Facile Synthesis of Yolk–Shell Bi@C Nanospheres with Superior Li-ion Storage Performances

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Abstract

Recently, exploring appropriate anode materials for current commercial lithium-ion batteries (LIBs) with suitable operating potential and long cycle life has attracted extensive attention. Herein, a novel anode of Bi nanoparticles fully encapsulated in carbon nanosphere framework with uniform yolk–shell nanostructure was prepared via a facile hydrothermal method. Due to the special structure design, this anode of yolk–shell Bi@C can effectively moderate the volume exchange, avoid the aggregation of active Bi nanoparticle and provide superior kinetic during discharge/charge process. Cycling in the voltage of 0.01–2.0 V, yolk–shell Bi@C anode exhibits outstanding Li⁺ storage performance (a reversible capacity over 200 mAh g^{−1} after 400 cycles at 1.25 A g^{−1}) and excellent rate capability (a capacity of 404, 347, 304, 275, 240, 199 and 163 mAh g^{−1} at 0.05, 0.1, 0.25, 0.5, 1.0, 1.8 and 3.2 A g^{−1}, respectively). This work indicates that rational design of nanostructured anode materials is highly applicable for the next-generation LIBs.

Keywords Yolk–shell structure · Li-ion batteries · Alloying type · Bismuth · Rate capability

1 Introduction

The excessive consumption of traditional fossil fuels causes daily increased energy crisis and environmental problems. Due to the high working potential and high energy density of lithium-ion batteries (LIBs), they have been widely applied in mobile phones, laptops, electric vehicles and other portable electronics [1–4]. Recently, the ever growing demand of energy storage devices results in a demand of high-performance LIBs with long cycle life. However, further enhancing their energy density and cycle life is hampered by the limited electrode materials. For example, the conventional graphite anode with a theoretical capacity of only 372 mAh g^{−1} cannot meet the demand of the next-generation LIBs

[5–7]. An obvious challenge here is the absence of suitable anode materials with superior rate performance and stable cycle life [8–10]. Among the current anode materials, transition metal compounds mostly with a high discharge/charge platform will seriously decrease the energy density in full cells [11–13], while carbon materials with low energy and volume density influence the integral cost–performance ratio of LIBs. Alloying-type anodes with high energy density, good electronic conductivity and appropriate discharge/charge platform have stimulated researchers' attention [14, 15]. However, the alloying process of these materials normally accompanies with serious volume fluctuation, which leads to severe structural deformation or pulverization and thereby rapid capacity decay [16–18].

Bismuth (Bi), a typical alloying-type anode, has an ultra-high volume energy density of 3800 mAh cm^{−3}, which is five times compared to that of graphite (756 mAh cm^{−3}) [19, 20]. Additionally, Bi also has advantages of both narrow band gap and high ion conductivity [21, 22]. And many works have been carried out to design appropriate Bi-based anodes for secondary batteries [19–25]. In the work of Li's group, nanosized Bi@NC was prepared via a simple carbonization of Zn-based organic framework and a subsequent replacement of Zn with Bi³⁺. The resulted N-doped carbon-embedded Bi@NC displayed a charge capacity of 285 mAh

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g^{-1} at 0.1 A g^{-1} over 100 cycles [24]. Ji and co-workers rationally designed Bi@NC nanorods via in situ carbonization and reduction of Bi_2S_3 @polypyrrole, and these Bi@NC nanorods achieved a high volumetric capacity over 1700 mAh cm^{-3} at 1.0 A g^{-1} after 500 cycles [25]. For the comparison of Li storage performance with commercial Bi, these modified Bi anodes are apparently enhanced, but for practical application the electrochemical performance still needs further to be improved. Carbon encapsulation and structure design are the most effective strategies to buffer the volume change and improve the structure stability [26]. Such method has been successfully applied in a few alloying-type anodes, including peapod-like $\text{Cu}_3\text{Ge/Ge@C}$ [27], 3D carbon-embedded Sb@C [28], yolk-shell Sb@C [29] and yolk-shell $\text{Sn}_4\text{P}_3\text{@C}$ [30]. Due to the special structure design and carbon encapsulation modification, the rate capability and cycling performance of these anodes are dramatically improved [31–33].

