mail address: [jhwu@hqu.edu.cn](mailto:jhwu@hqu.edu.cn) (J. Wu).

When a mirror is put on the bottom of Ni<sub>0.85</sub>Se CE, the PCE of the solar cell exceed 10%.

## 2. Experimental

### 2.1. Preparation of nickel selenide CE

The Ni<sub>0.85</sub>Se was prepared by modifying the methods reported in references [35–37]. 200 mg Na<sub>2</sub>SeO<sub>3</sub>·5H<sub>2</sub>O, 1200 mg NaOH, and 6 ml N<sub>2</sub>H<sub>4</sub>·H<sub>2</sub>O were dissolved in 10 ml deionized water to form a transparent solution A. 580 mg Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O was added to 10 ml n-octanol, and was heated at 60 °C in air, forming a clear solution B. Under stirring, the solution A and the solution B were mixed, and 15 mL oleic acid was added, which formed an emulsion. The emulsion was transferred to a stainless Teflon-lined autoclave (volume 50 mL), then sealed and heated at 160 °C for 24 h. The internal pressure of the autoclave was controlled by filling volume ratio of about 75%. After the reaction, the autoclave was cooled to room-temperature naturally. The product was dispersed in n-hexane of 40 mL, then ethanol of 20 mL was added to precipitate product by centrifugating. The obtained product (Ni<sub>0.85</sub>Se) was redispersed in n-hexane to form a well-distributed black ink with concentration of 0.05 g mL<sup>-1</sup>. The counter electrode was prepared by following steps. FTO conductive glass with size of 1.5 × 1.5 cm<sup>2</sup> was ultrasonic vibrated in acetone and isopropanol solution to wash away any impurities. The black Ni<sub>0.85</sub>Se ink was spin-casted on the clean FTO glass substrate at a rate of 2000 rpm for 20 s. Then, the FTO substrate was thermally treated at 80 °C for 30 min, after that the Ni<sub>0.85</sub>Se ink was second spin-casted on the first layer and dried at 80 °C for 30 min, thus a counter electrode with Ni<sub>0.85</sub>Se thickness of about 4 μm was obtained.

### 2.2. Fabrication of DSSC device

A underlayer of TiO<sub>2</sub> compact film was deposited on the FTO conductive glass by spin-casting the toluene solution of TiO<sub>2</sub> quantum dots [38] and subsequently sintered at 450 °C for 30 min in air. Then a layer of TiO<sub>2</sub> nanocrystalline film with a thickness of 10 μm was solidified on the TiO<sub>2</sub> compacted layer according to the reference [38]. After that, the TiO<sub>2</sub> film was transformed in an ethanol solution of N719 dye (2.5 × 10<sup>-4</sup> mol L<sup>-1</sup>) for 24 h to obtain dye-sensitized TiO<sub>2</sub> photoanode. The dye-adsorbed TiO<sub>2</sub> photoanode and the Ni<sub>0.85</sub>Se counter electrode or Pt counter electrode was clipped together. One drop of liquid electrolyte was injected into the interspace between the two electrodes, and a dye-sensitized solar cell thus was fabricated. The liquid electrolyte contained 0.4 M sodium iodide, 0.1 M tetrabutyl ammonium iodide, 0.5 M 4-tert-butylpyridine, and 0.05 M iodine in acetonitrile solution. The diagram for the fabricated solar cells and irradiation model is shown in Fig. 1.

### 2.3. Measurements

Transmittance was measured by a Lamda 950 UV/Vis–NIR spectrophotometer. The morphologies of Ni<sub>0.85</sub>Se were observed with a field emission scanning electron microscopy (FESEM, SU8010, Hitachi, Japan) at an acceleration voltage of 5 kV. The crystal structures of samples were recorded by X-ray diffraction (XRD) with Cu Kα (λ = 1.5418 Å) radiation, (Smart Lab 3 kW, Rigaku, Japan). Photovoltaic tests were carried out by measuring the Current–Voltage (J–V) characteristic curves on a Keithley 2400 source meter under the illumination of a simulated solar light coming from an AAA solar simulator (Newport-94043A, USA) equipped with a Xe lamp (450 W) and an AM1.5G filter. The light intensity was calibrated with a reference Si solar cell (Oriel-91150). The cyclic voltammetry (CV) curves were recorded from –0.6 to +1.2 V and back to –0.6 V on a conventional CHI660E setup with a three-electrode system which comprise an Ag/AgCl reference electrode, a Pt counter electrode, and a working electrode of the as-prepared CE. The scan rate is 50 mV s<sup>-1</sup> and the electrolyte contained an anhydrous acetonitrile solution consisting of 0.1 M LiClO<sub>4</sub>, 10 mM LiI and 1 mM I<sub>2</sub>. Electrochemical impedance spectroscopy (EIS) measurements were carried out in a Zennium electrochemical workstation (IM6) by applying an AC voltage of 5 mV amplitude and at 0 V bias in the frequency range

between 100 mHz and 100 kHz at room temperature. Tafel polarization curves were recorded on the same workstation by assembling symmetric cell consisting of FTO/Ni<sub>0.85</sub>Se|redox electrolyte|Ni<sub>0.85</sub>Se.

