Synthesis of PbO Nanostructures

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Black-phosphorus-analog lead monoxide (PbO), as a new emerging 2D material, has rapidly gained popularity due to its unique optical and electronic properties. Both theoretical prediction and experimental confirmation have revealed that PbO exhibits excellent semiconductor properties, including a tunable bandgap, high carrier mobility, and excellent photoresponse performance, which is undoubtedly of great interest to explore its practical application in a variety of fields, especially in nanophotonics.

lead monoxide   black phosphorus analogs   composites

1. Introduction

In recent years, the controlled synthesis of PbO nanocrystals in a series of shapes, including 0D NPs, 1D nanostructures (e.g., NWs, NRs, etc.), 2D NSs, and even 3D hierarchical nanostructures, has achieved significant progress. The synthetic methods, such as liquid-phase exfoliation (LPE) and hydrothermal or solvothermal methods, are generally divided into two categories: the “top-down” strategy and the “bottom-up” strategy. The “top-down” strategy is usually used to exfoliate layered bulk PbO crystals into 0D NPs or 2D single- or few-layered NSs under driving forces, such as sonication and scotch tape, due to their weak van der Waals interaction between neighboring stacked layers. The typical “top-down” strategies include LPE and mechanical cleavage. Note that the “bottom-up” strategy usually depends on the rational design and controlled synthesis of PbO nanostructures from the precursors under certain conditions, while the typical “bottom-up” technique for preparing high-quality PbO nanostructures is the hydrothermal method or the solvothermal method.

2. Zero-Dimensional PbO NPs

NPs (with a size range from 1 nm to 100 nm) have attracted extensive attention because of their versatile applications in (opto)electronics, catalysts, sensors, and biomedicines [1][2][3][4][5][6][7][8][9][10][11][12][13][14][15][16][17]. In 2019, Shur et al. [18] reported the formation of PbO NPs by laser ablation in hot water using Pb as a model metal. The PbO nanoparticles in a classical spherical shape can be observed immediately after only the laser ablation treatment, and the spherical NP shape rapidly changes to octahedra, rods, or plates when heated by laser ablation in water. Additionally, in 2018, a group successfully fabricated PbO quantum dots (QDs) with an LPE method [5]. The transmission electron microscopy (TEM) of the as-prepared PbO QDs showed an average lateral size of 3.2 ± 0.9 nm and an average thickness of 2.5 ± 0.5 nm, which corresponds to 4 ± 1 layers. The high-resolution TEM (HRTEM) image of the PbO QDs displays a clear lattice fringe of 0.20 nm, which is well assigned to the (200) plane of the β-PbO crystal [19]. Moreover, Chen et al. [20] demonstrated that PbO NPs were successfully synthesized at the water/air interface in the condition of the Langmuir films of poly(N-vinylcarbazole). A large quantity of round PbO NPs with a diameter of several nanometers can be observed. It should be pointed out that, if the temperature rises up to 40–50 °C, the size of the as-prepared NPs sharply increases and the crystallinity also improves [20].

3. One-Dimensional PbO Nanostructures

The 1D PbO nanostructures are fascinating systems, similar to other 1D nanostructure systems (e.g., 1D Te NWs [21], 1D Se nanotubes (NTs) [3], 1D Bi nanobelts [22], 1D ZnO NRs [23][24], and PtCu NWs [25]), for studying physicochemical properties due to their anisotropic character. For example, a facile alkylamine-mediated thermolysis strategy was reported for the fabrication of high-quality PbO NWs through the thermal decomposition of lead carboxylate in the presence of hexadecylamine (HDA) [26]. The uniform PbO NWs, with an average length of several micrometers and an average diameter of 7.1 nm, were successfully achieved in a high yield, and closer observation revealed that the as-prepared PbO NWs displayed the nature of a single crystal with a clear lattice fringe of 0.28 nm, which can be assigned to the (200) plane of the β-PbO crystal [26]. Additionally, Wang et al. [27] employed porous anodic aluminum oxide (AAO) templates to prepare a PbO NW array by a sol–gel method. The SEM image shows that the as-prepared PbO NWs were successfully grown in the nanochannels of the AAO templates, and all the NWs parallelly aligned to each other, showing an excellent vertical orientation on the AAO template to form an array. The average diameter of these as-prepared nanowires is ~80 nm, consistent with the channel diameter of the AAO template. Furthermore, Jia et al. [28] successfully synthesized single-crystalline PbO NRs with a hydrothermal approach. The TEM image shows that some NRs are parallel to each other, and that shorter NRs assemble beside the longer ones. The SAED pattern shows that the rod-shaped crystal grows along the (100) direction. Because of the size-dependent effect on the performance (the absorption property and bandgap energy (E_g) [5][29][30]), Bi nanostructures with different sizes can be readily synthesized by facilely tuning the reaction conditions (e.g., the reaction temperature and reaction time).

