



Fabrication and electrochemical properties of periodic pillared-layer nickel hybrid electrode



Xing Hu^{*}, Jianlong Jiang, Fan Zhang, Zhiyuan Ling

Department of Electronic Materials Science and Engineering, South China University of Technology, Guangzhou 510640, People's Republic of China

ARTICLE INFO

Article history:

Received 4 August 2013

Received in revised form 15 September 2014

Accepted 22 September 2014

Available online 11 October 2014

Keywords:

Electrochemical capacitor

Nickel

Anodic alumina

Photonic crystal

ABSTRACT

A periodic pillared-layer nickel hydroxide/nickel hybrid electrode was fabricated by electroforming nickel on an interconnected anodic alumina photonic crystal template followed by cyclic voltammetry. The XRD results confirmed the α -Ni(OH)₂ grown in-situ on the nanoporous nickel. The periodic pillar-layered hybrid electrodes showed the ultrahigh volumetric capacitance of 1387 F cm⁻³ and a large areal capacitance of 2.51 F cm⁻² calculated from the discharge curves (at discharge current densities of 5 mA cm⁻²) and it also showed good cycling stability (no loss of capacitance after 1000 cycles at a scan rate of 10 mV s⁻¹). The enhanced capacity performance for the layered hybrid electrode is mainly attributable to the optimized nanostructure of the current collector.

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1. Introduction

Electrochemical capacitors are currently receiving increasing interest due to their long-term cycling stability and high charging/discharging rates [1,2]. Among the electrochemical capacitors electrode materials, nickel oxides/hydroxides are the most promising candidates for alkaline supercapacitors due to their high theoretical specific capacitance, environmental friendliness, and low cost [3–7]. Unfortunately, an extremely high specific capacitance has been observed only for thin layers of electrode materials, which limits the materials' practical application. To achieve high areal capacitance, nickel foam is used for current collector to obtain high mass loading. For instance, both high specific capacitance and areal capacitance were reported for NiO nanorod/nickel foam (2018 F g⁻¹ and 4.4 F cm⁻²) [5], Ni(OH)₂/CoO nanowall/nickel foam (2374 F g⁻¹ and 11.5 F cm⁻²) [6] and Ni(OH)₂ nanoflake/carbon nanotube/nickel foam (3300 F g⁻¹ and 16 F cm⁻²) [7]. However, nickel foams with pore diameters and nickel wire diameters of tens of micrometers waste large amounts of space that could be used for volumetric capacitance.

In the few past decades, anodic aluminum oxide film with controllable pore size and high pore density has attracted much attention for synthesizing coaxial nanowire (NiO/Ni) [8] or coaxial nanotube (MnO₂/carbon) [9]. Although the arrays of one dimensional (1D) heterogeneous nanostructure can get high volume capacitance, their own drawbacks still exist. For example, when 1D nanomaterials are fabricated with high aspect ratios without interconnections, they tend to have a high electrical resistance due to their small diameters

and become mechanical unstable with aggregation [10]. Due to the structural interconnectivity, three dimensional (3D) nanostructures can provide a solution to the problems of 1D nanomaterial [10]. To get both high volumetric capacitance and areal capacitance, an optimization for the structure of current collector is necessary for the following: minimize the diameter of the current electrode while not decreasing the electronic conductivity, minimize the void space while not increasing pore resistance to the electrolyte and maximize the thickness of current collector while keeping excellent mechanical stability.

To fabricate electrode material with 3D nanostructures, a periodic interconnected anodic alumina film was obtained from branched AAO templates by chemical etching [11]. 3D interconnected carbon material was negative replicated from the periodic interconnected AAO template, but it didn't show good electrochemical performance due to poor conductivity [11]. In this work, 3D interconnected periodic pillared-layer nickels were obtained by electroforming nickel [12, 13] on a periodic interconnected anodic alumina, and α -Ni(OH)₂ was grown in-situ on the nanoporous nickel after cyclic voltammetry.

2. Experimental

High-purity aluminum sheets were first anodized at 40 V in 0.3 M oxalic acid at 0 °C for 10 min to obtain a protecting film and then periodically anodized in 0.3 M oxalic acid at 30 °C. An anodized film with multiple periodicities was produced by stepping the voltage down from 40 V to 4 V in 270 s and then increasing the voltage sinusoidally from 4 V to 40 V in 40 s. The basic component of the electroforming solution was Ni(NH₂SO₃)₂·4H₂O, as described in Ref. [13]. Residual aluminum and anodic alumina were removed by

^{*} Corresponding author. Tel./fax: +86 20 87111224.
E-mail address: Huxing@scut.edu.cn (X. Hu).

