Materials Letters 157 (2015) 205–208

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

Synthesis and characterization of single-crystalline vanadium pentoxide by the low-temperature of glycothermal method

Tzu Hsuan Chiang *, Tso-Ming Chen

Department of Energy Engineering, National United University, 2, Lienda, Nan-Shi Li, Miaoli, Taiwan 36003, ROC

ARTICLE INFO

Article history:
Received 16 January 2015
Received in revised form
27 April 2015
Accepted 23 May 2015
Available online 27 May 2015

Keywords:
Ceramics
Structural
Powder technology
Glycothermal

ABSTRACT

Single-crystalline vanadium pentoxide (V_2O_5) powder was prepared by a low-temperature of glycothermal method in which ammonium metavanadate (NH_4VO_3) solution was mixed with ethylene glycol, heated at 100 °C for 1 h, and annealed at 300 °C for 1 h. X-ray diffraction (XRD) and transmission electron microscopy (TEM) characterizations were coordinated with orthorhombic V_2O_5. The smallest rice-shaped V_2O_5 particles, with sizes of 2–3 μm, were obtained when the powders were annealed at 400 °C for 1 h. The low temperature of the glycothermal method used in preparing orthorhombic V_2O_5 was simpler to use than other methods.

© 2015 Elsevier B.V. All rights reserved.

1. Introduction

Currently, there are many methods of preparing V_2O_5 powders, including the hydrothermal method [1,2], solvo-hydrothermal method [3], sol–gel [4], glycothermal [5], electrospinning method [6], and co-precipitation [7]. However, these methods are complex and require expensive equipment. One study was conducted by Ragupathy et al. [8], and they used NH_4VO_3 mixed with ethylene glycol, refluxed the solution for 2 h at 190 °C with constant stirring and purging with N_2 gas until a black-colored vanadyl ethylene glycolate (VEG) precipitate was obtained. Then, the precipitate was calcined at 600 °C for 3 h. In another study by Fu et al. [5], sodium orthovanadate (Na_3VO_4) was mixed with ethylene glycol, and HCl was used to maintain the value of pH = 1. Then, the mixture was heated to a temperature of 120 °C for several hours. A blue powder was formed, and it was calcined at 600 °C for 2 h in an atmosphere of air to form urchin-like microstructures of V_2O_5. The above two methods have the advantage of only taking a short time to prepare the precipitates, but they have the disadvantage that the calcination temperatures had to be higher for the formation of orthorhombic V_2O_5.

In this study, we used a different glycothermal method of preparing V_2O_5 powder than was used by Ragupathy et al. [8] and Fu et al. [5]. The synthesis temperatures and times of the V_2O_5 powders were lower and shorter, respectively, (heating at 100 °C for 1 hr and sintering at 300 °C for 1 h) than those used by Ragupathy et al. [8] and Fu et al. [5]. The lower synthesis temperature and shorter time can save energy and costs. Therefore, this study focuses on used the lower temperature glycothermal reaction to synthesize V_2O_5 powder and simultaneously investigated the characteristics of V_2O_5 powder with respect to different heating temperatures.

2. Experimentation

2.1. Preparation and characterization analysis of V_2O_5

The ammonium metavanadate (NH_4VO_3, supplied by Sigma-Aldrich Co., Ltd.) was dissolved in one part of distilled water at 80 °C for 2 h, and 250 ml of 0.1 M NH_4VO_3 solution was prepared and mixed with 50 ml of ethylene glycol (provided by Showa Chemical Co., Ltd.). When the mixture was heated at 100 °C for 1 h, a precipitate was produced. After centrifugation, the solution was poured off, and the precipitate was washed with distilled water, placed in a dish, and placed in the oven at 80 °C for 24 h. As a result, V_2O_5 hydrate powder was obtained, and it was treated at different sintering temperatures, ranging from 200 to 600 °C for 1 h. The microstructures were observed using JEOL JED 2300 field emission scanning electronic microscopy (SEM), and the selected area electron diffraction (SAED) of the samples was analyzed, and high-resolution transmission electron microscopy (HRTEM) images were obtained using a Philips TECNAI 20 microscope. The X-ray diffraction (XRD) data collected over the 2θ range of 10–100° by a Rigaku TTRAX III rotating anode diffractometer with a Ni-

