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Nanostructured hexagonal cobalt oxide plates and their electrochemical properties

Xing Zhou [a,b,*], Feng Chen [a], Feng Cao [a], Wei Shen [a], Jiali Liu [a], Xingwang Xu [a]

[a] School of Chemistry, Biology and Materials Engineering, Suzhou University of Science and Technology, Suzhou 215009, China
[b] Shanxi Province Key Laboratory of Functional Nanocomposites, North University of China, Taiyuan 030051, China

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ABSTRACT

Hexagonal Co_3O_4 plates have been synthesized using a simple solvothermal method by tuning the NaOH concentration and initial Co^{2+} ion sources. XRD and HRTEM analyses revealed pure phase of Co_3O_4 plates. SEM and TEM studies showed the Co_3O_4 materials possess hexagonal shapes with average thickness of 23 nm and edge length of 110 nm approximately. The effects of reacting NaOH concentration and initial Co^{2+} ion sources were investigated by a series experiments. It was found that the NaOH concentration played an important role for the formation of such novel cobalt oxide. When the NaOH concentration of the mixture was as low as 0.2 M, the morphology of final products can changed from hexagonal plates to particles. The observed better electrochemical properties of the hexagonal Co_3O_4 plate were attributed to the unique hexagonal plate architecture. Due to the specific structure and electrochemical properties, these hexagonal Co_3O_4 plates are expected to have potential applications as candidates for catalysis, sensor and energy storage.

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1. Introduction

The control over the morphology of the materials with well defined shape and uniform size remains an important goal of present synthetic chemistry, because shape- and size- controlled nanomaterials are promising candidates as active components in a wide range of technological applications [1,2]. On this basis, much attention has been paid to synthesize nanomaterials with novel shapes which possess attractive physical and chemical properties [3–5]. In the past decade, tremendous research efforts have been directed to the synthesis of size- and shape- selective metal-oxide nanomaterials, because of its wide range of applications [6–10].

Cobalt oxide (Co_3O_4) nano-materials have been widely studied due to their excellent properties and potential applications as electrochemical, magnetic, and catalytic application [11–13]. For instance, supercapacitors, various sensors, CO oxidation, oxidation of trace ethylene, etc., because of their exceptional chemical and physical properties [14–16]. For further promoting their performance, many efforts have been paid toward the fabrication of various Co_3O_4 nano-structures with nanoparticles, nanotubes, nanorods, nanosheets, and mesoporous nanowires [17–23]. A lot of investigations have discovered that there is a close correlation between shape and performance of Co_3O_4. For example, Wang et al. successfully prepared mesoporous Co_3O_4 materials, which showed a high specific capacitance of 427 F g^{-1} at charge/discharge current density of 1.25 A g^{-1} [11]. Xia et al. prepared mesoporous Co_3O_4 monolayer hollow-sphere array by electrodepositing from aqueous solution containing cobalt precursor and exhibits a specific capacitances of 358 F g^{-1} at 2 A g^{-1} [24]. However, it is still challenging to develop facile, simple, and reliable synthetic approaches for hexagonal cobalt oxide plates with designed shapes.

Herein, we demonstrate a simple solvothermal method to synthesize hexagonal cobalt oxide plates depending on the control over reaction conditions. Our method is a direct synthesis method of Co_3O_4 via the production of $Co(OH)_2$ as an intermediate. These as-prepared hexagonal cobalt oxide plates were composed of many nanoparticles. The crystal structure, morphology, and electrochemical performances of the hexagonal cobalt oxide plates were further investigated carefully.

2. Experimental procedure

2.1. 1 Materials and methods

All reagents were analytical grade and used without further purification. In a typical synthesis, 4 mmol of $Co(NO_3)_2 \cdot 4H_2O$ and 0.6 mmol of Cetyltrimethyl Ammonium Bromide (CTAB) were

* Corresponding author at: School of Chemistry, Biology and Materials Engineering, Suzhou University of Science and Technology, Suzhou 215009, China.
E-mail address: xzhou@mail.usts.edu.cn (X. Zhou).