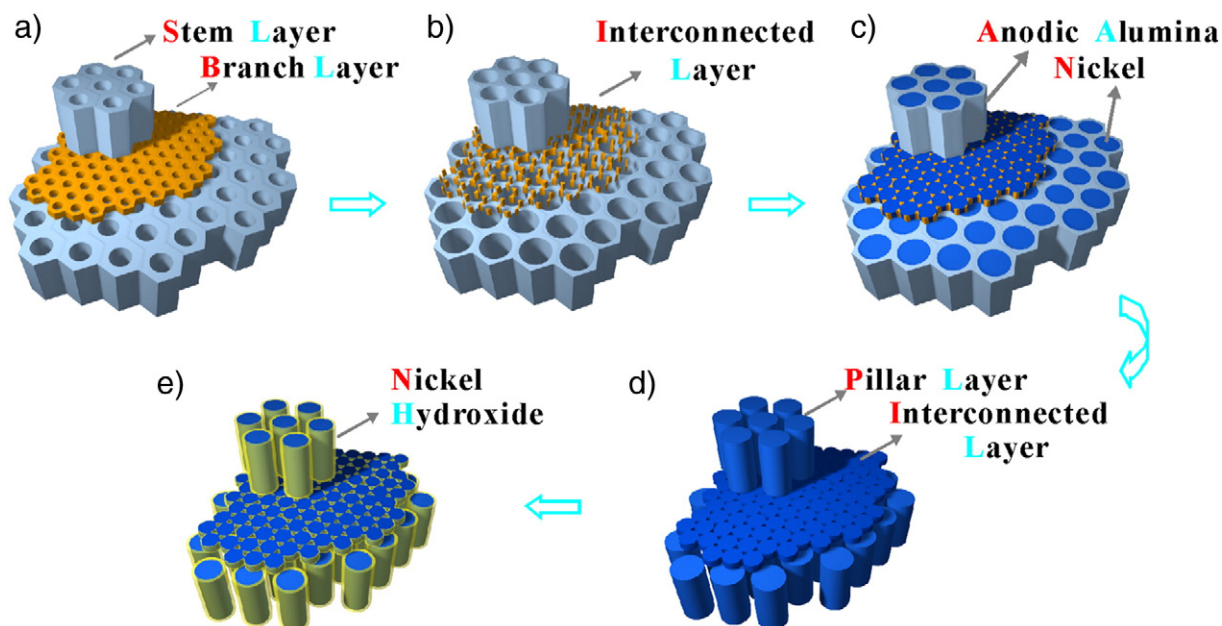


Fig. 1. Process used to fabricate periodic layered hybrid electrodes from anodic alumina photonic crystal template: (a) branched anodic alumina; (b) interconnected anodic alumina; (c) electroformed nickel-anodic alumina; (d) pillar-layered nickel; (e) layered nickel hydroxide–nickel hybrid electrode.

immersing the film in a 6 M KOH solution. To obtain nickel hydroxide for supercapacitor applications, a Ni electrode was anodized by performing cyclic voltammetry for 3 cycles between 0 and 0.5 V at a scan rate of 1 mV s^{-1} .

Cyclic voltammetry was conducted using an electrochemical workstation (CS 300) in 6 M KOH in a three-electrode cell system using a saturated calomel electrode (SCE) as the reference electrode and a graphite plate as the counter electrode. Charge–discharge cycling was performed from -0.1 to 0.4 V (SP-150, Bio-Logic). The areal capacitance (AC) value was calculated using half of the integrated area of the cyclic voltammetry curve or by integrating the discharge current over

time to obtain the charge and subsequently dividing the charge by the area of the electrode and the width of the potential window. The volumetric capacitance (VC) value was calculated by dividing the AC value by the porous layer thickness.

The morphology of the membranes was examined with a LEO1530VP field-emission scanning electron microscope equipped with an X-ray energy-dispersive spectroscopy (EDS) attachment. The phase compositions were identified by X-ray diffraction (XRD) using $\text{CuK}\alpha$ radiation (Rigaku D/max-III A). X-ray photoelectron spectroscopy (XPS) analyses were carried out with an Escalab 250 spectrometer (Thermo-VG Scientific).

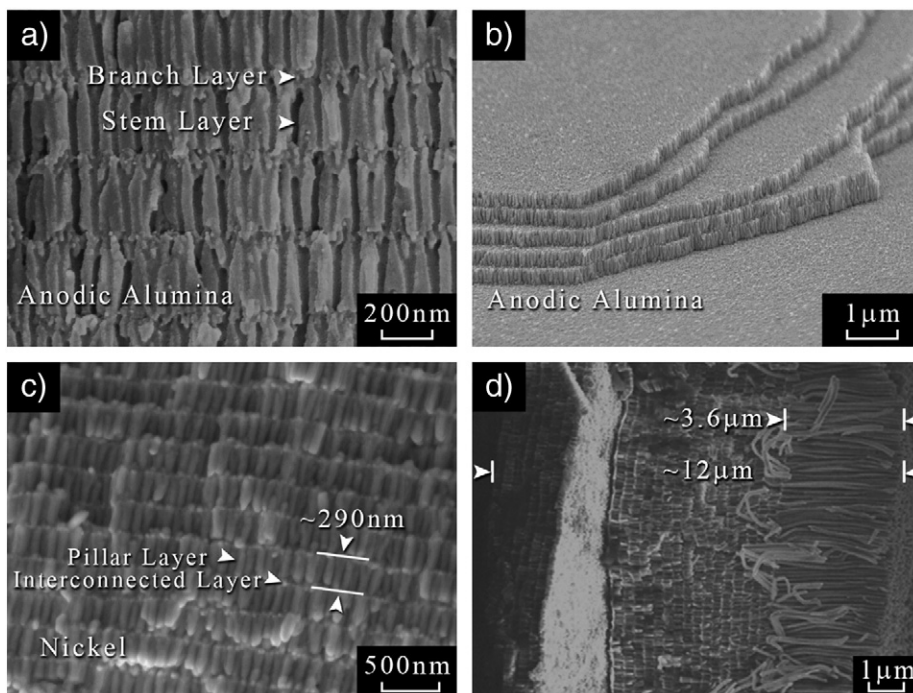


Fig. 2. SEM image of fractured (a) branched anodic alumina, (b) interconnected anodic alumina, (c) and (d) layered nickel.

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