



High rate capability and long cycle-life of nickel oxide membrane electrode incorporated with nickel and coated with carbon layer via *in-situ* supporting of engineering plastic for energy storage application



Xiaoning Zhao, Naijie Wang, Yongtao Tan, Ying Liu, Lingbin Kong, Long Kang, Fen Ran*

State Key Laboratory of Advanced Processing and Recycling of Non-ferrous Metals, Lanzhou University of Technology, Lanzhou 730050, PR China

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ABSTRACT

In this article, the nickel oxide membrane electrode incorporated with nickel and coated with carbon layer (M-NiO/Ni@CL) is prepared through phase-separation method followed by thermal annealing process. The effects of thickness and feeding ratio of precursor components on the structures and electrochemical behaviors are also investigated. The electrode membrane of M-NiO/Ni@CL with the thickness of 67 μm exhibits the highest specific capacitance of 375 F/g at a current density of 0.5 A/g, and even retains 353 F/g when the current density increasing 10 times (94.1%). In addition, an asymmetrical supercapacitor (ASC) cell based on the as-synthesized material of M-NiO/Ni@CL (positive electrode) and the activated carbon (negative electrode) is developed. The ASC device presents a high specific capacitance of 41.9 F/g at a current density of 0.5 A/g based on the total mass of cell within a voltage range of 0–1.6 V; more importantly, it shows an ultralong cycling stability, with 93.8% of capacitance retention after 5000 cycles. The high rate capability and the long-cycle life of NiO electrode are mainly due to the unique structure of membrane, the quantum-dots incorporation of nickel, and the thin layer of carbon coating.

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1. Introduction

With the growing serious environmental pollution and energy crisis, there is an emergency demand for developing clean, renewable and efficient sources of energy, as well as new technologies associated with energy storage and conversion [1]. In the past years, considerable efforts have been devoted to the development of alternative energy storage devices in response to supply the depletion [2]. Supercapacitors, also called electrochemical capacitors, are regarded as a new promising candidate for energy storage because of their excellent characteristics, i.e., high power density, fast charging-discharging capability, good cycle stability, and safe operation. And in recent years, supercapacitors have been applied in many areas, for instance, hybrid electric vehicles, portable electronics, mobile electronic devices, stand-by power systems, and so on [3–9].

Based on the energy storage mechanism, supercapacitors can be divided into two families: electric double-layer capacitors (EDLCs)

and pseudocapacitors [10–12]. EDLCs store charges in a thin double layer located at the interface between the electrolyte and the electrode [13], while pseudocapacitors is mainly based on the fast and reversible faradic (redox) reactions, which occurs on the surfaces of the electrode materials or the interface between electrode material and electrolyte [14]. From the materials point of view, there are three kinds of materials have been used in supercapacitors, that is, carbon, conducting polymer, and transition metal oxide [15,16]. All of these materials have their own advantages and disadvantages. Carbon material shows long cycle-life and good conductivity, but low specific capacitance. Conducting polymer always exhibits high flexibility, but poor cycle stability and relative low rate-capacity. As for the transition metal oxide, they possess ideal capacitor performance, low cost, and environmentally friendly nature, but poor electrical conductivity [17–20]. Therefore, it is necessary to develop better materials for supercapacitors electrodes, which exhibit good conductivity, large capacity, long-cycle life, and excellent rate capability.

As for the transition metal oxide, nickel oxide (NiO) is considered as an attractive candidate for the next generation of supercapacitors because of low cost, easy synthesis, and high theoretical

* Corresponding author.

E-mail addresses: ranfen@163.com, ranfen@lut.cn (F. Ran).

specific capacitance [21–23]. However, like other transition metal oxides, the low electronic conductivity of NiO significantly blocks electron transport and decelerates the redox reactions, resulting in poor rate capability. To address this issue, many studies have been devoted; among them, one effective strategy is combining the nanosized NiO with electrically conductive materials, such as graphene, activated carbon, carbon nanotubes, and other carbon materials [24–27]. For this purpose, Yi and his co-workers synthesized CNT@NiO composites through a chemical bath deposition method followed by thermal annealing, which shows remarkable rate capability and better cycle stability [28]. More recently, Tao and his co-workers prepared mesoporous NiO/carbon composite by a solution combustion process [29]. Nevertheless, some electrochemical performance like rate capability and cycle-life of the NiO electrode needs further improvement and strengthening.

In this work, we reported a novel and facile method combining liquid-liquid phase-separation and thermal annealing method to fabricate hierarchical porous nickel oxide membrane incorporated with nickel and coated with thin carbon layer (M-NiO/Ni@CL). Liquid-liquid phase-separation technique, which was reduced by immersion precipitation, is widely used in the fabrication of biomedical membranes [30], porous carbon [31], and flexible electrode materials [32]. The prepared M-NiO/Ni@CL used as electrode material would exhibit excellent rate capability, long cycle life, and high capacitance, attributing to the outstanding electric conductivity of carbon and ideal theoretical specific capacitance of NiO. The effects of thickness and feeding ratio of precursor on the structure and electrochemical performance were also studied in detail.

