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Short Communication

Mass production of Nickel@Carbon nanoparticles attached on single-walled carbon nanotube networks as highly efficient water splitting electrocatalyst

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ABSTRACT

Herein, we develop a direct current arc discharge method which enables large-scale synthesis of nickel@carbon attached single-walled carbon nanotube networks as an electrocatalyst for highly efficient water splitting. Mass amount of Ni@C/SCN (~80 g) could be easily obtained. After optimization, the catalyst exhibits a superior performance of electrochemical water splitting, which allows a current density of 10 mA cm⁻², with an overpotential of only 260 mV for OER and 198 mV for HER. The electrolyzer can achieve a current density of 10 mA cm⁻² at 1.8 V.

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Introduction

Hydrogen and oxygen production from water splitting actuated by the demand for renewable sources of energy is a dramatic eco-friendly pathway for renewable energy storage [1,2]. Currently, precious metals are the most commonly used electrocatalysts for efficient water splitting (e.g., Platinum (Pt)

for HER, Ruthenium dioxide (RuO₂) or Iridium oxide (IrO₂) for OER), however, high cost and low earth abundance greatly limit their widespread applications [3–6]. Demanded by these challenges, lots of efforts have been devoted to developing cost-efficient alternative catalysts including sulfides, selenides, phosphides, and many other nonprecious metal compounds [7–26]. Among them, metal nanoparticles encapsulated in graphite carbon layers (M@C) such as Fe@C,

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Co@C and Ni@C have attracted intense research interests due to their high activity and low cost [14,15,27–32]. Nevertheless, the synthetic methods of these Metal@Carbon materials generally include complicated synthesis and purification steps [33–38], which is not only very tedious and time consuming, but also not applicable for large scale production (Table S1). Therefore, the mass production of efficient noble metal free electrocatalysts that can be potentially used for future industrial application remains a challenge. Previously, direct current (DC) arc method is widely used for mass-producing carbonaceous nanomaterials, including fullerene, carbon nanotubes and graphene [39–41]. This inspires us to extend this method for mass production of Metal@Carbon materials, which can act as a highly efficient electrocatalyst for water splitting.

Results and discussion

In a typical procedure, the anode was a graphite rod in which a hole was drilled and filled with a powder mixture of Yttrium Nickel alloy and graphite powders, then, an arc was created between two electrodes by a current of 80 ampere (Figure S1 for a schematic view). After consuming the anode rod, the raw product was collected. After simple treatment with hydrogen peroxide and hydrochloric acid, the sample was calcined at an atmosphere of ammonia. The final product was collected. The detailed steps could be obtained from the experimental section in the supporting information.

Here, mass amount of raw product (~80 g) could be easily obtained (Fig. 1a). The raw product was first characterized by transmission electron microscopy (TEM). Fig. 1b shows a representative TEM image of the raw product produced by our method. A large amount of Ni particles with a uniform size of 10–30 nm covered in multi-layered graphene layers (black arrow in Fig. 1b) can be seen to evenly distribute in single-walled carbon nanotube (SWCNTs, red arrow in Fig. 1b) networks (Fig. 1c for schematic). In view of the existence of the structure of core-shell, we denoted the raw product, intermediate product and final product as Ni@C/SCN, Ni@C/SCN-A and Ni@C/SCN-AN respectively. After optimization, the structure of Ni@C and the networks of SWCNTs were maintained which could be observed from the TEM image of the final product (Ni@C/SCN-AN, Figure S2). SWCNTs network could significantly improve the conductivity of material because of the high conductivity of SWCNTs and the network characteristics. Excellent electrical conductivity is conducive to the electronic conduction of electrocatalytic reaction. After post-treatment, the yield of the Ni@C/SCN-AN sample is about 50% of that of the Ni@C/SCN-A sample.