4. Two-Dimensional PbO NSs

Two-dimensional NS materials, atomically thin sheets, have attracted tremendous interest due to their fascinating properties [31][32][33][34][35][36][37]. Inspired by the huge success of graphene and black phosphorus, a series of 2D materials have been exploited and demonstrated to have great potential in many applications, such as energy [38][39][40][41], catalysis [25][42][43][44], sensors [45][46][47][48], nanophotonics [37][49][50][51][52][53], and biomedicines [54][55][56]. Although great progress in 0D and 1D PbO nanostructures has been achieved, much less is known about 2D PbO NSs, their synthetic strategies, and their fascinating performance so far. In 2020, Fu et al. [57] developed a chemically clean route for synthesizing 2D PbO NSs by laser ablation. The SEM image of the as-fabricated PbO NSs shows a mean size of 1.5 μm in the planar dimension. The NS displays a hexagonal shape with a regular edge, as well as a smooth surface, and the SAED pattern indicates that the NS shows a (002)-terminated surface with a single-crystal nature. Moreover, in 2020, high-quality PbO NSs were also successfully synthesized by the thermal decomposition of lead carboxylate, the same as the abovementioned PbO NWs, without a higher HDA content [26]. The uniform PbO NSs, with a lateral size of ~300 nm, were successfully obtained at the initial reaction time of 30 min, and typical square-like NSs are clearly observed, indicating the successful synthesis of the 2D PbO nanostructures. In addition, in 2018, a group employed an LPE method to successfully fabricate circular PbO NSs in an isopropanol (IPA) solvent [30]. The TEM image of the circular 2D PbO NSs shows that the diameter of these as-prepared PbO NSs range from ~260 nm to ~400 nm. The HRTEM image exhibits that a clear fringe lattice of ~0.263 nm can be indexed to the (200) plane of the β-PbO crystal [30]. It should be pointed out that the preparation conditions in the LPE, such as the sonication power and time, as well as the solvent and sonication temperature, have a crucial effect on the size and morphology (for NSs or NPs) of the final PbO product.

5. Three-Dimensional PbO Hierarchical Heterostructures

Because of relatively easier manipulation and large-scale preparation compared to 0D, 1D, and 2D nanostructures, 3D hierarchical nanostructures have become more appealing in many fields, such as catalysis [58][59][60], sensors [61][62], optoelectronics [63][64], etc. Although low-dimensional (0D, 1D, and 2D) PbO nanostructures have achieved great progress in recent years, 3D hierarchical PbO nanostructures are also significant due to their unique properties [20][65][66]. For example, Behnoudnia and Dehghani [65] successfully synthesized 3D PbO nanostructures by the thermal decomposition of PbC_2O_4 nanostructures at 420 °C for 30 min. Additionally, Chen et al. [20] reported that 3D PbO nanostars and nanodendrites with several hundreds of nanometers were successfully synthesized when the subphase concentration of round or irregular PbO NPs was 1×10^{-4} mol L^{-1}. It can be seen that the as-synthesized 3D PbO nanostars contain a couple of nanodendrites, and each nanodendrite is composed of a trunk and several branches that are nearly vertical to the predominant trunk. The SAED pattern shows that the as-synthesized 3D PbO nanostars display regularly arranged spots, which can be assigned to those of the β-PbO, suggesting that the as-obtained nanostars are single-crystalline.

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