Supplementary Information

Synergic antimony-niobium pentoxide nanomeshes for high-rate sodium storage

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Fabrication of the synergic Sb-Nb$_2$O$_5$ nanomeshes

Nb$_2$O$_5$·nH$_2$O nanosheets were firstly synthesized via a hydrothermal treatment using NbCl$_5$ as precursor. Typically, 5 mmol NbCl$_5$ was added into 50 mL NH$_3$·H$_2$O aqueous solution (pH 8), forming a translucent white dispersion. After magnetic stirring for 2 h, a hydrothermal treatment at 240 ºC was carried out for 24 h. The Nb$_2$O$_5$·nH$_2$O nanosheets were obtained by washing with deionized water, centrifuging and freeze-drying. Then 20 mg Nb$_2$O$_5$·nH$_2$O nanosheets were suspended in 50 mL ethanol containing 50 uL NH$_3$·H$_2$O (28 wt.%) and 0.2 g cetyltrimethyl ammonium bromide (CTAB). After ultrasonically treating for 0.5 h, 60 mg SbCl$_3$ dissolved in 0.2 mL ethanol was added dropwise into the abovementioned dispersion under magnetic stirring. During this process, antimony source was deposited on the surfaces of CTAB modified Nb$_2$O$_5$·nH$_2$O nanosheets. An annealing treatment was subsequently carried out at 550 ºC under Ar atmosphere, generating SbNbO$_4$ nanosheets, which maintain the morphology of the Nb$_2$O$_5$·nH$_2$O nanosheets. Finally, the resulting SbNbO$_4$ nanosheets were calcinated under reducing atmosphere of H$_2$/Ar (10 vol% of H$_2$) at 600 ºC for various time from 10 min to 3 h. A typical annealing time is 3 h, thus the synergic Sb-Nb$_2$O$_5$ nanomeshes were obtained.

Structural characterizations

The morphology, microstructure and crystal structure of the samples were investigated by SEM (Zeiss Supera 55), TEM (JEOL 2100F), XRD (Rigaku D/MAX2200pc) measurements. The compositions of the samples were inspected by XPS (Thermo Escalab 250XI), Raman
(Renishaw Invia) and TG (NETZSCH STA 449 F3 Jupiter). The nitrogen physisorption measurements were performed on a Quantachrome QDS-MP-30 analyzer (USA) at 77 K.

