

Full Length Article

Low-temperature direct synthesis of mesoporous vanadium nitrides for electrochemical capacitors



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ABSTRACT

Mesoporous vanadium nitrides are directly synthesized by a one-step chemical precipitation method at a low temperature (70 °C). Structural and morphological analyses reveal that vanadium nitride consist of long and slender nanowhiskers, and mesopores with diameters of 2–5 nm. Compositional analysis confirms the presence of vanadium in the VN structure, along with oxidized vanadium. The cyclic voltammetry and charge-discharge tests indicate that the obtained material stores charges via a combination of electric double-layer capacitance and pseudocapacitance mechanisms. The vanadium nitride electrode exhibits a specific capacitance of 598 F/g at a current density of 4 A/g. After 5000 charge-discharge cycles, the electrode has an equivalent series resistance of 1.42 Ω and retains 83% of its initial specific capacitance. This direct low-temperature synthesis of mesoporous vanadium nitrides is a simple and promising method to achieve high specific capacitance and low equivalent series resistance for electrochemical capacitor applications.

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1. Introduction

The increasing need for the development of renewable and sustainable energy storage systems has generated a large demand for emerging technologies, including secondary batteries, electrochemical capacitors (ECs), and fuel cells. Among these energy storage systems, ECs are unique because of their high power density, short charge time, and long and stable cycle life [1–5].

ECs are generally classified as electric double-layer capacitors (EDLCs) and redox capacitors according to their working mechanism [6]. In EDLCs, which are based on carbonaceous materials, the capacitance is attributed to charge accumulation at the electrode-electrolyte interface. Redox capacitors exhibit pseudocapacitance due to the oxidation-reduction reactions that occur at the electrode surface. Various transition metal oxides and conducting polymers have been investigated as electrode materials for redox capacitors [7–15]. Thus, there is an increasing need for the development of advanced electrode materials having high specific surface area and good electrical conductivity.

Ruthenium oxide (RuO₂) exhibits a high specific capacitance (up to 720 F/g) [7], but the high cost hinders the large-scale commercial production of RuO₂ as a redox capacitor. Low cost materials such as manganese oxide (MnO₂) have been extensively studied to overcome high cost of RuO₂ [10,11,13,15–18]. However, poor electrical conductivity compared to RuO₂ limits the power capability of the MnO₂-based electrodes.

Recently, transition metal nitrides such as nickel nitride (Ni₃N), titanium nitride (TiN), molybdenum nitrides (MoN and Mo₂N), and vanadium nitride (VN) have received much attention as alternatives to metal oxides for electrochemical capacitor applications because of their excellent electrical conductivity and specific capacitance [19–23]. Among these, VN is considered to be a good candidate for electrode materials due to its high theoretical capacity (>500 F/g), fast redox Faradic response, and high hydrogen evolution overpotential [24–26].

Vanadium nitrides are typically obtained by the reduction of V₂O₅ powders using ammonia at high temperatures [27,28]. Calcination of V₂O₅ xerogels under an ammonia or nitrogen atmosphere was also reported to yield VN powders [29,30]. In previous studies on the synthesis of vanadium nitrides, V₂O₅ was used as the vanadium source due to its layered structure, while short chain amines or long chain ammonium were used as templates or intercalates

