

Fabrication of β -Ni(OH)₂ and NiO Hollow Spheres by a Facile Template-free Process

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Supplementary Information

S1. DSC and TG curves of Ni(OH)₂ hollow microspheres

Differential scanning calorimetric analysis (DSC) and thermogravimetric analysis (TG) of the as-prepared Ni(OH)₂ hollow microspheres were carried out on a simultaneous TG-DTA/DSC apparatus (NETZSCH STA 449C, Germany) with a heating rate of 10 °C min⁻¹ in flowing air (Fig. S1). The TG curve in Fig. S1 shows that there is rapid weight loss from 243 to 327 °C. The endothermic nature in the DSC curve along with the rapid weight loss in the TG curve suggests that Ni(OH)₂ rapidly decomposes in this temperature range. The weight of the sample continuously decreases up to ~600 °C, after which no obvious weight loss is further observed. The total weight loss below 600 °C was measured to be 19.6 %, in good agreement with the theoretical value of 19.5 % calculated from the decomposition reaction of Ni(OH)₂ to NiO, indicating that complete decomposition of Ni(OH)₂ is attainable at 600 °C.

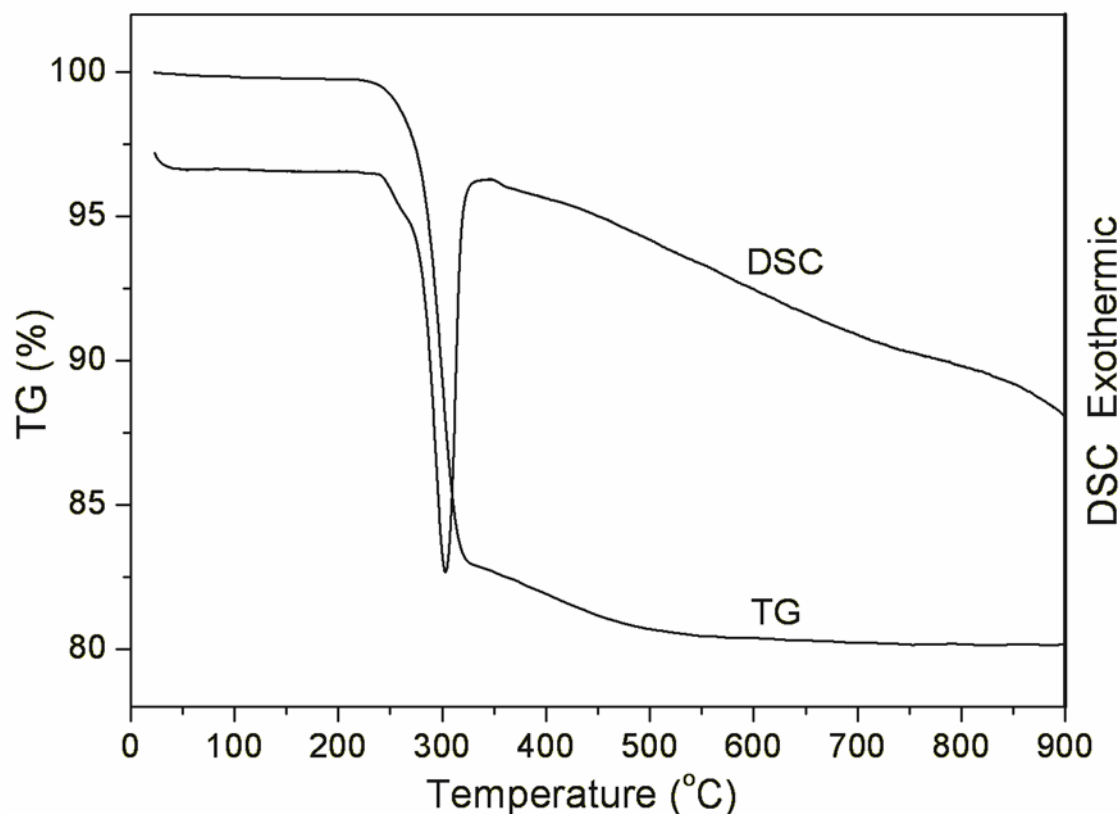


Fig. S1 Differential scanning calorimetric analysis (DSC) and thermogravimetric analysis (TG) curves of the as-prepared Ni(OH)₂ hollow microspheres.

