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LETTER

Synthesis of Y_2O_3 with nestlike structures

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Fabrication of nano- to microscopic-scale inorganic materials with special morphology is of great interest for materials chemistry due to their importance in basic scientific research and potential technological applications [1–4]. Very recently, self-assembly of nanostructured building blocks has become an active research field in materials synthesis. Strategies based on self-assembly have a number of advantages: First, they can carry out many of the most difficult steps in nanofabrication that involve atomic-level modification of structure. Second, they create stable constructs having a low level of defects and can be applied to many structures and materials [5].

Yttrium oxide (Y_2O_3) is an important engineering material in many fields due to its chemical and physical properties, such as high permittivity [6], high melting point, a relatively large band gap energy etc. For example, Y_2O_3 could be used as a waveguide material due to its high refractive index, or as a buffer layer for ferroelectrics and superconductors because of its low lattice-mismatch with silicon [7]. More applications of Y_2O_3 may emerge if shape-controlled nanocrystals could be achieved with high complexity [8, 9]. Very recently, Y_2O_3 nanotube [10, 11] and microprism [12] have been prepared for potential engineering applications. In this letter, we present first synthesis of Y_2O_3 nestlike microstructure through a facile hydrothermal process followed by subsequent heat treatment of the precursor at 500 °C for 2 h. The nestlike $Y(OH)_3$ and Y_2O_3 nanostructures exhibit unique geometrical shapes, which may lead to important applications in scientific research and technological applications.

All of the chemical reagents used in this experiment were analytical grade and used without further purification. In a typical procedure for preparing $Y(OH)_3$ nestlike crystal, 0.2 g of $Y(NO_3)_3 \cdot 6H_2O$ and 1.0 g of glycine were dissolved in 40 ml of deionized water, then $NH_3 \cdot H_2O$ (25 wt%) was added dropwise into the above solution under constant stirring with adjusting the pH value to 9.6. The solution was then sealed into a 50 ml Teflon-lined autoclave, followed by hydrothermal treatment at 170 °C for 5 days in an electric oven. After the treatment, white products were collected by filtration, washed with distilled water and absolute ethanol several times, and dried at room temperature for 24 h. Nestlike Y_2O_3 could be obtained by thermal decomposition of the as-synthesized $Y(OH)_3$ powder at 500 °C for 2 h.

The XRD patterns of all samples were recorded on a Rigaku/Max-3A X-ray diffractometer with CuKα radiation ($\lambda = 0.15418$ nm). Microstructures of the products were obtained by scanning electron microscopy (SEM, JEOL, JSM-6330F). The TEM images were recorded on a JEOL-200CX TEM, using an accelerating voltage of 200 kV.

Figure 1 displays the XRD patterns of the as-prepared $Y(OH)_3$ and Y_2O_3. All of peaks of the XRD pattern in Fig. 1a can be indexed as a pure hexagonal phase of $Y(OH)_3$ (JCPDS 83-2042). However, the reported value for the 100% peak of a fully ordered hexagonal is 16.3° in contrast to the present study where the maximum peak was observed at a 2θ of 41.8°, which may be related to this special structure. In Fig. 1b, all the diffraction peaks can be indexed as a