* Corresponding author. Fax: +886 37382402.
E-mail address: thchiang@nuu.edu.tw (T. Hsuan Chiang).

http://dx.doi.org/10.1016/j.matlet.2015.05.112
0167-577X/© 2015 Elsevier B.V. All rights reserved.

filtered, Cu-K radiation source (wavelength of 1.54 Å). Fourier transform infrared (FTIR) spectra were obtained with a JASCO FT/IR-470 plus spectrometer in the wavelength range from 400 to 4000 cm^{-1} and with a resolution of 4 cm^{-1}. The gas chromatography–mass spectrometry (GC–MS) used a model 5890 A gas chromatograph, a model Trio-2000 mass selective detector, and a HP 5890 II MS chemstation. The extracted compounds were separated using TG-WAXMS capillary column (30 m × 0.25 mm i.d., 0.25 μm film thickness). The GC temperature program was as follows: initial temperature was 50 °C, increased to 250 °C at a rate of 10 °C/min and held for 10 min. The injection temperature was 240 °C, the injection sample volume was 0.1 μl, and the split ratio was 70:1. Helium (99.999%) was used as the carrier gas with a flow rate of 1 ml/min. Electron impact ionization (EI) with nominal electron energy of 70 eV was used.

3. Results and discussion

3.1. Formation of V_2O_5 powders

The ethylene glycol has the ability to oxidize in glycothermal reaction. The reaction mechanism is propounded, the intermediate products as acetaldehyde should be identified by a GC–MS. The result of GC obtained that retention time appeared at 2.99 min for acetaldehyde and 9.76 min for ethylene glycol, as shown Fig. 1(a), which the EI mass spectramolecular ions, $[M]^+\cdot$, were m/z 31, 33, 43 and 62 for ethylene glycol, and m/z 29 and 44 for acetaldehyde, as shown Fig. 1(b). The results obtain confirm some part of ethylene glycol can be oxidized to acetaldehyde. Then acetaldehyde continue to oxidize to acetic acid, as shown in Eq. (2) [9] and acetic acid decomposes to form acetate ions (CH_3COO^-) and hydrogen ions (H^+), as shown in Eq. (3). The NH_4VO_3 consisted of vanadate (VO_3^-) and NH_4^+, as shown in Eq. (3). Then, VO_3^- and H^+ from Eq. (2) reacted to form vanadium pentoxide hydrates ($V_2O_5\cdot H_2O$), as shown in Eq. (4). When the powder was annealed at 300 °C for 1 h and above, orthorhombic V_2O_5 was formed.

$$2CH_2OHCH_2OH + O_2 \rightarrow 2CH_3CHO + 2H_2O \quad (1)$$

$$2CH_3CHO + O_2 \rightarrow CH_3COOH + 2H_2O \rightarrow CH_3COO^- + H^+ \quad (2)$$

$$NH_4VO_3 \rightarrow VO_3^- + NH_4^+ \quad (3)$$

$$2VO_3^- + 2H^+ \rightarrow V_2O_5\cdot nH_2O \xrightarrow{\Delta} V_2O_5 \quad (4)$$

