Supporting Information

Ultralong Sb_2Se_3 Nanowire-Based Free-Standing Membrane Anode for Lithium/Sodium Ion Batteries

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EXPERIMENT SECTION

Preparation of Sb_2Se_3 Nanowires

Sb_2Se_3 nanowires were obtained through a facile hydrothermal process with a slight modification. [S-1] In brief, 0.2 mM of $Sb(CH_3COO)_3$ and 0.3 mM of Na_2SeO_3 were dissolved to 75 mL of distilled water. Then 1.92 mM of hydrazine hydrate (80 wt%) was added drop-wise under constant stirring. Subsequently, the resulting solution was transferred into a 100 mL Teflon-lined autoclave. The autoclave was sealed, heated, and maintained at 120 °C for 36 h. The as-obtained product was filtered, washed with distilled water and alcohol several times, and then dried at 70 ^oC for 12 h in vacuum.

For controlling experiments on the concentration of hydrazine hydrate, the amount of hydrazine hydrate were 0.24 mM, 0.48 mM and 0.96 mM for sample C1, C2 and C3 respectively, without changing any other experimental parameters.

For controlling experiments on morphology evolution, the hydrothermal time were set as 6 h, 12 h and 24 h without changing any other experimental parameters.

Fabrication of Ultralong Sb_2Se_3 Nanowire Based Free-Standing Membrane

The ultralong Sb_2Se_3 nanowire based free-standing membrane was fabricated *via* a facile suction process. In brief, a designed amount of Sb_2Se_3 nanowires were dispersed in distilled water and treated by an ultrasound process for ~2 h. Then, the black suspension was filtrated through a poly (vinylidene fluoride) membrane filter (pore size: 220 nm). The membrane was first put in ambient condition for several

hours until it can be carefully separated from the membrane filter. The final membrane was put in 70 ^{o}C oven for 12 h to evaporate the water before its use.

Materials Characterization

Field emission scanning electron microscopy (FESEM) images and energy dispersive X-ray spectra (EDS) were collected with a JEOL-7100F microscope. Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) images were recorded by using a JEM-2100F STEM/EDS microscope. X-ray diffraction (XRD) patterns of sample were obtained with a D8 Advance X-ray diffractometer, using Cu Kα radiation (λ=1.5418 Å). Raman spectra were acquired using a Renishaw RM-1000 laser Raman microscopy system. Brurauer–Emmerr–Teller surface area was measured using a Tristar II 3020 instrument by adsorption of nitrogen at 77 K.

Electrochemical Performance Measurements

For the preparation of ultralong Sb_2Se_3 nanowire based free-standing membrane electrode, the membrane was carefully cut into pieces with an area of 0.4 – 0.8 cm^{-2}. The average mass loading was 1.5 – 2.0 mg cm^{-2}. The comparison conventional electrodes were prepared by mixing the Sb_2Se_3 nanowires, carbon black and poly(vinylidene fluoride) (PVDF) at the weight ratio of 80: 10: 10. The slurry was casted onto Cu foil and dried under a vacuum oven at 70 ^{o}C overnight. The electrochemical measurements were carried out by assembly of 2016 coin cells in a glove box filled with pure argon gas. For lithium storage performance test, Li metal

foil was used as the counter electrode and reference electrode, and a Celgard 2400 microporous membrane as the separator. The electrolyte was a solution of 1 M $LiPF_6$ in the ethylene carbonate/dimethyl carbonate (1:1 in volume). Galvanostatic charge-discharge tests were performed at a potential range of 0.01 – 3 V *vs.* Li/Li^+ using a multichannel battery testing system (LAND CT2001A). Cyclic voltammetry (CV) and electrochemical impedance spectra (EIS) were tested with an electrochemical workstation (Autolab PGSTAT 302N and CHI600E). All of the measurements were carried out at room temperature.

With respect to sodium performance test, Na metal foil was used as the counter electrode and reference electrode, a glass fiber as the separator. The electrolyte was a solution of 1 M trifluomethanesulfonate ($NaCF_3SO_3$) in diethyleneglycol dimethylether (DEGDME). Galvanostatic charge-discharge tests were performed at a potential range of 0.01 – 2 V *vs.* Na/Na^+ using a multichannel battery testing system (LAND CT2001A). Cyclic voltammetry (CV) and electrochemical impedance spectra (EIS) were tested with an electrochemical workstation (Autolab PGSTAT 302N and CHI600E). All of the measurements were carried out at room temperature.

