Chinese Journal of Catalysis 36 (2015) 432–438

available at www.sciencedirect.com

ScienceDirect

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

Article

Hydrothermal synthesis and photocatalytic properties of tantalum pentoxide nanorods

Juxia Li, Weili Dai *, Junqing Yan, Guangjun Wu, Landong Li, Naijia Guan #

Key Laboratory of Advanced Energy Materials Chemistry (Ministry of Education), College of Chemistry, Nankai University, Tianjin 300071, China

ARTICLE INFO

Article history:
Received 24 July 2014
Accepted 29 August 2014
Published 20 March 2015

Keywords:
Tantalum pentoxide
Nanostructure
Crystal growth
Photocatalytic property
Anisotropic growth

ABSTRACT

Tantalum pentoxide (Ta_2O_5) nanorods were hydrothermally synthesized using polyethylene glycol (PEG) as a guiding agent. The nanorods were characterized by X-ray diffraction, scanning and transmission electron microscopies, and diffuse reflectance ultraviolet-visible and photoluminescence spectroscopies. The effects of crystallization duration and $Ta_2O_5/Sr(OH)_2$ ratio on the product morphology were investigated, and a growth mechanism was proposed. Phase-pure Ta_2O_5 nanorods with controlled morphology were formed in the presence of PEG and $Sr(OH)_2$, which was necessary to form the nanorods. $Sr(OH)_2$ induced the surface dissolution and re-growth of Ta_2O_5. PEG induced the anisotropic growth of Ta_2O_5 by acting as a capping agent. The products were used to photocatalytically degrade rhodamine B under ultraviolet irradiation. The catalytic activity directly correlated with the length-diameter ratio of the Ta_2O_5 nanorods. A maximum apparent reaction rate constant of 0.156 min^{-1} was obtained. The Ta_2O_5 nanorods were stable during photocatalytic reaction and could be recycled several times without loss of activity.

1. Introduction

Semiconductor photocatalysis has attracted much interest for its potential in air pollution, solid waste and water pollution treatments [1–4]. Various semiconductors have been explored, with tantalum pentoxide (Ta_2O_5) being attractive because of its high photocatalytic activity, dielectric constant, refractive index, and chemical stability [5–10]. Nanomaterials with well-defined crystal structures and morphologies have been applied in various areas because of their size and structure-dependent properties [11–13]. Much effort has focused on one-dimensional (1D) nanostructures, such as nanorods and nanowires [14–24]. These exhibit different physical and chemical properties to their bulk counterparts.

Nanoparticulate Ta_2O_5 has been prepared by sol-gel [25,26], hydrothermal [27–29], solvothermal [30,31], hot filament metal vapor deposition [32], and microemulsion [33,34] methods. Most of these methods yield poorly crystalline Ta_2O_5 nanoparticles. Subsequent high-temperature calcination is also generally required to yield the final product, which can lower the surface area and decrease the catalytic activity. Hydrothermal synthesis can yield nanostructures with high crystallinity, high purity and narrow size distribution [35]. The product morphology can be controlled by adjusting the hydrothermal synthesis conditions. A few tantalates have been hydrothermally prepared at low temperature [36–41]. The hydrothermal synthesis of nanoparticulate Ta_2O_5 is difficult because Ta_2O_5 is much more inert than TiO_2 [34,42]. Ta_2O_5 nanowires [27] and nanoflowers [28] were recently hydrothermally prepared using hydrofluoric acid, which is impractical for green syntheses and

* Corresponding author. Tel: +86-22-23509140; Fax: +86-22-23500341; E-mail: weilidai@nankai.edu.cn
# Corresponding author. Tel: +86-22-23509140; Fax: +86-22-23500341; E-mail: guannj@nankai.edu.cn
This work was supported by the Collaborative Innovation Center of Chemical Science and Engineering (Tianjin).
DOI: 10.1016/S1872-2067(14)60215-1 | http://www.sciencedirect.com/science/journal/18722067 | Chin. J. Catal., Vol. 36, No. 3, March 2015

industrial application. Gömpel et al. [29] hydrothermally prepared Ta_2O_5 nanorods without using hydrofluoric acid, instead using an expensive tantalum(V) *n*-butoxide source. A hydrothermal synthesis of Ta_2O_5 nanoparticles using an economical Ta source in the absence of hydrofluoric acid is required.

In the present study, 1D Ta_2O_5 nanorods with tunable morphology were hydrothermally synthesized with the assistance of polyethylene glycol (PEG) and $Sr(OH)_2$. Ta_2O_5 powder was used as the Ta source. The effect of the synthesis parameters on the product morphology was investigated, and a growth mechanism was proposed. The photocatalytic performance of the Ta_2O_5 nanorods in the photodegradation of rhodamine B (RhB) under UV irradiation was investigated. The nanorods exhibited high activity, a high apparent reaction rate constant, and good recyclability.