3.2. Effects of temperature on the morphologies

V_2O_5 powders without thermal treatment and after sintering from 200 to 300 °C had irregular cubic shape morphologies, as shown in Fig. 2(a–c). The surface of the powders without thermal treatment were smooth, and the colors of the powders were close to brown-black. When the powders were annealed at 200 and 300 °C for 1 h, the color of the powders changed to brown, and the surfaces of the powders had cracks. When the powders were annealed at 400 °C, the surfaces of the powders decomposed into rice-shaped particles with 2–3 μm as the smallest particles size, as shown in Fig. 2(d), and the color of powders changed to burnt orange. The powders were annealed at 500 °C for 1 h, which caused the particles to increase in size (5–13 μm) until they were larger than the size of the particle annealed at 400 °C, as shown in Fig. 2(e), and the color of the powders changed to orange. When the powders were annealed at 600 °C for 1 h, they formed larger particle sizes of 10–35 μm, and the color of the powders changed to tangerine, as shown in Fig. 2(f). When the powders were annealed at temperatures up to 500 °C, the grains became more closely compacted, which enhanced the surface diffusion and mobility of the species and led to the smaller grains' joining together to grow into larger grains.

3.3. Structure characteristics of V_2O_5 powders

The powders without thermal treatment were sintered at 200–600 °C for 1 h, and the results of XRD are as shown in Fig. 3(a).

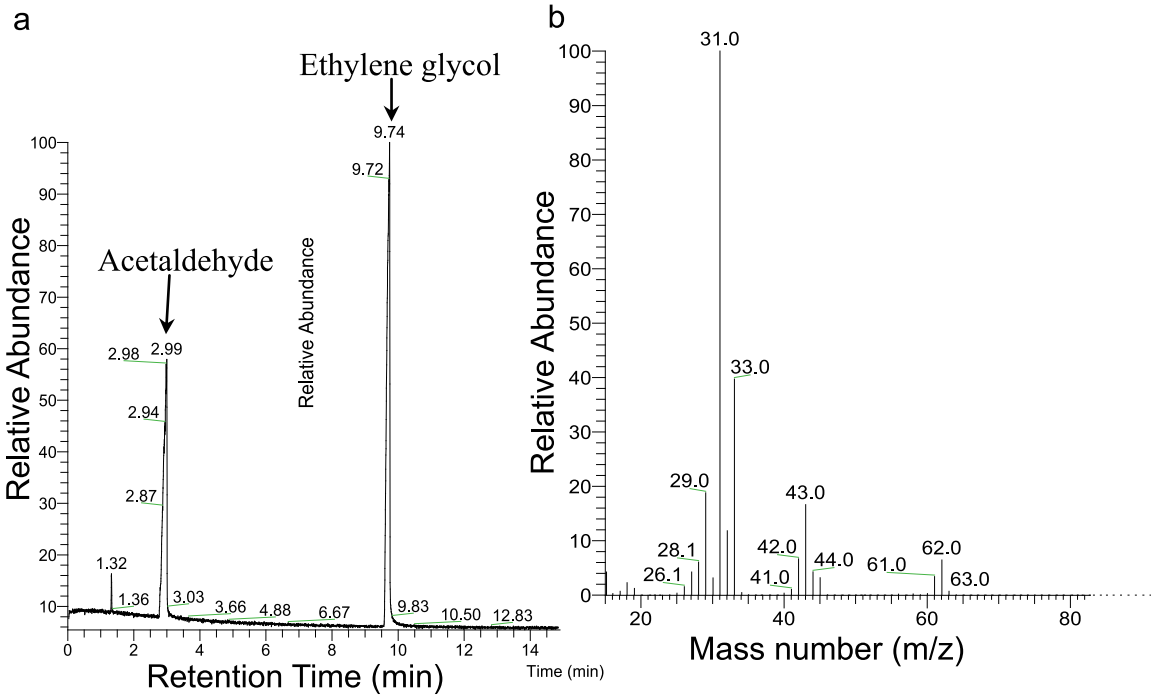

Fig. 1. (a) GC–MS chromatograms (b) Electric Ionization Mass Spectra of mixture.

Download English Version:

https://daneshyari.com/en/article/8018105

Download Persian Version:

https://daneshyari.com/article/8018105

Daneshyari.com