Steel Mesh Reinforced Ni(OH)₂ Nanosheets with Enhanced Oxygen Evolution Reaction Performance

Jin Liang,* Haiqi Shen and Jie Kong

Abstract

Water splitting is one of the most promising technology to solve the current energy and environmental issues. The high overpotential and sluggish kinetics of the oxygen evolution reaction (OER) seriously hinders the energy conversion efficiency of the full water splitting. Therefore, it is of great significance to develop high-active and stable OER catalytic materials for improving the energy transfer efficiency and durability. In this work, we prepared the Ni(OH)₂/stainless steel wire mesh (SSWM) self-supporting electrode by a simple one-step method. This in-situ growth method can accurate controllability for improving activity of the catalysts, efficaciously inhibit the self-aggregation of Ni(OH)₂, and the obtained two-dimensional array of oriented nanosheets can quickly promote the acquisition of electrolytes and gas release. The integrated Ni(OH)₂/SSWM electrodes possess prominent conductivity, abundant porous structure and high specific surface area. As OER catalysts, this obtained electrodes exhibit the relatively low overpotential of 223 mV and low Tafel slope of 25.2 mV dec⁻¹ at 10 mA cm⁻². Moreover, the integrated Ni(OH)₂/SSWM electrodes possess stable long-term performance (60 hours). Therefore, this research provides a novel strategy for large-scale production of highly active integrated OER electrodes.

Keywords: Ni(OH)₂/SSWM; Self-Supporting; Oxygen Reduction Reaction; Catalysis.

Received date: 7 February 2021; Accepted date: 15 April 2021.

Article type: Research article.

1. Introduction

Water splitting could large-scale product highly purified hydrogen which is one of the most important means to relieve energy crisis and environmental pollution problem.^[1] Hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) as the two pivotal reactions, which decide the rate and efficiency of hydrogen production by water splitting. Compared with HER, OER involving four-electron reactions possess the lower reaction rate and higher reaction energy barrier. So, high-efficiency and stable OER catalysts can be the key factor to accelerate reaction rate and improve energy conversion efficiency.^[2] Currently, precious metal catalysts (such as Ir, Ru and Pd) have been demonstrated that they can prove the reaction rates and low the overpotentials of OER. But the high price of these metals limits the large-scale applications of water splitting.^[3] Therefore, the catalysts with abundant storage, high active and stable performance are urgent to be explored.

Transitional metal-based materials have been considered as the promising candidates for the ideal OER catalysts.^[4-5] Nickel-based material, as one member of traditional metal family, has been widely studied as OER catalysts for their high efficiency, stability and relatively low-price.^[6-10] such as, Ni-Fe hydroxide,^[11] Ni-Co hydroxide,^[12] Ni-Te and Ni(OH)₂/nickel foam.^[13,14] But for most of the current relative materials, there are three main issues needed to be solved. First, the most semi-conducting transitional metal-based materials are poor-conductivity and adverse to electron transport during the OER reaction. Second, transitional metal-based catalysts are easy to be self-aggregation, which tent to cover each other, resulting to reducing the active sites of the catalysts. Third, the polymer binders need to be added for pasting active materials on the conductive metal in the traditional OER test. The added binders can weaken the conductivity of the electrode and effect the activity and stability of the OER catalysts.^[5]

In this study, a simple and inexpensive synthesis method was used to prepare a self-supported integrated electrode (Fig. 1). Ni(OH)₂ nanosheets were in-situ grown on the surface of stainless-steel mesh substrate (SSWM) by one-step hydrothermal method. The SSWM substrate contributed several advantages. First, SSWM could effectively inhibit the self-aggregation of Ni(OH)₂, guaranteeing the adequate

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Fig. 1 Schematic illustration: the preparation method of $\text{Ni}(\text{OH})_2/\text{SSWM}$.

exposed active sites of catalysts. Second, SSWS can be considered as an adherent for crystal growth, and the uniform stencil surface orientation is in favor of oriented ultra-thin 2D nanosheet arrays. Third, the conductive substrate could strengthen the conductivity of the whole electrodes, accelerating reaction rate and improving energy conversion efficiency. Moreover, this in-situ growth promotes rapid access to electrolytes and rapid release of gases, resulting in effective utilization of active sites. In OER catalytic applications, the overpotential of the $\text{Ni}(\text{OH})_2/\text{SSWS}$ was 223 mV at 10 mA cm^{-2} . These self-supported electrodes exhibited not only excellent electrocatalytic activity but also outstanding stability (60 hours).

