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Fabrication of single-crystalline Co_3O_4 nanorods via a low-temperature solvothermal process

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Abstract

Co_3O_4 nanorods with average diameter and length of ~50 nm and 1 μm were successfully prepared via a simple surfactant-assisted solvothermal method at 160 °C for 12 h. The formation of Co_3O_4 nanorods is attributed to alcoholysis of cobalt ions dispersed in ethanol in the presence of a capping agent—CTAB. The composition and purity of the sample were characterized by X-ray diffraction (XRD). Transmission and scanning electron microscopy images show that the particles are homogenous and have the shape of rods. The mechanism of forming Co_3O_4 nanorods is also discussed.
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Keywords: Nanorods; Solvothermal; Cobalt oxide; Chemical synthesis

1. Introduction

In the past few years, one-dimensional (1D) nanomaterials have attracted considerable attention due to their unique optical, electrical and magnetic properties and potential applications in nanodevices [1–3]. Intensive work has been directed towards the synthesis of 1D nanomaterials, such as nanorods, nanowires and nanotubes. Co_3O_4, a mixed valence compound with a normal spinel structure, is the stablest phase in the Co–O system and one of the most important transitional metal oxides that has a gas-sensing behavior and solar energy reflecting properties [4,5]. Due to its function in the reduction of SO_2 with CO [6,7], ammonia oxidation [8] and the reduction of NO with methane [9], cobalt oxide can also be used as an effective catalyst in environmental protection and chemical engineering process. Furthermore, it is also a traditional precursor of an anode material in Li-ion rechargeable battery [10].

Various methods have been developed to prepare Co_3O_4, such as sol–gel route, reduction–oxidation route, gel hydrothermal oxidation, homogeneous precipitation, chemical spray pyrolysis, chemical vapor deposition and cobalt salt decomposition [11–17]. However, most of the attention has been focused on the synthesis of cobalt oxide nanoparticles. Because the properties and applications of Co_3O_4 are greatly influenced by its size, shape and size distribution, preparation of cobalt oxide

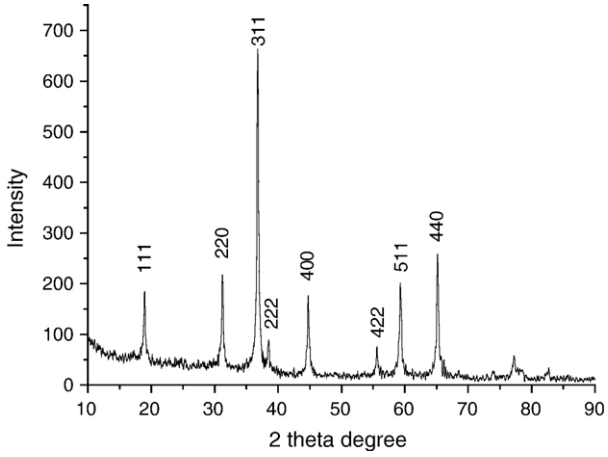

Fig. 1. XRD pattern of as-prepared Co_3O_4 nanorods.

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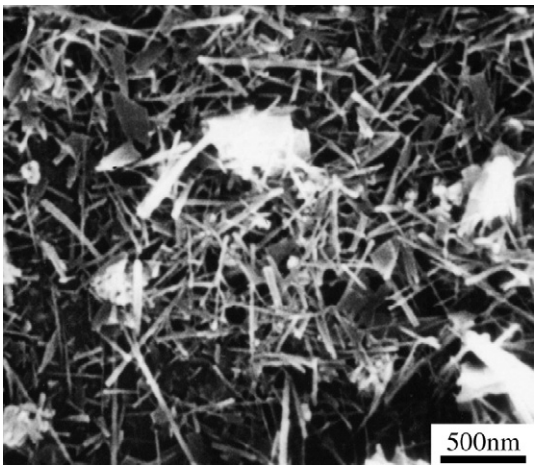

Fig. 2. SEM image of Co_3O_4 nanorods.

of different sizes and morphologies including tubes, rods, films, hollow spheres and cubic single crystals in nanoscale was also intensively conducted [18–23]. However, reports of 1D cobalt oxide are relatively few [19,20]. Furthermore, high temperature and complex steps are often needed in their synthesis. Then it will be of great significance if temperature could be decreased and steps be simplified. Heath and LeGoues pioneered the use of solvothermal synthesis for generating semiconductor nanowires [24]. This method was later exploited by Qian et al. to process a rich variety of materials [25]. In this letter, a moderate temperature solvothermal strategy was applied for the growth of single-crystalline Co_3O_4 nanorods with the assistance of surfactants.