S2. Ni(OH)₂ microspheres synthesized at lower temperatures

In addition to the description in the main text, the Ni(OH)₂ microspheres can also be prepared at temperatures below 100 °C. In a typical experiment, 5.0 mmol of Ni(NO₃)₂·6H₂O and 2.0 g of glycine as well as 2.0 g of Na₂SO₄ salt were dissolved in 25 mL of deionized water, then 10 mL of NaOH (5 M) was added dropwise into the above solution under magnetic stirring to form a clear blue solution. The solution was then transferred to a glass bottle and kept inside an oil bath at 60-100 °C without stirring for 24h. After the treatment, green Ni(OH)₂ products were collected by filtration, washed three times with deionized water, and dried at room temperature for 24 hours. Fig. S2 shows the typical SEM images and XRD pattern of Ni(OH)₂ microspheres synthesized at 80 °C for 24 h. Investigations showed that at low synthesis temperature some joined Ni(OH)₂ microspheres can be occasionally observed in as-prepared samples (indicated with arrows in Fig. S2a). The Ni(OH)₂ prepared at 80 °C is still well-crystalline (Fig. S2c), identified as the single phase β-Ni(OH)₂ with well-crystalline hexagonal structure (*a*=0.3126 nm, *c*=0.4605 nm, JCPDS file No. 14-0117).

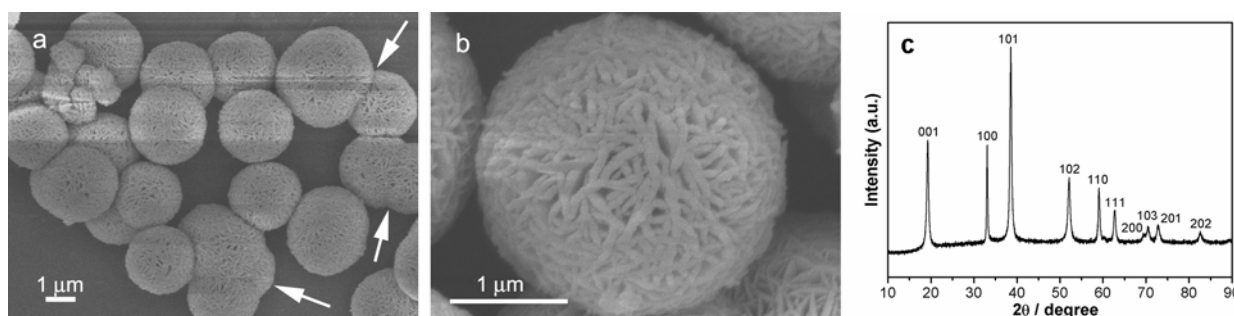


Fig. S2 (a)-(b) SEM images of Ni(OH)₂ spheres (prepared at 80 °C, 24h) observed under different magnifications; (c) XRD pattern of the Ni(OH)₂ (prepared at 80 °C, 24h).

S3. XRD patterns of the Ni(OH)₂ products with different reaction time

The formation process of Ni(OH)₂ microspheres built from β-Ni(OH)₂ nanosheets is indicated in the following series of experiments (5.0 mmol of Ni(NO₃)₂·6H₂O and 2.0 g of glycine as well as 2.0 g of Na₂SO₄ salt were dissolved in 25 mL of deionized water, then 10 mL of NaOH (5 M) was added dropwise into the solution under magnetic stirring so that a clear blue solution was formed. The resulting solution was sealed into Teflon-lined autoclave. After that, the autoclave was transferred to an electric oven at 100 °C and kept for 30 min, 1h, and 24 h respectively). Fig. S3 displays the X-ray diffraction (XRD) patterns of the Ni(OH)₂ prepared at 100 °C with reaction time of 30 min, 1h and 24h respectively. As shown in Figure S3, the crystallinity of the as-prepared Ni(OH)₂ gradually increases with the reaction time, which indicates that Ostwald ripening (crystallites grow at the expense of the smaller ones) is the underlying mechanism in this hollowing process.

