



Short communication

Nickel promoted cobalt disulfide nanowire array supported on carbon cloth: An efficient and stable bifunctional electrocatalyst for full water splitting

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ABSTRACT

Development of efficient noble metal-free bifunctional electrocatalysts for hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) is an ongoing challenge. Herein, we report the development of nickel promoted cobalt disulfide nanowire array supported on carbon cloth (Ni_{2.3%}-CoS₂/CC) as an efficient bifunctional electrocatalyst for water splitting with superior activity and good durability in basic media. This Ni_{2.3%}-CoS₂/CC electrode delivers 100 mA cm⁻² at overpotential of 231 mV for HER and 100 mA cm⁻² at overpotential of 370 mV for OER. The water electrolyzer based on Ni_{2.3%}-CoS₂/CC only requires a cell voltage of 1.66 V to afford 10 mA cm⁻², implying the great potential for water splitting applications.

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1. Introduction

Great concerns about environmental pollution and global energy crisis, human being is extremely urgent to search sustainable carbon-free energy sources. Hydrogen is widely considered as an ideal alternative for the cleanness, efficiency and renewability [1–5]. Water electrolysis provides us a simple approach for producing highly pure hydrogen by means of hydrogen evolution reaction (HER) [6–9]. HER is one of the two half reactions of the water splitting reaction which occurs at cathode, and the other reaction is oxygen evolution reaction (OER) at anode. Both reactions contribute to the overall efficiency of water splitting [10]. The below equation shows the practical operational voltage (E_{op}) for water splitting:

$$E_{op} = \eta_{anode} + \eta_{cathode} + \eta_{others} + 1.23 \text{ V}$$

where the theoretical minimum voltage for water splitting is 1.23 V, however, the overpotential (η) is inevitably required to overcome the intrinsic activation barriers [11]. η_{others} could be decreased by electrolytic cell design, whereas η_{anode} and $\eta_{cathode}$ are related to electrode catalysts [7,8]. Therefore, to make the splitting process cost-effective and more valuable, efficient HER and OER catalysts are of crucial importance to decrease the overpotential. Pt-based materials for HER and Ru- or

Ir-based materials for OER are recognized as the most effective ones [12,13]. Unfortunately, the low abundance and high cost of these noble metals hinder their application for large-scale applications. To this end, increasing research efforts have been focused on discovery of cheap, earth-abundant noble metal-free electrocatalysts to replace these noble metal based catalysts [8].

The overall water splitting process needs to be performed in either strongly acidic or alkaline electrolytes to achieve the lowering overpotential [14]. In acidic media, the anodic catalysts with reasonable activity are largely limited by using acid-insoluble precious metals [15, 16]. On the other hand, earth-abundant first-row transition metals have emerged as strong candidates for both HER and OER catalysts in alkaline media and commercial water electrolyzers are operated in alkaline water [7]. Besides, one should bear in mind that a bifunctional catalyst conducting HER and OER in the same electrolyte will simplify the system and reduce the cost. In light of the abovementioned facts, it is highly desirable to develop first-row transition metal based bifunctional catalysts for both HER and OER in strongly alkaline media.

Recently, CoS₂ has been reported as high-efficiency HER catalysts [17–19]. It is suggested that the crystal structure of CoS₂ is similar to the catalytic environment of hydrogenase [20,21]. Based on the fact that certain hydrogenase needs the participation of nickel ions to complete the catalytic process of hydrogen production, we safely hypothesize that Ni promoted CoS₂ will lead to outstanding efficiency for HER [22]. More importantly, the corresponding OER performance has not yet well-documented in the literature [21,23,24]. We thus represent a

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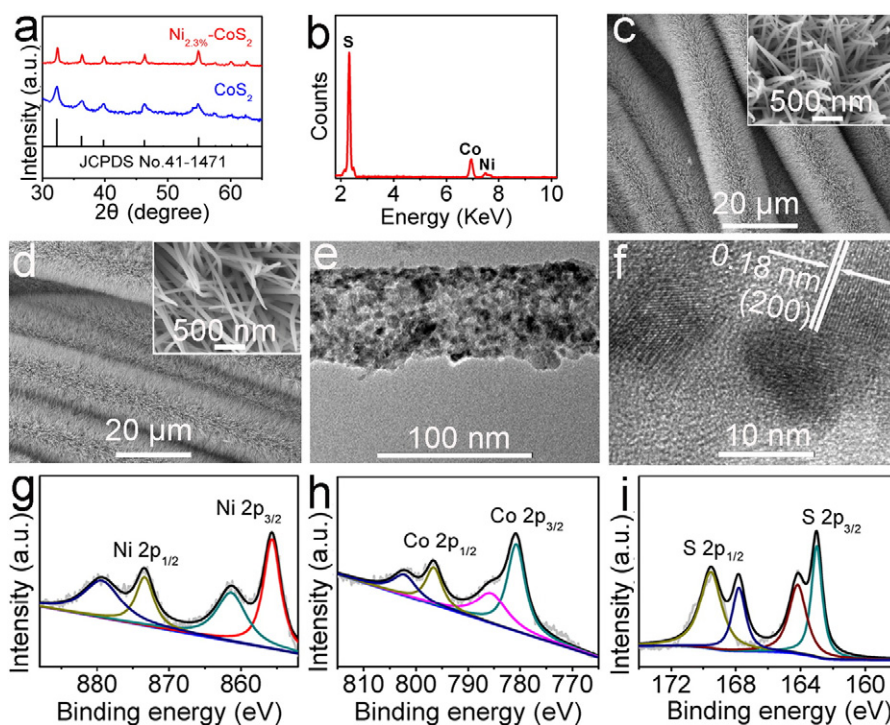