Herein, uniform yolk-shell Bi@C nanospheres have been rationally designed via a facile hydrothermal and subsequent annealing treatment. Due to the special design, such kind of Bi@C anode has multiple advantages: (1) Yolk-shell structure endows more void space to moderate the volume exchange; (2) the framework of continuous carbon shell effectively avoids the aggregation of Bi nanoparticles during the repeated cycles; and (3) nanosized Bi significantly shorts the diffusion distance of Li^+ , further reduces the energy barrier for Li^+ transportation and accelerates the electrochemical reaction kinetics. Possessing these merits, yolk-shell Bi@C anode exhibits outstanding cycling stability (a high reversible capacity over 200 mAh g^{-1} after 400 cycles at 1.25 A g^{-1}) and outstanding rate performance (a capacity of 404, 347, 304, 275, 240, 199 and 163 mAh g^{-1} at 0.05, 0.1, 0.25, 0.5, 1.0, 1.8 and 3.2 A g^{-1} , respectively.)

2 Experimental

2.1 Materials Synthesis

Bi@C precursor was prepared via a typical hydrothermal method [34]. $6.0 \text{ g C}_6\text{H}_{12}\text{O}_6$, $2.0 \text{ g Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ and 1.0 g PVP ($M_w = 40,000$) were dissolved in 20 mL ultrapure water with strong magnetic stirring. Then, $10 \text{ mL CH}_3\text{COOH}$ was poured into the above solution and stirred for another 20 min . After that, the above homogeneous solution was poured into a 100 mL Teflon-lined stainless-steel autoclave and maintained at 200°C for 1 h . After naturally cooling down to room temperature, the precipitate was centrifuged with ultrapure water and ethanol for three times and dried at 60°C for 12 h in a vacuum oven. Finally, yolk-shell Bi@C nanospheres were obtained by annealing the precursor in a Ar-flowing tube furnace at 600°C for 2 h with 3°C min^{-1} .

2.2 Materials Characterization

The morphology and structure information of Bi@C nanospheres was characterized by a field emission scanning electron microscope (FESEM, Zeiss Gemini DSM 982). The crystal structure of Bi@C composites was examined using a PANalytical Empyrean X-ray diffraction (XRD) equipment at 40 kV and 45 mA with CuK_α radiation. The detailed microstructure information of Bi@C nanospheres was further revealed by a transmission electron microscope (TEM, JEOL 2100 Co., Japan) with an operating voltage of 200 kV .

2.3 Electrochemical Tests

The working electrode was prepared by mixing Bi@C nanospheres with sodium alginate and conductive carbon black (with a weight ratio of 70:15:15) in a mortar with appropriate ultrapure water to homogeneous slurry and then coated on a Cu foil and dried at 100°C for overnight in a vacuum oven. After that, the dried Cu foil was cut into 12-mm disks with a mass loading of $1.5\text{--}1.8 \text{ mg cm}^{-2}$. The coin cell (CR2016) with Bi@C anode was assembled in an Ar-filled glove box with H_2O and O_2 below 0.1 ppm . 1 M solution of LiPF_6 in a mixture of diethyl carbonate/ethylene carbonate (DEC/EC, 1:2 by vol) containing $10 \text{ wt\%}$ addition of fluoroethylene carbonate (FEC) was used as the electrolyte, Li foil as a counter-electrode and Celgard2400 as a separator. Cyclic voltammetry (CV) profiles were recorded by Gamry Interface 1000 electrochemical workstation in the voltage of $0.01\text{--}2.0 \text{ V}$ at different scan rates. Galvanostatic discharge/charge testing was measured from 0.01 to 2.0 V , with a Land 2001A multichannel battery testing system.

3 Results and Discussion

The fabrication process of yolk-shell Bi@C nanospheres is schematically depicted in Fig. 1a. As shown in the figure, the anode of yolk-shell Bi@C was prepared by a first hydrothermal crystallization and subsequent annealing in Ar-flowing tube furnace. It is worth noting that the annealing process induces the transformation of glucose-derived carbon compound into an amorphous carbon coating layer. The detailed crystalline information was revealed by XRD characterization, and the results are provided in Fig. 1b. It is obvious that the diffraction peaks of products well match with the criterion JCPDS card No.05-0519 with a typical rhombohedral phase. Obviously, all the distinct XRD peaks can be indexed to the crystalline plane of (003), (012), (104), (106), (015), (113), (202), (024), (107), (116) and (122). The Raman spectrum (Fig. 1c) displays the graphitic G band