We further studied the Ni@C structures in Ni@C/SCN and Ni@C/SCN-AN by high-resolution TEM (HRTEM, Figure S3). It revealed that the Ni nanoparticle is encapsulated in dozens of graphene layers with a d-spacing of 0.21 nm which corresponds to the (111) plane of Ni metal, indicating that nickel metal state is preserved in the final product Ni@C/SCN-AN. The materials were also studied by X-ray diffraction (XRD, Fig. 2a). The three well resolved peaks at 2θ of 44.5° , 51.8° , and 76.4° can be unambiguously assigned to the (111), (200), and (220) planes of the Ni nanoparticles, respectively. This result

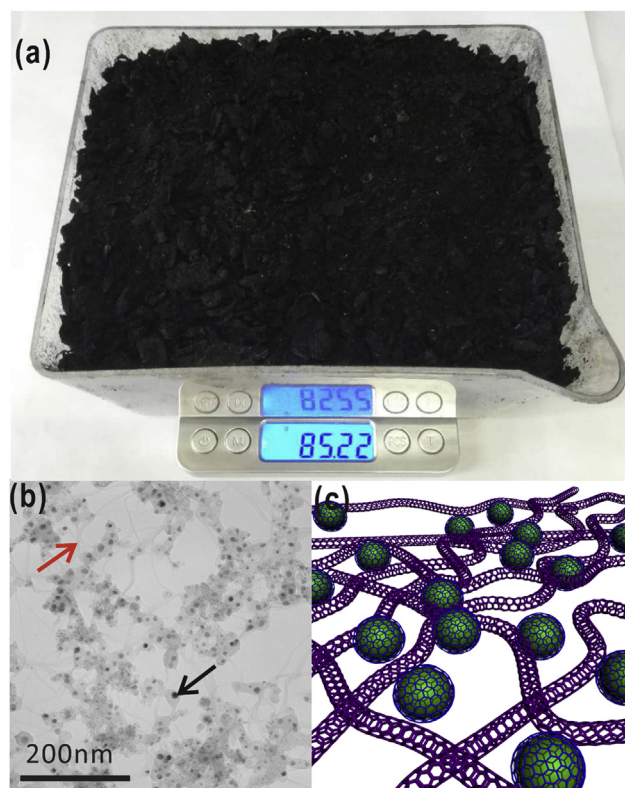


Fig. 1 – The photograph of masses of the raw product (a); TEM image (b) and schematic picture (c) of raw product.

further illustrates that post-treatment did not cause any damage to the metallic state of nickel. The presence of carbon nanotube is reflected by a peak at 26.6° due to graphite C (111) diffraction. In order to obtain the weight content of nickel metal, we conducted a thermogravimetric analysis (TGA) in an air atmosphere with a ramping rate of 10 K/min (Fig. 2b). A residual mass of 36% was obtained from TGA analysis of the Ni@C/SCN sample and the counterpart of Ni@C/SCN-AN is 75.11%. Consider that the SWCNTs and the carbonaceous impurities were completely burned off near 1000°C in air and the residual material is metal oxide, the Ni contents in the Ni@C/SCN and Ni@C/SCN-AN composites could be easily determined to be 28.3% and 59% from the residual mass, respectively. With the increasing of Ni content, the activity of electrocatalytic reaction will also increase. High nickel content means more active sites which is closely related to the catalytic activity. In addition, Ni@C/SCN-AN sample has better thermal stability than Ni@C/SCN sample (Fig. 2b).

We use X-ray photoelectron spectroscopy (XPS) measurements to prove the nitrogen doping (Figure S4). In the final sample (Ni@C/SCN-AN), the content of nitrogen is 7.37% (Table S4) with three kinds of nitrogen (pyridinic-N, graphitic-N, pyrrolic-N). The N-doping has been proved to be an effective way to regulate the electronic structure and electrocatalytic property of graphene through the electron interaction [14]. So, we also believe that nitrogen doping contributes to OER performance.

The OER performance of Ni@C/SCN, Ni@C/SCN-A, Ni@C/SCN-AN and IrO_2 sample was measured in 1 M KOH solution

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