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Short Communication

In-situ synthesis of carbon-coated β -NiS nanocrystals for hydrogen evolution reaction in both acidic and alkaline solution

Jianfang Zhang ^a, Yan Wang ^{a,*}, Jiewu Cui ^{a,**}, Jingjie Wu ^b, Xia Shu ^a,
 Cuiping Yu ^a, Huizhong Bai ^a, Mengyuan Zhai ^a, Yongqiang Qin ^a,
 Hongmei Zheng ^a, Yanwu Zhu ^{c,d}, Yong Zhang ^{a,d,e,f}, Yucheng Wu ^{a,d,e,f,***}

^a School of Materials Science and Engineering, Hefei University of Technology, Hefei, 230009, China

^b Department of Chemical and Environmental Engineering, University of Cincinnati, Cincinnati, OH, 45221, United States

^c School of Chemistry and Chemical Engineering, Hefei University of Technology, Hefei, 230009, China

^d Base of Introducing Talents of Discipline to Universities for Advanced Clean Energy Materials and Technology, Hefei, 230009, China

^e China International S&T Cooperation Base for Advanced Energy and Environmental Materials, Hefei, 230009, China

^f Key Laboratory of Advanced Functional Materials and Devices of Anhui Province, Hefei, 230009, China

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ABSTRACT

The electrocatalytic hydrogen evolution reaction (HER) performance of carbon-coated β -NiS (NiS@C) nanocrystals has been investigated for the first time. The NiS@C nanocrystals were synthesized via a facile solvothermal method followed by in-situ carbonization process with the solvent (ethylene glycol) as the carbon source. Benefited from the well-dispersed NiS nanocrystals and the formation of carbon layer on NiS nanocrystals, the NiS@C nanocrystals displayed a high HER activity in 0.5 M H₂SO₄, which needed an overpotential of 85 mV to achieve current density of 10 mA cm⁻¹ with a Tafel slope of 46 mV dec⁻¹, as well as long term stability for HER. Additionally, the NiS@C nanocrystals also showed efficient electrocatalytic activity under alkaline media.

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* Corresponding author.

** Corresponding author.

*** Corresponding author.

E-mail addresses: stone@hfut.edu.cn (Y. Wang), jwcui@hfut.edu.cn (J. Cui), ycwu@hfut.edu.cn (Y. Wu).

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Introduction

Hydrogen is considered as a clean energy sources alternative to the fossil fuel that help resolve the increasingly serious environmental problems [1–3]. Hydrogen production by water electrolysis using suitable electrocatalysts is a promising and effective approach to generate hydrogen from abundant water [4–6]. It is known that the Pt-group metals possess the highest electrocatalytic activity towards hydrogen evolution reaction (HER). However, the scarcity and high cost of Pt limit their practical applications [7,8]. Therefore, the first and most important step is to search highly efficient non-noble metals based HER electrocatalysts with low overpotential and long-term durability.

To date, the first-row transition metals based electrocatalysts have been extensively explored as promising candidates for HER [9–13]. Among them, Ni-based electrocatalysts have been widely studied for water splitting. Especially nickel sulfides (eg. Ni₃S₂, NiS₂, NiS, etc.) have great potential application for HER due to their low cost, earth abundance, and good electron transport ability [14–20]. It has been reported that the catalytic performance of NiS and NiS-based nanostructures display superior catalytic activity toward HER and oxygen evolution reaction (OER) [21–23]. However, few NiS-based catalysts show satisfactory long term stability due to unstable structure in strongly acidic or alkaline media. On the other hand, for the practical application in industry, the HER electrocatalysts should work efficiently under a broad pH range of electrolyte [24–27]. Most of NiS exhibited higher performance in acidic solution than alkaline solution because higher concentration of H⁺ in acidic solution facilitates the HER process [28,29]. Besides, it is more challenging to produce hydrogen in alkaline solution due to it suffers from high overpotential and instability [29,30]. Therefore, it is highly desirable to develop highly active and durable HER electrocatalysts under both acidic and alkaline media.

