Contents lists available at ScienceDirect

Electrochemistry Communications

journal homepage: www.elsevier.com/locate/elecom

Short communication

Facile synthesis of thin NiFe-layered double hydroxides nanosheets efficient for oxygen evolution

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ARTICLE INFO

ABSTRACT

Article history: Received 13 October 2015 Received in revised form 5 November 2015 Accepted 10 November 2015 Available online 14 November 2015

Keywords: Layered double hydroxides Nanosheets Oxygen evolution reaction Activity Stability

Ultrathin NiFe-layered double hydroxide (LDH) nanosheets are facilely and cost-effectively fabricated. The as-prepared NiFe-LDH nanosheets are highly active and stable electrocatalysts for water oxidation in alkaline environment, exhibiting a much higher oxygen evolution activity and lower overpotential with the enhanced stability, much superior to the previously reported LDH-derived catalysts. This work provides a facile methodology for the fabrication of thin nanosheets, which are promising in the renewable energy conversion.

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1. Introduction

Electrochemical water splitting offers one of the most attractive approaches in the realization of efficient energy conversion and storage [1–3]. In oxygen evolution reaction (OER), the overall efficiency of the complicated OER is severely influenced by the energy loss and large overpotential. To date, OER catalysts based on precious metal oxides RuO₂ or IrO₂ are most effective, but the high price and scarcity make them impractical to use on a large scale. Besides, it has been reported that the RuO₂ and IrO₂ catalysts display poor stability in alkaline solution [4]. Toward this end, it is crucial to develop highly active and cost-effective OER catalysts based on earth-abundant metals. Cophosphate and Ni-borate catalysts have been reported to display good performance for OER in neutral and weakly basic medium [5,6], where it required ~400 mV overpotential to reach 10 mA cm⁻². Perovskite Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O₃ catalyst is also efficient for OER in alkaline medium, which are even more active than IrO_2 and RuO_2 [7,8]. Recently, the low-cost NiFe-derived layered material, spinel and oxides were reported to display promising performances of OER in alkaline solutions [9,10]. It has been proposed that layered metal hydroxides are the active centers [11,12].

Corresponding author. E-mail address: kai_yan@brown.edu (K. Yan). to stack and lack good control over the hierarchical nanostructures. Herein, we have developed a facile, and cost-effective route to synthesize ultrathin NiFe-LDH nanosheets in the mixed solvent of ethanol and isopropanol. The obtained nanosheets are highly active and stable catalysts for OER in alkaline conditions, where they exhibit the enhanced oxygen evolution activity in comparison with other LDHderived electrocatalytic materials reported to date. The fabricated thin nanosheets display more active sites and improve the electronic

The skeleton of layered double hydroxides (LDHs) was composed of a regular assembly of interlayer anions and positively charged metallic-

hydroxide layers in which the main layers are formed by MO₆ octahedra

that share edges [13,14]. Due to this unique structural property, LDHs

exhibit promising nature as high-performance OER catalysts. However,

LDHs still suffers from poor electronic conductivity and electron trans-

portation. To reduce these drawbacks, the conductivity and activity of

LDHs has been further enhanced by coupling to a carbon nanotube,

graphene-like network or by exfoliation [14–18]. Among them, exfoliation method appears more attractive due to the relatively easy synthesis

and good performances observed in OER. The resulted LDHs by exfolia-

tion methods often display thin nanosheets with better conductivity

and large space between the layers. However, the synthesis still requires

multiple steps of LDH precursor synthesis, anion-exchange, exfoliation in

high boiling point solvents (e.g., formamide or N-methylpyrrolidone) and

final removal of these high boiling points solvent. The removal of high

boiling point solvents often associates with the utilization of excessive

ester and low yield of LDHs. Besides, the resulted LDHs plates often tend











Fig. 1. Schematic procedure of the facile and cost-effective synthesis NiFe-LDH nanaosheets.

conductivity. This work presents a novel strategy to design low-cost and high-performances OER electrocatalysts that are attractive for the application in transformation of renewable energy sources.

2. Experimental

2.1. LDH nanosheet synthesis

Thin LDH nanosheets were synthesized in two steps of coprecipitation and delamination. In the first step, LDH precursor was synthesized by a classic co-precipitation method at room temperature as our previous report [19]. The second step of delamination was performed, where 100 mg LDHs were dispersed into the 200 mL mixed solvent of ethanol and isopropanol with the volume ratio of 1 at 60 °C under the stirring condition of 1000 rpm for 12 h. The resulting suspension was centrifugated at 5000 rpm for 5 min and dried at 80 $^\circ\mathrm{C}$ under vacuum condition.

2.2. Characterizations

The powder X-ray diffraction (XRD) patterns for qualitative phase analysis were collected on a Phillips PW 1050-3710 transmission diffract meter with Cu K α radiation ($\lambda = 1.5406$ Å). The data were collected in the range of 8–65° with a step width of 0.05/20. Scanning electron microscopy (SEM) analysis was performed on FEI Helios—FIB operating at an acceleration voltage of 10 kV. TEM analysis was investigated on Philips CM 20 instrument at 120 kV. HR-TEM was performed on JEOL-2010 at 200 kV to investigate their structural features. Nitrogen adsorption–desorption was measured using a Micromeritics ASAP 2020 sorption analyzer at 77 K, and the samples were activated under a vacuum of 0.01 mbar at



Fig. 2. XRD patterns (a) of the NiFe-LDHs: before (i) and after exfoliation (ii); XPS analysis of the chemical state of Ni (b) and Fe (c); (d) N₂ adsorption-desorption isotherm curve and pore size distribution (Inset) of the fabricated NiFe-LDH.

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