Yolk-shell SiO@Co₉S₈ particles encapsulated in carbon fibres by electrostatic spinning for lithium-ion battery anodes

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1. Experiment.

1.1 Material synthesis

Polyvinylpyrrolidone (PVP, Mw=58000), polyacrylonitrile (PAN, Mw=150,000), N,N-dimethylformamide (DMF, 99.9%), cobalt nitrate hexahydrate $(Co(NO_3)_2 \cdot$

6H₂O), 2-methylimidazole, and thioacetamide (TAA, 99%). All reagents were used without further purification. 1.2. Characterization of materials

The morphology and structure of the samples were measured using scanning electron microscopy (SEM, Verios G4, USA) and field emission transmission electron microscopy (FETEM, FEI Talos F200X). The crystalline nature of the samples was analyzed by x-ray powder diffraction (XRD, Rigaku, Cu-Ka) at a scanning rate of 4°C/min. The elements of the composites were analyzed by x-ray photoelectron spectroscopy (XPS, Kratos, USA). The composition and phase structure of the final products were further determined by Raman spectrophotometer (Alpha 300R).

1.3. Electrochemical characterization

The prepared electrode materials were cut into 10 mm diameter discs using a slicer and the mass of each disc was measured using a balance; the electrode materials were prepared without the need for conductive agents and adhesives. The CR2016 button cell was assembled in a glove box filled with Ar atmosphere ($O_2 < 23$ ppm and $H_2O < 1$ ppm) using 1M LiPF6 as the electrolyte (VEC: VDMC: VMC = 1:1:1, containing 5% FEC additive). Firstly, the electrode pads were placed into the positive shell, and 45 ul of organic electrolyte was dripped to fully moisten them, and the PP diaphragm was lightly placed on the positive shell, and then the spacer and lithium metal foil were placed on the negative shell. Use tweezers to fasten the positive and negative shells, and after applying pressure in the press, the assembly of the CR2016 button cell was completed. After

resting for a period of time, its electrochemical performance was tested. A multi-channel quiescent current system Land (Land CT200IA) was used for electrochemical testing. The test voltage range was set between 0 and 3.0 volts. Cyclic voltammetry (CV) tests were performed on button cell batteries using a multi-channel electrochemical workstation from Gamry, USA, to study the polarization of lithiumion batteries during the reaction process. The voltage range was set between 0 and 3.0 V with a scan rate of $0.1 \text{ mV} \cdot \text{s}^{-1}$. This test method can help to investigate the performance characteristics and electrochemical behavior of the battery.



2. TEM and EDS images of SiO@ZIF-67 particles.



Fig.S3. TEM and EDS images of SiO CF.



Fig.S4. TEM and EDS images of SiO@Co CF.



g.S5. HRTEM image of SiO@Co CF (a). HRTEM images of SiO@Co $_9S_8$ CF (b and c).



Fig.S6. SEM images of the cross-section of SiO@Co₉S₈CF electrode.



Fig.S7. SiO@Co₉S₈ CF pictures taken with a camera in a natural environment.



Fig.S8. XPS spectra of SiO@Co₉S₈CF films in C 1s and N 1s (a and b).



Fig.S9. Cycling performance of a full cell assembled from SiO@Co₉S₈ CF electrodes after 300 cycles at a current of 0.5 $A \cdot g^{-1}$.



Fig.S10. SEM images of SiO@Co₉S₈CF electrode before cycling (a and b); SEM images of SiO@Co₉S₈CF electrode after 15 cycles at 0.2 A·g⁻¹ (c and d); TEM and EDS images of SiO@Co₉S₈CF electrode before cycling (e); TEM and EDS images of SiO@Co₉S₈CF electrode after 15 cycles at 0.2 A·g⁻¹ (f). SEM images of SiO CF electrode before cycling (g and h); SEM images of SiO CF electrode after 15 cycles at 0.2 A·g⁻¹ (i and j).