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Rationally Designed Three-Layered Cu₂S@Carbon@MoS₂ Hierarchical Nanoboxes for Efficient Sodium Storage

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Abstract

Hybrid materials, integrating the merits of individual components, have been proven as ideal structures for efficient sodium storage. However, the construction of hybrid structures with decent physical/electrochemical properties is still challenging. Herein, we report the elaborate design and synthesis of hierarchical nanoboxes composed of three-layered Cu₂S@carbon@MoS₂ as anode materials for sodium-ion batteries. Through a facile multistep template-engaged strategy, ultrathin MoS₂ nanosheets are grown on nitrogen-doped carbon-coated Cu₂S nanoboxes to realize the Cu₂S@carbon@MoS₂ configuration. The rational design can efficiently shorten the diffusion path of electrons/Na⁺ ions, accommodate the volume change of electrodes during cycling, enhance the electric conductivity of the hybrids, and offer abundant active sites for sodium uptake. By virtue of these structural and compositional advantages, these three-layered Cu₂S@carbon@MoS₂ hierarchical nanoboxes manifest excellent electrochemical properties in terms of decent rate capability and stable cycle life.

Keywords: MoS₂, Cu₂S, nitrogen-doped carbon, nanoboxes, sodium-ion batteries

Sodium-ion batteries (SIBs) have attracted widespread attention from both academic and industrial battery communities in recent years, in view of the fascinating merits of natural abundance and low cost of sodium resources.^[1-4] Tremendous efforts have been put in exploring suitable anode materials with desirable electrochemical properties. Owing to the large compositional diversity and decent electrochemical reversibility, metal sulfides have been shown as attractive anode materials for SIBs.^[5, 6] For example, Co_xS ,^[7, 8] Cu_xS ,^[9, 10] Sn_xS ,^[11, 12] MoS_2 ,^[13, 14] and Fe_xS ^[15, 16] have been reported to deliver high reversible capacity. However, metal sulfides still face some challenges for practical applications, including poor electric conductivity, huge volume changes, and sluggish ionic transport kinetics.^[17, 18] Rational designs in composition and structure have been proven as effective strategies to address these issues. In particular, compared to single metal sulfides, the incorporation of different metal sulfides into one smart hybrid nanostructure has been proposed to facilitate the electrochemical reactions due to the preserved advantages of individual components and the possible synergetic effects (i.e., enhanced electric/ionic conductivity, electrochemical activity, and mechanical stability).^[19-22] For example, we have designed hierarchical CuS@CoS_2 double-shelled nanoboxes with decent electrochemical performance.^[23]

Owing to the good electric conductivity and low cost, carbonaceous materials, especially the nitrogen-doped carbon, have been widely adopted to decorate the electrode materials thus facilitating the electrochemical properties.^[24, 25] On the other hand, hierarchical hollow nanostructures with high surface area and low-dimensional nanoscale building units have shown prominent advantages for sodium storage.^[26-28] The hollow configurations can help to buffer the strain during sodium insertion/extraction, and the permeable nanoshells can promote the transport of electrons/ Na^+ ions and offer a large contact area for full infiltration of electrolyte.^[29, 30]

On account of the aforementioned considerations, the integration of these strategies into one alliance may greatly contribute to the creation of new nanostructures with boosted sodium storage capability. Herein, we report the delicate design and fabrication of three-layered

Cu₂S@carbon@MoS₂ hierarchical nanoboxes through a facile multistep template-engaged strategy. Starting from Cu₂O nanocube templates, CuS nanoboxes are formed via the sequential sulfidation and etching processes (**Figure 1**). Afterwards, the CuS nanoboxes are coated with polydopamine (PDA) and annealed to form the Cu₂S@carbon nanoboxes. Lastly, the hierarchical Cu₂S@carbon@MoS₂ nanoboxes are obtained by growing a layer of MoS₂ on the Cu₂S@carbon nanoboxes. The as-synthesized three-layered Cu₂S@carbon@MoS₂ hollow architectures with hybrid shells and nanosized building blocks fully integrate the advantages of each component. As expected, the Cu₂S@carbon@MoS₂ hierarchical nanoboxes manifest superior electrochemical properties with high specific capacity and stable cycle life.

