Facile Preparation of 1st-Row Transition Metal Chalcogenides as Hydrogen Evolution Catalysts in Water

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I. Introduction

The combination of rising energy demands, anthropogenic climate change, and diminishing fossil fuels have dictated the search for carbon-neutral and sustainable energy sources one of the most urgent tasks in the scientific community. Among various renewable energy sources, solar energy is a promising target due to its gigantic amount. However, its nature of diurnal variation, intermittence, and unequal distribution requires efficient capture, conversion, and storage. Thus, solar-drive water splitting to produce hydrogen and oxygen has been widely regarded as an attractive approach where hydrogen acts as the energy carrier. Nature evolves hydrogenases enzymes to catalyze the conversion between proton and hydrogen in neutral aqueous media. Three types of hydrogenase cofactors, binuclear iron/iron, iron/nickel, and mononuclear iron, have been identified. Inspired by the active sites of hydrogenases, herein we describe the facile preparation of nickel-sulfide films as competent and robust hydrogen evolution catalysts in neutral aqueous media.

II. Methods

With the use of fluorine-doped tin oxide (FTO) as both the working and auxiliary electrodes and a Ag/AgCl reference electrode, a nickel-sulfide film can be formed on FTO from nickel nitrate and thiourea precursors via a process of potentiodynamic deposition. After rinsing thoroughly with water and acetone, the dried nickel-sulfide film was annealed under nitrogen for three hours. The morphology and valence states of the prepared nickel-sulfide film were characterized via scanning electron microscopy (SEM) and X-ray photoelectron spectroscopy (XPS). The proton reduction performance was evaluated in pH 7 phosphate buffer via various electrochemical techniques, including polarization, Tafel plot, Nyquist plot, and controlled potential electrolysis.

III. Results

Figure 1A displays the SEM images of annealed nickel-sulfide film on FTO, implying its amorphous nature. XPS data summarized in Figure 1B confirms the valence states of +2 and -2 for nickel and sulfur, respectively. It demonstrates that the major composition of the nickel-sulfide film is NiS. As depicted in Figure 1C, negligible current response was observed for blank FTO working electrode. However, once the nickel-sulfide film was deposited on FTO, a sharp rise in current was apparent when the potential was scanned more negative than -0.8 V vs Ag/AgCl. The resistance of the catalytic film was measured via electric impedance spectroscopy whose results are included in Figure 1E. The robustness of the nickel-sulfide film was confirmed by its controlled potential electrolysis (Figure 2).

IV. Conclusions

A facile preparation method of electrodynamic deposition allows us to generate nickel-sulfide films on FTO electrodes. Various characterization techniques were utilized to confirm its amorphous nature and major composition of NiS. This film exhibits competent and robust catalytic performance for the hydrogen evolution reaction, representing a promising catalyst candidate for electrocatalytic water splitting.

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