Fig. 1 **a** Schematic illustration of the fabrication of yolk–shell Bi@C nanospheres; **b** XRD patterns of Bi@C nanospheres; **c** Raman spectrum of yolk–shell Bi@C nanospheres; **d**, **e** low- and high-magnification SEM images of Bi@C composites

and defect-induced D band in the yolk–shell Bi@C with an area ratio of the D band to G band (I_D/I_G) about 1.35, indicating considerable defects in the carbon shell. SEM was conducted to analyze the morphology and microstructure of Bi@C sample. As depicted in Fig. 1d, the final Bi@C displays a well uniform nanosphere shape with a rough surface. The high-magnification image (Fig. 1e) reveals that Bi@C spheres have an average diameter of about 600 nm and Bi exists as nanoparticles distributed in the core of sphere. Through the above analysis, the core–shell structure has been successfully prepared via this rationally designed hydrothermal route. The uniform carbon-coated Bi@C nanospheres could be promising with stable Li-ion storage performance.

Additionally, TEM was applied to further reveal the structure information of these yolk–shell Bi@C nanospheres. As depicted in Fig. 2, TEM images of Bi@C nanospheres with different magnifications (Fig. 2a–c) clearly exhibit that all Bi@C nanospheres have a diameter of approximately 600 nm, which is well consistent with the SEM results. It can

be observed that most of Bi exists as nanoparticles with the size of about 100 nm, and embeds in the core of the nanosphere with abundant of voids between nanoparticles. The thickness of carbon layer can also be observed with about 150 nm. It is worth noting that large amount of ultrafine Bi nanoparticles distributed in the carbon shell is attributed to the Bi diffusing into carbon materials during annealing process at 600 °C, resulting in abundant void spaces. High-resolution TEM (HRTEM) was also carried out to further analysis the detailed structure information of yolk–shell Bi@C spheres. Figure 2d,e reveal that dense Bi nanoparticles are fully encapsulated in the carbon framework. Figure 2f displays a crystalline lattice of a big Bi particle with a d -spacing of 3.28 Å, which can be indexed to the (012) plane of rhombohedral Bi.

The Li^+ storage electrochemical performance of these yolk–shell Bi@C nanospheres was measured with coin cells (CR2016). Figure 3a displays the initial three CV profiles of Bi@C anode at 0.1 mV s^{-1} . Apparently, the initial CV profile is different from others at the cathodic scan fraction,

Fig. 2 a–c Low- and high-magnification TEM images of yolk–shell Bi@C nanospheres; d–f the corresponding HRTEM images of Bi@C

and the cathodic peaks could be ascribed to the formation of solid electrolyte interface (SEI) combined with the alloying reaction of Bi to form Li_3Bi [35]. The distinct anodic peak around 0.9 V is corresponding to de-alloying of Li_3Bi . During the second and third cathodic scans, three reduction peaks around 0.78 V, 0.65 V and 0.46 V are associated with the multistep lithiation of Bi and result in Li_3Bi [36, 37]. These well-overlapped CV curves indicate the highly reversible lithiation/delithiation process of yolk–shell Bi@C. The first three capacity–voltage profiles are provided in Fig. 3b, which directly show a high initial discharge/charge capacity of 821 and 470 mAh g^{-1} at 0.1 A g^{-1} , respectively, with a Coulombic efficiency of 57.2%. Normally, the discharge/charge voltage platforms of these profiles are well consistent with the CV analysis. The capacity–voltage profiles at different current densities (Fig. 3c) display a similar shape with a little variation in the voltage platforms, which can be ascribed to the increased polarization influence at high current densities. Besides, it can be observed that the capacity of yolk–shell Bi@C of 404, 347, 304, 275, 240, 199 and 163 mAh g^{-1} sustained at 0.05, 0.1, 0.25, 0.5, 1.0, 1.8 and 3.2 A g^{-1} , respectively. The corresponding rate performance (Fig. 3d) displays stable cycling performance at each current density, revealing the high structure stability of these yolk–shell Bi@C nanospheres. The cycling performance was also measured by galvanostatic discharge/charge testing. Figure 3e exhibits yolk–shell Bi@C anode cycling in the voltage of 0.01–2.0 V, achieving a high reversible capacity

over 272 mAh g^{-1} after 80 cycles at 0.5 A g^{-1} . Figure 3f displays the long cycle performance for 400 cycles at a high current density of 1.25 A g^{-1} , and the current Bi@C anode can still achieve a reversible capacity of 272 mAh g^{-1} . The superior storage performance of Bi@C anode is benefitted from the particular yolk–shell structure, which endows enough interspace for the volume change during the Li-ion insertion/extraction. Furthermore, the outer carbon layer effectively restrains the aggregation of active Bi nanoparticles and keeps the nanostructure from collapsing, thus resulting in a stable Li^+ storage performance.

