Supplementary Information

Facile synthesis of one-dimensional peapod-like Sb@C submicron-structures†

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Experimental

Preparation of Sb$_2$O$_3$ submicron-wires (Sb$_2$O$_3$ SWs). The synthesis of Sb$_2$O$_3$ SWs was reported previously. Typically, 400 mg of poly(vinyl pyrrolidone) (PVP, Alfa Aesar, $M_w = 58,000$ g/mol) was first dissolved in a mixture of 0.6 mL ethylenediamine (EDA, Aldrich) and 80 mL deionized water (DI-water). Then, 150 mg of Sb powder (~ 100 mesh, Aldrich) was added into the PVP/EDA/DI-water mixture under vigorous stirring. After continuous stirring for 5 hrs at room temperature, the mixture solution was filtered and washed with DI-water and EtOH repeatedly. Finally, Sb$_2$O$_3$ SWs were collected after dried at 60 °C overnight.

Preparation of one-dimensional peapod-like Sb@Carbon submicron-structures (Sb@C). 100 mg of Sb$_2$O$_3$ SWs was dispersed into an aqueous solution of glucose (30 mL, 0.1 M) under sonication and then transferred into a 50 mL Teflon-lined stainless steel autoclave and kept in an oven at 180 °C for 4 hrs. The product was collected by filtration, washed several times with DI-water and EtOH, and dried at 60 °C overnight. The intermediate product (Sb$_2$O$_3$@C) was then heated in a tube furnace at 500 °C under N$_2$/H$_2$ for 2 hrs to give the Sb@C.
**Characterization.** X-Ray diffraction (XRD) patterns were collected using a Rigaku Ultima IV Diffractometer with Cu Kα irradiation ($\lambda = 1.5406$ Å). WITec confocal Raman with a 514 nm laser source was used to collect the Raman spectra. For ease of comparison, the XRD and Raman results are normalized to the intensity of the strongest peak. A Shimadzu TGA-50 thermogravimetric analyzer was used to study the carbon content in the Sb@C under flowing Air at a heating rate of 10 °C/min from ambient temperature to 800 °C. The morphology was examined by field-emission scanning electron microscopy (FESEM) using an FEI QUANTA 600F environmental SEM. Transmission electron microscopy (TEM), high-resolution TEM (HRTEM), and high angle annular dark field scanning TEM (HAADF-STEM) measurements were carried out on an FEI Titan 80–200 microscope coupled with a HAADF detector and an EDX spectrometer.

**Electrochemical Measurements:** The electrodes consist of 80 wt% Sb@C, 10 wt% carbon black (Super-P) and 10 wt% CMC. The mixture was mixed in water before being casted onto a Cu foil current collector by a doctor blade. The electrodes were dried at 120 °C under vacuum for 12 hrs. A typical loading of active mass is 1.5 mg/cm$^2$. The electrochemical tests were performed in 2032 coin cells. The cells have a sodium pellet as the counter electrode and a glass-fiber separator, and the solution (1 M) of NaPF$_6$ in ethylene carbonate (EC)/ diethyl carbonate (DEC) (1:1 in volume) as the electrolyte. Galvanostatic cycling was conducted on an Arbin BT2000 system at room temperature.
Figure S1 Raman spectra of (a) bulk Sb powder, (b) Sb$_2$O$_3$ SWs, and (c) Sb@C.

![Raman spectra](image)

Figure S2 (a) TGA curve of Sb@C tested from RT to 800 °C with a heating rate of 10 °C min$^{-1}$ in a flowing air. (b) XRD pattern of the product collected after the TGA measurement. The XRD pattern of the product collected after the TGA measurement can be indexed to the orthorhombic phase of Sb$_2$O$_4$ (JCPDS No. 80-0231). Determined by the TGA result, the carbon content in the Sb@C is around 25%.

![TGA and XRD](image)
Figure S3 EDX spectra of (a) bulk Sb, (b) Sb$_2$O$_3$ SWs, (c) Sb$_2$O$_3$@C, and (d) Sb@C. (Au/Pd particles used as a conductive coating for FESEM observations)

Figure S4 FESEM images of Sb$_2$O$_3$@C.
**Figure S5** FESEM images of Sb@C.

**Figure S6** Low-magnification TEM images of Sb@C.

**Figure S7** (a) First and second discharge/charge curves of the Sb@C electrode at a current density of 10 mA/g in the range of 0.01–2 V. (b) Capacity–cycle number curve of Sb@C.