

Synthesis and TEM observation of fluffy hollow carbon spheres by FeCl₃ catalyzed solvent-thermal reaction

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Abstract

Fluffy hollow carbon spheres with the diameter of 0.3–1.8 μm and shell thickness of 30–80 nm were successfully synthesized via FeCl₃ catalyzed solvent-thermal reaction in a sealed autoclave at 350 °C for 3 h, with CaC₂ and CCl₄ as carbon sources and reactants. XRD and TEM observations verify that the fluffy hollow carbon spheres are composed of curved and disordered carbon flakes with the interlayer instance of approximate 0.35 nm. Furthermore, MCNTs with Fe–Mn–Cr nanorods encapsulated are also observed. A possible growth mechanism for the fluffy hollow carbon sphere is proposed.

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Keywords: Hollow carbon spheres; Solvent-thermal reaction; Electron microscopy; Microstructure

1. Introduction

Onion-like carbon spheres were first synthesized by Iijima [1] in 1987. Recently, spherical carbon is becoming a hot and fashionable subject in carbon field due to its potential applications in many industrial fields as catalyst supports [2], lithium-ion secondary batteries [3], drug delivery [4], energy storage medium [5], injectable scaffolds for tissue regeneration [6], etc. Many methods have been developed to produce spherical carbon, mainly including chemical vapor deposition [7,8], self-generated template approach [9], hydrothermal reaction [10] and arc discharge [11].

Hollow spherical carbon exhibits unique properties with low weight, thermal insulation, and high compressive strength. However, reports about hollow carbon structure are

very limited. In this paper, we describe a new route for synthesizing fluffy hollow carbon spheres by FeCl₃ catalyzed solvent-thermal reaction with CaC₂ and CCl₄ as carbon sources. This is the first report where CaC₂ was used as a

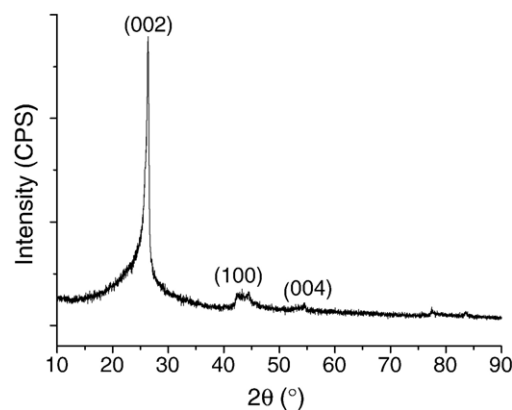


Fig. 1. XRD pattern of the washed product.

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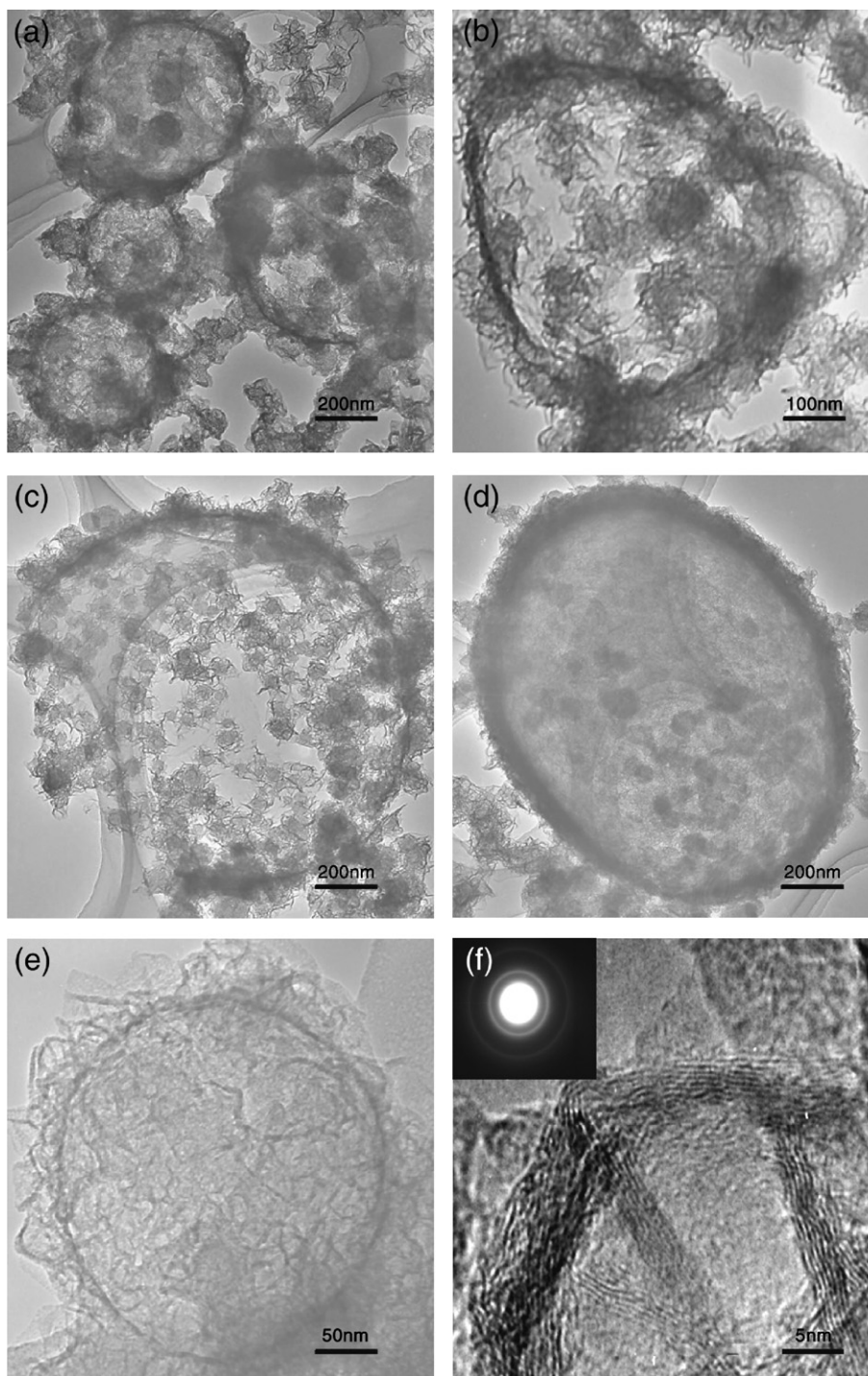
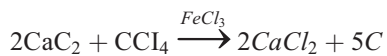


Fig. 2. TEM images of fluffy hollow carbon spheres (a–e) and HRTEM image of thin edge (f) and SAD pattern.

carbon precursor to get hollow spherical carbon. The reaction can be represented as



2. Experiment

In this experiment, 14.1 g of CaC_2 powder (experimental reagent, >95%), 10 ml of CCl_4 (A.R grade purity, >99%) and

0.9 g of FeCl_3 powder (A.R grade purity, >99%) were put into a 100 ml stainless steel autoclave. The air in the autoclave was excluded by argon gas. The temperature change was recorded through a thermocouple installed in the autoclave. The autoclave was sealed and heated to reaction temperature and kept at 350 °C for 3 h. After the reaction, the autoclave was cooled to room temperature naturally. The light and cotton-shape dark product was carefully collected and washed with dilute hydrochloric acid, absolute ethanol

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