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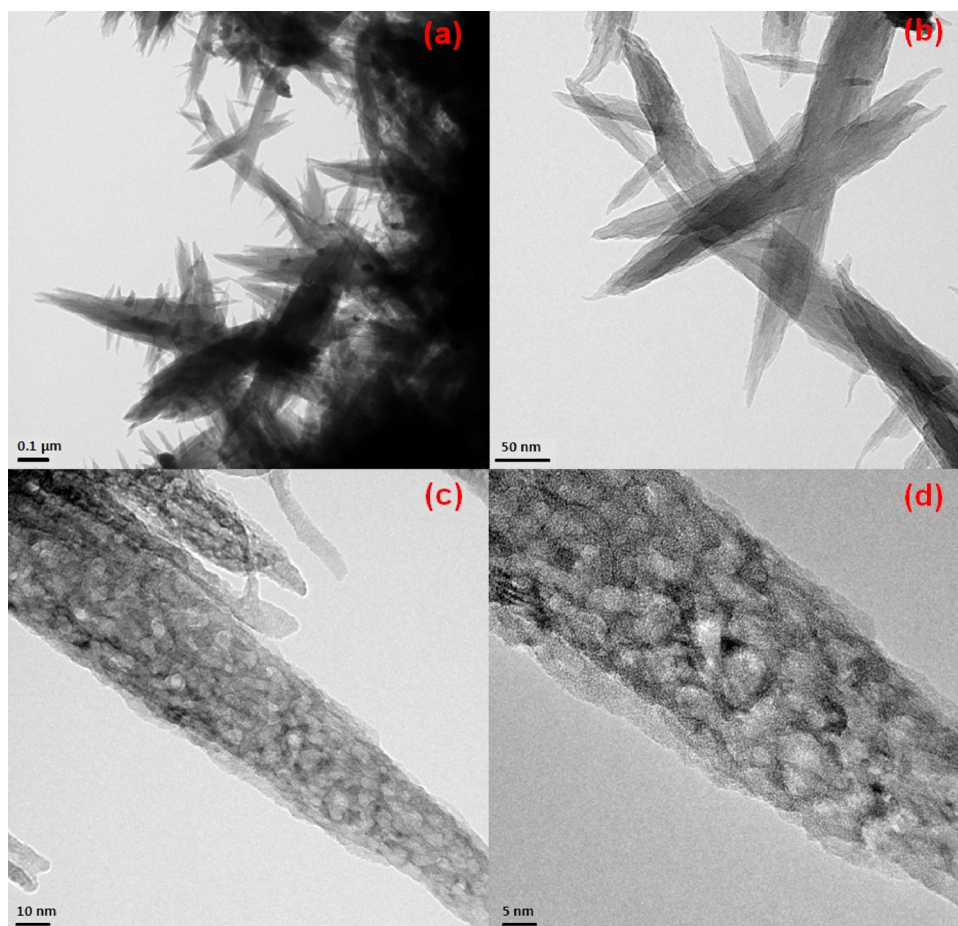


Fig. 1. TEM images of the vanadium nitrides obtained by one-step chemical precipitation.

[31–33]. However, these methods require high temperatures and long durations. It is necessary to develop a simple and cost-effective approach to synthesize vanadium nitrides.

In this study, we report on a low-temperature direct method to obtain the mesoporous vanadium nitrides via a one-step chemical precipitation route. The structural, morphological, compositional, and electrochemical properties of the vanadium nitrides were investigated in order to evaluate them as electrode materials for electrochemical capacitors.

2. Experimental

2.1. Synthesis of the vanadium nitrides

Vanadium tetrachloride (VCl_4 , Aldrich) and urea (NH_2CONH_2 , Sigma-Aldrich) were used as the vanadium and nitrogen sources, respectively. 20 mL of a 20 mM vanadium tetrachloride solution was mixed with 40 mL of a 83 mM urea solution. The mixture was stirred at room temperature for 5 min and ultrasonicated for 30 min. Then, it was oven-aged at 70 °C for 2 h. The resulting product, which was the vanadium nitrides, was collected, cleaned thoroughly with double-distilled water and ethanol, and dried in an oven at 50 °C for 6 h.

2.2. Fabrication of the vanadium nitride electrode

The vanadium nitride electrode was fabricated by combining the active material with a conducting additive and a binder. First, a mixture of the synthesized vanadium nitride powders (85 wt%)

and Super-P carbon black (MMM Carbon, 5 wt%) were homogenized in an agate mortar. To this, 10 wt% PTFE (polytetrafluoroethylene, 60 wt% dispersion in water, Sigma Aldrich) binder was added to form a slurry. The slurry was ultrasonicated to ensure thorough mixing of the components, and uniformly coated onto a Ni foil current collector (110 μm thick) using a film applicator. The slurry-coated electrode was dried in an oven at 80 °C for 6 h.

2.3. Materials characterization

The morphology and structures of the vanadium nitrides were examined by a transmission electron microscope (TEM, FEI Morgagni) and an X-ray diffractometer (XRD, Rigaku). The TEM was operated at an acceleration voltage of 300 kV. XRD used a Cu-K α radiation source ($\lambda = 0.15418$ nm) at 40 kV and 150 mA. Compositional analysis was carried out by X-ray photoelectron spectroscopy (XPS, Thermo Scientific K-Alpha ESCA). The X-ray source was monochromatized Al-K α radiation at 1486.6 eV. The surface area and pore size of the vanadium nitrides were analyzed by nitrogen adsorption-desorption measurements (Micromeritics, ASAP 2010). Samples were outgassed at 200 °C for 4 h prior to measurements. The surface area and pore size of the samples calculated based on the Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) desorption methods.

2.4. Electrochemical analysis

A standard three-electrode cell system (Princeton Applied Research, VSP) was used to analyze the electrochemical properties

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