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Short communication

Porous nickel disulfide/reduced graphene oxide nanohybrids with improved electrocatalytic performance for hydrogen evolution

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ABSTRACT

Single porous nickel disulfide (NiS_2) nanoballs and nanohybrids of NiS_2 with reduced graphene oxide (NiS_2/rGO) were successfully prepared by a simple hydrothermal process in the absence or presence of graphene oxide. NiS_2/rGO nanocomposites exhibit remarkable electrocatalytic performance for hydrogen evolution from water splitting due to the plentiful active sites in the porous NiS_2, the improved conductivity and the positive synergetic effect between NiS_2 and rGO. The nanocomposites displayed superior activity for the hydrogen evolution reaction (10 mA cm^{-2} vs. −200 mV, Tafel slope of 52 mV dec^{-1}) and an excellent electrocatalytic stability.

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1. Introduction

The growing energy demands and excessive consumption of non-renewable fossil fuels have already resulted in serious environmental pollution and energy crisis [1]. It is urgent to explore green and sustainable energy resources. As the most attractive molecular fuel, hydrogen is considered as the next-generation alternative energy carrier duo to the inherent advantages such as high energy capacity, sustainable and pollution-free [2]. Among various systems of hydrogen evolution reaction (HER) [3], water electrolysis based on electrocatalysts is most attractive. Numerous inorganic materials have been researched for electrocatalytic HER from water, but none could match both the stability and performance of platinum so far. However, the large-scale application of the noble metals is difficult to realize because of the scarcity and high cost. As one of most economic and competitive strategy, much more earth-abundant materials with high activity have been exploited to replace the precious metal catalysts in the HER application [4]. During the past few years, transition metal dichalcogenides (TMD) based electrocatalysts have attracted tremendous attention for HER including MoS_2 [5], WS_2 [6,7], CoS_2 [8], FeS_2 [9], NiS_2 [10] and CoPS [11]. Unfortunately, the gap of the HER activity of TMDs compared to Pt is obvious, which the essence can attribute to the limited active sites and intrinsic low electrical conductivity.

Porous nanomaterials can provide more active sites and/or rich edge sites to achieve high-efficiency HER by providing a larger surface area compared with the general nanomaterials, which can overcome the intrinsic defect for the limited active sites [12,13]. For instance, Wang et al. reported the trimetallic nanoporous structure PtPdRu with hollow cavity and porous dendritic shell, which shown the competitive activity for oxygen reduction reaction [14]. Jin et al. demonstrated the $NiSe_2$ nanosheets with porous structure through a simple selectively etching method also exhibited excellent catalytic activity for the HER compared to the same nanosheets without porous [15]. The second effective strategy to improve the electrocatalytic activity is decoration of electrocatalyst on graphene or CNTs through the enhancement of the electrical conductivity [16]. A simple one-step solvothermal process was proposed by Rout et al. [17] for fabrication of MoS_2 nanosheets on the surface of graphene. And the synthetic MoS_2/rGO nanohybrids exhibited superior electrocatalytic in the HER (Tafel slope ~ 41 mV dec^{-1}).

Herein, we develop a rapid and simple hydrothermal method to prepare porous NiS_2/rGO nanohybrids. Thanks to the rich active sites from porous structures and the merging of excellent electrical conductivity medium graphene, the as-prepared nanocomposites display competitive electrocatalytic activity and stability for the HER, matching the activity of MoS_2/rGO [15] and WS_2/rGO [6].

2. Experimental

2.1. Preparation of porous NiS_2/rGO nanohybrids

In a typical experiment, 10 mmol of anhydrous $NiCl_2$ (Sigma-Aldrich, 99.98%), 12 mmol of thioacetamide (Shanghai Chemical Factory of China, AR), 15 mL ethylenediamine (Shanghai Chemical Factory of

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China, AR) and 0.02 g poly(vinylpyrrolidone) (PVP, Shanghai Chemical Factory of China, AR) were dissolved in 10 mL of distilled water, respectively. Then 15 mL of 8.0 mg mL^{-1} graphene oxide (exfoliated from natural graphite using a modified Hummers method reported elsewhere [17]) in absolute ethanol was added to the above solution. Under magnetic stirring for 30 min, the mixture solution was transferred to a stainless steel autoclave (50 mL) and maintained at 180 °C for 24 h. After cooling naturally, the precipitate of NiS_2/rGO were washed with distilled water and absolute ethanol three times each and dried in a vacuum oven at 50 °C overnight. Single porous NiS_2 was synthesized with the same method as the NiS_2/rGO without introduction of graphene oxide to the reaction system.

2.2. Sample characterization

The morphologies of the samples were characterized by FE-SEM with EDS (Hitachi S-4800) and TEM (JEOL JEM-2100F). XRD measurements were carried out on a BRUKER D8 Advance X-diffractometer with Cu Kα radiation. Raman spectra of the samples were measured on a Raman microscope (Renishaw inVia). The BET surface area was measured using the nitrogen gas adsorption-desorption method (TriStar II 3020) at 77 K.

