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Antimony trioxide microstructures: 3D grass-like architectures and optical properties

Limin Song[a], Shujuan Zhang[b,*,1], Qingwu Wei[a]

[a] College of Environment and Chemical Engineering & State Key Laboratory of Hollow-Fiber Membrane Materials and Membrane Processes, Tianjin Polytechnic University, Tianjin 300160, PR China
[b] College of Science, Tianjin University of Science & Technology, Tianjin 300457, PR China

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ABSTRACT

Antimony trioxide (Sb_2O_3) micro and nanostructures with various shapes have shown novel physical and chemical properties essential for technological applications. In the present study, we report the hydrothermal growth of a novel three-dimensional (3D) Sb_2O_3 grass-like microstructure by the direct reaction between $SbCl_3$ and NaOH in a solution. The as-prepared 3D Sb_2O_3 microstructures consist of numerous microbelts with sharp tips and lengths of up to approximately 10 μm, as well as widths of around 1.0 μm. Other tunable architectures, including rose-like, cubic, belt-like, and bundle-like units, have also been prepared by changing the experimental conditions. The growth mechanism of Sb_2O_3 microstructures with grass shapes is speculated and analyzed in detail. Their optical properties are also surveyed using photoluminescence (PL) spectroscopy. A broad PL emission is revealed, suggesting potential applications in electronics and optoelectronics.

1. Introduction

Over the past decades, micro and nanostructured materials with various shapes, have attracted a great deal of attention because of their potential applications in micro and nanoscale devices [1,2]. Such shapes include belts, wires, tubes, walls, trees, flowers, and so on. Simultaneously, processes that fine tune the size and shape of these materials have gained considerable interest for developing complex multifunctional materials due to their expanding properties. Many efforts have been made to transform different morphologies of micro and nanoscale materials into complex structures [3–5]. Sb_2O_3 is an important semiconducting material widely used as a catalyst, conductive material, and functional filler. Sb_2O_3 is also used in optical, optoelectronic, and magnetic materials. The highly efficient flame-retardant synergistic capability of Sb_2O_3 makes it invaluable in plastics, paints, adhesives, and so on [6,7]. To date, several methods for synthesizing Sb_2O_3 materials have been reported [8]. The methods include hydrothermal [9–12], vacuum evaporation [13], γ-ray radiation–oxidization [14], hybrid induction and laser heating [15], and thermal reduction [16,17] and oxidation [18]. However, the selective synthesis of complex three-dimensional (3D) Sb_2O_3 microstructures with various shapes remains a challenge.

In the past few years, the preparation of Sb_2O_3 using the Sb^{3+} ions and NaOH as the raw materials by hydrothermal method has been reported widely [9–12], but the synthesis of Sb_2O_3 with 2D or 3D structures are rarely reported by fine-tuning experimental conditions. In the current work, we directly synthesized high-quality Sb_2O_3 microstructures with various shapes via a simple hydrothermal route. The changed microstructures include 3D grass-like, rose-like, cubic, belt-like, and bundle-like Sb_2O_3. The influences of different reaction parameters, including concentration, solvent, surfactant, reaction temperature, and time, on the size and shape of the formed Sb_2O_3 products were investigated in detail. The possible mechanisms leading to 3D grass-like Sb_2O_3 microstructures were proposed. Photoluminescence (PL) spectroscopy was used to study their emission properties, and the results revealed their potential applications in light-emission micro and nanodevices.

2. Experimental

2.1. Chemicals

All the reagents were purchased from Tianjin Guangfu Chemical Reagent Company. All the reagents used in the experiments were analytical grade and used without further purification. Double distilled water was used throughout the experiments.

* Corresponding author. Tel.: +86 22 60600658; fax: +86 22 60600658.
E-mail address: zhangshujuan@tust.edu.cn (S. Zhang).
[1] Co-first author.

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Table 1
Sb_2O_3 morphology obtained under different reaction conditions.

$SbCl_3$ (mol/L)	NaOH (mol/L)	Surfactant/solvent	T (°C)	Reaction time (h)	Morphology and shape
0.05	1.5	Distilled water	180	24	3D grass-like microstructures
0.05	0.75	Distilled water	180	24	Ultrahigh-aspect-ratio microwires
0.05	3	Distilled water	180	24	Ultrahigh-aspect-ratio microwires
0.05	4.5	Distilled water	180	24	Microbelts
0.05	6	Distilled water	180	24	Nanocubes
0.05	1.5	Distilled water	160	24	Microbelts
0.05	1.5	Distilled water	200	24	Bundle-like microbelts
0.05	1.5	Methanol	180	24	Rose-like microstructures
0.05	1.5	Cetyltrimethylammonium bromide (CTAB)/distilled water	180	24	Bundle-like microbelts
0.05	1.5	Sodium dodecyl sulfate (SDS)/distilled water	180	24	Bundle-like microbelts
0.05	1.5	Distilled water	180	3	Microbelts
0.05	1.5	Distilled water	180	9	Microwires
0.05	1.5	Distilled water	180	15	Grass-like microstructures consisting of microwires

2.2. Synthesis of 3D grassr-like Sb_2O_3 microstructures

In a typical hydrothermal procedure for 3D grassr-like Sb_2O_3 microstructures, 0.17 g (0.05 mol/L) $SbCl_3$ and 0.9 g NaOH (1.5 mol/L) were dissolved in 15 mL of deionized water under continuous stirring, and then a Cd sheet (1 cm × 1 cm) was placed into the above solution to collect products. The Cd sheet surface polished with sandpaper. The Cd sheet was then washed with deionized water to neutral pH and dried in air. The above mixture was transferred into a 25 mL Teflon-lined autoclave. The solution was heated to 180 °C, and maintained for 24 h. After completing the reaction, the autoclave cooled down naturally. The Cd sheet at the bottom of the autoclave was collected and washed repeatedly with water and alcohol to remove the unreacted ions. Finally, the resulting Cd sheet was dried in air.

