



Fabrication of V_2O_3/C core–shell structured composite and VC nanobelts by the thermal treatment of VO_2/C composite

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ABSTRACT

Belt-like V_2O_3 encapsulated into carbon tubes (V_2O_3/C) core–shell structured composite and vanadium carbide (VC) nanobelts have been successfully synthesized by the thermal treatment with VO_2/C core–shell structured composite through adjusting the heating temperature for the first time. The amorphous carbon on the surface of VO_2 plays a dual role in this thermal process, namely as the reductant to reduce VO_2 to V_2O_3 or VC, and as the carbon precursor for the V_2O_3/C carbon shell and VC. The as-obtained samples were respectively characterized by X-ray powder diffraction, energy-dispersive X-ray spectrometer, Raman spectrum, elemental analysis, Fourier transform infrared spectroscopy, scanning electron microscopy, transmission electron microscopy and Brunauer–Emmett–Teller. V_2O_3/C was successfully synthesized at 700–900 °C for 2 h, and VC nanobelts were successfully prepared at 1000 °C for 2 h. The as-obtained V_2O_3/C composite and VC nanobelts contain C–H groups, which will facilitate the linkage of catalytic species or polymers to the surface in their potential applications. V_2O_3/C composite has higher specific surface area than that of VC due to the amorphous carbon coated on the surface of V_2O_3 . Furthermore, the thermal stability of VC in air was investigated by Thermo-Gravimetric/Differential Thermal Analyzer, revealing that it had good thermal stability and oxidation resistance below 335 °C in air.

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1. Introduction

Vanadium oxides and their related compounds had attracted much attention in the past decades due to their promising applications in the cathode materials for reversible Li-ion batteries, catalysts, optical switching and sensors [1–9]. Of the family of vanadium oxides, vanadium (III) oxide (V_2O_3) displays a metal-to-insulator transition (MIT) as the function of temperature, pressure and doping concentration [10,11]. It exhibits a first-order transformation from a monoclinic antiferromagnetic insulator (AFI) phase to a rhombohedral paramagnetic metallic (PM) at near -120 °C, with a jump in the resistivity of about seven orders of magnitude. And a broad second-order phase transition occurs at about 170 °C from the metallic to a semiconducting state [11,12]. These properties allow V_2O_3 and doped V_2O_3 to have wide potential applications in different areas, including temperature sensors, optical device,

conductive composite polymer and catalyst [11,13–15]. Therefore, in the past decades, many approaches had been explored to prepare V_2O_3 . However, the synthesis of V_2O_3 is mainly focused on the bulk or sphere-like powders. The fabrication of V_2O_3 with belt-like morphology has been comparatively rarely reported [16–18]. Therefore, the fabrication of V_2O_3 with belt-like morphology stimulates great interest and is a meaningful challenge for materials scientists. Besides, it was reported [13] that V_2O_3 can be easily oxidized to high-valence vanadium oxides in the air atmosphere, which may restrict its applications. In this paper, belt-like V_2O_3/C composite was synthesized, which may improve the oxidation resistance of V_2O_3 and expand its application in future.

In the previous decades, the transition metal carbides had attracted a great interest owing to their outstanding physical and chemical properties, including high melting point, high thermal conductivity, high hardness, high strength even at high temperatures and high chemical resistance [19–23]. Among them, vanadium carbide (VC) is a very important industrial material and it can be widely applied to cutting materials, abrasive and anti-wear materials [19,24,25]. So far, various methods have been reported to prepare VC and some approaches have been widely used for its production, such as, high temperature reactions [26], mechanical

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alloying [27], gas reduction-carburization [28], temperature programmed reaction [23], aluminothermic reduction of vanadium oxide [22], the carburization of vanadium oxide with an organic reagent [21], hydrothermal process [29] and so on. However, the synthesis of VC using the precursor vanadium oxide with high valence and C composite has not been reported to the best of our knowledge.

As is well known, vanadium oxides with high valence can react with active carbon to form vanadium oxides with low valence or vanadium carbides. Thus, the idea that the synthesis of VC using vanadium oxides with high valence and carbon core-shell structured composite is practical, which is not reported before. In the previous reports, the synthesis of $V_3O_7 \cdot H_2O/C$ [30,31] and VO_2/C [16] composites had been reported. In this paper, we chose VO_2/C composite to synthesize VC. The synthesis of VO_2/C composite was according to the reference [16], however, the synthetic route was greatly modified to fabricate the VO_2/C composite with controllable carbon shell through the addition of the surfactant (SDS), which is very important to prepare VC nanobelts. In our experiments, it was found that belt-like V_2O_3/C core-shell structured composite was the intermediate product of VC. Herein, a novel route was developed to fabricate V_2O_3/C core-shell structured composite and VC by the thermal treatment with the precursor VO_2/C composite through adjusting the heating temperature. It was found that the amorphous carbon on the surface of VO_2 can play a dual role in this thermal process, namely as the reductant to reduce VO_2 to V_2O_3 or VC, and as the carbon precursor for the V_2O_3/C carbon shell and VC.

2. Experimental

2.1. Materials

Vanadium pentoxide (V_2O_5), ethanol, D-(+)-glucose ($C_6H_{12}O_6 \cdot H_2O$) and sodium lauryl sulfate (SDS) with analytical grade were purchased from Sinopharm Chemical Reagent Co., Ltd and used without any further purification.