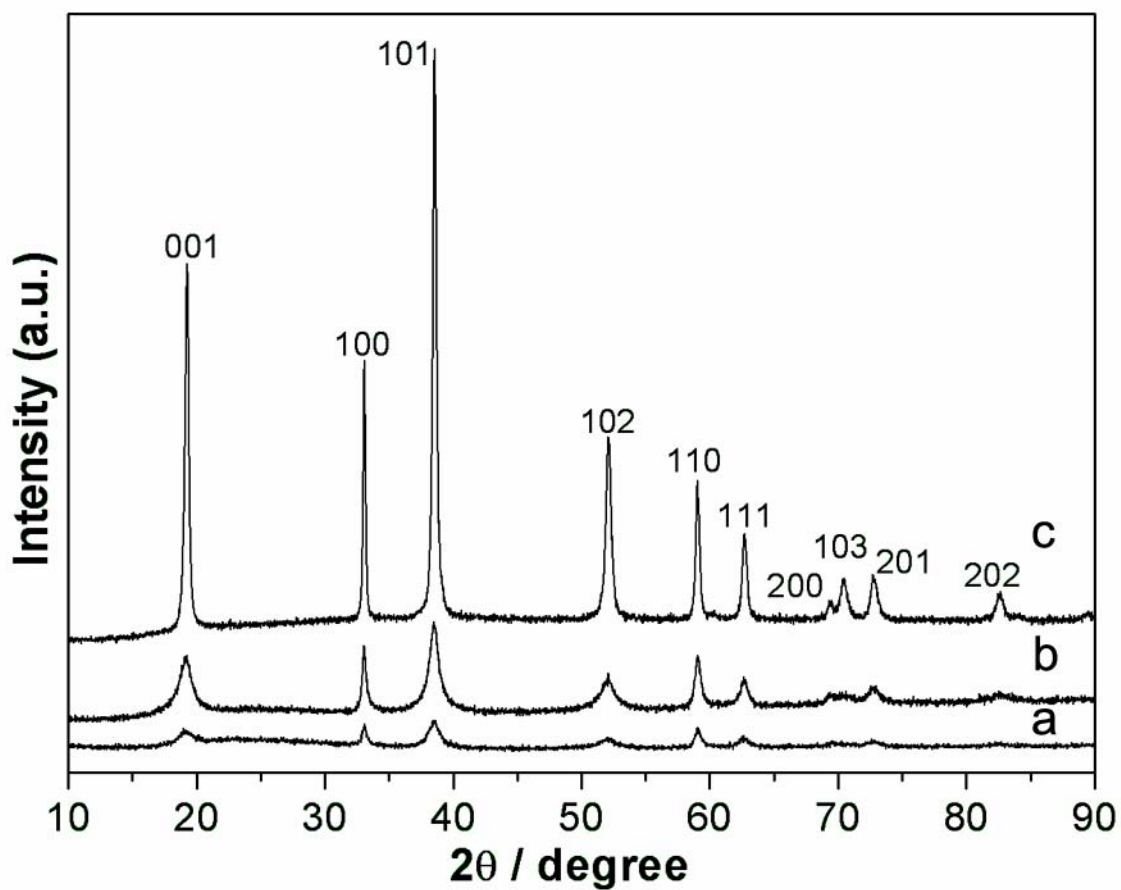


Fig. S3 XRD patterns of the Ni(OH)_2 prepared at 100°C with different reaction time: (a) 30 min; (b) 1 h; (c) 24 h (final product).