2 Experiment

2.1 Material preparation

$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, sodium citrate (SCT) were bought from Beijing Yinuokai Technology Co., Ltd. Hexamethylenetetramine (HMT) was purchased from Guangdong Guanghua Technology Co., Ltd. The agents purchased above are directly used without post-processing. SSWM ($1 \times 2 \text{ cm}^2$) was successively treated by acetone, ethanol and deionized water to remove the oxide layer.

2.1.1 Synthesis of $\text{Ni}(\text{OH})_2/\text{SSWM}$

In a typical $\text{Ni}(\text{OH})_2/\text{SSWM}$ preparation process, one piece of SSWM was added to the solution containing 0.073g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.25 mmol), 0.035g of hexamethylenetetramine (0.25 mmol HMT), 0.074g of trisodium citric (0.25 mmol SCT) and H_2O (40 mL). Then, the upper solution was transferred to a round bottom flask (100 mL) and heated in oil bath at 90 °C for 12 hours. After cooling down to room temperature, the production was rinsed with deionized water/ethanol and dried at room temperature. At last, $\text{Ni}(\text{OH})_2/\text{SSWM}$ could be successfully obtained.

2.1.2 Synthesis of NiO/SSWM

The obtained $\text{Ni}(\text{OH})_2/\text{SSWM}$ composites were placed in the tube furnace and annealed at 350 °C with a rate of $5 \text{ }^\circ\text{C min}^{-1}$ under air. After 3 h, the system naturally cooled down to room temperature, NiO/SSWM can be harvested.

2.1.3 Synthesis of $\text{Ni}(\text{OH})_2$ Powder

$\text{Ni}(\text{OH})_2$ powder were prepared by the same method of $\text{Ni}(\text{OH})_2/\text{SSWM}$ without SSWM.

2.1.4 Synthesis of $\text{Ni}(\text{OH})_2\text{-SSWM}$

10 mg of $\text{Ni}(\text{OH})_2$ powder was weighed and added to 50 μL electrolyte solution and 1 ml ethanol. After 30 minutes of sonication, a uniformly dispersed solution was obtained. Take 48 ml of the solution and evenly coat it on the SSWM. After natural drying, $\text{Ni}(\text{OH})_2\text{-SSWM}$ electrode can be obtained.

2.2 Characterizations

The morphology and structure information of the materials prepared was characterized by scanning electron microscope (SEM, Verios G4), transmission electron microscope (TEM) and selected area electron diffraction (SAED) images (JEOL JEM-2100). X-ray powder diffraction (XRD) was applied to measure crystal structures and composition information of the obtained sample (Rigaku Mini Flex 600). X-ray photoelectron spectroscopy (XPS) measurements were performed on the test equipment (Kratos).

2.3 Electrochemical Measurements

The electrochemical tests were operated on the CHI660E electrochemical workstation (Shanghai Chenhua) applying a three-electrode electrochemical cell. All experiments were performed at room temperature in 1 M KOH aqueous solution. For the OER performance testing, SSWM-based electrodes, Ag/AgCl and Pt electrodes were employed as working electrodes, reference electrodes, and counter electrode respectively. All the overpotential (η) relative to the reversible hydrogen electrode (RHE), can be recorded and calculated by the equation: $E(\text{RHE}) = E(\text{Ag} / \text{AgCl}) + 0.197 + 0.059 \text{ pH}$. Linear sweep voltammetry (LSV) curves were performed at a scan rate of 5 mV s^{-1} . The Cyclic Voltammograms (CVs) test is performed under a voltage window of -0.05 V - 0.05 V at the different scan rates. The electrochemical surface area (ESCA) was determined from the double-layer capacitance (Cdl) of the materials by a simple CV method. The electrochemical impedance spectroscopy (EIS) of the samples was measured at 1.507 V (vs RHE) over a frequency range of 0.1-100000 Hz in 10 mV.

Fig. 2 SEM images of SSWM substrates (a and b) and Ni(OH)₂/SSWM (c and d).

