



Vanadium oxide nanorings: Facile synthesis, formation mechanism and electrochemical properties[☆]



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A. Nanostructures

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ABSTRACT

This paper describes the hydrothermal synthesis of vanadium oxide nanorings and nanobelts from aqueous precursors without using any template. These nanorings are formed via the acidification of sodium metavanadate solution. A simple ion intercalation/de-intercalation process occurs under mild hydrothermal conditions leading to the self-rolling of exfoliated vanadium oxide nanobelts. The structure and morphology of the products are characterized by XRD, SEM, TEM and charge-discharge measurement. XRD pattern reveals that the products consist of V_2O_5 nanorings and $Na_{0.3}V_2O_5$ nanobelts evidenced by SEAD pattern and EDS. Highly magnified TEM images exhibit nanorings made of nanoribbons of width about 300 nm and thickness of about 60 nm. Electrochemical analyses revealed that the V_2O_5 nanorings/nanobelts delivers an initial lithium-ion intercalation capacity of 280 mAh g^{-1} and reaches a stabilized capacity of 200 mAh g^{-1} at a current density of 100 mA g^{-1} .

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1. Introduction

1-D nanostructured transition metal oxides exhibits a unique class of materials because of their redox activity, which is connected with outstanding electrochemical and catalytic properties. Among transition metal oxides, layered vanadium pentoxide ($E_g = 2.8 \text{ eV}$) and its compounds allow a wide range of practical applications such as lithium batteries [1–6], catalysis, [7,8], electro-chromic devices [9], supercapacitors [10], actuators [11], sensors [12] etc, due to their outstanding structural flexibility combined with chemical and physical properties.

V_2O_5 is a typical intercalation compound with a layered crystal structure that can be reversibly intercalated and de-intercalated between the layers has been extensively studied as a cathode

material for rechargeable lithium batteries because of its low cost, abundance, easy synthesis and high energy density [13]. However, the development of rechargeable lithium batteries with V_2O_5 as a cathode material has been limited due to its poor structural stability, low electronic and ionic conductivity, and slow electrochemical kinetics [14,15]. It is well known that the structure and morphology of V_2O_5 can strongly influence its electrochemical performance. In recent years, a lot of research has been focused on the synthesis and fabrication of 1-D nanostructured V_2O_5 . It has been demonstrated that V_2O_5 nanorods, nanotubes, nanowires, nanoribbons, etc, are regarded as promising active lithium intercalation cathode materials because they provide shorter path lengths for both electronic and Li ionic transport [16].

$V_2O_5 \cdot nH_2O$ and cation intercalated V_2O_5 [$M_{0.3}V_2O_5 \cdot nH_2O$ ($M = Li^+, Na^+$)] have been extensively studied for their electrochemical properties. They consist of V_2O_5 bilayers whose interlayer space is occupied by water molecules ($V_2O_5 \cdot nH_2O$) and cations ($Na_{0.3}V_2O_5 \cdot nH_2O$). In the case of $Na_{0.3}V_2O_5 \cdot nH_2O$, the structural anisotropy of the particles was shown to induce a better

[☆] Electronic supplementary information's (ESI) are available: SEM images for the variations of experimental parameters and charge-discharge profile.

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electrochemical response, compared to the analogous compound synthesized by solid state reactions [17,18].

Although various methods are available for the synthesis of low dimensional V_2O_5 nanostructured electrode materials [19–25], many of them suffer from the limits of high temperatures, special equipment's and/or special experimental conditions. The hydrothermal synthesis remains interesting because it is a powerful tool to transform transition metal oxides into high quality nanostructures and nanostructured vanadium oxides in different morphologies besides lower temperature involved in the cost effective environmentally benign process.

A common preparative procedure for nanostructured vanadium oxide materials from molecular clusters to 1-D and 2-D layered compounds involves the hydrothermal treatment of a vanadium precursor (Ex. NH_4VO_3 , Vanadium alkoxides, V_2O_5 or $VOSO_4$). Nanotubes are obtained via a self-rolling process while amazing morphologies such as nanospheres, nanoflowers and even nanorichins are formed via the self-assembling of nanoparticles [26]. Control over the nucleation and growth may be crucial in the tailoring of the size, shape, surface structure and consequently to the final properties of the resultant VO_x materials.

Increasing attention is being nowadays paid to ring [27–32] like nanomaterials because of their size, special morphology-related properties and potential nanoscale applications [31]. This paper reports on the synthesis and the electrochemical performance of V_2O_5 nanorings and $Na_{0.3}V_2O_5$ nanoribbons obtained under hydrothermal conditions. The mechanism for the formation of nanorings is also discussed.

