Superlattices and Microstructures 48 (2010) 154–161

Contents lists available at ScienceDirect

Superlattices and Microstructures

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

A facile and novel synthetic route to $Ni(OH)_2$ nanoflowers

Zhipeng Cheng [a,*], Jiming Xu [a], Hui Zhong [a], Dong Li [a], Pengcheng Zhu [a], Yi Yang [b]

[a] Jiangsu Key Laboratory for Chemistry of Low-Dimensional Materials, Huaiyin Normal University, Huaian 223300, China

[b] National Special Superfine Powder Engineering Research Center, Nanjing University of Science & Technology, Nanjing 210094, China

ARTICLE INFO

Article history:
Received 15 June 2009
Received in revised form
13 April 2010
Accepted 19 May 2010
Available online 15 June 2010

Keywords:
Nanomaterials
Microstructure
$Ni(OH)_2$
Nanosheet

ABSTRACT

$Ni(OH)_2$ nanoflowers with thin nanosheets as building blocks were synthesized using a homogeneous precipitation method with NH_4F as a complexing agent and a NH_4OH solution as an OH^- supplier in an aqueous $NiCl_2$ solution. A series of techniques, including X-ray diffraction, fourier transform infrared spectroscopy, thermogravimetric analysis, high-resolution transmission electron microscopy, field-emission scanning electron microscope, and UV–Vis spectroscopy, was used to characterize the nanoflowers. Investigations on the formation process of the flowers indicated that NH_4F is vital to the formation of the special flower-like microstructures.

1. Introduction

In recent years, controlling the shape of transition metal hydroxide nanostructures has become an increasingly interesting research topic because of its strong correlation with electronic, optical, magnetic, and catalytic properties of nanoparticles [1–6]. $Ni(OH)_2$ nanostructures have been intensively studied for their numerous applications, such as positive electrode materials in alkaline secondary batteries and as catalyst precursors [7–10].

Numerous chemical methods have been developed for the controlled synthesis of $Ni(OH)_2$ nanostructures with various shapes, such as nanowires, nanodisks, nanotubes, etc. For example, $Ni(OH)_2$ nanowires and nanodisks were prepared under hydrothermal conditions (160 °C) for 34 h using ethanol [11]. $Ni(OH)_2$ nanotube arrays were fabricated using porous alumina membranes as hard templates [12]. Recently, a few studies were conducted on the synthesis of flower-like $Ni(OH)_2$ nanostructures because of their special structure, large surface area, and superior electrochemical properties.

* Corresponding author. Tel.: +86 0517 83525091; fax: +86 0517 83525377.
E-mail address: wanwanshun@126.com (Z. Cheng).

0749-6036/$ – see front matter
doi:10.1016/j.spmi.2010.05.013

For instance, hierarchical $Ni(OH)_2$ flowers were obtained by reacting nickel dimethylglyoximate and NaOH in solution via the hydrothermal route at 120 °C for 4 h [13]. Porous β-$Ni(OH)_2$ flowers were prepared using $NiCl_2$ and hexamethylenetetramine (HMTA) via the hydrothermal route at 180 °C for 12 h [14]. Flower-like $Ni(OH)_2$ nanoarchitectures were synthesized with ethylenediamine in aqueous $NiCl_2$ solution through a mild hydrothermal reaction at 90 °C for 12 h [15]. $Ni(OH)_2$ nanoflowers have also been synthesized by a reverse micelle/microemulsion method in a water-in-oil system at 140 °C for 12 h [16]. Moreover, flower-shaped $Ni(OH)_2$ nanostructures have been synthesized using $NiCl_2$ and ammonium hydroxide at 65 °C in 4 h via an aqueous solution route [17]. However, previous reports indicated that a higher temperature and pressure, as well as a longer reaction time, is usually required to obtain flower-like $Ni(OH)_2$ nanostructures. Thus, developing a simple and effective method to synthesize flower-like $Ni(OH)_2$ at low temperatures and short reaction times is still necessary.

In this paper, we report a facile and novel approach for the fabrication of $Ni(OH)_2$ nanoflowers, with thin nanosheets as building blocks, using a homogeneous precipitation method in the presence of NH_4F. Furthermore, a possible mechanism for the formation of $Ni(OH)_2$ nanoflowers is proposed and discussed.

2. Experimental procedures

2.1. Fabrication of $Ni(OH)_2$ *nanoflowers*

All reagents were of analytical grade and used without further purification. Typically, 3.5 g $NiCl_2 \cdot 6H_2O$ and 1.5 g NH_4F were dissolved in 500 mL deionized water until the solution became green and homogeneous. Subsequently, a 2 M aqueous solution of NH_4OH was added dropwise until the pH value of the mixture was adjusted to 8.0. The above solution was then heated to 60 °C for 20 min without stirring. Finally, $Ni(OH)_2$ flowers were obtained following centrifugation, washing with absolute alcohol, and drying in a vacuum at 40 °C for 6 h.

2.2. Characterization of $Ni(OH)_2$ *flowers*

Phase identification via X-ray diffraction (XRD) was conducted on a Bruker Advance D8 X-ray diffractometer using Cu Kα radiation. The morphology was observed by a field-emission scanning electron microscopy (FE-SEM, LEO 1530VP), transmission electron microscopy (TEM, Tecnai 10), and high resolution transmission electron microscopy (HRTEM JOEL-2010). The chemical structure was measured with a Bruker vector-22 Fourier transform infrared (FTIR) spectrophotometer using KBr. The optical absorption spectrum was recorded on a GBC UV/VIS 916 spectrophotometer.

3. Results and discussion

Fig. 1 shows the XRD pattern of the as-prepared $Ni(OH)_2$ sample. The pattern shows that α-$Ni(OH)_2$ coexists with β-$Ni(OH)_2$. One of the phases is indexed to the (006), (101), (009), and (110) planes of α-$Ni(OH)_2$, while the other is indexed as hexagonal β-$Ni(OH)_2$.

Fig. 2 shows the typical FTIR spectrum of the as-prepared $Ni(OH)_2$ sample. The narrow and sharp peak at 3664 cm^{-1} is due to the υ_{O-H} stretching vibration, which confirms the brucite structure of the $Ni(OH)_2$ phase. Peaks at 3448 and 1645 cm^{-1} are assigned to the $\upsilon(H_2O)$ stretching vibration and $\delta(H_2O)$ bending vibration of water molecules adsorbed onto the product. The peak at about 650 cm^{-1} is due to the δ_{Ni-O-H} vibration. The peak at 511 cm^{-1} corresponds to the δ_{O-H} of the hydroxyl groups. Fig. 2 shows mixed characteristics of α-$Ni(OH)_2$ and β-$Ni(OH)_2$. The bands at about 650 and 511 cm^{-1} are characteristic of α-$Ni(OH)_2$ and β-$Ni(OH)_2$, respectively, and are consistent with the XRD result in Fig. 1.

Fig. 3 illustrates the TGA curve of the as-prepared $Ni(OH)_2$ sample. Two weight loss regions can be observed in Fig. 3. The first region is below 300 °C, where structurally bonded water was removed. The second region is between 300 and 800 °C, where the sample decomposed to NiO. The weight losses of