Fig. 1. (a) XRD patterns of $\text{Ni}_{2.3\%}\text{-CoS}_2$ and CoS_2 scratched down from CC. (b) EDX spectrum of $\text{Ni}_{2.3\%}\text{-CoS}_2$. SEM images of (c) $\text{Ni}_{2.3\%}\text{-CoS}_2$ and (d) CoS_2 . (e) TEM image of $\text{Ni}_{2.3\%}\text{-CoS}_2$ nanowire. (f) HRTEM image of $\text{Ni}_{2.3\%}\text{-CoS}_2$ nanowire. XPS spectra in the (g) Ni 2p, (h) Co 2p and (i) S 2p for $\text{Ni}_{2.3\%}\text{-CoS}_2/\text{CC}$.

robust Ni promoted CoS_2 electrocatalyst not only for HER but also for OER. To the best of our knowledge, it is the first example of Ni promoted CoS_2 as a bifunctional HER and OER electrocatalysts for overall water splitting in alkaline media.

2. Experimental

2.1. Reagents and materials

All reagents were used as received. Pt/C (20 wt.%) and Nafion (5 wt.%) were purchased from Sigma-Aldrich. $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$, $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$, urea and KOH were purchased from Beijing Chemical Corporation. Carbon cloth (CC) was purchased from Wuhan Instrument Surgical Instruments business and cleaned by sonication sequentially in acetone, ethanol and deionized water. The deionized water was obtained through a Millipore system.

2.2. Preparation of $\text{Ni}_{2.3\%}\text{-CoS}_2/\text{CC}$

$\text{Ni}_{2.3\%}\text{-CoS}_2/\text{CC}$ (2.3 wt.% Ni-doped CoS_2 , details of calculation at the end of this section) precursor was synthesized by a simple hydrothermal method. Typically, 0.3 mmol $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 6.0 mmol $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 36.0 mmol urea were dissolved in 40 mL deionized water. Then the above solution and cleaned CC (2 cm × 3 cm) were transferred to a 50 mL Teflon-lined stainless-steel autoclave and maintained at 120 °C for 6 h. After the autoclave cooled down, the CC with precursor was taken out, washed with deionized water and dried. To obtain $\text{Ni}_{2.3\%}\text{-CoS}_2/\text{CC}$, the substrates grown with precursor was immersed in 25 mM $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ solution (40 mL) and reacted at 120 °C for 8 h. The loading mass of $\text{Ni}_{2.3\%}\text{-CoS}_2/\text{CC}$ is 0.97 mg cm^{-2} . The Ni content was calculated using the equation: $\text{Ni (wt\%)} = [\text{n}_{\text{Ni}} \cdot \text{Mr}(\text{Ni})] / [\text{n}_{\text{Ni}} \cdot \text{Mr}(\text{Ni}) + \text{n}_{\text{Co}} \cdot \text{Mr}(\text{CoS}_2)]$, with Mr being the relative formula mass and n_x being the molar amount of the respective precursor. $\text{Ni}_{1.2\%}\text{-CoS}_2/\text{CC}$, $\text{Ni}_{3.5\%}\text{-CoS}_2/\text{CC}$ and $\text{Ni}_{4.6\%}\text{-CoS}_2/\text{CC}$ were prepared similarly by adjusting the amount of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ to 0.15, 0.45 and 0.60 mmol, respectively. The CoS_2

nanowire array was also synthesized under the same condition, except for just adding $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$.

2.3. Preparation of RuO_2 and Pt/C loaded electrodes

To a solution of 0.01 mol $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ in 100 mL deionized water at 100 °C, 1 mL KOH (1.0 M) was added and stirred for 45 min. The mixture was centrifuged for 10 min and filtered. The precipitate was washed with deionized water and dried. The dry product was calcined in air at 300 °C for 3 h to afford RuO_2 . 20 mg Pt/C or RuO_2 and 10 μL 5 wt.% Nafion solution were dispersed in 990 μL water/ethanol (V:V = 1:1) by 30 min sonication to form a catalyst ink. Then 48.5 μL catalyst ink was loaded on CC with a loading mass of 0.97 mg cm^{-2} [25].

2.4. Characterization

X-ray diffraction (XRD) patterns were recorded on a RigakuD/MAX 2550 diffractometer with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$). The X-ray photoelectron spectroscopy (XPS) measurements were performed on an ESCALABMK II X-ray photoelectron spectrometer using Mg as the exciting source. Scanning electron microscopy (SEM) images were taken on a XL30 ESEM. Transmission electron microscopy (TEM) measurements were made on a HITACHI H-8100 electron microscopy (Hitachi, Tokyo) with an accelerating voltage of 200 kV. Inductively coupled plasma optical emission spectrometry (ICP-OES) analysis was performed on Thermo Scientific iCAP6300.

2.5. Electrochemical measurements

Electrochemical measurements were performed with a CHI 660D electrochemical analyzer (CH Instruments, Inc., Shanghai) in a standard three-electrode setup at ambient temperature. $\text{Ni}_{2.3\%}\text{-CoS}_2/\text{CC}$ was used as working electrode, a graphite rod as counter electrode and a saturated calomel electrode (SCE) as the reference electrode. In all measurements, the SCE reference electrode was calibrated with respect to reversible hydrogen electrode (RHE). In 1.0 M KOH,

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