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Supporting information for

A simple synthesis of nanoporous Sb/C with high Sb content and dispersity as advanced anode for sodium ion batteries

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Experimental Section

Synthesis of Sb/C composites: The Sb/C composite was prepared by direct ball milling of commercial Sb_2O_3 powders (Sinopharm Chemical Reagent, China, 99.6% in purity) and CaC_2 (Macklin Inc, China, 97% in purity). Pre-ball milling of CaC_2 was carried out because the as received CaC_2 is too large in particle sizes. For c-Sb/C and a-Sb/C, the mass ratio of Sb_2O_3 and CaC_2 was set to be 1:1 and 1:2 respectively. The weight ratio of milling balls to reagent powders was selected to be 20:1. The milling vial was filled with Ar and set in a shaking miller (QM-3A, Nanjing, China). The rotation speed of the miller was set to 1000 rpm and the milling time was fixed to 10 h. The obtained product was leached in 0.1M HCl for 5h and then washed with purified water 3 times. After the washing process, the samples were dried under vacuum at 60 ^{o}C overnight.

Materials characterizations: Powder XRD patterns were collected on a Bruker D8 X-ray diffractometer with Cu-Kα radiation. The microstructures and morphologies were characterized by SEM (FEI Quanta 200) and TEM (JEOL JEM-2010FEF, 200 kV). Elemental mapping was conducted by TEM (Tecnai G2 F30, FEI, 300 kV) equipped with an X-ray energy dispersive spectrometer. The N_2 sorption isotherms were collected using a Micromeritics ASAP 2020 Analyzer (Norcross, GA) at 77 K. X-Ray photoelectron spectroscopy (XPS) analysis was carried out with a Thermo Fisher ESCALAB 250Xi) with an Al Kα X-ray source. Raman characterization was carried out using a RM-1000 Laser Confocal Raman Microspectroscopy (Renishaw, England) employing a 514.5 nm laser beam. The electronic conductivity of the sample pellet prepared by die pressing (20 MPa) was measured using an RTS-8 four-probe measurement system (4 Probes TECH).

Electrochemical characterizations: The electrochemical measurements of Sb/C composites were carried out using 2016 type coin cell and a Celgard 2400 separator. The working electrode was made by coating slurry of 80 wt % active material, 10 wt% CMC binder and 10 wt % Super P on copper foil substrate. The mass loading of the active material within the film was 0.9-1.1 mg cm^{-2}. The electrode film was dried under vacuum at 80 ^{o}C overnight before tests. A homemade sodium foil was served as the counter and reference electrode. The electrolyte was a solution of 1 M $NaClO_4$ in ethylene carbonate (EC)/dimethyl carbonate (DMC) (1:1 by volume) with 5% fluoroethylene carbonate (FEC) additive. All the cells were assembled in a glove box with water/oxygen content lower than 1ppm. The galvanostatic charge/discharge test was conducted on BTS 55 Battery Testing System (Nawere, China). Cyclic voltammetric measurements were carried out with coin cell at a scan rate of 0.1 mV s^{-1} using a CHI660a potentiostat (CHI instruments, Shanghai). Electrochemical impedance spectra were recorded by a CHI660a potentiostat (CHI instruments, Shanghai) with oscillation amplitude of 5 mV at the frequency range from 100 m Hz to 100 kHz.

