



In-situ confined formation of NiFe layered double hydroxide quantum dots in expanded graphite for active electrocatalytic oxygen evolution



Jinxue Guo^a, Xiaoyan Li^a, Yanfang Sun^b, Qingyun Liu^c, Zhenlan Quan^a, Xiao Zhang^{a,*}

^a College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, China

^b College of Science and Technology, Agricultural University of Hebei, Cangzhou 061100, China

^c College of Chemistry and Environmental Engineering, Shandong University of Science and Technology, Qingdao 266590, China

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ABSTRACT

Development of noble-metal-free catalysts towards highly efficient electrochemical oxygen evolution reaction (OER) is critical but challenging in the renewable energy area. Herein, we firstly embed NiFe LDHs quantum dots (QDs) into expanded graphite (NiFe LDHs/EG) via in-situ confined formation process. The interlayer spacing of EG layers acts as nanoreactors for spatially confined formation of NiFe LDHs QDs. The QDs supply huge catalytic sites for OER. The in-situ decoration endows the strong affinity between QDs with EG, thus inducing fast charge transfer. Based on the aforementioned benefits, the designed catalyst exhibits outstanding OER properties, in terms of small overpotential (220 mV required to generate 10 mA cm⁻²), low Tafel slope, and good durable stability, making it a promising candidate for inexpensive OER catalyst.

1. Introduction

Electrochemical oxygen evolution reaction (OER) provides a clean and competitive solution for energy conversion and storage via electrolysis of water, which is also key enabler in other renewable energy conversion devices of rechargeable metal-air batteries and fuel cells [1,2]. Unfortunately, OER usually suffers from high overpotential and low efficiency due to its sluggish kinetics [3]. OER requires noble metal based catalysts (IrO₂/RuO₂) to achieve low overpotential and highly efficient output [4]. However, the scarcity and high cost of these noble metals cannot afford the widespread applications. Extensive efforts must be made to explore economically acceptable and sufficiently active catalysts.

Layered double hydroxides (LDHs) have triggered considerable research interests as low cost and highly efficient alternatives towards OER, due to their specific two-dimensional lamellar structure and diversity [5,6]. However, the OER performances of LDHs are hindered by their poor electrical conductivity and limited number of active sites. To realize improved OER activity, various carbon materials have been employed to combine with LDHs for enhanced electrical conductivity, such as CNTs [7,8], carbon quantum dots [9], and graphene [10,11]. On the other hand, great efforts have been paid on obtaining more active sites [5,6,10]. It is well known that, decreasing the size of catalysts could obtain more exposed catalytic sites [12]. Different methods have been used to synthesize various functional inorganic

nanoparticles towards their applications [13–23]. Tang and co-workers have anchored nanometer-sized NiFe LDHs on nitrogen-doped graphene framework via in-situ formation for pursuing more active sites and enhanced catalytic activity [10]. Notwithstanding these efforts, however, the precise control of nanosized NiFe LDHs hybridization with conductive substrate to realize increased active sites and enhanced electrical conductivity is still badly needed on the way for exploring advanced OER electrocatalysts.

The 2D geometry of graphite is visibly well-matched with the layered structure of LDHs and there is also a charge compatibility between the positively charged LDH and negatively charged graphite [24]. The large distance between expanded graphite sheets compared with LDH platelets implies that it may be possible to form an open network allowing access of the reactants to the active LDH sites which is helpful for the high rate conversion of the reactants [25].

Herein, NiFe LDHs quantum dots (QDs) embedded in expanded graphite (NiFe LDHs/EG) has been prepared as novel electrocatalyst for oxygen evolution. The synergism between Ni and Fe assures high catalytic activity of composite catalyst. The EG improves the electrical conductivity. The interlayer spacing of EG layers serves as nanoreactors for spatially confined formation of NiFe LDHs QDs. The small size of QDs endows abundant active sites, and the in-situ formation process assures the strong affinity and fast charge exchange between QDs with EG. Therefore, the designed NiFe LDHs/EG possesses combined advantages of huge active sites and fast electron transfer, which

* Corresponding author.

E-mail address: zhx1213@126.com (X. Zhang).

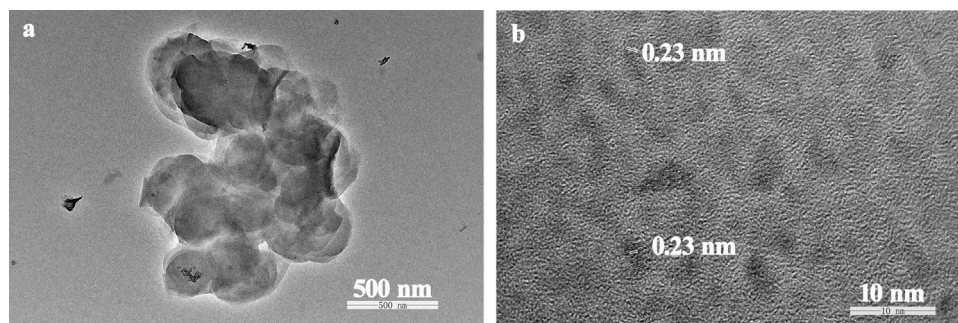


Fig. 1. (a) TEM and (b) HRTEM images of NiFe LDHs/EG.