2. Experimental

All chemicals used in this work were of analytical reagent grade, obtained from the commercial market and used without further purification. X-ray diffraction patterns were measured using a Rigaku D/max-IIB X-ray diffractometer at a scanning rate of 4° per minute with 2θ ranging from 10 to 90°, using Cu Kα radiation ($\lambda = 1.5418$ Å). Photomicrographs were obtained using a JEM-2010 transmission electron microscope (TEM), working at 160 kV and a JEOL JSM-840 scanning electron microscope (SEM) working at 20 kV.

The experimental process is very simple. In a typical process, 1.1 g, 1.2 g, 1.3 g, 1.4 g and 1.5 g of $Co(NO_3)_2 \cdot 6H_2O$ were dissolved in 10 ml absolute ethanol respectively, then 1.0 g of CTAB was added to the solutions. The mixture was magnetically stirred until it became homogenous. Then they were transferred into 18 ml autoclaves, sealed and kept at 160 °C for 24 h. After that, the autoclaves were allowed to cool to room temperature naturally. The obtained precipitation was then filtered, washed with absolute ethanol to remove ions and possibly remaining surfactant in the final products, and dried at 80 °C in air for 6 h. Control experiment was done similarly by varying the amount of CTAB at 0 g, 0.5 g and 1.0 g.

3. Results and discussion

Fig. 1 shows the XRD pattern of Co_3O_4 nanorods. All the diffraction peaks can be indexed to a cubic phase with lattice constant of $a = 8.0722$ Å. No impurity peaks are observed.

Fig. 2 is a typical SEM image of Co_3O_4 nanorods. As can be seen, the sample was composed of uniform rod-like structures with diameter of 50 nm and length of 1 μm. JEM-2010 transmission electron microscope at 160 kV was also employed to examine the morphology of the nanorods. Samples were prepared by placing drops of diluted ethanol dispersed on the nanocrystalline surface of cooper grids, which were purchased commercially. Fig. 3a gives the TEM image of the sample, clearly confirming that the products consist of uniform rod-like Co_3O_4 particles and the diameter and length of the nanorods are in good agreement with those observed in SEM. SAED pattern (Fig. 3b) obtained by focusing the electron bean on an individual nanorod indicates its single crystal nature.

As is known some oxygen can be dissolved in water, it is in charge of the oxidation of Co(II). Keeping the amount of ethanol and CTAB at

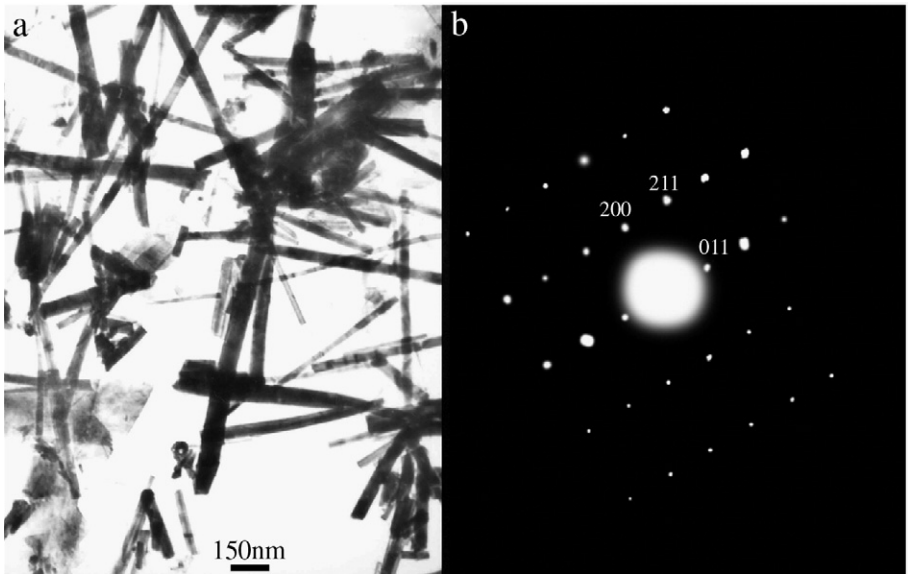

Fig. 3. a. TEM image of Co_3O_4 nanorods. b. SAED pattern of Co_3O_4 from a single nanorod.

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