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# Self-assembly of indium phosphide with an urchin-like architecture through a hydrothermal route

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ABSTRACT

Urchin-like InP architectures were synthesized by hydrothermal route at 160 °C. The products were characterized with scanning electron microscopy, X-ray diffraction, spectrofluorometry. Results show that the hollow urchin-like InP microspheres with diameters of 40–70 μm were composed by numerous microrods with a diameter of about 0.5–2 μm. The obtained InP samples were of high purity zinc blende (cubic) structure. According to the analysis on the effects of reaction time on the morphologies, a possible formation mechanism of hollow urchin-like InP architectures was proposed. The optical properties of the urchin-like InP hollow microspheres were also investigated at room temperature, and results show that the urchin-shape InP microspheres displayed a strong emission at 599 nm, which is quite different from that observed in all previous reports related to the InP nano/micro structures.



## 1. Introduction

Indium phosphide (InP), as an important III–V semiconductor with a direct band gap of 1.35 eV at room temperature, has attracted increasing attention because of their significant applications in fiber optical communications, high-speed electronic devices, and optoelectronic devices, such as high-speed logic circuits, millimetre-wave sources and amplifiers [1–4]. In recent years, different morphologies of InP, such as nanowire [5,6], nanotube [7], sphere-like [8], and peanut-like [9], were fabricated and widely applied. However, there have been rare, if any, reports on the fabrication of InP with a novel urchin-like architecture so far in the literature.

In this paper, we report on the synthesis of urchin-shaped 3D InP structures through a hydrothermal route. The structures, morphologies, and optical properties of produces were studied. Based on the characterization of the products, a possible growth mechanism of the urchin-like InP structures was proposed.

## 2. Experimental

In a typical synthesis of urchin-like InP architecture, 2 mmol of $InCl_3 \cdot 4H_2O$ was dissolved in 15 mL of NaOH aqueous solution (1.0 mol/L). Then 1.7 mmol of cetyltrimethylammonium bromide (CTAB), 20 ml of n-octane, and 2 ml of n-hexanol were added into the solution under vigorous stirring for about 60 min to form microemulsion solution. Afterward, 9 mmol of white phosphorus ($P_4$) and 3.5 mmol of iodine ($I_2$) were added in order. Finally the newly formed microemulsion solution was transferred into a 50 mL stainless Teflon-lined autoclave and heated at 160 °C for certain time and then cooled to room temperature naturally. The product was filtered and washed with xylene, ethanol, dilute HCl (0.3 mol/L) and distilled water several times. Finally, white precipitate was obtained by centrifugation and following drying at 60 °C.

Crystal structures, morphologies and compositions of the prepared samples were characterized by using X-ray diffractometer (XRD, Cu Kα, λ = 0.1541874 nm), field-emission scanning electron microscope (FESEM, JEOL-6300) equipped with an energy dispersive X-ray spectrometer (EDS). Photoluminescence (PL) spectrum was recorded on an Edinburgh FLSP920 fluorescence spectrometer (excited with a He-Cd line at 325 nm).

## 3. Results and discussion

### 3.1. Morphology and structure

Fig. 1 shows different magnification SEM images of the samples prepared at 160 °C for 24 h. It can be seen from Fig. 1(a) that these urchin-like spheres, with diameters ranging from 40 to 70 μm, were hollow, as shown by an arrow. Enlarged image of microurchin in Fig. 1(b) shows that the urchin-like sphere InP was composed of many microrods with shape-tips. The InP microrods radiated from the center of the crystals, forming an urchin-shaped spherical structure. Fig. 1(c) shows the high magnification SEM image of a typical InP microurchin. The InP microrods are about 0.5–2 μm in width and several micrometers in length. The inset is EDS spectrum of the InP microurchins. The strong