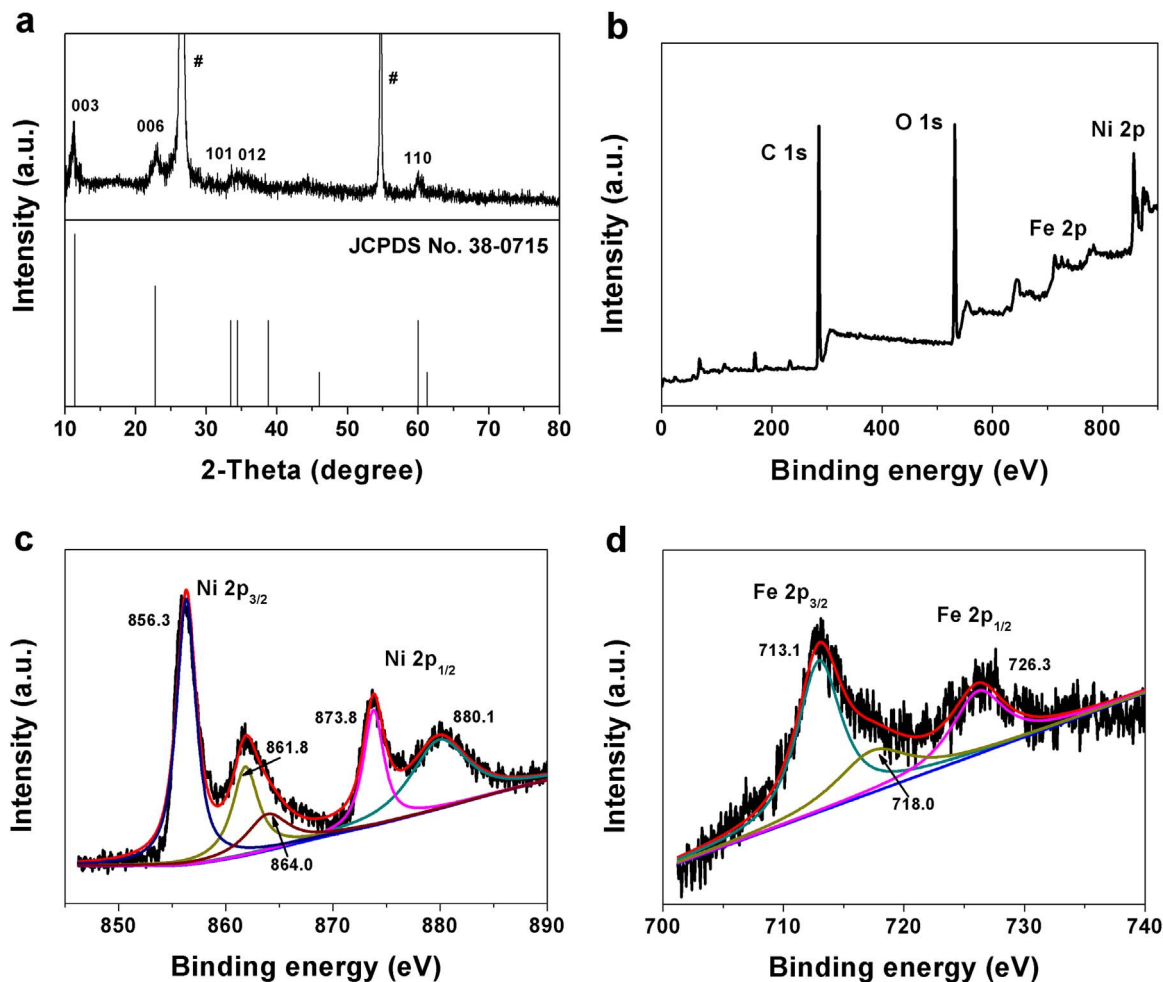


Fig. 2. (a) XRD pattern and (b) survey XPS spectrum of NiFe LDHs/EG. High resolution XPS spectra of (c) Ni 2p and (d) Fe 2p of NiFe LDHs/EG.

exhibits ultralow overpotential of 220 mV at a current density of 10 mA cm^{-2} for OER. The present sample also delivers relatively good catalytic durability, which needs further improved.

2. Experimental

All chemicals and materials are purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai city, China) and used without further treatment. Natural graphite powder (1 g) is dispersed in 3 g of concentrated H_2SO_4 (75%), which is then added with 0.5 g of KMnO_4 . The mixture is stirred for 1 h at 60°C . The precipitate is washed with water till to neutral. The obtained powder is heated in a tube furnace at 800°C for 2 min under Ar with a heating rate of 2°C every minute to prepare EG. To obtain NiFe LDHs/EG, EG (0.15 g), $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

(1 mM), $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (1 mM), sodium citrate (0.2 mM), and 0.5 g of sodium lauryl sulfate (SDS) are dispersed in 100 mL of water for ultrasonic of 1 h. The precipitation is washed with water to remove the metal ions adsorbed on the surface of EG, which is then redispersed in 100 mL of water which contains 3.5 mM urea. The dispersion is transferred into 150 mL of autoclave for heating treatment at 160°C for 48 h. After naturally cooled to room temperature, the final product of NiFe LDHs/EG is collected by centrifugation, washed with water and ethanol, and dried at 60°C for 24 h. Pristine NiFe LDHs is obtained via the similar hydrothermal method at 160°C for 48 h under autogenous pressure without the addition of EG.

NiFe LDHs/EG is characterized with scanning electron microscope (SEM) using a JEOL JSM-7500F, transmission electron microscope (TEM) on a FEI Tecnai G2 F30, X-ray photoelectron spectrum (XPS)

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