ADDITIONAL FIGURES

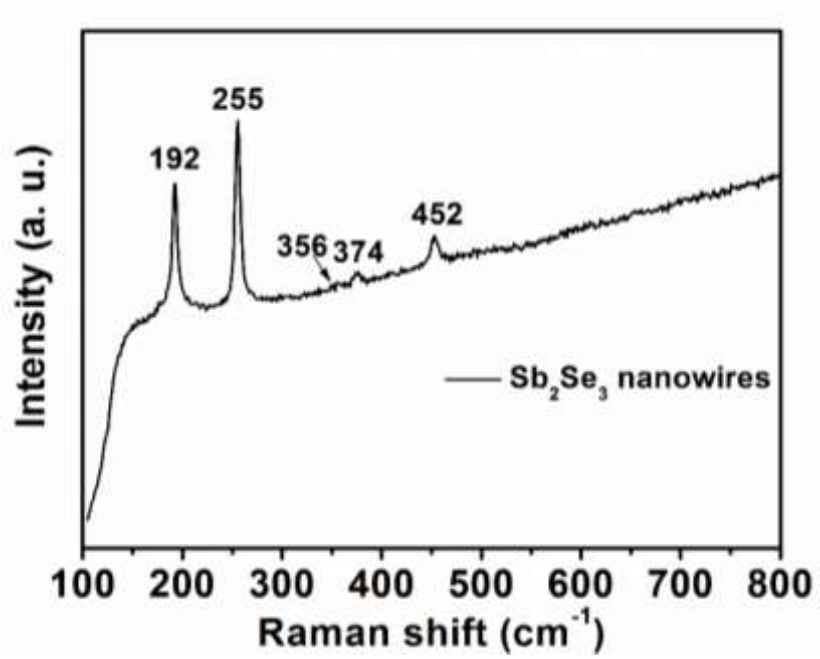

Figure S1 Raman spectrum of Sb_2Se_3 ultralong nanowires.

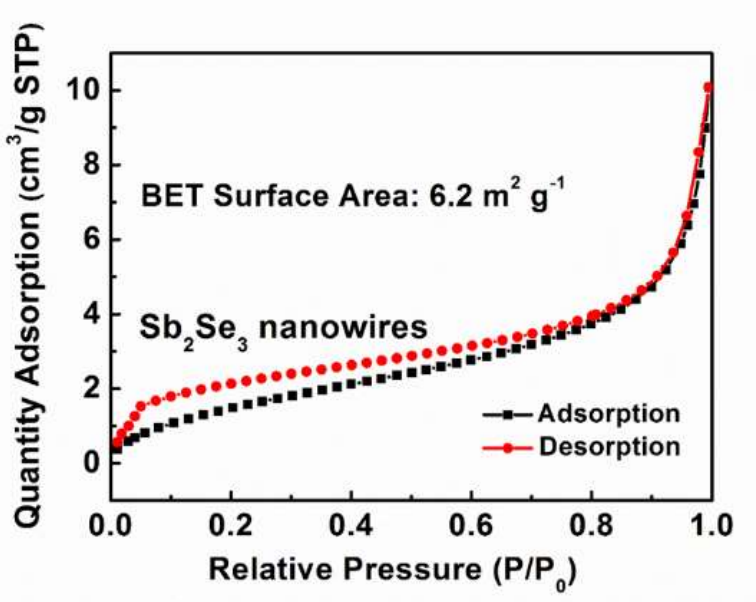

Figure S2 Nitrogen adsorption–desorption isotherm curve of Sb_2Se_3 nanowires product.