To further explore the origin of outstanding Li^+ storage performance that yolk–shell Bi@C nanospheres possess, CV profiles at various scan rates from 0.2 to 2.0 mV s^{-1} were carried out to calculate the Li^+ diffusion coefficient and the results are provided in Fig. 4a. Obviously, these CV profiles display similar skeleton with a slight deflection of the oxidation/reduction peaks, and this could be associated with the increased polarization influence at high scan rates. According to previous theory, the peak current (I_p) and the scan rate (ν) obey the following Randles–Sevcik equation [38]:

$$I_p = 2.69 \times 10^5 A C D_{\text{Li}^+}^{1/2} n^{3/2} \nu^{1/2}, \quad (1)$$

where C is the shuttle concentration (mol cm^{-3}), A is the electrode area (cm^2), D_{Li^+} is the Li^+ diffusion coefficient, n is the transferred electrons at the oxidation peaks ($n=3$) and ν is the scan rate (V s^{-1}). By fitting $\nu^{1/2}$ vs I_p (Fig. 4b),

Fig. 3 Li⁺ storage performance of yolk-shell Bi@C nanospheres: **a** CV curves at 0.1 mV s⁻¹; **b** the first three discharge/charge profiles; **c** discharge/charge profiles at different current densities from 0.05 to 3.2 A g⁻¹; and **d** the corresponding rate performance; **e**, **f** cycling performance at 0.5 A g⁻¹ and 1.25 A g⁻¹, respectively

we can obtain the fitted straight line with a slope of about 0.13. Thus, the calculated Li⁺ diffusion coefficient of the yolk-shell Bi@C is approximately $6.9 \times 10^{-7} \text{ cm}^2 \text{ s}^{-1}$. These data suggest that such yolk-shell Bi@C anode with a high Li⁺ diffusion coefficient, facilitating the Li⁺ diffusion, thus results in superior rate capability. Furthermore, electrochemical impedance spectroscopy (EIS) analysis was performed to measure the diffusion kinetics of yolk-shell Bi@C anode. As exhibited in Fig. 4c, two semicircles in the high- and medium-frequency region represent the charge transfer resistance at the two-phase interface and the diffuse resistance in the carbon shell, respectively, and the slope line in the low-frequency region is the Warburg impedance of Li⁺ diffusing into the electrode. To better understand the electrochemical performance level of such yolk-shell Bi@C anode, the detailed comparison of rate capability with previously reported Bi-based anodes for LIBs is provided in

Fig. 4d. Evidently, this yolk-shell Bi@C anode exhibits a better rate capability than previously reported Bi-based anodes [16, 24, 35–37]. The outstanding Li⁺ storage performances are closely linked to that of the yolk-shell structure which endows more interspace to mitigate the volume variation, and carbon shell framework effectively avoids the aggregation of Bi nanoparticle during the discharge/charge cycles. Furthermore, nanosized Bi significantly reduces the diffusion distance of Li⁺, further decreases the energy barrier for Li⁺ transportation and accelerates the electrochemical reaction kinetics.

Fig. 4 **a** CV curves of yolk-shell Bi@C nanospheres from 0.2 to 2.0 mV s⁻¹; **b** the fitting curve of the peak current (*I*_p) vs scan rate (*v*^{1/2}); **c** EIS curve of yolk-shell Bi@C nanospheres after 80 cycles at 0.5 A g⁻¹; **d** the rate capability comparison of this yolk-shell Bi@C anode with reported Bi-based materials

4 Conclusion

In summary, uniform yolk-shell Bi@C nanospheres have been successfully prepared via a facile hydrothermal crystallization and annealing treatment. The yolk-shell Bi@C anode exhibits a capacity of 200 mAh g⁻¹ over 400 cycles at 1.25 A g⁻¹ and displays a capacity of 404, 347, 304, 275, 240, 199 and 163 mAh g⁻¹ at 0.05, 0.1, 0.25, 0.5, 1.0, 1.8 and 3.2 A g⁻¹, respectively. The outstanding Li⁺ storage performance of yolk-shell Bi@C is ascribed to that the coated carbon framework effectively prevents the aggregation of active Bi nanoparticle, palliates the magnitude volume exchange and protects the nanostructure from destroying during cycling. Such a simple design strategy lays a solid foundation for the synthesis of nanostructured materials and broadens a way for the development of battery materials.

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