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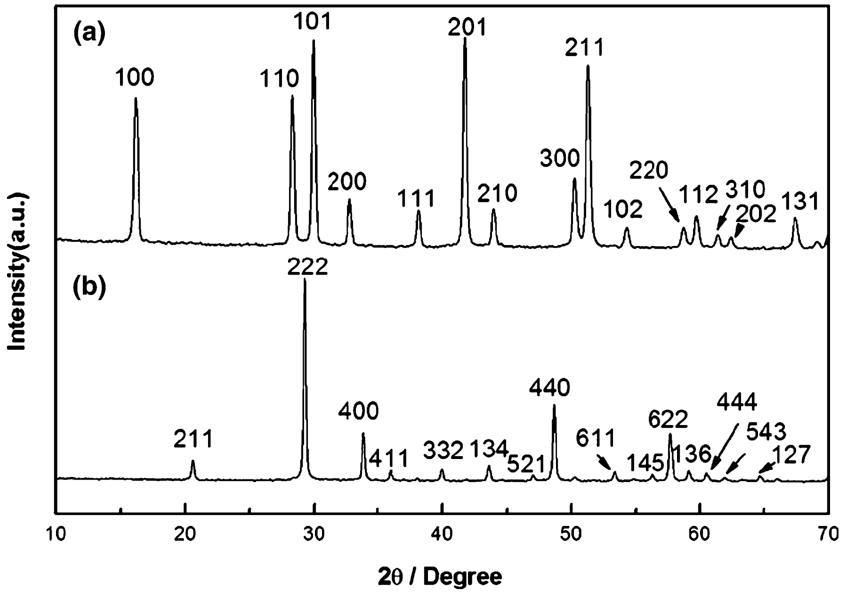

Fig. 1 XRD patterns of (**a**) the as-prepared $Y(OH)_3$ powder and (**b**) Y_2O_3 prepared by thermal decomposition of the $Y(OH)_3$ powder at 500 °C for 2 h

pure cubic phase of Y_2O_3 with a calculated lattice constant of $a = 1.0602$ nm (JCPDS 88-1040).

The morphology and structure of the as-synthesized $Y(OH)_3$ powder were further investigated by SEM. As shown in Fig. 2a, the overall morphology of the samples indicates that there exists a great deal of uniform nestlike structures. Further magnified image (Fig. 2b) shows that the diameter of the nestlike structure is about 2 μm. Close observation (Fig. 2c) reveals that these nestlike products are actually composed of nanosheetlike structures.

Nestlike Y_2O_3 structures were obtained from the nestlike $Y(OH)_3$ particles by calcination at 500 °C for 2 h. The general morphology of the as-prepared Y_2O_3 samples is clearly shown in Fig. 3a. Careful observation (Fig. 3c) shows that the nestlike Y_2O_3 nanostructures consist of nanosheets in a highly close-packed assembly. It is interesting that both Y_2O_3 and its precursor almost possess the same morphology; even such a transformation has a chemical process.

To understand the formation process of the nestlike structures, time-dependent experiments were carried out by quenching the Teflon-lined autoclave using cold water at different reaction stages. A series of TEM and SEM images in Fig. 4 show the morphology at different reaction stages corresponding to the reaction time of 10, 24, 50, 70 h, respectively. When the reaction continued to 10 h, the intermediate products were mainly nanoparticles with the diameter less than 10 nm (Fig. 4a). When prolonging the reaction time to 24 h, an examination of the intermediate products showed cubic-like particles (Fig. 4b). It appears that the cubic-like particles have rough surfaces and are composed of smaller nanoparticles, which indicate that the growth of cubes is based on the aggregation of nanoparticles rather than the normal concept for crystal growth that is typically thought to occur via atom-by-atom addition to an existing nucleus or template. When increasing the reaction time to 50 h, cubic-like particles began to aggregate and some nanosheets were formed. When we further prolonged the reaction time to 70 h, some nestlike structures had readily formed and the newly formed crystallites were self-assembled on the existing nuclei to form the nestlike structure.

Regarding the growth process, it is believed that glycine plays a crucial rule in ensuring the formation of these shapes because only aggregated nanoparticles were obtained without glycine. Usually, absorption of ions or molecules played an important role in the aggregation process, which was directional [13, 14]. Without glycine, the nanoparticles grow randomly through strong interactions between the nanoparticles themselves to reduce the interfacial energy. Evidently, glycine molecules preferentially adsorb to different crystal faces of $Y(OH)_3$ to produce anisotropic nanoparticles, which is favorable to ordered aggregates. Further studies are necessary to understand the exact formation mechanism of these shapes because it may open up new opportunities to fabricate more complex nanostructured materials.

In summary, a novel nestlike $Y(OH)_3$ structures have been prepared via a facile hydrothermal process.