**Electrochemical measurements**

Electrochemical experiments were carried out using standard CR2032 type coin cells. The working electrodes were prepared by mixing the active materials, acetylene black and Carboxy Methylated Cellulose (CMC) in deionized water, coated uniformly on pure Cu foil, and dried at 80 °C under vacuum for 12 h. The loading mass of the active materials on the electrode is 0.4–0.6 mg cm$^{-2}$. Pure sodium foil was used as the counter electrode, glass fiber as the separator, and a solution of 1 M NaClO$_4$ in ethylene carbonate (EC)/Propylene carbonate (PC) (1:1 by volume) with 5% Fluoroethylene carbonate (FEC) addition as the electrolyte. The cells were assembled in an argon-filled glove box. Galvanostatical discharge-charge experiments were tested on a Land CT2001A battery test system at different current densities in a voltage range of 0.01-2.0 V. And the specific capacity was calculated based on the mass of the entire active materials. The volumetric capacity is calculated by multiplying gravimetric capacity by electrode density based on the electrode only. In order to acquire an accurate density value, the cross-section SEM investigation was carried out on electrode with high loading mass of 1.45 mg. And the diameter of the electrode is 1.2 cm with area of 1.13 cm$^2$. Moreover, a pressure of 10 MPa was performed on the electrode before cross-section survey and galvanostatical discharge-charge tests for the calculation of volumetric capacity. Electrochemical impedance spectroscopy (EIS) measurements of the electrodes were carried out on an electrochemical workstation (CHI760E). The impedance spectra were obtained by applying a sine wave with amplitude of 5.0 mV over the frequency range from 100 kHz to 0.01 Hz. Fitting of impedance spectra to the proposed equivalent circuit was performed by the code Zview.
Figure S1. TEM image of Nb$_2$O$_5$·nH$_2$O nanosheets.
Figure S2. HRTEM image of Nb$_2$O$_5$·nH$_2$O nanosheets.
Figure S3. SEAD pattern of Nb$_2$O$_5$·nH$_2$O nanosheets.
Figure S4. TEM image of SbNbO$_4$ nanosheets.
Figure S5. HRTEM image of SbNbO$_4$ nanosheets.
Figure S6. SEAD pattern of SbNbO₄ nanosheets.
Figure S7. XRD patterns of the SbNbO$_4$ nanosheets with different decomposition time.
Figure S8. SEM image of 600°C decomposition for 10 min.
Figure S9. SEM image of 600°C decomposition for 1 hour.
Figure S10. Low-magnified SEM image of Sb-Nb$_2$O$_5$ nanomeshes with magnification of 2,000.
Figure S11. Magnified SEM image of Sb-Nb$_2$O$_5$ nanomeshes.
Figure S12. Nitrogen adsorption/desorption isotherms and pore size distribution (inset) of the synergic Sb-Nb$_2$O$_5$ nanomeshes, which shows a BET specific surface area of 20.4 m$^2$ g$^{-1}$. 
Figure S13. Optical photograph of the synergic Sb-Nb$_2$O$_5$ nanomeshes produced in large scale.
Figure S14. SEM image of the Sb-Nb$_2$O$_5$ nanomeshes produced in large scale.
Figure S15. TEM image of the synergic Sb-Nb$_2$O$_5$ nanomeshes.
Figure S16. Magnified TEM image of the synergic Sb-Nb$_2$O$_5$ nanomeshes.
Figure S17. HRTEM image of the synergic Sb-Nb$_2$O$_5$ nanomeshes with Sb lattice fringes.
Figure S18. HRTEM image of the synergic Sb-Nb$_2$O$_5$ nanomeshes with Nb$_2$O$_5$ lattice fringes.
Figure S19. Raman spectrum of the synergic Sb-Nb$_2$O$_5$ nanomeshes.
Figure S20. a) The XRD pattern demonstrates the product after Thermogravimetric (TG) measurement to be Sb$_2$O$_4$. b) TG profile of the synergic Sb-Nb$_2$O$_5$ nanomeshes. The content of Sb is calculated to be 68.6 wt.% based on the following equation: Sb (wt.%) = 100% × (2 × molecular weight of Sb) / (4 × molecular weight of O) × (weight increase after oxidation).
Figure S21. Discharge and charge capacities of the initial 10 cycles at 0.1 A g⁻¹.
Figure S22. Discharge and charge capacities of the additive acetylene black at 0.1 A g$^{-1}$. 
Figure S23. Charge-discharge profiles of the first two cycles at 0.1 A g\(^{-1}\) of Nb\(_2\)O\(_5\)·nH\(_2\)O nanosheets.
Figure S24. Charge-discharge profiles of the first two cycles at 0.1 A g\(^{-1}\) of SbNbO\(_4\) nanosheets.
Figure S25. *Ex situ* TEM image of the synergic Sb-Nb$_2$O$_5$ nanomeshes after 100 discharge-charge cycles at 5.0 A g$^{-1}$ with well-maintained microstructure.
Figure S26. *Ex situ* XRD patterns of the Sb-Nb$_2$O$_5$ nanomeshes at different discharge-charge voltages.
**Figure S27.** Selected charge-discharge profiles of the synergic Sb-Nb$_2$O$_5$ nanomeshes at a current density of 0.05 mA cm$^{-2}$, delivering a reversible volumetric capacity of 1001 mAh cm$^{-3}$. 
Figure S28. Cross-section SEM image of electrode with mass loading of 1.45 mg.
Figure S29. Modified Randles equivalent circuit for the electrode/electrolyte interface of the synergic Sb-Nb$_2$O$_5$ nanomeshes, Nb$_2$O$_5$·nH$_2$O nanosheets, SbNbO$_4$ nanosheets and Sb particles. $R_e$, $R_f$ and $R_{ct}$ stand for the electrolyte resistance, surface resistance and charge-transfer resistance, respectively. $C_f$ and $C_{dl}$ are the surface capacitance and double-layer capacitance, respectively. $Z_w$ represents the Warburg impedance related to the diffusion of sodium ions into the bulk electrodes.
Figure S30. (a) Cyclic voltammograms of Sb particles at the sweep rates of 0.1 to 10 mV s\(^{-1}\). (b) Plots of log \(i\) versus log \(v\) of both anodic and cathodic peaks (circles) and b-value determination (lines) according to the relationship: \(i=a v^b\).
Figure S31. (a) Cyclic voltammograms of Nb$_2$O$_5$·nH$_2$O nanosheets at the sweep rates of 0.1 to 10 mV s$^{-1}$. (b) Plots of log ($i$) versus log ($v$) of both anodic and cathodic peaks (circles) and b-value determination (lines) according to the relationship: $i=a v^b$. 
Figure S32. CV profiles with capacitive current separation of Sb-Nb$_2$O$_5$ nanomeshes at 0.1 mV s$^{-1}$, delivering 44% capacitive contribution of the total capacity.
Figure S33. Capacitive and diffusion controlled capacity contributions of Sb-Nb$_2$O$_5$ nanomeshes at various sweep rates ranging from 0.1 to 5 mV s$^{-1}$. 
Table S1. Progress on synthesis of Sb-based and Nb-based materials and their electrochemical performances for sodium storage