Cu₂O nanocubes synthesized through a reduction method are adopted as the starting templates. The Cu₂O nanocubes are highly uniform with an average size of about 600 nm (**Figure 2a**, e and Figure S1a-c, Supporting Information (SI)). Powder X-ray diffraction (XRD) (Figure S1d, SI) and energy-dispersive X-ray (EDX) (Figure S2, SI) analyses indicate the phase purity of the Cu₂O nanocubes. When the Cu₂O nanocubes are immersed in a proper amount of Na₂S solution, the Cu₂O@CuS core-shelled nanocubes are formed.^[31, 32] Field-emission scanning electron microscopy (FESEM) and transmission electron microscopy (TEM) images (Figure 2b, f and Figure S3, SI) show that the as-generated particles maintain the cubic shapes, while EDX spectrum confirms the presence of S element in the nanocubes (Figure S4, SI). Afterwards, the Cu₂O core can be selectively dissolved by sodium thiosulfate solution to generate the CuS nanoboxes. Most of the diffraction peaks in the XRD pattern of the obtained product correspond to hexagonal CuS (Figure S5a, SI), and the elemental composition of Cu and S is further confirmed by the EDX spectrum (Figure S5b, SI). The cubic nanostructure is well preserved after the etching of the Cu₂O core (Figure 2c and Figure S6a, b, SI). The hollow interior is further elucidated by TEM and FESEM observations (Figure 2g and Figure S6c, d, SI). A uniform PDA layer is then coated on the CuS nanoboxes to achieve the CuS@PDA nanoboxes. FESEM and TEM images reveal that the CuS@PDA product well inherits the nanobox

structure but the surface becomes slightly rough (Figure S7, SI). EDX spectrum also suggests the successful coating of PDA layer on the CuS nanoboxes (Figure S8, SI). During a subsequent high temperature treatment, the PDA layer is carbonized to form nitrogen-doped carbon, which can simultaneously reduce CuS into Cu₂S.^[33, 34] The nanobox structure is well preserved (Figure 2d, h and Figure S9a-c, SI), and the elemental mapping images clearly confirm the coating of N-doped carbon on the structure (Figure S9d, SI). The XRD pattern and EDX results (Figure S10, SI) validate the transformation of CuS@PDA into Cu₂S@carbon.

Through a facile solvothermal method, ultrathin MoS₂ nanosheets are uniformly grown on the Cu₂S@carbon nanoboxes. The XRD pattern of as-prepared product matches well with those of tetragonal Cu₂S and hexagonal MoS₂ (Figure S11a, SI), and EDX spectrum reveals a Mo/Cu molar ratio of 0.41: 1 in the obtained product (Figure S11b, SI). The D and G bands in the Raman spectrum confirm the existence of carbon (Figure S12, SI).^[35] The contents of carbon and nitrogen are determined to be around 22.9 and 2.1 wt%, respectively, via EDX analysis. The panoramic FESEM images show that the obtained Cu₂S@carbon@MoS₂ particles inherit the cubic structure with a rough surface (**Figure 3a, b** and Figure S13a, SI). The closer observations clearly reveal that the hierarchical shells are composed of randomly assembled ultrathin nanosheets (Figure 3c and Figure S13b, c, SI). The well-preserved hollow configuration of the Cu₂S@carbon@MoS₂ nanoboxes is confirmed by TEM examinations (Figure 3d, e and Figure S13d, SI). No noticeable interlayer gaps can be discerned between the different layers (Figure 3f), indicating that the three layers are highly allied. Consistent with the XRD result, a set of visible lattice fringes with an interplanar distance of 0.62 nm can be assigned to the (002) plane of MoS₂ (Figure 3g). Elemental mappings (Figure 3h) of a single Cu₂S@carbon@MoS₂ nanobox demonstrate that the Cu, S, Mo, C, and N elements are uniformly distributed throughout the entire nanoparticle. For comparison, bare MoS₂ nanosheets are synthesized through a similar method (Figure S14 and S15, SI).