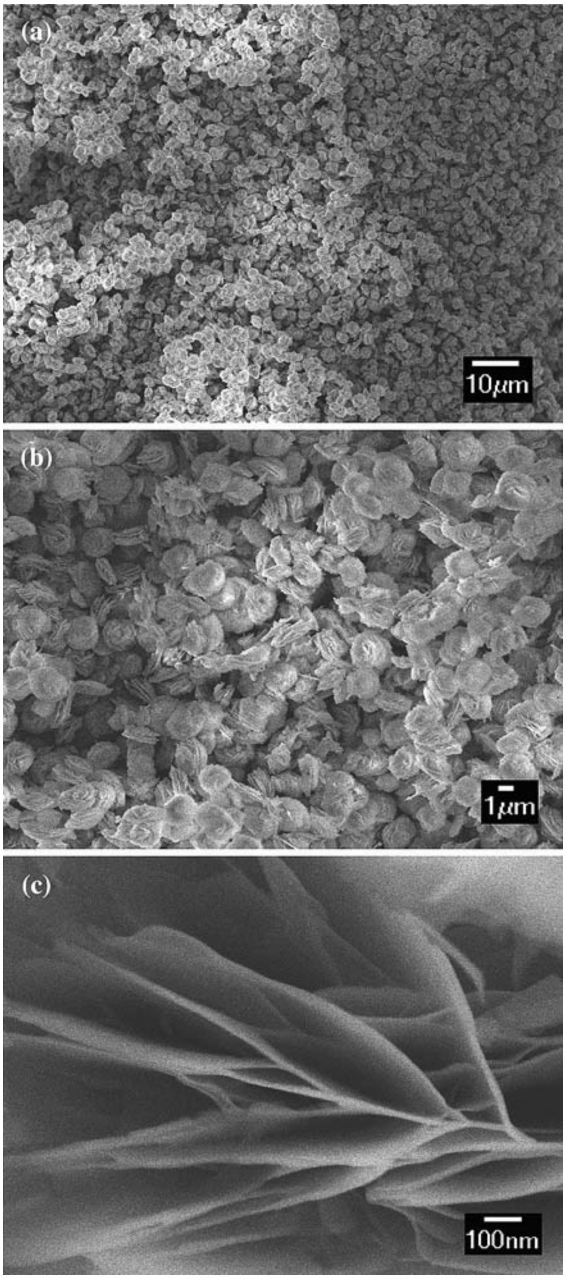

Fig. 2 SEM images of $Y(OH)_3$ at different magnification

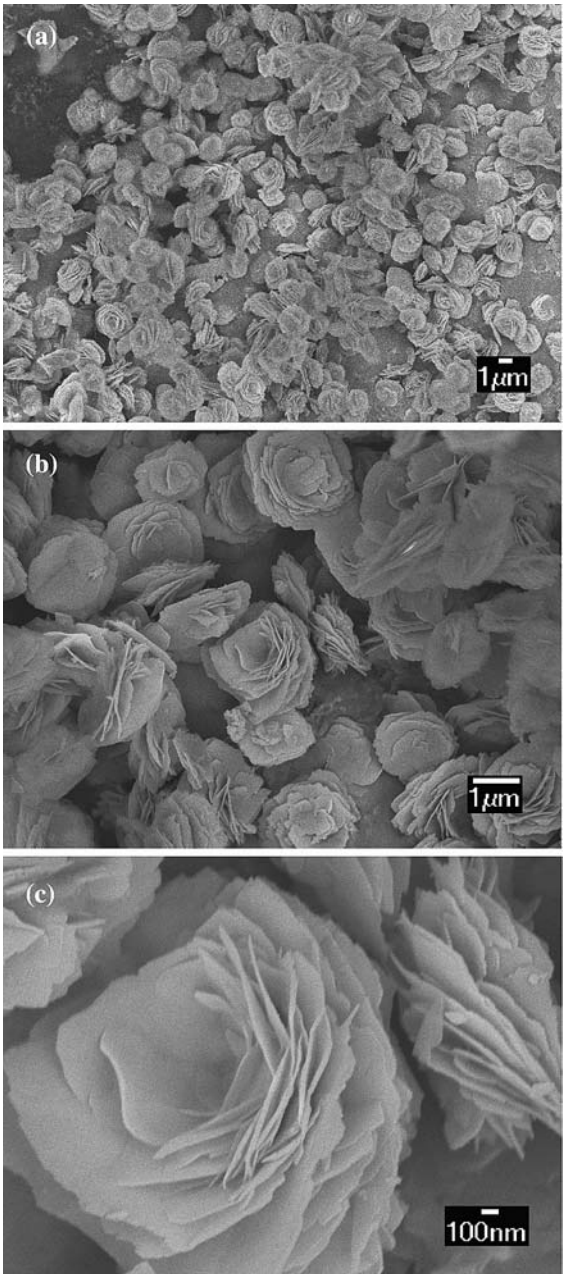

Fig. 3 SEM images of Y_2O_3 at different magnification

Y_2O_3 with the similar morphology was obtained by the thermal decomposition of nestlike $Y(OH)_3$ at 500 °C for 2 h. From a technological point of view, these obtained nestlike structures may have potential applications in many