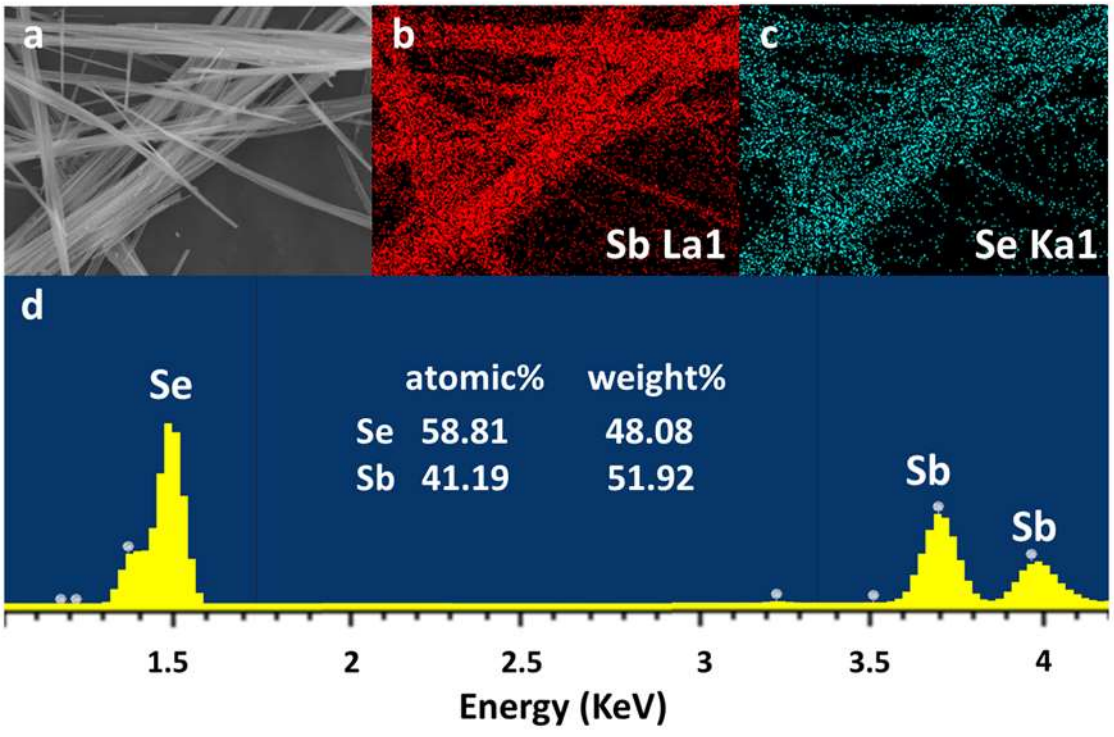

Figure S3 SEM image (a) and corresponding elemental mapping images (b and c); EDX spectrum (d) of Sb_2Se_3 ultralong nanowires.