2.3. Electrochemical measurements

Electrochemical measurements were collected through a three-electrode system on an electrochemical workstation (Chenhua, CHI660E). All the measurements were carried out in 30 mL of 0.5 M H_2SO_4 electrolyte. 4 mg of NiS_2/rGO powder and 30 μL Nafion solution (Sigma Aldrich, 5 wt%) were dispersed in 2 mL water isopropanol solution with a volume ratio of 3:1 by sonicating for 1 h. Then, 20 μL (60 μg catalyst) of the dispersion was dripped onto a glassy carbon electrode as the working electrode, an Ag/AgCl (in 3 M KCl solution) electrode as the reference electrode and a graphite rod (Alfa Aesar, 99.9995%) as a counter electrode. The electrochemical stability of the catalyst was evaluated (0.10 V ~ −0.4 V vs. RHE) with a scan rate of 50 mV s^{-1}. The Nyquist plots were measured in the same electrochemical workstation with an overpotential of 200 mV.

3. Results and discussion

Fig. 1 displays the morphologies of NiS_2 and NiS_2/rGO through the comparing of SEM and TEM images. Clearly, nanoballs-like morphology NiS_2 with rough surface and plenty of nanopores possesses the uniform grains size (~100 nm) from Fig. 1a and b. After merging graphene oxide as high electrical conductivity medium, the observed folds and wrinkles over the entire spherical surface are identified as graphene from graphene oxide reduction process from Fig. 1c and d, indicating the conformal wrapping and bridging of NiS_2 spheres by graphene matrix. Meanwhile, it has also been demonstrated that the graphene oxide has no influence on the nucleate and grow stages of NiS_2 nanoball due to the maintaining of the average diameter and pore size of the NiS_2 nanospheres in the nanocomposites. The HRTEM images (the inset of Fig. 1b and d) indicates that the lattice spacing of the samples are ~0.28 nm which are well corresponding to the (200) plane of NiS_2 [18]. This observed nanoporous structures from Fig. 1 might be explained as follows. The formation of the NiS_2 nanoporous structures can be attributed to the loss of the organic component at high temperatures ($NiS(en)_{0.5} \rightarrow NiS_2$), which similar to the reported literature regarding the formation of porous ZnS nanostructures [19].

Fig. 2 shows the XRD patterns for NiS_2 and NiS_2/rGO samples. Compared with the standard diagram (JCPDS No. 65-3325), the NiS_2 curve reveals an excellent agreement with the main peaks which center around 2θ values of 27.16 (111), 31.47 (200), 35.30 (210), 38.81

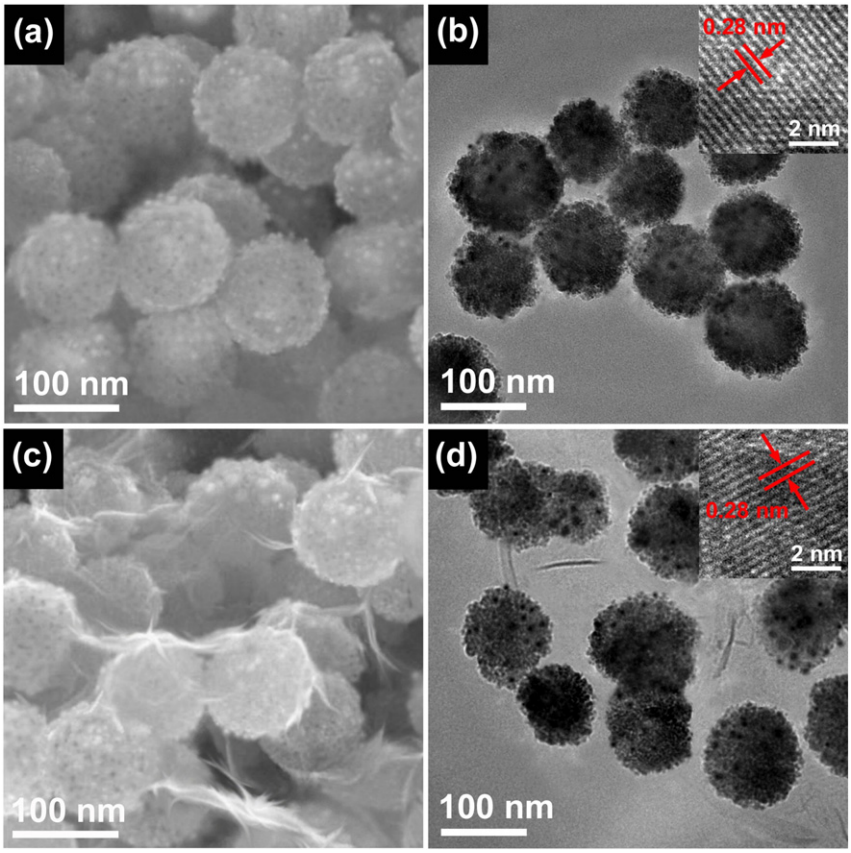

Fig. 1. FE-SEM images of (a) NiS_2, (b) NiS_2/rGO. TEM images of the as-synthesized (c) NiS_2 and (d) NiS_2/rGO and the corresponding HR-TEM images (insets).

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