

Facile Synthesis of Vanadium Pentoxide Nanorod by Hydrothermal Route and their Characterization

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Abstract: In the present study, we report a simple and reproducible method to produce V_2O_5 nanorod composed of stacked platelets are fabricated through a facile, low-cost, and energy-saving approach. The preparation procedure involves a room-temperature precipitation of precursor in aqueous solution and subsequent calcinations. The structure of the V_2O_5 nanowires was investigated with X-ray diffraction and transmission electron microscopy.

Keywords: Vanadium pentoxide nanorod, hydrothermal synthesis.

1. Introduction

Nanoscale 1D oxide particles have gained increasing technical importance in classical applications such as catalysts, passive electronic components, and ceramic materials [1]. Vanadium pentoxide V_2O_5 is widely used as a catalyst [2], [3] and [4], as a cathode material for solid-state batteries [5], [6] and [7], in windows for solar cells [8] and [9] for electrochromic devices [10] and for electronic and optical switches [11].

Especially, nanostructured V_2O_5 , such as nanotubes, [14] nanoparticles, [12, 13] nanowires, [12, 11] and mesoporous materials, [13] have demonstrated superior lithium insertion/extraction performances. Nonetheless, the associated problems, like low packing density and high reactivity of electrolyte at the electrode surface, always lead to the low volumetric energy density, poor cycling stability, and safety hazards of the battery. [11-14]

Fabrication of hierarchical microspheres composed of nanostructures provides a feasible strategy to overcome these limitations. [2] As a matter of fact, hierarchical hollow V_2O_5 microspheres, [8] porous V_2O_5 microspheres, and yolk-shell V_2O_5 microspheres [10] have been reported with much improved lithium storage properties.

Various chemical methods have been adopted for the

preparation of V_2O_5 nano particles including gas-phase methods, sol-gel methods, evaporative decomposition of solutions, and wet chemical synthesis. Besides, the adopted solvent and vanadium sources are always quite expensive.

In this study, synthesis and characterization of V_2O_5 nanorods have been investigated using simple hydrothermal reaction of vanadium acetate hydrated.

2. Experimental Section

Synthesis of V_2O_5 nanorods

In the first step V_2O_5 nanorods were grown by a simple hydrothermal reaction of vanadium acetate hydrated (Sigma Aldrich, USA) and cetyltrimethyl ammonium bromide (CTAB) (AR Grade, Sigma Aldrich, USA) were used without further purification. CTAB assisted low temperature hydrothermal process: 0.5 g of CTAB and 2 g of sodium hydroxide (NaOH) were dissolved in 50 ml of distilled water to form a transparent solution A under stirring. 3 g of vanadium acetate was dissolved with 50 ml of deionized water to form a transparent solution B. The solution C was transferred into a 100 ml Teflon lined stainless steel autoclave and sealed tightly.

Hydrothermal treatments were carried out at 300°C for 20 h. After that, the autoclave was allowed to cool down naturally. After the reaction, the white products

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were harvested by centrifugation and throughout washing with deionized water, and were finally dried at 50°C in air.

Materials characterization

Crystallographic phases of all the products were investigated by powder X-ray diffraction (Bruker, D8-Advance XRD, $\text{CuK}\alpha$, $\lambda=1.5406 \text{ \AA}$). Morphologies of samples were examined by field-emission scanning electron microscope (FESEM; JEOL, JSM-6700F) and transmission electron microscope (TEM; JEM-2010, 200 kV).

3. Result and Discussion

The XRD pattern of the as-obtained V_2O_5 nanorods are shown in Fig. 1.

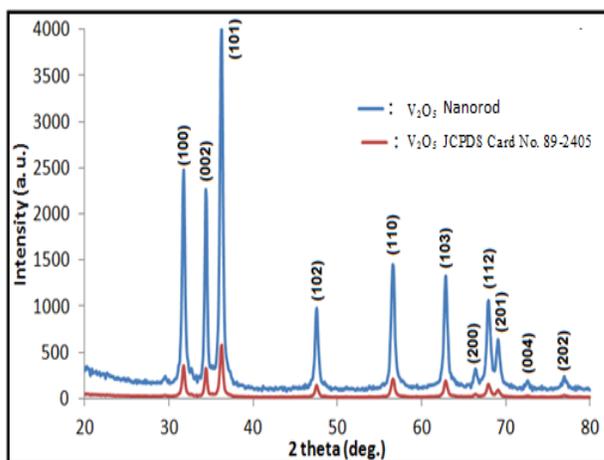


Fig.1: XRD pattern for the V_2O_5 rods and standard V_2O_5 .