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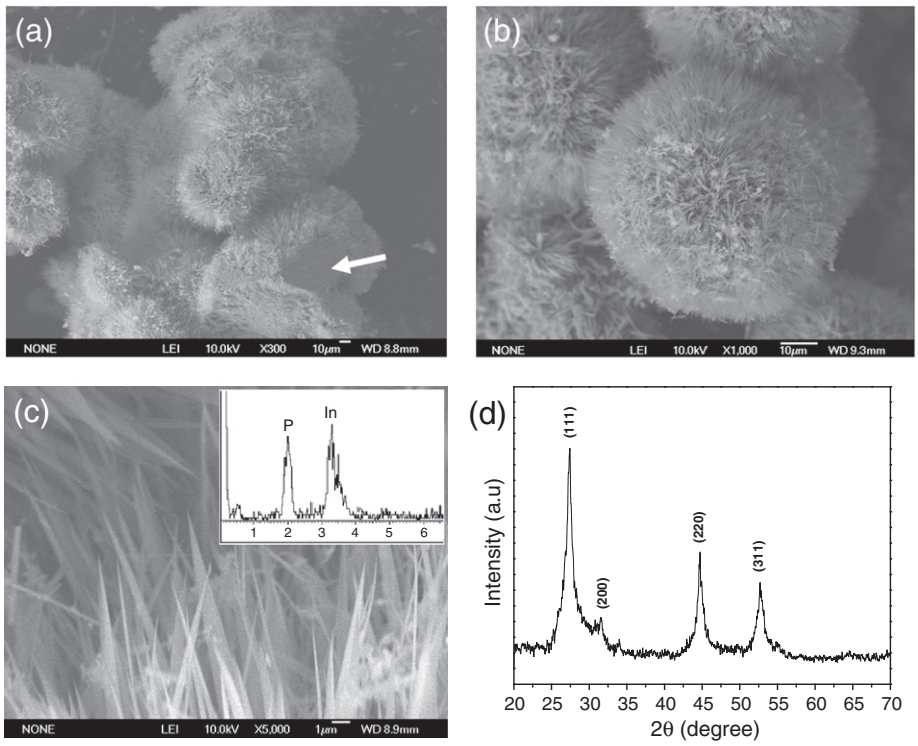


**Fig. 1.** (a–c) SEM images with different magnification of the samples prepared at 160 °C for 24 h. The inset is EDS spectrum. Fig. 1(d). XRD pattern of InP microurchins.

peaks of In and P were clearly present, and no other peaks from impurities were detected. This confirmed that the synthesized product was high purity InP. Fig. 1(d) is the XRD patterns of the as-synthesized sample. All the peaks were well indexed as zinc blende (cubic) structure of InP (JCPDS no.32–0452, a = 0.5869 nm) and the sample had high phase purity.

### 3.2. Formation of the urchin-like InP structure

To investigate the formation mechanism of the urchin-like InP structure, time-dependent growth was investigated by hydrothermal reaction at 160 °C for 12 h, 18 h, and 24 h, and the morphologies of the samples are shown in Fig. 2. After reaction at 160 °C for 12 h

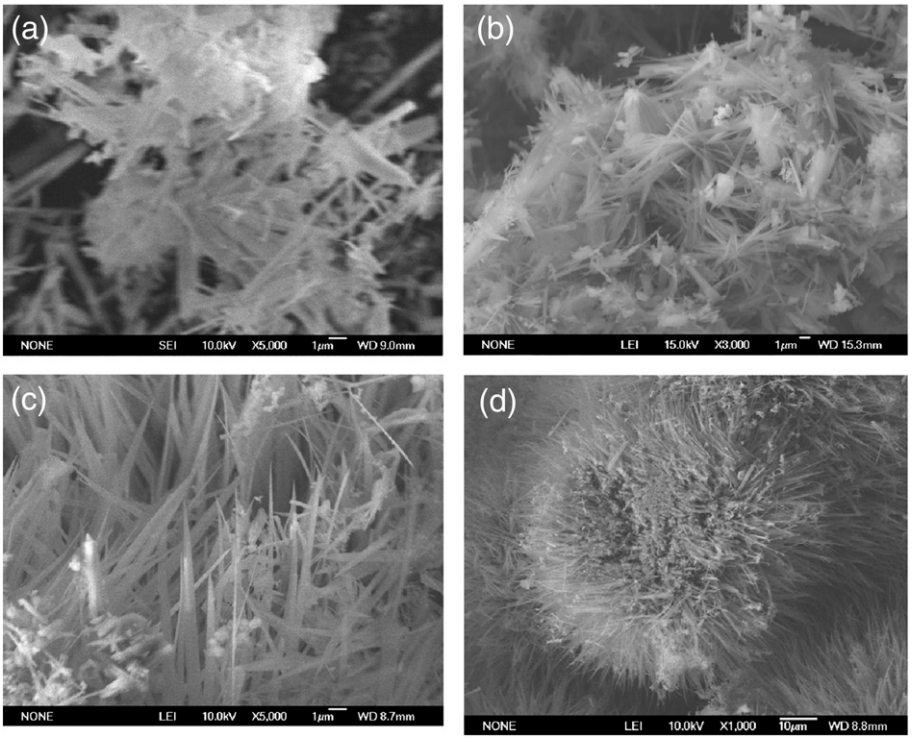


**Fig. 2.** SEM images of urchin-like InP microspheres synthesized at 160 °C for 12 h (a), 18 h (b) and 24 h (c). Low-magnification image of urchin-like InP microspheres synthesized at 160 °C for 24 h(d).