3. Results and Discussion

The morphology and surface structure of the samples can be analyzed by scanning electron microscope (SEM), transmission electron microscope (TEM). As shown in Fig. 2a and b, the surface of the steel mesh substrate was smooth and flat without other impurities. After the one-step reaction, it can be clearly observed (Figs. 2c and d) that the SSWM became rather rough and unsmooth. For the magnification SEM images (Fig. 3a), two-dimensional Ni(OH)₂ nanosheets tightly

grew on the surface of the three-dimensional steel mesh substrate. From partially further enlarging image of the Ni(OH)₂/SSWM (Fig. 3b), Ni(OH)₂ nanosheets closely fitted the steel mesh substrate, and those ultra-thin Ni(OH)₂ with were evenly and vertically arranged on the substrate, which further formed interpenetrating network architecture benefiting to facilitate electron conduction. The SSWM substrates can effectively relieve the aggregation of Ni(OH)₂ nanosheets. In addition, this urchin-thin nanosheet structure

Fig. 3 SEM images of Ni(OH)₂/SSWM (a and b); TEM images of Ni(OH)₂/SSWM (c and d) and SAED (e).

Fig. 4 XRD pattern (a); XPS spectra of Ni(OH)₂/SSWM: full scan spectrum (b), Ni 2p spectra (c) and O 1s spectra (d).

can increase the electrochemical area and provide more exposed active sites. Also, the rough surface may reduce the interaction between solids and gases, releasing the air bubbles adhered to the electrode surface. From the TEM images (Figs. 3 c and d), it can be observed that the thickness of the Ni(OH)₂ nanosheet is about 6 nanometers, which further demonstrated the ultra-flaky structure formed on the substrate surface. The typical selected area electron diffraction (Fig. 3e) showed the obtained nanosheets possess several diffraction rings corresponding to the (003), (006), (010) crystal plane of Ni(OH)₂ respectively.

X-ray diffraction (XRD) was used to characterize the crystalline structure of the samples. Fig. 4a shows the XRD pattern of the Ni(OH)₂, in which peaks at distinct diffraction peaks at 11.3°, 22.7°, 33.4°, and 59.9° corresponding to (003), (006), (010), (110) crystal planes. The tested results can be assigned to the Ni(OH)₂ (JCPDS No: 38-0715).^[16] To better understand the chemical state of the catalysts, XPS was applied to deeply analyze the relative information of the obtained materials. As displayed in Fig. 4b, C, O, and Ni peaks can be clearly observed in the full scan spectrum, which indicates that the sample contains those three elements. In the XPS spectrum of Ni 2p (Fig. 4c), the peaks at 856.3 eV and 879.6 eV can be belong to the Ni 2p_{3/2} and Ni 2p_{1/2} orbits, and the self-selected energy interval between the two peaks is 17.7 eV. In addition, there are satellite peaks

of Ni at 879.6 eV and 862.0 eV, which are matched with the characteristics of Ni(OH)₂ in the literature, which further proves that the product on the steel mesh substrate is Ni(OH)₂.^[12] In the characterization of the XPS spectrum of O 1s (Fig. 4d), peaks can be obviously observed at 530.9 eV,

which is mainly resulted from oxygen in Ni(OH)₂. In addition, certain peak intensities were also observed at 531.7 eV and 532.6 eV. The former may be caused by the hydrogen bond interaction between hydroxyl groups in Ni(OH)₂,^[11] and the latter may be resulted from NO₃⁻ ions intercalated between Ni(OH)₂ lattice layers.^[15,17]

The evaluation of OER performance was performed in a 1 M KOH solution in a three-electrode system. Self-supporting integrated Ni(OH)₂/SSWM was measured as the working electrode, while Ag/AgCl electrode and Pt sheet (1×2 cm²) were applied as reference electrode and counter electrode, respectively. In order to make corresponding comparisons, the corresponding properties of Ni(OH)₂-SSWM, NiO/SSWM, and substrate SSWM were characterized at the same conditions. Fig. 5a shows the polarization curves of different prepared catalysts at 5 mV s⁻¹ and their corresponding overpotential values at 10 mA cm⁻² displayed in Fig. 5b. The Ni(OH)₂/SSWM exhibited only 223 mV to achieve the current density of 10 mA cm⁻², which is the lowest overpotential among the NiO/SSWM (309 mV), Ni(OH)₂-SSWM (414 mV), and SSWM (329 mV). And electrocatalytic kinetics of the obtained electrodes were further verified by the Tafel slope. As calculated in Figs. 5c and d, the Tafel slopes were 54.5 mV dec⁻¹, 41.6 mV dec⁻¹, 35.6 mV dec⁻¹, corresponding to NiO/SSWM, Ni(OH)₂-SSWM and SSWM, respectively. However, the Tafel slope of Ni(OH)₂/SSWM was only 25.2 mV dec⁻¹, showing excellent catalytic activity. The main reason for this phenomenon is firstly attributed to a stencil substrate with a three-dimensional network structure and good conductivity. The conductive substrate provides an excellent path for the transfer and transmission of charge. Secondly, the