All the diffraction peaks could be indexed to hexagonal wurtzite V_2O_5 (JCPDS Card No. 89-2405, $a = 0.3249 \text{ nm}$, $c = 0.5205 \text{ nm}$) with high crystallization. No characteristic peaks were observed for other impurities such as V or $\text{V}(\text{OH})_2$. The crystallites sizes of the V_2O_5 rods and tripods are estimated using the Scherrer formula.

The average values of grain sizes are 18 nm for the V_2O_5 rods. FESEM analysis of the intermediate products performed during the course of growth helps us to understand how the nano rod-assembled. FESEM sample collected at different stages of the hydrothermal process. At the initial stage (reaction time = 30 minutes), nucleation starts and numerous nanoparticle seeds are observed, along with irregular shaped aggregates (Fig. 2a).

As the reaction progresses to 60 minutes, the nanoparticles grow and form individual nano rod structures that are observed as the primary products (Fig. S2b). These primary products, as the petals of the flower, begin to coalesce around the same central core after reaction for 80

minutes (Fig. 2c). As the reaction time prolonged to 100 minutes, self-assembly of the belts continued, which ultimately comprise the final rod (Fig. 2d).

Upon above observations, a plausible mechanism for the evolution of nano rod-assembled is proposed (Fig. 2).

- (i) In the initial stages nucleation of primary nanoparticles and minimize the overall surface energy of the system.
- (ii) Subsequently, growth of the nanoparticles into nanobelts due to their propensity for One-dimensional growth the nanoparticles into nanobelts due to their propensity for accompanying oriented attachment to form nanobelts.
- (iii) Self-assembly of the nanobelts around a central core and formation of nano rod-assembled through the Ostwald ripening process.

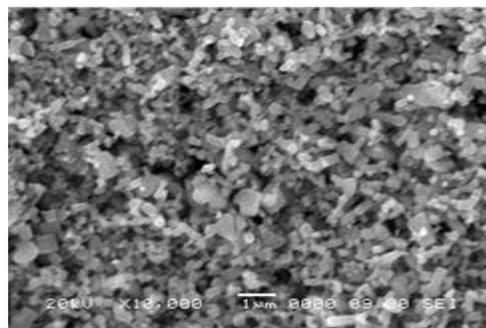


Fig.2 (a)

Typical TEM images of the V_2O_5 nanorods are shown in Fig. 3(a-d).

V_2O_5 nanorods with diameters ranging from 20 to 60 nm have been synthesized conveniently with this method. The length of the V_2O_5 nanorods is about 400 nm, the materials maintained their morphology even after annealing at 500°C for 1 h in air. Fig. 5.3(d) shows the SAED pattern of V_2O_5 rod which reveal that the individual rod is single crystalline in nature preferentially grow along the [0001] direction (the c axis).

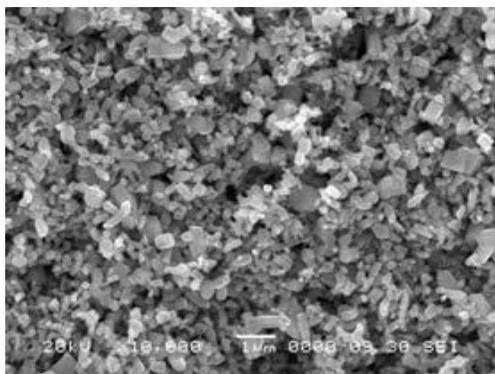


Fig.2 (b)

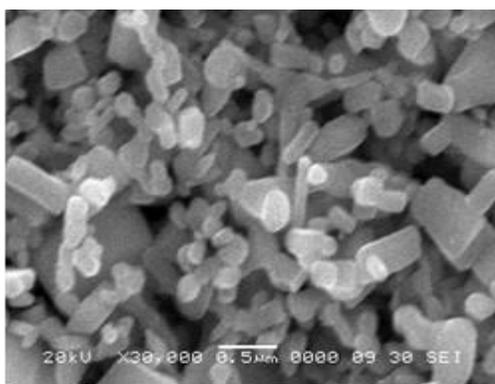


Fig.2 (c)