2.2. Synthesis of V_2O_3/C composite and VC

The synthesis of V_2O_3/C composite and VC was performed in three steps. First, VO_2 nanobelts was prepared according to our previous report [32], and the detailed information was presented in the Supplementary data. Second, 0.40 g of the above VO_2 nanobelts and 0.01 g of SDS were dispersed into the glucose solution (4.00 g of glucose and 50 mL of distilled water) in a 100 mL beaker under ultrasonic for 20 min, and then the mixture was stirred vigorously for 1 h by magnetic stirrer. After the solution became suspension, they were transferred into a 100 mL Teflon Lined stainless steel autoclave, which was sealed and maintained at 180 °C for 4 h. After cooling to room temperature naturally, the products were filtered off, washed with distilled water and absolute ethanol several times, and dried in vacuum at 75 °C. VO_2/C composite was successfully synthesized according to the analyses of XRD, Raman and TEM, as shown in the Supplementary data. Third, the above products (VO_2/C) were heated in a tube furnace with 5 °C/min heating rate under a flow of argon gas at 700–1000 °C for 2 h to synthesize V_2O_3/C composite and VC.

2.3. Characterization

X-ray powder diffraction (XRD) was carried out on D8 X-ray diffractometer equipment with Cu $K\alpha$ radiation, $\lambda = 1.54060 \text{ \AA}$. The morphology and dimension of the products were observed by the scanning electron microscopy (SEM, Quanta 200) and the transmission electron microscopy (TEM, JEM-100CXII). The Raman spectrum was taken on a RM-1000 spectrometer (Confocal Raman

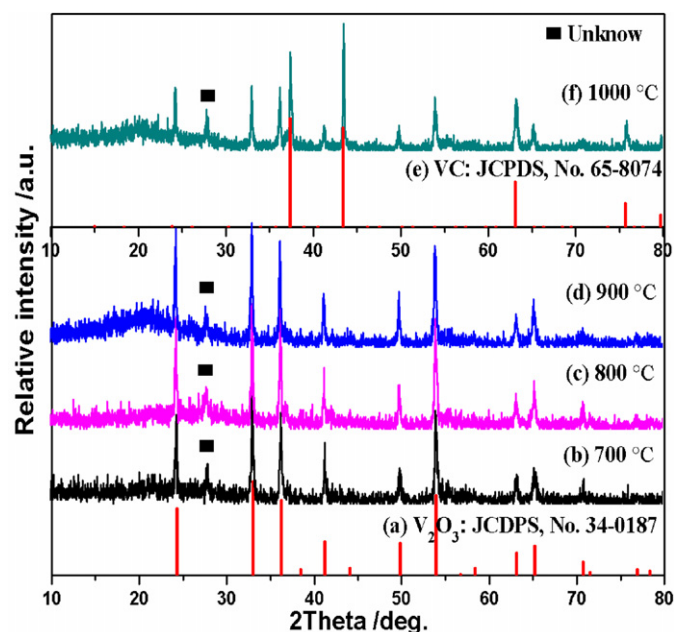


Fig. 1. XRD patterns of the resulting products obtained at different reaction temperatures for 2 h: (a) V_2O_3 , JCPDS, no. 34-0187; (b) 700 °C; (c) 800 °C; (d) 900 °C; (e) VC, JCPDS, no. 65-8074; (f) 1000 °C.

Microspectroscopy) with an argon-ion laser at an excitation wavelength of 514.5 nm. The elemental analysis (EA) of the sample was carried out using a Vario EL III equipment (Germany) with a TCD detector to analyze the element of C, H and N. Fourier transform infrared spectroscopy (FT-IR) pattern of the solid samples was measured using KBr pellet technique and recorded on a Nicolet 60-SXB spectrometer from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} . Thermo-Gravimetric/Differential Thermal Analyzer (TG/DTA) was performed on SETSYS-1750 (AETARAM Instruments). About 10 mg of the as-obtained sample was heated in an Al_2O_3 crucible in nitrogen atmosphere from ambient temperature to 650 °C at a constant rise of temperature (10 °C/min). The specific surface area and the pore size distribution were examined by Brunauer–Emmett–Teller (BET) N_2 gas adsorption method at 77 K and carried out on a JW-BK equipment. Pore volume and average pore size were calculated by Barrett–Joyner–Halenda (BJH) formula.

3. Results and discussion

The phases of the samples obtained at different annealed temperatures ranging from 700 to 1000 °C for 2 h were evaluated by XRD, as shown in Fig. 1. All the diffraction peaks from Fig. 1b–d can be readily indexed to the rhombohedral crystalline phase (space group: R-3c 167) of V_2O_3 in agreement with the literature value (JCPDS, no. 34-0178, $a = 4.920 \text{ \AA}$, $c = 13.883 \text{ \AA}$) [33], whose plots are shown in Fig. 1a. These results indicate that VO_2 can be transformed to V_2O_3 at 700–900 °C for 2 h. However, when the reaction temperature increases to 1000 °C, a new phase is observed, as depicted in Fig. 1f. All the new diffraction peaks can be readily assigned to the cubic phase (space group: $Fm-3m$, no. 225) of VC in agreement with the literature value (JCPDS, no. 65-8074, $a = 4.164 \text{ \AA}$) [34], whose plots are represented in Fig. 1e. We also observe that a little of V_2O_3 remains in the sample. Thus, the sample obtained at 1000 °C for 2 h consists of major VC and minor V_2O_3 detected by XRD, which is denoted as VC in the following discussion. The peak located at 27–28° in Fig. 1b–d and f, is unknown due to our current knowledge. However, a relatively weak and broadened peak ranging from

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