Electronic Supporting Information

Two-dimensional hybrid nanosheets of few layered MoSe₂ on reduced graphene oxide as anode for long-cycle-life lithium-ion batteries

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Experimental

Materials synthesis

GO was synthesized using modified Hummer's method.^{1,2} Briefly, 0.6 g graphite and 3.0 g KMnO₄ disolved in 30 mL concentrated sulfuric acid, the mixture was transferred into a 50 mL Teflon lined stainless steel autoclave, the reaction was carried out in ice bath for 2 hours and subsequently sealed and heated at 80 °C for another 2 hours in an oven. After cooling, diluting and centrifuging, the obtained GO was dispersed in deionized water to gain GO solution with concentration of 2.5 mg mL⁻¹

The MoSe₂/rGO hybrids were prepared by a facile solvothermal approach. 0.2419 g Na₂MoO₄ 2H₂O and 10 mL GO solution mixed in 15 mL DMF and ultrasonic dispersion homogeneously. Stoichiometric Se powder was dissolved into 10 mL of 80% N₂H₄•H₂O in the open air stirring for about 1 hour forming the hydrate-Se solution. Subsequently, the hydrate-Se solution was added into Na₂MoO₄ solution slowly, and then transferred to a 50 mL autoclave and kept in an electrical oven at 200°C for 24 hours. Finally, the precipitates were collected by centrifugation and washed with deionized water and anhydrous ethanol several times. To obtain the final products, the precipitates were annealed at 500°C for 4 h under an argon atmosphere. The synthesis procedures of the bulk MoSe₂ are similar to that of MoSe₂/rGO hybrids, but without adding GO solution in the solvothermal process.

Materials Characterizations

X-ray diffraction (XRD, Rigaku D/max 2500) measurements were performed to investigate the crystallographic phase of the as-synthesized MoSe₂/rGO hybrids. TG trace of MoSe₂/rGO hybrids was obtained using a combined DSC/TGA instrument (TGA, NETZSCH STA 449C thermobalance).

The temperature and heat flow were calibrated using standard materials (indium and zinc) in air at a heating rates of 10°C min⁻¹. Raman spectra of powder samples were recorded on Raman microscope (Horiba Jobin Yvon, Lab Ram Aramis) with a laser excitation wavelength of 532 nm. The X-ray photoelectron spectroscopy (XPS) analysis was performed on an AXIS-ULTRA DLD-600W system. The morphologies and sizes of the as-prepared products were characterized by scanning electron microscopy (SEM, Quanta FEG 250), transmission electron microscope (TEM, JEOL JEM-2100F) images and High-resolution transmission electron microscope (HRTEM).

Electrochemical measurements

The electrochemical properties were carried out via stainless-steel coin cells (CR 2016). Cathode electrodes were obtained with 80% as-synthesized MoSe₂/rGO hybrids, 10% acetylene black, and 10% polyvinylidene fluoride (PVDF) binder. The cells were assembled in a glove box (Mbraun, Germany) filled with ultra-high purity argon using polypropylene membrane as the separator, Lithium metal as the anode, and 1M LiPF₆ in ethylene carbonate/dimethyl carbonate (EC/DMC) (1:1 v/v) as the electrolyte (Shenzhen Capchem Technology Co., Ltd.). Cyclic voltammetry (CV) of MoSe₂/rGO coin cell was tested using an electrochemical workstation (CHI660C, China) at a scan rate of 0.1 mV s⁻¹ in the voltage range of 0.01 V – 3 V (vs. Li/Li⁺). The galvanostatic charge/discharge experiments were studied in a potential range of 0.01 V – 3 V (vs. Li/Li⁺) using a multichannel battery testing system (Land CT 2001A). The electrochemical impedance spectrometry (EIS) was performed on a ZAHNER-IM6ex electrochemical workstation (ZAHNER Co., Germany) in the frequency range of 100 kHz to 10 m Hz on a cell. The loading of the MoSe₂/rGO cathode material for coin cell test is about 1-2 mg. The specific capacity and current density are calculated on the basis of the weight of

MoSe₂/rGO hybrids

Table S1. $R_{\rm f}$ and $R_{\rm ct}$ values of the MoSe₂/rGO hybrids and bulk MoSe₂ electrodes obtained bysimulation of equivalent circuit model.

Samples	$R_{ m f}\left(\Omega ight)$	$R_{\rm ct}\left(\Omega ight)$
MoSe ₂ /rGO hybrids	4.29	28.02
Bulk MoSe ₂	31.81	186.5



Figure S1. SEM images of bulk MoSe₂



Figure S2. The cyclic voltammetry (CV) curves of the selected cycles for bulk $MoSe_2$ at a scan rate of 0.1 mV s⁻¹.



Figure S3. Charge-discharge curves of the first three cycles of bulk MoSe₂ at 100 mA g⁻¹.



Figure S4. Cycling performance and coulombic efficiency of bulk MoSe₂.



Figure S5. Cycling performance and coulombic efficiency of MoSe₂/rGO hybrids at 2 and 5 A g⁻¹.



Figure S6. Nitrogen adsorption-desorption isotherms of MoSe₂ and MoSe₂/rGO.

Notes and references

1. C. Bao, L. Song, W. Xing, B. Yuan, C. A. Wilkie, J. Huang, Y. Guo and Y. Hu, *J. Mater. Chem.*, 2012, **22**, 6088-6096.

2. W. S. Hummers Jr and R. E. Offeman, J. Am. Chem. Soc., 1958, 80, 1339-1339.