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dissolved in 60 mL of ethanol with intensive stirring to form a burgundy homogeneous solution. Then 0.4 M of aqueous NaOH solution was added by dropwise into above solution at room temperature until the pH value was 12, during which dark green solution appeared. The mixture was then heated to 65 °C for 45 min with mechanical stirring under reflux conditions. After that, the mixture was sealed in a 100 mL Teflon-lined stainless steel autoclave at 190 °C for 6 h. After the reaction was completed, the resulting black products were collected, rinsed three times with distilled water and EtOH, and then vacuum-dried at 50 °C for 4 h. The obtained black powder was placed into an alumina boat, and heated in a furnace to 350 °C with a rate of 2 °C min^{-1}, and maintained for 3 h under atmospheric pressure.

2.2. Characterizations

The morphology and structure of the samples were investigated by scanning electron microscopy (SEM, Hitachi, S3400N) and transmission electron microscopy (TEM, Hitachi, HT7700, 100 kV). The crystal structure of the samples were recorded with X-ray powder diffraction (XRD, Bruker, D8 Advance, Cu-Kα radiation, λ=1.5406 Å). High resolution transmission electron microscopy (HRTEM) image were taken on a Hitachi HT-7700 transmission electron microscope at an accelerating voltage of 100 kV. Electrochemical measurements were carried out using an electrochemical working station (RST5200, Suzhou, China) in a three-electrode at room temperature. 3 M aqueous solution of KOH was used as electrolyte. A platinum foam electrode (1 cm × 2 cm) and a saturated calomel electrode (SCE) served as the counter electrode and reference electrode, respectively.

3. Results and discussion

The structural composition details of the products obtained from 190 °C for 6 h have been studied by XRD analyses. Fig. 1a showed the XRD results for the successful synthesis products. Several diffraction peaks at 2 theta=31.01°, 36.58°, 37.75°, 44.62°, 55.39°, 59.13°, 65.05°, and 68.42° correspond to the (220), (311), (222), (400), (422), (511), (440), and (531) crystal planes of the cobalt oxide (Co_3O_4, JCPDS No. 42-1467), respectively. There are no other peaks for impurities, indicating the acquisition of Co_3O_4 sample with a pure phase [22]. Fig. 1b displayed a representative SEM image of the sample with a panoramic view, from which the hexagonal cobalt oxide plates were observed on a large scale. The magnified SEM image (Fig. 1c) indicated that the Co_3O_4 contains uniformly dispersed hexagonal plate like with average thickness of 23 nm and edge length of 110 nm approximately, respectively. The microstructure of these Co_3O_4 nanowires was further investigated by TEM technique. As shown in Fig. 1d, the image was taken from a few arbitrary-selected hexagonal Co_3O_4 plates. The edge length was about 60–120 nm, and a large amount of interconnected nanoparticles could be clearly seen in the magnified TEM image (Fig. 1e). The fine structure of the hexagonal plates has been studied by high-resolution TEM (HRTEM). Fig. 1f shows the corresponding HRTEM taken from the circled position (Fig. 1e) of the as-prepared sample. The lattice spacing was calculated to be 0.244, 0.242 and 0.233 nm, correspond to the lattice planes of (222), (222), and (311) in Co_3O_4. Many lattice spacings with different orientations could be observed, demonstrating very good crystallinity. On the other hand, the HRTEM results also shown that the hexagonal Co_3O_4 plates were composed of nanoparticles.

