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Article

Rapid Synthesis of Lead Oxide Nanorods by One-step Solid-state Chemical Reaction at Room Temperature

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A simple and facile method was reported to synthesize lead oxide nanorods. Nanorods of lead oxide were obtained directly from grinding solid metal salt and sodium hydroxide in agate mortar with the assistance of a suitable nonionic surfactant in only one step, which is different from the result of hydroxide in solution. The product has been characterized by XRD, TEM and SEM. The formation mechanism of rod-like morphology is discussed and the surfactant plays an important soft-template role in modifying the interface of solid-state reaction and according process of rod-formation.

Keywords nanomaterial, nanorod, lead oxide, solid-state reaction

Introduction

The field of nanostructured materials is a fascinating area of chemistry and materials science because of their novel properties that differ from those of bulk materials and potential application in both mesoscopic research and the development of nanodevices.[1,2] One-dimensional (1D) materials are an important category of nanostructured materials and have been widely researched yielding various special structures such as nanotubes, nanorods, nanowires and nanobelts *etc*.[3-5] Considerable efforts have been placed on the synthesis of 1D nanostructured materials using laser ablation, template, molecular beam epitaxy and other methods.[6-8] However, these preparation methods usually require complex process control, very high temperature or long synthesis time.

Due to their electrical and magnetic properties, metal oxide semiconductor materials are of both fundamental and technological importance.[9] Many oxide 1D nanomaterials have been reported in past few years.[10-12] However, very little work has been focused on the study of lead oxide nanomaterials. It is known that lead oxide acts as positive active masses for lead-acid batteries[13] and plays a unique structural role in acting both as a network modifier and network former in a glass.[14] So study on synthesis and novel properties of lead oxide nanorods is still a new area for the nanostructured materials.

Here we report a simple and rapid route for the synthesis of lead oxide with controllable morphology by means of one-step solid-state reaction with the assistance of a suitable nonionic surfactant, polyethylene glycol (PEG) 400. The influence of different surfactant was discussed and the surfactant plays an important soft-template role in the process of solid-state reaction. To the best of our knowledge, studies on lead oxide nanorods obtained by one-step solid-state reaction have not been reported. Compared with the methods reported previously, this synthesis method has shown many advantages such as low temperature, one-step process, mild reaction conditions and low energy consumption.[15-20] Thus, this method may provide a new path to fabricate one-dimensional nanostructured materials with a convenient, economical and environmentally friendly way.

Experimental

All the reagents are analytically pure and were used without further purification. Manipulations and reactions were carried out in air.

Our procedure for synthesizing PbO nanorods is as follows. 0.02 mol of solid $PbCl_2$ was weighed and ground for about 5 min in agate mortar, then 5 mL of polyethylene glycol (PEG) 400 was added. After mixing completely, 0.03 mol of solid NaOH was added to the mixture, which was ground for 50 min. Finally, the mixture was washed with distilled water and EtOH in ultrasonic bath. The product was dried in air.

XRD was taken on a MAC science MXP18AHF X-ray diffractometer with graphite-monochromatized Cu Kα radiation (λ＝0.154056 nm), employing a sampling width of 0.02°. TEM images were measured on a

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Hitachi H-600 transmission electron microscopy with an accelerating voltage of 100 kV. SEM image was performed on a LEO 1430VP scanning electron microscopy.

Results and discussion

Figure 1 shows the XRD patterns of product prepared by solid-state reaction at room temperature. All of the diffraction peaks of the product can be indexed as lead oxide with orthorhombic phase (JCPDS Card file No. 38-1477), although the relative intensity of lead oxide is not consistent with that of bulk lead oxide. The most intensive peak of bulk lead oxide is (1 1 1). However, for our sample that is (0 2 0), although the other peaks are consistent with those of bulk lead oxide. The particle sizes calculated from the XRD peak widths by Deby-Scherrer equation are about 12 nm. No characteristic peaks of impurities, such as NaCl, PEG, and by-products were observed.