<table>
<thead>
<tr>
<th>Materials</th>
<th>Synthesis method</th>
<th>Reversible capacity a)</th>
<th>Rate capability b)</th>
<th>Ref.</th>
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<tbody>
<tr>
<td>Sb-Nb$_2$O$_5$ nanomeshes</td>
<td>Hydrothermal and heat treatment</td>
<td>558/0.1</td>
<td>190/10</td>
<td>This work</td>
</tr>
<tr>
<td>Sb/SiOC</td>
<td>Sonication and pyrolysis</td>
<td>510/0.05 C</td>
<td>453/20 C</td>
<td>40</td>
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<td>Sb@TiO$_{2-x}$ nanotubes</td>
<td>Solvothermal, hydrolysis and heat treatment</td>
<td>601/0.066</td>
<td>312/13.2</td>
<td>22</td>
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<tr>
<td>Few-layer antimonene</td>
<td>Liquid-phase exfoliation</td>
<td>642/0.066</td>
<td>429/3.3</td>
<td>42</td>
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<tr>
<td>Sb nanosheets</td>
<td>Liquid-phase exfoliation</td>
<td>463/0.062</td>
<td>82/2.5</td>
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<tr>
<td>Sb/C fibers</td>
<td>Electrospinning and heat treatment</td>
<td>422/0.1</td>
<td>88/6</td>
<td>11</td>
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<tr>
<td>TiNb$_2$O$_7$/graphene</td>
<td>Freeze dying and heat treatment</td>
<td>311/0.025</td>
<td>146/0.5</td>
<td>41</td>
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<tr>
<td>Nb$_2$O$_5$@C/rGO</td>
<td>Sol-gel and heat treatment</td>
<td>285/0.025</td>
<td>109/3</td>
<td>19</td>
</tr>
<tr>
<td>G-Nb$_2$O$_5$ nanosheets</td>
<td>Nanocasting and heat treatment</td>
<td>230/0.05</td>
<td>100/2</td>
<td>20</td>
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<tr>
<td>a-H-Nb$_2$O$_5$ film</td>
<td>Anodization and heat treatment</td>
<td>185/0.1</td>
<td>84/2</td>
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a) The reversible capacity is summarized as capacity/corresponding current density. b) The rate capability is summarized as capacity/corresponding current density. The unit of capacity is mAh g$^{-1}$, and the unit of current density is A g$^{-1}$.
**Table S2.** Kinetic parameters of the synergic Sb-Nb$_2$O$_5$ nanomeshes, Nb$_2$O$_5$·nH$_2$O nanosheets, SbNbO$_4$ nanosheets and Sb particles.

<table>
<thead>
<tr>
<th>Sample</th>
<th>$R_c$</th>
<th>$R_{ct}$</th>
<th>$\sigma_w$</th>
<th>$D$</th>
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<tr>
<td></td>
<td>$\Omega$</td>
<td>$\Omega$</td>
<td>$\Omega$ s$^{-1/2}$</td>
<td>cm$^2$ s$^{-1}$</td>
</tr>
<tr>
<td>synergic Sb-Nb$_2$O$_5$ nanomeshes</td>
<td>10.8</td>
<td>49.7</td>
<td>490.4</td>
<td>1.15×10$^{-13}$</td>
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<tr>
<td>Nb$_2$O$_5$·nH$_2$O nanosheets</td>
<td>12.1</td>
<td>151.7</td>
<td>1176.2</td>
<td>2.00×10$^{-14}$</td>
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<tr>
<td>SbNbO$_4$ nanosheets</td>
<td>13.5</td>
<td>128.6</td>
<td>584.2</td>
<td>8.13×10$^{-14}$</td>
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<tr>
<td>Sb particles</td>
<td>13.1</td>
<td>129.3</td>
<td>746.8</td>
<td>4.97×10$^{-14}$</td>
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