2.3. Characterization of 3D grass-like Sb_2O_3 microstructures

The phase composition of the as-prepared product was determined using a Rigaku D/max 2500 powder diffractometer (XRD) with a Cu Kα radiation of wavelength of 1.5406 Å and were analyzed from 25° to 65° (2θ) with a graphite monochromator. The morphology and size of the as-prepared products were observed by a Hitachi S-4800 scanning electron microscope (FESEM) with energy-dispersive X-ray spectroscopy (EDS). Microstructure of the products was characterized by a Hitachi 7650 transmission electron microscope (TEM) operated at an accelerating voltage of 100 kV. The groups on the samples were studied by infrared absorption spectroscopy using a Bruker Tensor37 Fourier transform infrared spectrometer (FT-IR). UV/vis spectra were recorded on a HP8453 spectrophotometer at room temperature. The ability of emission

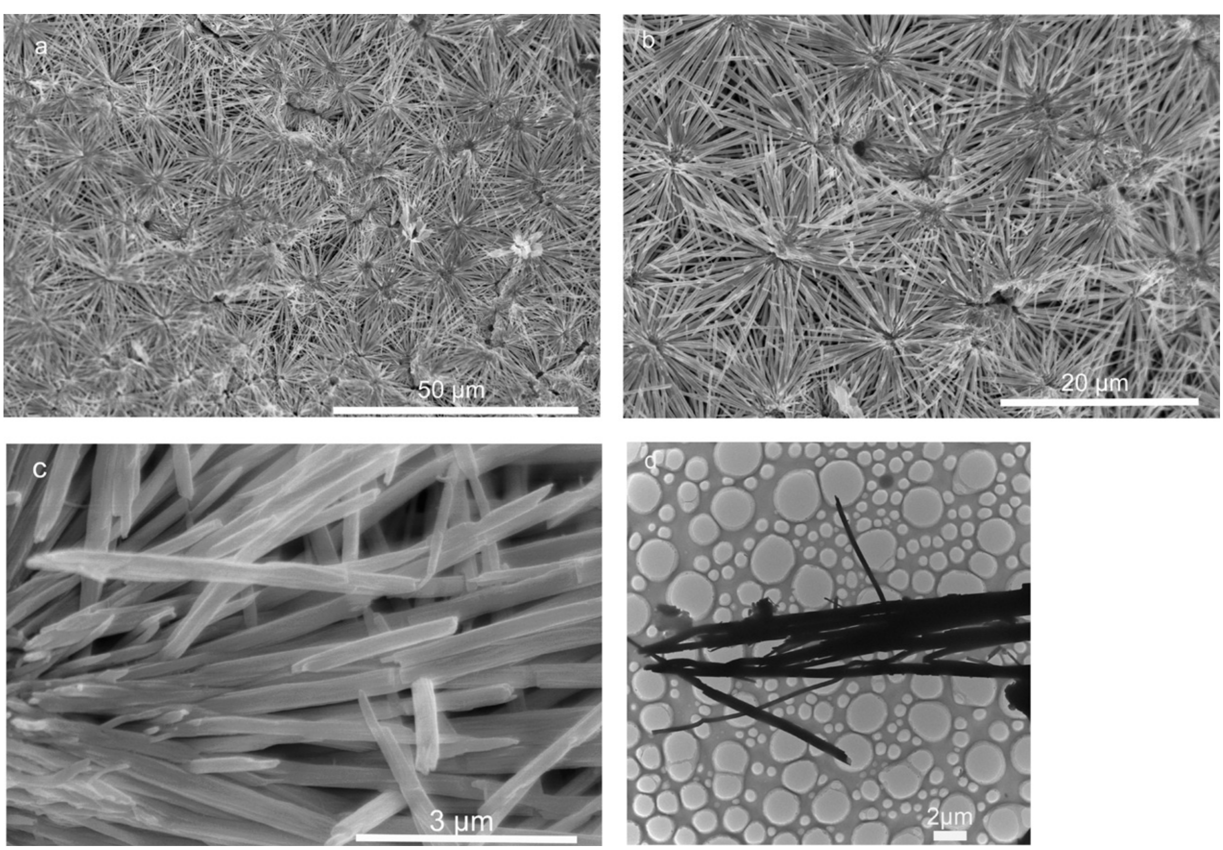

Fig. 1. FESEM and TEM images of typical 3D grass-like Sb_2O_3 microstructures at 180 °C for 24 h (0.05 mol/L $SbCl_3$ and 1.5 mol/L NaOH).