2. Experimental

2.1. Chemicals

Polyether sulfone (PES, Ultrason E6020P, $M_w = 58,000$ Da) was obtained from BASF, Germany. Activated carbon was purchased from Shenyang Kejing Auto-instrument Co. Ltd. $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ was purchased from Tianjin Beichen Founder Reagent Factory and $\text{NH}_3 \cdot \text{H}_2\text{O}$ was obtained from Tianjin BASF Chemical Co., Ltd. All the other chemicals (analytical grade) were obtained from Sinopharm Chemical Reagents Co. Ltd., China, and used without further purification.

2.2. Synthesis of nickel hydroxide ($\text{Ni}(\text{OH})_2$)

$\text{Ni}(\text{OH})_2$ was prepared by a facile-improved precipitation method [33]. In a typical procedure, 40 g of $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ was dissolved in 141 mL deionized water in a glass beaker with magnetic stirring at room temperature. Then the nickel chloride hydrate solution was slowly adjusted to $\text{pH} = 9$ by dropwise addition of 5 wt% $\text{NH}_3 \cdot \text{H}_2\text{O}$ at a temperature of 20 °C, controlling the addition time to more than 2 h. The resulting suspension was stirred at this temperature for an additional 3 h. Finally the solid was filtered, washed with a copious amount of distilled water, and dried at 60 °C for 6 h.

2.3. Preparation of ultrathin porous nickel hydroxide membrane (M-Ni(OH)₂/PES)

Porous nickel hydroxide membrane (M-Ni(OH)₂/PES) was prepared by a phase-separation technique. In a typical synthesis, $\text{Ni}(\text{OH})_2$ mixed with conducting graphite was firstly dispersed in dimethylacetamide (DMAC) under magnetic stirring and ultrasonication to obtain a homogeneous turbid solution. Then, PES was dissolved in the solution and stirred for 24 h. The casting solution

was prepared as membrane by spin coating (Spin Coater, KW-4A) at 20 °C at a rotating speed of 500 rpm/min for 15 s, which was immediately immersed in a coagulation bath of deionized water at 20 °C. Then, the ultrathin hybrid polymer-nickel hydroxide membrane was transferred to a water bath and immersed for 24 h to remove the residual solvent. Finally, the membrane was dried in vacuum oven at 60 °C for 24 h. In the experimental process, PES, $\text{Ni}(\text{OH})_2$, and conducting graphite with mass ratio of 1: 3: 1 was added in the casting solution. And the thickness and nanostructure of porous membrane were tuned by adjusting solid content in casting solutions; the membranes with different thickness of 62, 67, 72, and 84 μm were prepared, respectively. Moreover, the membranes with different mass ratio basing on PES, $\text{Ni}(\text{OH})_2$ and conducting graphite of 1: 2: 1 and 1: 4: 1 were also prepared, respectively.

2.4. Preparation of hierarchical porous nickel oxide membrane incorporated with nickel and coated with carbon layer (M-NiO/Ni@CL)

M-NiO/Ni@CL was prepared by a simple heat-treatment process. Typically, the prepared porous nickel hydroxide membrane was pre-oxidized at 250 °C for 6 h in air and carbonized under a flowing nitrogen atmosphere in a tube furnace equipped at 700 °C for 2 h to fabricate M-NiO/Ni@CL. We chose the thermal annealing condition basing on TGA curve of PES (Fig. S1).

2.5. Materials characterization

The microstructures and morphologies were characterized by field emission scanning electron microscope (SEM, JSM-6701F, JEOL, Japan) and transmission electron microscope (TEM, JEM-2010, JEOL, Japan). The photographs of the membrane were taken using a camera (PowerShot S100V, Canon, Japan). The crystal structure was analyzed by X-ray diffraction (XRD Bruker, D8 Advance, Germany). The surface area was calculated using the Brunauer-Emmett-Teller (BET) equation. Pore size distribution was calculated by the Barrett-Joyner-Halenda (BJH) method using the adsorption branch of the isotherm. The surface chemical composition of the samples was analyzed by X-ray photoelectron spectroscope (XPS, physical Electronics UK).

2.6. Electrode preparation

For the preparation of M-NiO/Ni@CL electrode, M-NiO/Ni@CL power, acetylene black, and conducting graphite at a mass ratio of 80: 7.5: 7.5 (wt%) was added in an agate mortar until a homogeneous black powder was obtained [34]. To this mixture, polytetrafluoroethylene (5 wt%) was added with a few drops of ethanol. The resulting paste was pressed at 10 MPa to nickel foam that served as a current collector then dried at 80 °C for 12 h. Each electrode contained 4 mg of the electroactive material and had a geometric surface area of 1 cm^2 .

2.7. Electrochemical characterization

All electrochemical measurements were conducted in a classical three-electrode glass cell setup at room temperature. The synthesized sample, platinum foil electrode (1.5 × 1.5 cm) and a saturated calomel electrode (SCE) served as working electrode, counter electrode and reference electrode, respectively. The performance of electrochemical properties was tested using cyclic voltammetry (CV), and galvanostatic charging-discharging (GCD) on a land cell tester.

The electrochemical measurement of the asymmetric

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