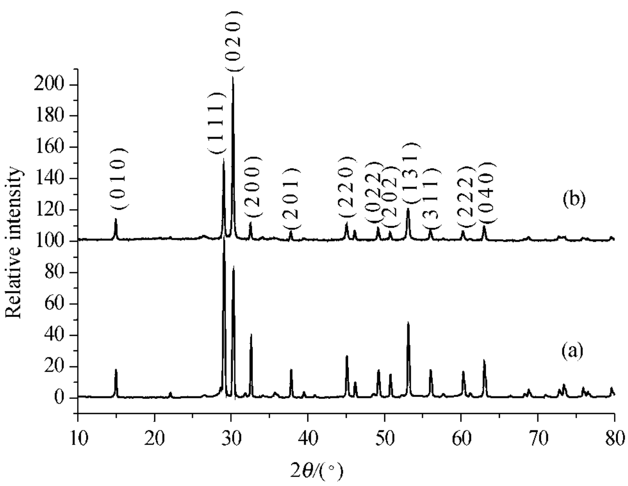

Figure 1 XRD patterns of (a) bulk PbO and (b) PbO nanorods prepared by solid-state reaction.

Figure 2 illustrates the typical TEM images of product prepared by solid-state reaction at room temperature. As can be seen from Figure 2 that the product was comprised of nanorods. The nanorods have a diameter from 40 to 140 nm and a length of up to 10 μm. The SEM image (shown in Figure 3) also indicates the same result.

Unlike the reactions in the solution, solid-state reactions were carried out in non-aqueous solution. So it may be preformed in different mechanism from the solution reaction and different products may be obtained. Here, oxides were obtained via grinding solid salts with sodium hydroxide in one step, which is different from the results of hydroxides in solution.

In the experiment, the surfactant plays an important role in the process of shape-formation. When no surfactant or no suitable surfactant was used in the reaction process, only nanospheres were obtained. After adding a suitable surfactant to the reaction mixture, the rod-like

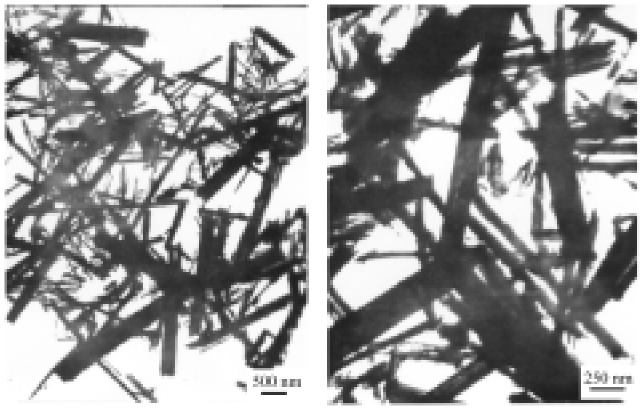

Figure 2 TEM images of PbO nanorods prepared by solid-state reaction.

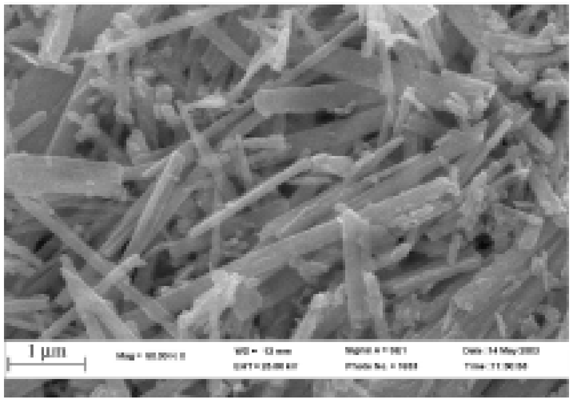

Figure 3 SEM image of PbO nanorods prepared by solid-state reaction.

structure was observed. Here, PEG 400 was selected as the suitable surfactant and added to the reaction system. The growth process was well controlled through PEG 400, so it can be thought that the surfactant may act as a soft template and modifies the interface of solid-state reaction facilitating growth of the nanocrystallites in good orientation.[21] Other surfactants such as Tween-60 and Span-80 were also tested in our experiments. However, no nanorods were formed. It is very important to select suitable surfactant for the shape of product in our present work.

Conclusions

Lead oxide nanorods have been successfully prepared by one-step solid-state method at room temperature. The key to the formation of the rod-shape is the assistance of a suitable surfactant. In addition, the present synthesis method is a simple and facile approach for synthesis of one-dimensional materials. With the development of solid-state reaction techniques for morphology control, this method is expected to synthesize other nanostructured materials with desired morphology.

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