2. Experimental

2.1. Hydrothermal synthesis of Ta_2O_5 nanorods

All chemicals were purchased from Alfa Aesar and used as received. In a typical synthesis, 2 mmol of Ta_2O_5 powder (99.99%) was dispersed in anhydrous ethanol, and a solution of $Sr(OH)_2$ was added dropwise under stirring. PEG (M_w = 300, 50 mg) with certain deionized water was added. The mixture was transferred to a polytetrafluoroethylene-lined autoclave (30 mL) for static crystallization at 200 °C for 5, 24, or 48 h. The resulting precipitate was collected by centrifugation and thoroughly washed with deionized water and ethanol. The product was dried at 80 °C overnight to yield Ta_2O_5 nanorods, which were denoted by Ta_2O_5-*x*-*y*, where *x* is the $Ta_2O_5/Sr(OH)_2$ molar ratio used during synthesis, and y is the reaction duration.

2.2. Characterization

X-ray diffraction (XRD) patterns were recorded on a Bruker D8 ADVANCE powder diffractometer using Cu-K_α radiation (λ = 0.1542 nm) at a scanning rate of 12°/min at 2θ = 10°–80°. Specific surface areas were determined through low-temperature N_2 adsorption-desorption isotherms, collected on a Quantachrome iQ-MP gas adsorption analyzer. Samples were dehydrated at 300 °C for 2 h prior to measurement. The total surface area was calculated via the BET equation. The morphologies and compositions were examined by field emission scanning electron microscopy (FE-SEM, Hitachi S-4800). Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) images were collected on a Philips Tecnai G2 20 S-TWIN microscope at 200 kV. A few drops of a sample suspension in ethanol were allowed to evaporate on a carbon-coated copper grid at ambient temperature. Ultraviolet-visible (UV-vis) diffuse reflectance spectra were recorded in the air against $BaSO_4$ at 200–700 nm using a Varian Cary 300 spectrophotometer. Photoluminescence (PL) spectra were recorded on a Spex FL201 fluorescence spectrophotometer. Samples were dry-pressed into self-supporting wafers and then excited by a 325-nm He–Cd laser at ambient temperature.

2.3. Photocatalytic degradation of RhB

The photocatalytic degradation of RhB was performed in a top-irradiation-type double-walled quartz cell cooled by water. A 250 W mercury lamp (λ_{max} = 365 nm) was used as the light source. About 0.1 g of catalyst was added to 200 mL of RhB solution (10 mg/L) in the quartz cell. The suspension was stirred in the dark until the RhB concentration was constant (~30 min), which indicated adsorption equilibrium. After commencing the photocatalytic reaction, aliquots were removed at regular time intervals and analyzed by UV-vis absorption spectrometry (Varian Cary 300).

3. Results and discussion

3.1. Effect of synthesis parameters on product morphology

The presence of PEG and $Sr(OH)_2$ during the hydrothermal synthesis of Ta_2O_5 nanostructures affects their resulting morphology. Figure 1 shows that irregular nanoparticles of large (~2 μm) and small (~200 nm) size were formed in the absence

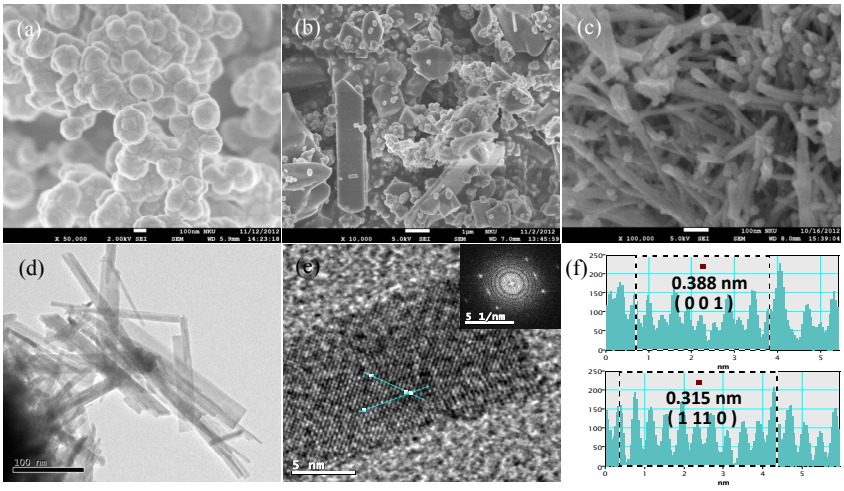

Fig. 1. SEM image of raw material Ta_2O_5 (a), synthesis product without PEG (b), and synthesis product with PEG (c); TEM (d) and HRTEM (e) images of synthesis product with PEG; (f) lattice analysis of TEM image. Hydrothermal synthesis conditions: $Ta_2O_5/Sr(OH)_2$ = 1/1, 200 °C, 48 h.