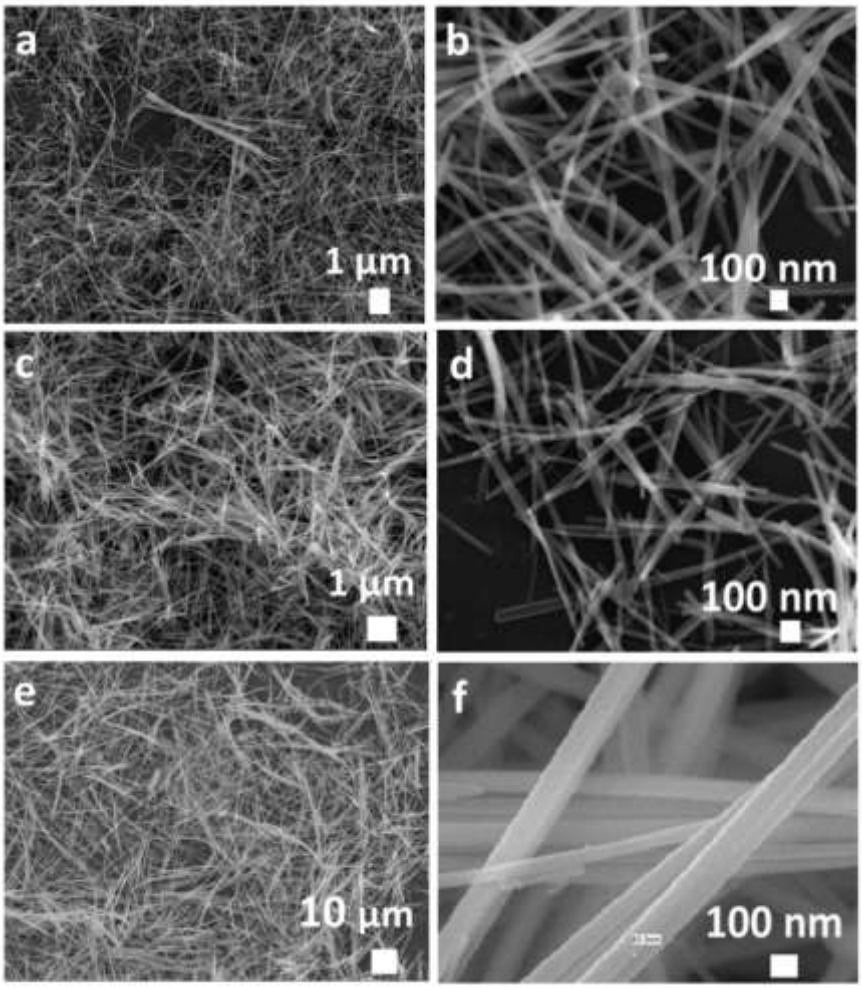

Figure S4 SEM images of Sb_2Se_3 products obtained at different concentrations of hydrazine hydrate: (a-b) 0.24mM, (c-d) 0.48 mM, and (e-f) 0.96 mM.

Figure S5 Digital photograph of the membrane fabricated with sample C1.

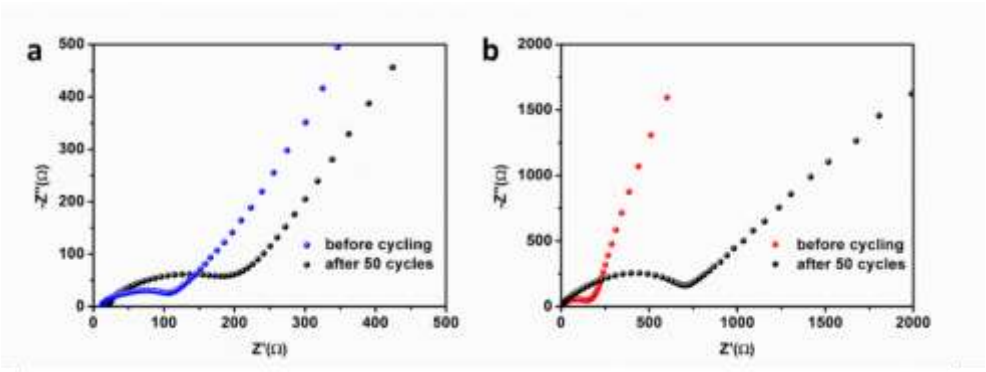

Figure S6 The Nyquist plots of the Sb_2Se_3 nanowire membrane before cycling and after 50 cycles (at a current density of 100 mA g^{-1}) for LIBs (a) and SIBs (b).

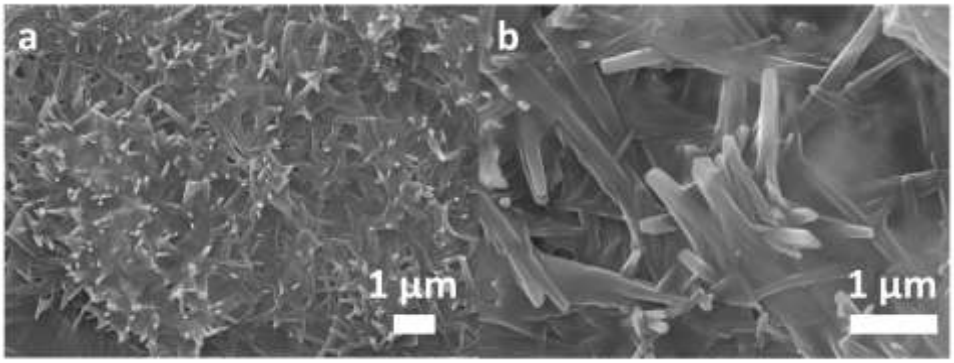

Figure S7 SEM images ((a) at low magnification, (b) at high magnification) of the Sb_2Se_3 membrane electrode after 50 cycles in LIBs at a current density of 100 mA g^{-1}. The cell was disassembled at the fully delithiated state.

