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High-rate capability and Superior cyclability of flower-like Sb₂S₃ anode for high capacity sodium-ion batteries

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Experimental Details

Materials Synthesis

All of the reactants and solvents were of analytical grade and used without further purification. Sb₂S₃ nanostructures were prepared *via* a facile polyol refluxing process. To get Sb₂S₃ microflowers, 3 mmol antimony trichloride and 2.5 mmol L-cysteine were mixed with 60 mL EG solution in presence of 6 mL ethylenediamine. The refluxing system was kept at 175 °C for 50 minutes. After cooled down to ambient temperature naturally, the resulted precipitation was collected by centrifugation and washed thoroughly with water and ethanol several times, vacuum dried at 80 °C overnight for characterizations. We have prepared comparable samples in a similar procedure to that described above, instead, the reaction time was reduced to 10 min, 20 min, 30 min, 40 min. The other one is in absence of ethylenediamine.

Materials Characterization

The morphologies and structures of the products were characterized by field-emission scanning electron microscopy (FESEM, LEO 1430VP, Germany). Transmission electron microscopy (TEM) images were recorded using a HRTEM (JEOL JEM-2010). The composition of the samples was analyzed using an energy dispersive X-ray spectrometer (EDS) attached to the SEM instrument. The powder X-ray diffraction (XRD) pattern was collected on a Bruker D8 Advance diffractometer equipped with Cu Ka radiation over the 20 range of 10-70°. The adsorption–desorption isotherms of nitrogen were measured by using automatic volumetric adsorption equipments (BELSORP mini II, BEL Japan, Inc.), and specific surface areas were calculated by non-localized density functional theory (NLDFT) using N₂-adsorption isotherms at 77 K.

Electrochemical Measurements

Electrodes were made by mixing active materials with acetylene black and carboxymethyl cellulose (CMC) sodium salt in de-ionized water in the ratio of 3:1:1. The electrode was fabricated by using 2032 coin cells with sodium metal as the counter electrode. One molar NaClO₄ in propylene carbonate with 5 wt% fluorinated ethylene carbonate (FEC) was used as the electrolyte. The glass microfiber filters (GF/D whatman) served as the separator. Galvanostatic charge/discharge cycles were performed on a LAND 2001A Battery Tester between 0.01 and 2.00 V at various current densities. Cyclic voltammetry measurements were carried out on an electrochemical workstation (CHI750D) in the potential range of 0.01-2.00 V vs. Na⁺/Na at a scan rate of 0.1 mV s⁻¹.



Fig. S1 N_2 adsorption-desorption isotherm of flowerlike Sb_2S_3 .



Fig. S2 SEM of Sb_2S_3 in variable reaction time a) 10 min, b) 20 min, c) 30 min, d) 40



Fig. S3 a) SEM of Sb₂S₃ in absence of ethylenediamine, b) XRD pattern of Sb₂S₃.



Fig. S4 a) The initial discharge-charge curves of the bulk Sb_2S_3 , b) cycling performance of bulk Sb_2S_3 at current rate of 20 mAh g⁻¹.



Fig. S5 a, b, c, d) SEM images of Sb_2S_3 on the surface of collector before electrochemical test.



Fig. S6 EDS images of Sb_2S_3 on the surface of collector after electrochemical test of 20 cycles.