## 3. Results and discussion

### 3.1. Phase, morphology and transparency

Fig. 2 shows the XRD pattern of the as-prepared nickel selenide powder and standard Ni<sub>0.85</sub>Se powder. The peak positions as-prepared nickel selenide powder can be well indexed to Ni<sub>0.85</sub>Se (JCPDS No. 18-0888), and these peaks are referred as (101), (102), (110), and other crystal faces of Ni<sub>0.85</sub>Se (JCPDS No. 18-0888). On the other hand, the compositions of obtained nickel selenide products were determined by EDX, indicating that the atomic ratio of Ni:Se was 0.831:1. The results indicate that Ni<sub>0.85</sub>Se with hexagonal structure [37] has been synthesized successfully. The nonstoichiometric nickel selenide contains more defects (mainly vacancies) within it [36], which is beneficial to enhance the electrocatalytic activity of counter electrode, and thus high photovoltaic performances for the DSSCs.

Fig. 3 displays the field emission scanning electron microscopy (FESEM) images of Ni<sub>0.85</sub>Se. It can be seen that the as-prepared Ni<sub>0.85</sub>Se exists in particles aggregated state, the particle size is about 200 nm. The aggregation exhibits a porous structure. As we known that the reduction reaction of triiodide ions occurs on the interface of counter electrode, the counter electrode with porous structure is conducive to improve the reaction speed and enhance electrocatalytic activity when it is used as a counter electrode in DSSC.

Fig. 4 shows the transmission spectrum of the as-prepared Ni<sub>0.85</sub>Se spin-coated on FTO glass substrate. It can be seen that the Ni<sub>0.85</sub>Se keep high optical transparency (>75% in the visible region). The transparency of Ni<sub>0.85</sub>Se allow sunlight back incident when used as counter electrode, which provide additional contribution to a high power conversion efficiency when put a mirror on the bottom of CE.

### 3.2. Photovoltaic performance

We assembled three dye-sensitized solar cells, one is the solar cell using Pt as counter electrode, the second is the solar cell using as-prepared Ni<sub>0.85</sub>Se as counter electrode, the third is using Ni<sub>0.85</sub>Se as counter electrode and attached a reflect mirror on the counter electrode bottom (Fig. 1b). The solar cells were irradiated with a simulated solar light with intensity of 100 mW cm<sup>-2</sup> (AM 1.5) from the front (photoanode) and the rear (counter electrode) of the solar cells, respectively. The photocurrent–voltage curves of the DSSCs were measured and shown in Fig. 5, and the corresponding photovoltaic parameters are summarized in Table 1.

From Fig. 5a, when irradiation from the front of the cell, the DSSC with Ni<sub>0.85</sub>Se counter electrode shows open-circuit voltage (V<sub>OC</sub>) of 0.780 mV, short-circuit current density (J<sub>SC</sub>) of 16.39 mA cm<sup>-2</sup>, and power conversion efficiency (PCE) of 8.88%;

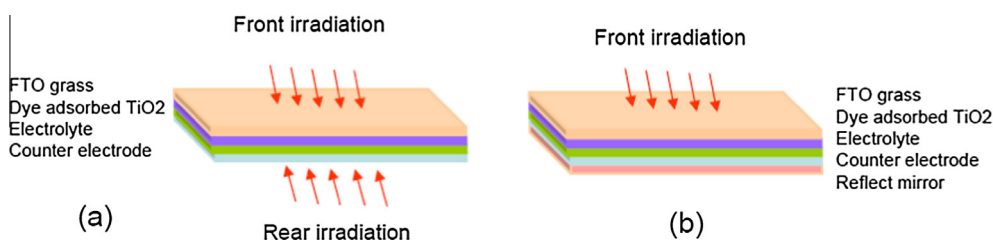


Fig. 1. The diagram for fabricated solar cells: (a) Irradiation from the front and the rear; (b) Putting a mirror on the back and irradiation from the front.

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