The Cu₂S@carbon@MoS₂ nanoboxes are used as anode materials for SIBs. The sodium storage behavior is first evaluated by cyclic voltammetry (CV) analyses (**Figure 4a**). During the initial cathodic scan, a broad peak at 1.85 V corresponds to the insertion of Na⁺ ions into MoS₂ and Cu₂S.^[36, 37] The following small peak at 0.92 V is related to the further insertion of Na⁺ into Na_xMoS₂ accompanied the formation of a solid electrolyte interphase layer.^[36, 38] A series of peaks at about 0.75, 0.66, and 0.52 V is attributed to the sequential phase transitions with the further intercalation of Na⁺ ions into the Na_xCu₂S structure and the conversion reaction to generate Cu and Na₂S.^[33, 39] The pronounced peak at 0.10 V is related to the conversion reaction (Na_xMoS₂ + Na⁺ → Mo + Na_xS).^[36, 40] In following cycles, two cathodic peaks at around 1.49 and 0.77 V emerge, corresponding to the insertion reaction and conversion reaction of Cu₂S and MoS₂, respectively.^[9, 36, 41] During the anodic scans, the oxidation peaks at 1.60 and 2.19 V are assigned to the desodiation of the Na_xCu₂S,^[9, 33, 41] while the peak at 1.94 V corresponds to the conversion from Na₂S and Mo to the MoS_x.^[36, 40]

Figure 4b displays the representative galvanostatic discharge-charge curves of the Cu₂S@carbon@MoS₂ nanoboxes at a current density of 0.05 A g⁻¹. The electrode delivers an initial charge capacity of 442 mAh g⁻¹ with nearly overlapped discharge-charge voltage profiles of the 2nd and 3rd cycles. The rate capability of the Cu₂S@carbon@MoS₂ nanobox electrode is explored with gradually increased current densities (Figure 4c). When tested at 0.05, 0.1, 0.2, 0.3, 0.5, 1.0, 2.0 A g⁻¹, average capacities of 430, 410, 386, 368, 359, 337, 316 mAh g⁻¹ can be delivered, respectively. Impressively, a decent capacity of 297 mAh g⁻¹ can be maintained with further increasing the current density to 3.0 A g⁻¹. The superb rate capability is also validated by the discharge-charge curves at different current densities (Figure 4d). On the contrary, the capacities of the Cu₂S@carbon nanoboxes and MoS₂ nanosheets show rapid decay with elevation of current density (Figure S16, SI). The rate performance of the Cu₂S@carbon@MoS₂ nanoboxes is also superior to some of the MoS₂-based electrodes.^[13, 42-45]

Moreover, the Cu₂S@carbon@MoS₂ nanobox electrode also demonstrates superb cycling stability. Specifically, the Cu₂S@carbon@MoS₂ nanoboxes manifest stable cyclability over 200 cycles at 0.3 A g⁻¹ with an average capacity fading of only 0.098% per cycle (Figure 4e), which is superior to some of reported MoS₂-based electrodes (Table S1, SI).^[13, 42-44, 46] In comparison, the capacities of the Cu₂S@carbon nanoboxes and MoS₂ nanosheets fade rapidly with cycling. Moreover, the nanobox structure of the Cu₂S@carbon@MoS₂ electrode can still be well preserved after prolonged cycling (Figure S17, SI). The above results highlight the greatly enhanced electrochemical performance of the Cu₂S@carbon@MoS₂ nanoboxes, which should benefit from the unique hierarchical hollow configurations accompanied by the synergetic effect of the three well-designed shells of the sandwich-like Cu₂S@carbon@MoS₂ nanoboxes. Specifically, the inner layer of Cu₂S nanobox backbone can accommodate the large volumetric change during repeated cycling to achieve stable cycling performance. The middle nitrogen-doped carbon could maintain the nanobox structures. Both the Cu₂S backbone and sandwiched nitrogen-doped carbon layer could enhance the electric conductivity of the Cu₂S@carbon@MoS₂ nanoboxes so as to effectively facilitate the charge transfer process as revealed by electrochemical impedance spectroscopy (EIS) spectra (Figure S18, SI). The outer layer of ultrathin MoS₂ nanosheets with high surface area enables the full infiltration of electrolyte and offers decreased diffusion path for electrons/Na⁺ ions to achieve high capacity. Besides, the three-layered alignment could enhance the structural stability of the hybrids, rendering greatly enhanced cycling performance.