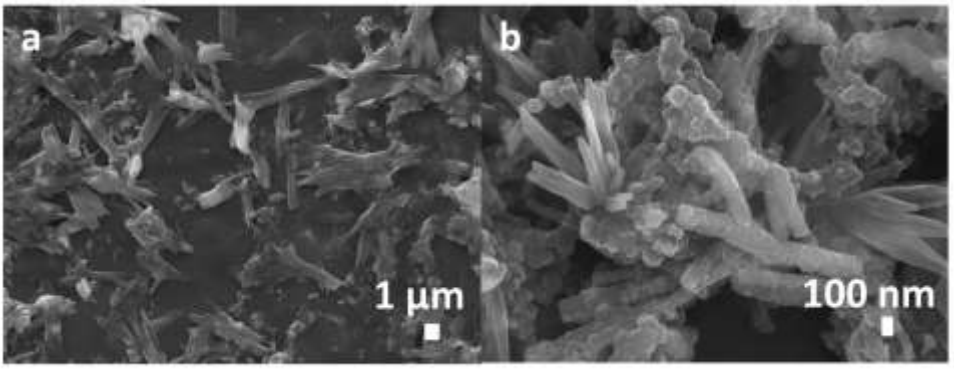

Figure S8 SEM images ((a) at low magnification, (b) at high magnification) of the Sb_2Se_3 membrane electrode after 50 cycles in SIBs at a current density of 100 mA g^{-1}. The cell was disassembled at the fully desodiated state.

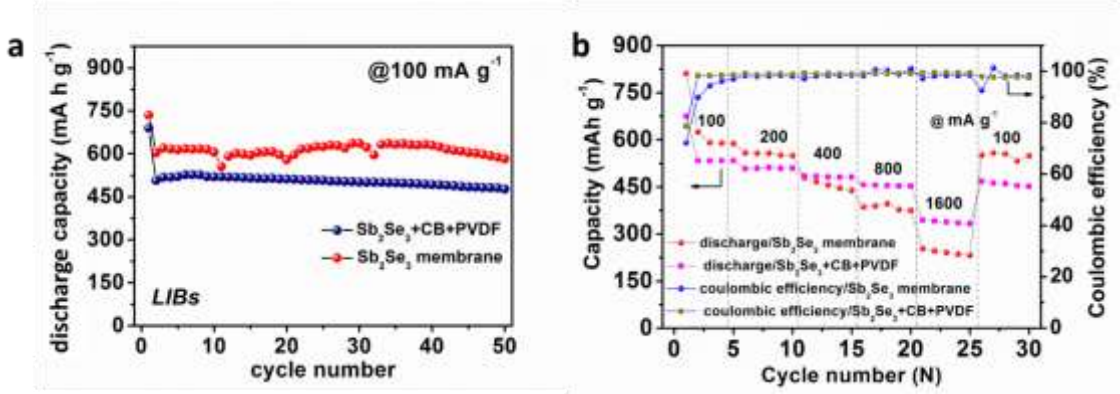

Figure S9 Comparison of lithium storage performances of Sb_2Se_3 membrane and conventional electrode with carbon black additive and PVDF binder: (a) cycling performance at a current density of 100 mA g^{-1}; (b) rate performance.

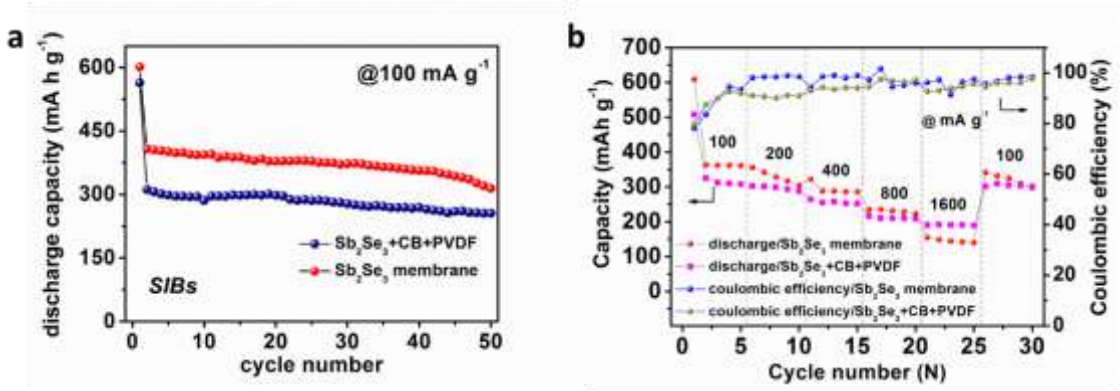

Figure S10 Comparison of sodium storage performances of Sb_2Se_3 membrane and conventional electrode with carbon black additive and PVDF binder: (a) cycling performance at a current density of 100 mA g^{-1}; (b) rate performance.

REFERENCE

[S-1] Ma, J.; Wang, Y.; Wang, Y.; Chen, Q.; Lian, J.; Zheng, W., Controlled Synthesis of One-Dimensional Sb_2Se_3 Nanostructures and Their Electrochemical Properties. *J. Phys. Chem. C* **2009,** *113*(31), 13588-13592.