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Synthesis of single-crystal β-$Ni(OH)_2$ nanodisks and α-Fe_2O_3 nanocrystals in C_2H_5OH–NaOH–$NH_3\cdot H_2O$ system

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Abstract

Circular β-$Ni(OH)_2$ nanodisks and rhombohedral and hexagonal α-Fe_2O_3 nanocrystals were prepared using the C_2H_5OH–NaOH–$NH_3\cdot H_2O$ system under hydrothermal conditions. The C_2H_5OH/H_2O solvent is an appropriate one for the growth of these two materials with their thermodynamically favored morphologies. The possible formation mechanisms are discussed.
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Keywords: $Ni(OH)_2$; Fe_2O_3; Hexagonal; Rhombohedral; Nanodisks; Nanocrystals; C_2H_5OH/H_2O solvent

1. Introduction

In recent years, synthesis and fabrication of low-dimensional nanostructured materials, such as nanorods, nanowires, nanoplatelets, and nanotubes, have received much research interest due to their outstanding physical and chemical properties [1–5].

β-Nickel hydroxide (β-$Ni(OH)_2$), as a electrical conductivity p-type semiconductor with a maximum theoretical specific capacity of 289 mA h/g [6], has received increasing attention because of its extensive applications, especially as a positive electrode active material in alkaline rechargeable Ni-based batteries [7–9]. A number of physical and chemical methods were employed to synthesize $Ni(OH)_2$ nanostructure because nanoscale $Ni(OH)_2$ was proved to have much better electrochemical performance than the bulk's material [10,11].

α-Ferric oxide (α-Fe_2O_3) is another of the most important transition metal compound semiconductors ($E_g = 2.1$ eV) which is environmentally friendly, nontoxic, and corrosion-resistant. It has been widely studied, owing to its applications in pigments, magnetic storage, nonlinear optics, gas sensing, and catalysis [12–14]. Yen et al. [15] started with γ-FeOOH; the α-Fe_2O_3 obtained grew from an original size of 5 nm to a size about 25 nm. They also found that α-Fe_2O_3 crystallites obtained by calcination of amorphous $Fe(OH)_3$ showed sizes less than 5 nm. Wen et al. [16] prepared vertically aligned iron oxide nanobelt and nanowire arrays on a large-area surface by direct thermal oxidation of iron substrates under a flow of O_2.

Recently, we reported the synthesis of oriented attachment CuO nanosheets via a C_2H_5OH–NaOH–$NH_3\cdot H_2O$ system [17]. Because of the layered structure and the anisotropic growth rate of CuO, the obtained CuO nanocrystals grew along certain directions and tended to have sheet-like morphologies. In this communication, $Ni(OH)_2$ nanodisks and Fe_2O_3 nanocrystals were also synthesized in the same system successfully. The phase compositions, morphologies, and crystallite sizes of the obtained products were characterized by XRD and TEM, respectively, and the influences of different materials' structures were discussed.

2. Experimental

2.1. $Ni(OH)_2$

A quantity of 25 ml of $Ni(NO_3)_2\cdot 6H_2O$ (1.00 M, in pure ethanol) was added to 25 ml of ammonia solution (25%) and 5 ml of aqueous NaOH (1.00 M). Then four sets of the solution mixture were transferred to Teflon-lined stainless steel autoclaves, which were then heated at 150 °C for 2, 6, and

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24 h and 180 °C for 48 h, respectively. After reaction, the autoclaves were cooled naturally and the products were washed with deionized water and absolute ethanol several times. The final products were dried in vacuum at 60 °C for 12 h.

2.2. Fe_2O_3

The Fe_2O_3 nanocrystals were synthesized in the same way except for substituting $Fe(NO_3)_3{\cdot}9H_2O$ for $Ni(NO_3)_2{\cdot}6H_2O$ and heating at 200 °C for 48 h.

A powder X-ray diffraction (XRD) pattern was collected on a D/Max 2550V X-ray diffractometer with Cu$K\alpha$ irradiation at $\lambda = 1.5406$ Å. The morphologies of the samples were observed through TEM (JEM-2010, 200 kV).

3. Results and discussion

3.1. $Ni(OH)_2$

X-ray powder diffraction patterns of $Ni(OH)_2$ nanocrystals are illustrated in Fig. 1. They correspond to the characteristic diffraction of the hexagonal β-$Ni(OH)_2$ (JCPDS No. 14-0117). Two other peaks are located at 2θ values of 15° and 30° in Fig. 1a, which is very weak, indicate that some impurities exist in the products, but obviously the main phase is still $Ni(OH)_2$. The pure phase of $Ni(OH)_2$ nanocrystals are obtained after 6 h of treatment. No peaks from any else phase and impurity are observed in Fig. 1b. And the diffraction peaks are sharper and stronger, which implies that the crystallinity of $Ni(OH)_2$ samples is improved by increasing the reaction time.