fields such as luminescent devices, catalysts. This simple method is expected to allow fabrication of other inorganic materials with controllable phase and shapes.

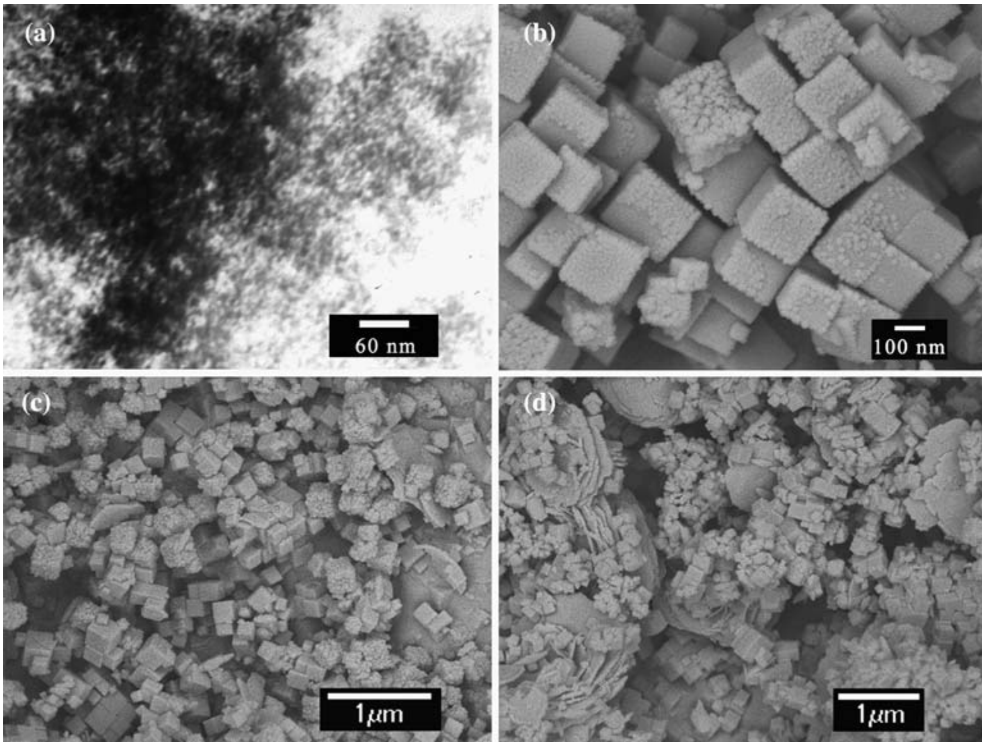

Fig. 4 (**a**) TEM image of intermediate product obtained at 10 h; SEM images of intermediate products obtained at different times: (**b**) 24 h, (**c**) 50 h, (**d**) 70 h

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