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Surfactant free hydrothermal formation of Pb_3O_4 nanorods

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Abstract

Lead oxide (Pb_3O_4) nanorods with average diameter of about 90 nm, average length of ∼1.5 μm and average crystallite size of about 42 nm, were prepared through a novel surfactant free hydrothermal route and were characterized with XRD, SEM and TEM investigations. Selected area electron diffraction pattern captured from a single nanorod with average diameter of 80 nm, confirmed the tetragonality of Pb_3O_4 lattice structure and ⟨2 1 1⟩ growth direction of the typical nanorods. The possible suggested mechanism is that the oriented attachment of Cl^--capped PbO_2 clusters and their subsequent decomposition under hydrothermal environment resulted in formation of Pb_3O_4 nanorods.

Keywords: Ceramics; Chemical synthesis; Nanofabrications

1. Introduction

Nanostructure one-dimensional (1D) materials have attracted an extensive attention due to their weird physical, optical, electronic and mechanical properties [1–3] and their novel applications in functional materials, nanodevices and nanotechnology [4–8]. Therefore, various 1D nanostructure materials, such as elemental carbon [9,10], sulfides [11,12], oxides [13], nitrides [14] and metals [15] have been prepared via different methods.

Due to their unique properties, lead oxides (including four basic types, i.e. PbO, PbO_2, Pb_2O_3 and Pb_3O_4) [16], have wide applications such as network-modifiers in luminescent glassy materials [17], pigments [18], storage batteries [1,19,20] and nanoscale electronic devices [21]. Various researchers have prepared 1D lead oxide nanostructures through different methods. Cao et al. [16], synthesized PbO_2 and Pb_3O_4 nanorods utilizing cetyltrimethylammonium bromide (CTAB) as the structure directing agent. Xi et al. [21] used poly vinyl pyrrolidone (PVP) as the surfactant for hydrothermal preparation of Pb_3O_4 microtubes. Also, PbO_2 nanobelts were synthesized by Pan et al. [22] through evaporation of PbO powders at 950 °C for 1 h under a pressure of 300 Torr, and subsequent deposition on the surface of alumina strip substrates. Although, Pb_3O_4 has fascinating physical properties due to its mixed valence band and unique electronic structure [23], investigations on preparing one dimensional nanostructures of this interesting material is limited.

Oriented attachment which involves direct self-assembly of adjacent nanocrystals along a specific crystallographic orientation, has been proposed by pioneers like Penn and Banfield [24] and Alivisatos [25], as an effective mechanism for the growth of anisotropic nanostructures [26]. Here, we prepared Pb_3O_4 nanorods via a novel surfactant free hydrothermal route and proposed a possible mechanism based on oriented attachment and subsequent decomposition of Cl^--capped PbO_2 polar clusters under hydrothermal conditions. It is expected that such investigation can be applied as a general route to prepare one-dimensional oxides without utilization of the surfactants.

2. Experimental

All the utilized materials were purchased from Merck, Germany and used as received without further purification. In a typical experiment, 1.66 g $PbCl_2$ powder was mixed vigorously at 70 °C for 1 h, with a 30 ml NaOH solution with a concentration of 10 M. The mixture was then put into a Teflon lined stainless steel autoclave with autogenous pressure control at 200 °C for 24 h without any shaking or stirring. After the reactions were completed, the autoclave was led to cool down to the room temperature, naturally. A white precipitate was obtained which was then centrifuged for 10 min at 1500 rpm. The obtained product was purified in the following cycle in order to remove the remaining alkaline solution and probable impurities; it was washed with deionized water, decanted and centrifuged, several times. Then it was washed with acetone and dried at 60 °C for 24 h in a drying oven. The obtained powder was characterized with XRD, SEM and TEM analyses. The powder X-ray diffraction pattern (XRD) of the resulted product was obtained by a Simens D500 diffractometer with Cu

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