In this communication, we designed a simple and effective approach to synthesize carbon-coated β-NiS nanocrystals to improve the activity and durability. The β-NiS nanocrystals are synthesized by a facile solvothermal method followed by carbon layer coating on β-NiS nanocrystals (NiS@C) via in-situ carbonization process. In this synthesis process, the ethylene glycol solution not only regulates the size of NiS nanocrystals, but also acts as carbon source for the formation of carbon layer. The presence of carbon layer on the surface of NiS nanocrystals increases the electric conductivity as well as the durability of NiS nanocrystals under both acidic and alkaline media. The synergistic effect of the NiS nanocrystals and carbon layer makes the enhanced electrocatalytic HER performance in acidic solution with an overpotential of only 85 mV to deliver a current density of 10 mA cm⁻². Remarkably, this NiS@C electrode also exhibits efficient electrocatalytic HER performance in alkaline solution. The performance shows no degradation after a long term chronoamperometry test for 20 h.

Experimental section

Synthesis of NiS@C nanocrystals

NiS@C nanocrystals were synthesized via two step reactions where in the first step the NiS nanocrystals was prepared by a

facile solvothermal method, followed by in-situ carbonization process. In a typical synthesis process, 2 mmol Ni(C₂H₄O₂)₂·4H₂O and 5 mmol thiourea were dissolved in 40 ml ethylene glycol-water mixture solution (volume ratio is 3:1). After ultrasonic for 30 min, the resulting solution was sealed in a 50 ml Teflon-lined autoclave and heated to 180 °C for 10 h, then cooled to room temperature naturally. The precipitate was centrifuged and dried in an oven at 70 °C overnight. The as-prepared product was then annealed at 600 °C for 2 h in Ar atmosphere with the heating and cooling rate of 2 °C min⁻¹.

Characterization

The morphologies of as-prepared samples were characterized using an SU8020 field-emission scanning electron microscopy (FESEM, Hitachi, Japan). The structures and crystalline phases were investigated by X-ray powder diffraction (XRD, Rigaku, Japan). High resolution transmission electron microscopy (HRTEM) images were observed using a JEM-2100F (JEOL, Japan) operating at 200 kV. X-ray photoelectron spectroscopy (XPS) spectra were recorded on ESCALAB250 (Thermo, American) using a monochromatic Al K α X-ray beam.

Electrochemical measurements

The electrochemical measurements were carried out using a CHI760E electrochemical workstation with three-electrode cell where a graphite rod and saturated calomel electrode (SCE) were used as counter and reference electrodes, respectively. The working electrode was prepared as follows. 5 mg of catalyst was dissolved in 1 ml of the mixture of NMP and H₂O (volume ratio is 2:1), and then 40 μ l Nafion solution was added in the mixture with ultrasonic treatment to obtain homogeneous solution. 10 μ l of as-prepared solution was placed on a glassy carbon electrode (5 mm) and dried in air. The mass of the catalyst loading on the electrode is about 0.2 mg cm⁻². N₂-saturated 0.5 M H₂SO₄ and 1.0 M KOH were used as electrolytes. All of the potentials with respect to SCE in this work were calibrated with reference to the reversible hydrogen electrode (RHE) by the equation: $E_{(RHE)} = E_{(SCE)} + 0.0591\text{pH} + 0.2415\text{ V}$.

Linear sweep voltammetry (LSV) measurements were conducted at a scan rate of 20 mV s⁻¹. Compensation for iR drop was used for all LSVs. The electrochemical impedance spectra (EIS) was carried out in a frequency range of 100 kHz and 0.1 Hz at an amplitude of 5 mV with overpotential from 100 to 200 mV. For the electrochemical active surface area to be evaluated, cyclic voltammetry was measured in a potential range of 0.1–0.2 V vs. RHE with various scan rates between 10 and 200 mV s⁻¹. The stability tests were carried out by performing CV for 1000 cycles in the potential range of –0.5–0.1 V vs. RHE and the chronoamperometry tests at an overpotential of 150 mV for 20 h.

Results and discussion

The X-ray diffraction (XRD) pattern (Fig. 1a) reveals characteristic diffraction peaks of β-NiS (JCPDS no. 12-0014). After carbon coating by calcination, the diffraction peaks of β-NiS

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