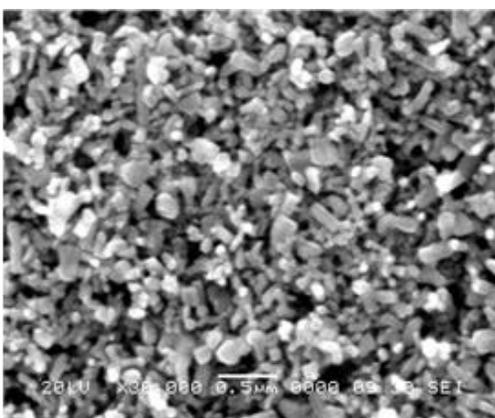


Fig.2 (d)

Fig.2: FE SEM images of the V_2O_5 particle (a, b), and (c, d) V_2O_5 nano rod.

The sharp spots in SAED pattern also indicate that the individual rod of V_2O_5 microstructure is single crystalline in nature. Thus, the TEM result clearly indicates that the ZnO nanorods are wurtzite structure of V_2O_5 .

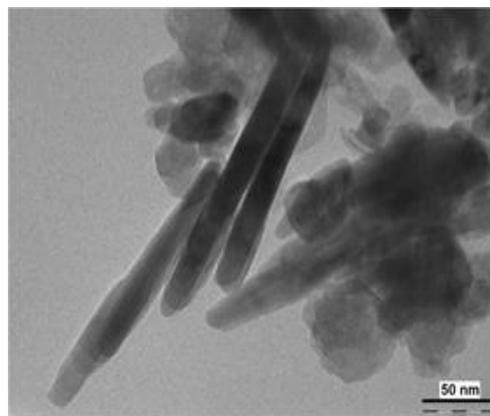


Fig.3 (a)

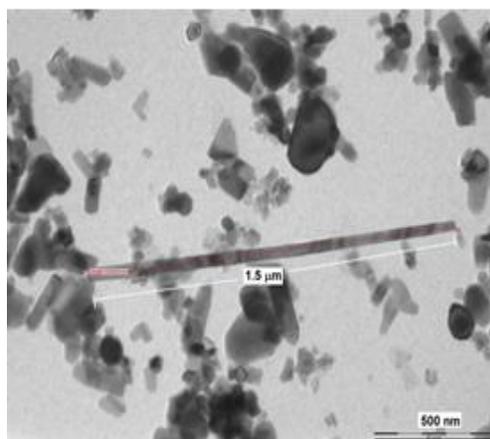


Fig.3 (b)

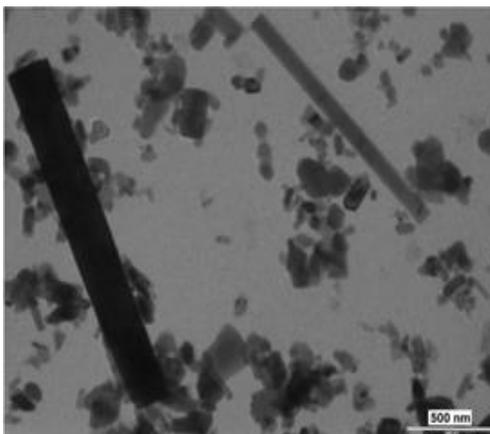


Fig.3 (c)

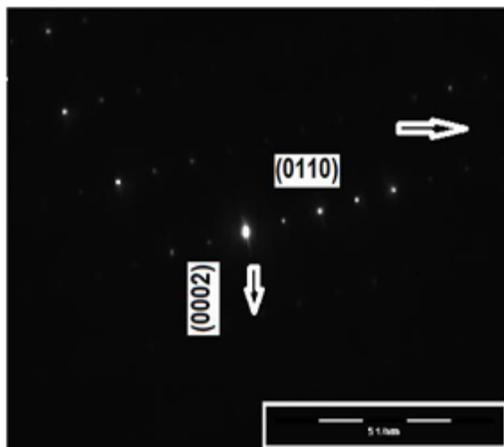


Fig.3 (d)

Fig.3: TEM images of the V_2O_5 nanorods (a-c) and (d) SAED pattern of single rod.

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4. Conclusion

In summary, we have prepared large scale V_2O_5 nanorods with different sizes and shapes have been successfully synthesized via simple hydrothermal route, using vanadium acetate and Cetyltrimmonium bromide (CTAB) as the reactants.

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