To gain some insights into the high performance of the Cu₂S@carbon@MoS₂ nanoboxes, the kinetic study based on CV tests at different scan rates is performed (**Figure 5a**). According to the equation between measured current (*i*) and scan rate (*v*): $i = av^b$, the *b* value can be derived from the relationship between the log values of *i* and *v*.^[23] The *b* value indicates the sodium storage behavior. More specifically, a *b* value of 0.5 signifies a feature of the diffusion-controlled process, whereas a *b* value of 1.0 indicates that the system is mainly controlled by the capacitive process. The results

show that the b value is higher than 0.65 for all the redox peaks (Figure 5b), indicating the dominant fast kinetics of capacitance-controlled behavior. The capacitive contribution in the Cu₂S@carbon@MoS₂ nanoboxes can be further determined by separating the capacitive capacity and diffusion-controlled capacity. For example, the capacitive contribution is prominent with a high value of around 73.1% at a scan rate of 1 mV s⁻¹ (Figure 5c). The proportion of capacitive capacity steadily rises with the elevating scan rate and finally attains 84.4% at 4 mV s⁻¹ (Figure 5d). The high-rate capability and capacitive behavior of the Cu₂S@carbon@MoS₂ nanoboxes might be ascribed to the structural features with a hierarchically hollow structure composed of ultrathin nanoparticles and nanosheets.

In summary, we have demonstrated a multi-step templating strategy for the fabrication of hierarchical three-layered Cu₂S@carbon@MoS₂ nanoboxes. The synthetic approach can readily tailor the structure and composition of the hierarchical hybrid materials. The hybrid conformations with integrated advantages of each component can facilitate the transport and storage of electrons/Na⁺ ions to achieve enhanced sodium storage properties. This work may provide some inspiration for the design and construction of advanced anode materials for sodium-ion batteries.

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Figures and Captions

Figure 1. Schematic illustration of the synthetic process of the three-layered $\text{Cu}_2\text{S}@ \text{carbon}@ \text{MoS}_2$ nanoboxes.

Figure 2. (a-d) FESEM images and (e-h) TEM images of the (a, e) Cu_2O nanocubes, (b, f) $\text{Cu}_2\text{O}@\text{CuS}$ nanocubes, (c, g) CuS nanoboxes, (d, h) $\text{Cu}_2\text{S}@\text{carbon}$ nanoboxes.

Figure 3. (a-c) FESEM images, (d-f) TEM images, and (g) HRTEM image of $\text{Cu}_2\text{S}@\text{carbon}@\text{MoS}_2$ nanoboxes, (h) elemental mapping images of a single $\text{Cu}_2\text{S}@\text{carbon}@\text{MoS}_2$ nanobox.

Figure 4. (a) The 1st to 5th CVs of the Cu₂S@carbon@MoS₂ nanobox electrode at a scan rate of 0.1 mV s⁻¹. (b) Typical discharge-charge voltage profiles of the Cu₂S@carbon@MoS₂ nanobox electrode at a current density of 0.05 A g⁻¹. (c) Rate performance of Cu₂S@carbon@MoS₂ nanobox electrode and (d) the corresponding discharge-charge voltage profiles. (e) Cyclic performance of the Cu₂S@carbon@MoS₂ nanoboxes, MoS₂ nanosheets, Cu₂S@carbon nanoboxes at a current density of 0.3 A g⁻¹.

Figure 5. (a) The CVs of the Cu₂S@carbon@MoS₂ electrode at various scan rates. (b) Determination of the *b* value using the relationship between peak current and scan rate. (c) Separation of the capacitive and diffusion currents in the Cu₂S@carbon@MoS₂ electrode at a scan rate of 1 mV s⁻¹ with the capacitive fraction shown by the shaded region. (d) Contribution ratio of the capacitive and diffusion-controlled charge versus scan rate.

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Three-layered hierarchical $\text{Cu}_2\text{S}@\text{carbon}@\text{MoS}_2$ nanoboxes are synthesized through a multistep template-engaged strategy. With the unique shell architecture and complex composition, these $\text{Cu}_2\text{S}@\text{carbon}@\text{MoS}_2$ nanoboxes exhibit enhanced electrochemical properties as an anode material for sodium-ion batteries.