2. Experimental

Hydrothermal process was carried out like our previous report to synthesis Ammonium vanadate nanorings or Cadmium

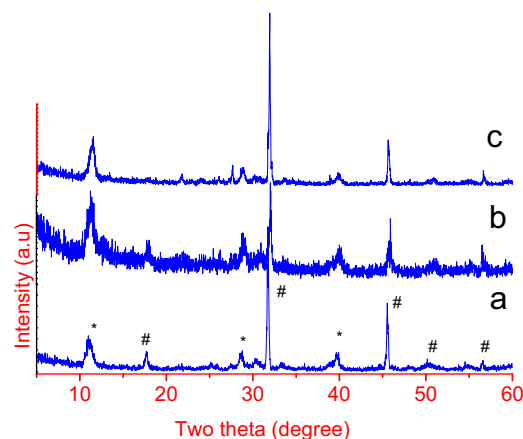


Fig. 1. PXRD patterns of the nanorings/nanoribbons prepared at (a) 130 °C for 2 days, (b) 150 °C for 2 days and (c) 110 °C for 3 days. (* = $Na_{0.3}V_2O_5$, # = V_2O_5).

carbonate nanorings [33,34]. 0.27 g $NaVO_3$ was dissolved in 20 mL distilled water taken in a 30 mL capacity Teflon tube. Two drops of HCl (pH \approx 3–4) was added to it to maintain acidic medium and stirred for 10 min. The resultant wine-red solution was subjected to hydrothermal treatment from 100 °C to 180 °C for 1–3 days. The reddish-brown non-adherent spongy-like bulky material was collected and washed with distilled water and absolute alcohol several times before being dried in air.

2.1. Characterizations

Powder X-ray diffraction data were recorded on Philips X'pert PRO X-ray diffractometer with graphite monochromatized Cu-K α (1.5418 Å) radiation. The Fourier transform infrared spectrum

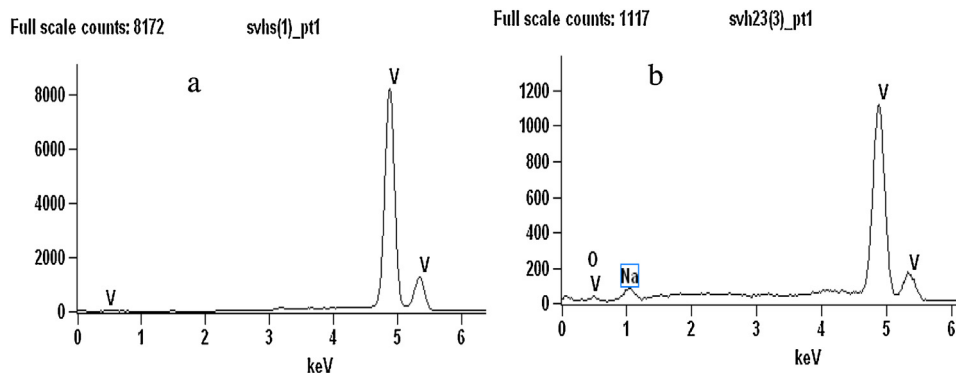


Fig. 2. EDS spectrum of (a) nanorings and (b) nanoribbons prepared at 110 °C for 3 days.

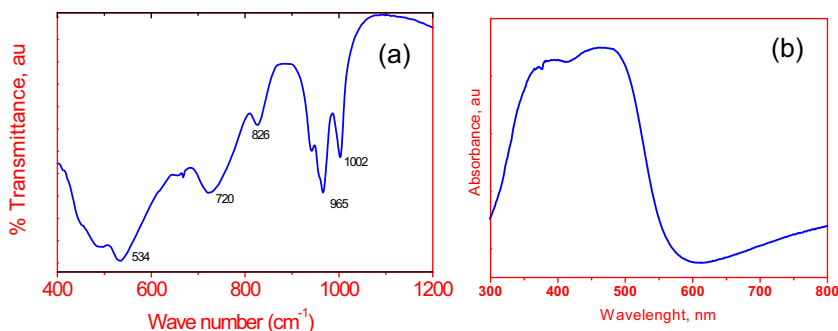


Fig. 3. (a) FTIR spectrum and (b) UV-vis spectrum of the V_2O_5 nanorings/nanoribbons prepared at 130 °C for 2 days.

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