The control of structure morphology can be realized by adjusting several parameters, such as the concentration of NaOH and the initial Co^{2+} ion sources. To study the hexagonal plate formation mechanism, the cobalt oxide samples have been prepared with different NaOH concentrations and investigated in detail to reveal the relationship between the morphology and the alkali concentration. The SEM images of the as-prepared samples are shown in Fig. 2. The sample obtained using NaOH concentration

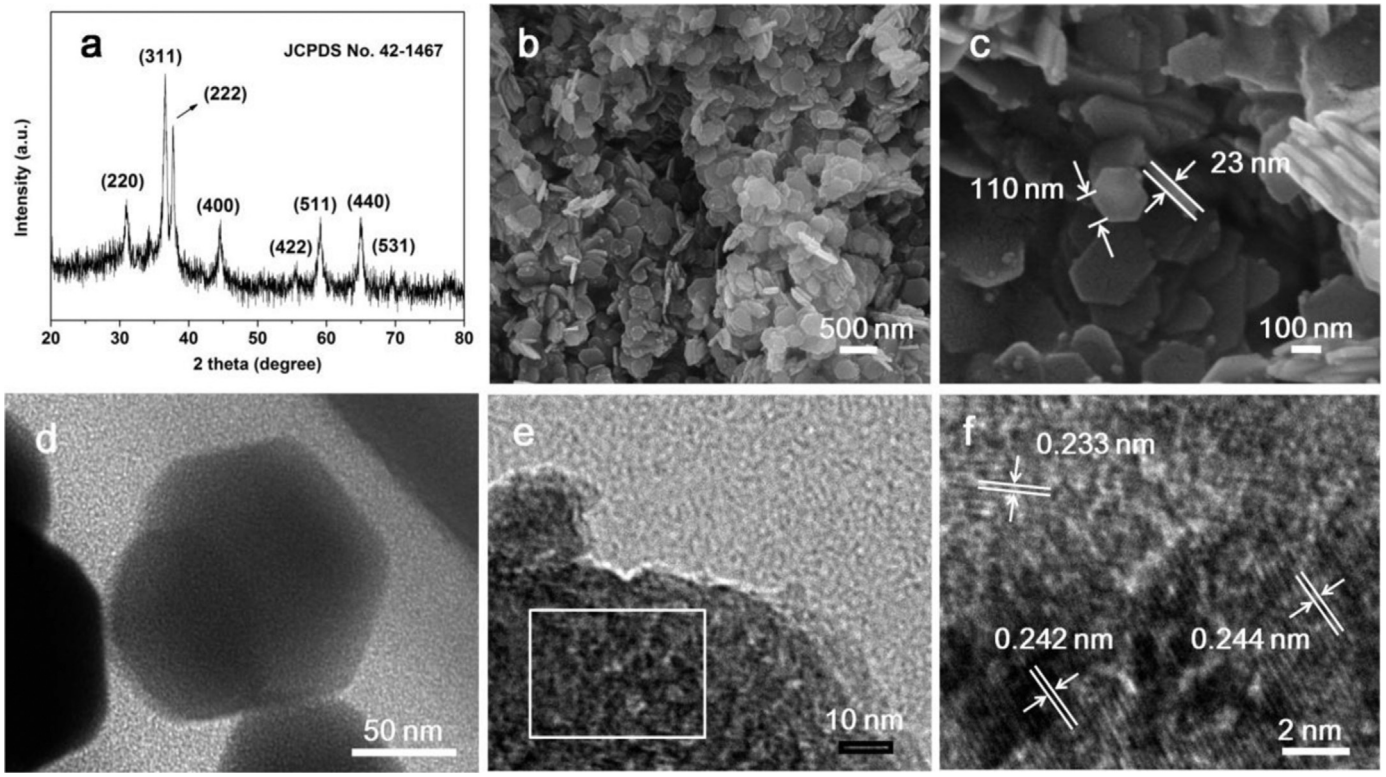

Fig. 1. XRD pattern (a), Low-magnification (b), and high-magnification (c) SEM images, TEM image (d), magnified TEM image (e), and HRTEM image (f) from the circled position in (e) of the as-prepared sample.

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