Fig. 5 OER performance of the obtained for catalysts. Polarization curve (a) of the electrode at a scanning speed of 5 mV s⁻¹, and the corresponding value of overpotentials (b); Tafel slope (c) and histogram (d). Plot capacitor current density versus different scan rates (e) and EIS curves (f).

ultra-thin nanosheet structure of Ni(OH)₂ can provide a larger active surface area, which can provide a very short diffusion path for ions benefiting to ion transmission. To verify the second reason, the electrochemically active surface areas (ECSA) were obtained by a cyclic voltammetry curve (Fig. 6). By plotting the relationship between the scanning rate and the current, and this value is proportional to the ECSA, which can reflect the size of the electrochemically active surface area to a certain extent.^[26] The Cdl value (Fig. 5e) of Ni(OH)₂/SSWM was 0.249 mF cm⁻², which was significantly higher than

NiO/SSWM (0.137 mF cm⁻²), Ni(OH)₂-SSWM (0.207 mF cm⁻²) and SSWM (0.231 mF cm⁻²), further validating the superiority of the combination of Ni(OH)₂ and stencil substrate.

EIS curves is an important and common characterization method to analyze the dynamic process of the electrochemical reaction.^[20,30] The smaller diameter of the semicircle of the low-frequency curve, the faster charge transfer in the electrode, that is, the lower the charge transfer resistance (R_{ct}).^[21,31] Intermediate are easier to form, which increases the kinetic

Fig. 6 CV curves of different samples: Ni(OH)₂/SSWM (a), NiO/SSWM (b), Ni(OH)₂-SSWM (c), and SSWM (d).

reaction rate to a certain extent, so the catalytic activity is also higher.^[22] The EIS curves of obtained samples were shown in Fig. 5f. It can be clearly observed from the spectrum that the curve radius had a large difference. In the low frequency region, the radius of the other samples is obviously higher than

that of Ni(OH)₂/SSWM, so it can be judged that the catalytic activity of Ni(OH)₂/SSWM is higher. The catalytic stability of the self-supported Ni(OH)₂/SSWM electrode was tested in 1.0 M KOH solution using chronopotentiometry (Fig. 7a). After more than 60 hours of testing, the overpotential of

Fig. 7 The Chronopotentiometry lines of Ni(OH)₂/SSWM for OER at 10 mA cm⁻² (a) and different current densities.

Ni(OH)₂/SSWM had only a slight change of 18.8 mV, illustrating the excellent stability of this electrode. Besides, the Ni(OH)₂/SSWM electrodes had further been measured at different current densities ($j = 10, 20, 30, 40$ and 50 mA cm^{-2}) and the results were shown in Fig. 7b, showing excellent stability. In addition, the morphology of the cycled Ni(OH)₂/SSWM was re-tested by SEM. Stable structure is beneficial to good cycle stability. Obviously, the morphology of the material was almost no change (Fig. 8), which is also the crucial factor for the excellent OER performance.

Fig. 8 SEM images of Ni(OH)₂/SSWM after testing.

4. Conclusion

In summary, we prepared Ni(OH)₂/SSWM self-supporting electrodes by a facile one-step method. The SSWM could effectively inhibit the self-aggregation of Ni(OH)₂, guaranteeing the adequate exposed active sites of catalysts. This substrate also can be considered as an adherent for crystal growth, and the uniform stencil surface orientation are in favor of oriented ultra-thin 2D nanosheet arrays. Meanwhile, the conductive substrate could strength the conductivity of the whole electrodes, accelerating reaction rate and improving energy conversion efficiency. More importantly, this in-situ growth strategy promoted rapid access to electrolytes and rapid release of gases, resulting in effective utilization of active sites. As the OER catalysts, Ni(OH)₂/SSWM delivered a remarkable performance.

Acknowledgements

The financial support from the Fundamental Research Funds for the Central Universities (No. 31020180QD128) is gratefully acknowledged. Shaanxi Association of Science and Technology (5111190012), Shaanxi Provincial Key R&D Program (2020KW-023), China Postdoctoral Science Foundation (2020M673474) and China Postdoctoral Science Foundation (2019TQ0262).

Supporting Information

Not Applicable.

Conflict of Interest

There is no conflict of interest.

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