Fig. 2 shows the TEM micrographs of $Ni(OH)_2$ nanocrystals with treatment time of (a) 2 h, (b) 6 h, and (c) 24 h at 150 °C and (d) 48 h at 180 °C, respectively. It can be seen from Fig. 2a that the products take on a flake-like shape and some of them have rolled to be needle-like. Estimated from the TEM image, the thickness of as-prepared nanoflakes is less than 10 nm. They aggregate in a severe condition because of the short reaction time, but it is improved after the time is prolonged to 6 h (Fig. 2b). In Fig. 3c, we can clearly see that the $Ni(OH)_2$ nanoflakes are of incongruous sizes and the morphologies are irregular. But their aggregations become very weak.

Further prolonging the treating time and increasing the reaction temperature to 48 h and 180 °C, the $Ni(OH)_2$ nanoflakes change to uniform and well-dispersed disklike nanocrystals with a diameter of ~200 nm (Fig. 2d). The HRTEM image and corresponding SAED pattern are shown in Figs. 2e and 2f. The perfect diffraction dots present hexagonal dot arrays and can easily be indexed to be the ED pattern with the incident electron beam along the [001] direction, which indicate the obtained $Ni(OH)_2$ nanodisks are equiaxial single-crystals and the

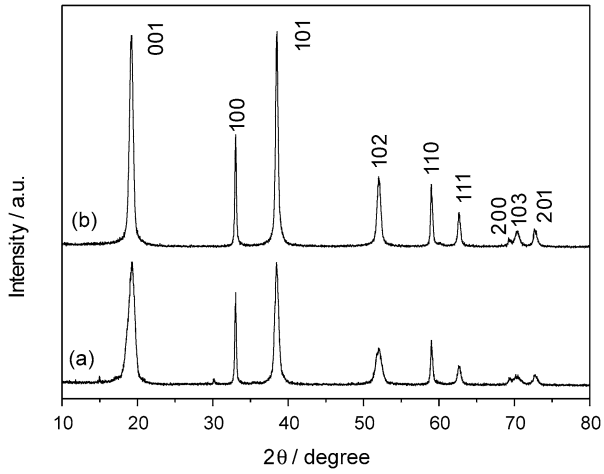

Fig. 1. XRD patterns of $Ni(OH)_2$ nanocrystals obtained by treating for (a) 2 h and (b) 6 h.

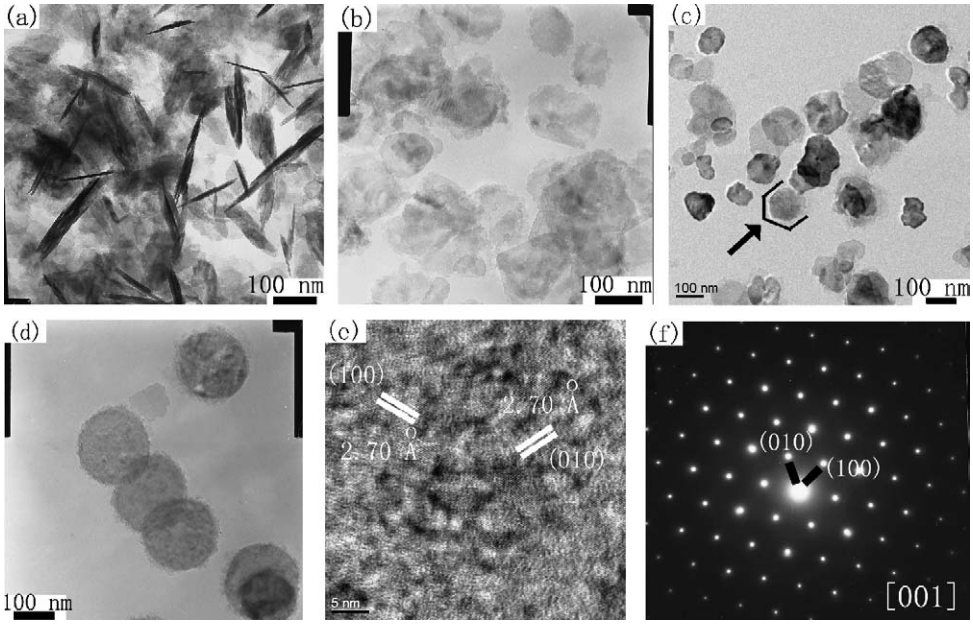

Fig. 2. TEM images of $Ni(OH)_2$ nanocrystals with treatment times of (a) 2 h, (b) 6 h, (c) 24 h at 150 °C and (d) 48 h at 180 °C, (e) and (f) are the HRTEM image and the corresponding SAED pattern of $Ni(OH)_2$ nanodisks in (d).

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