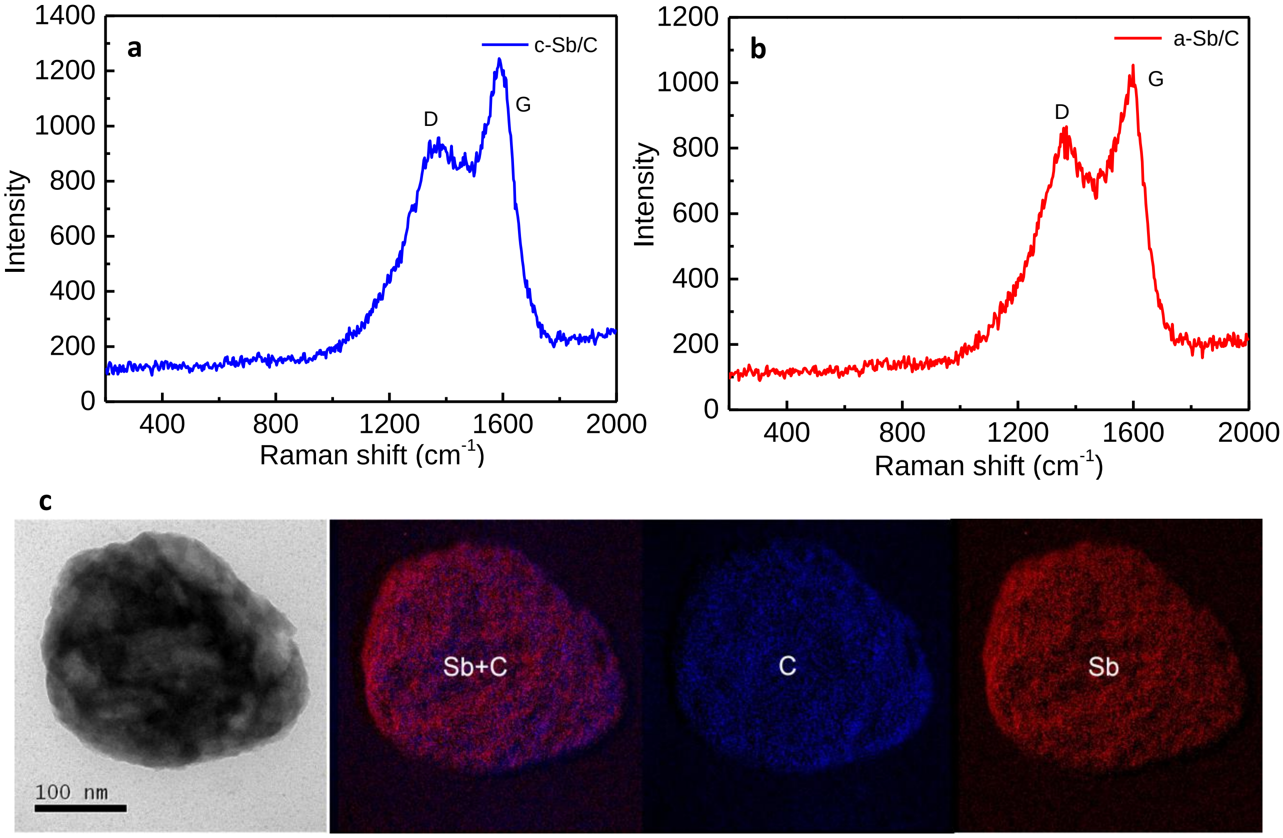

Fig. S1 (a) Raman spectrum of c-Sb/C; (b) Raman spectrum of a-Sb/C; (c) elemental mapping images of c-Sb/C.

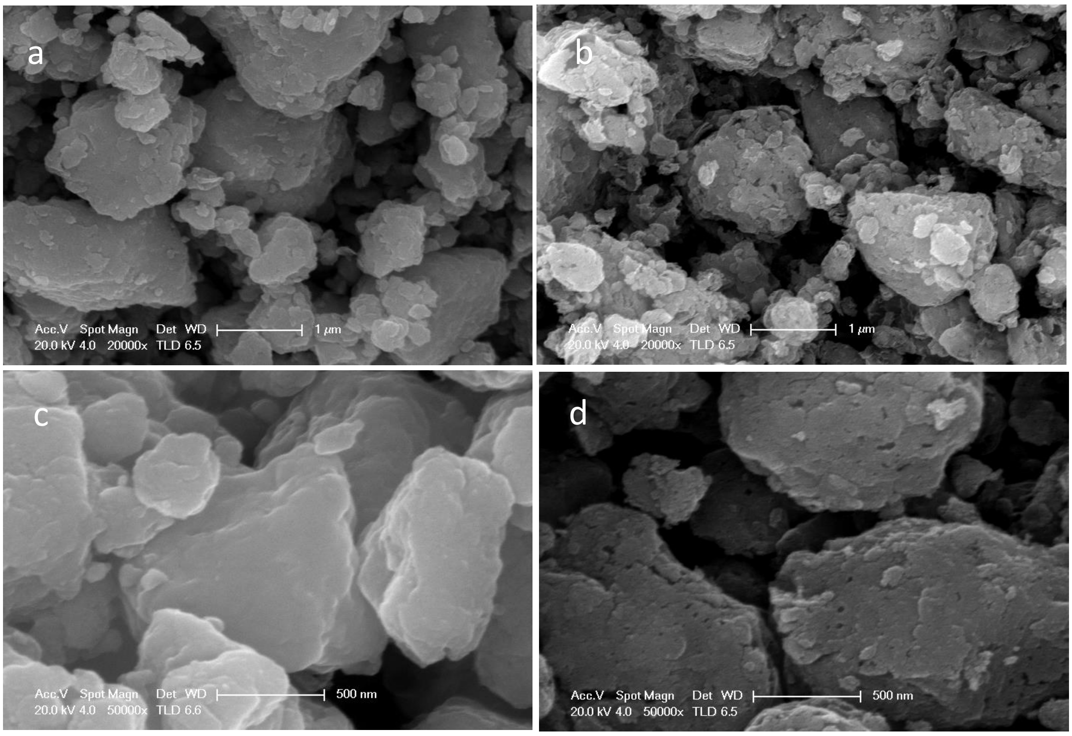

Fig. S2 SEM images of c-Sb/C (a, b) and a-Sb/C (c,d)

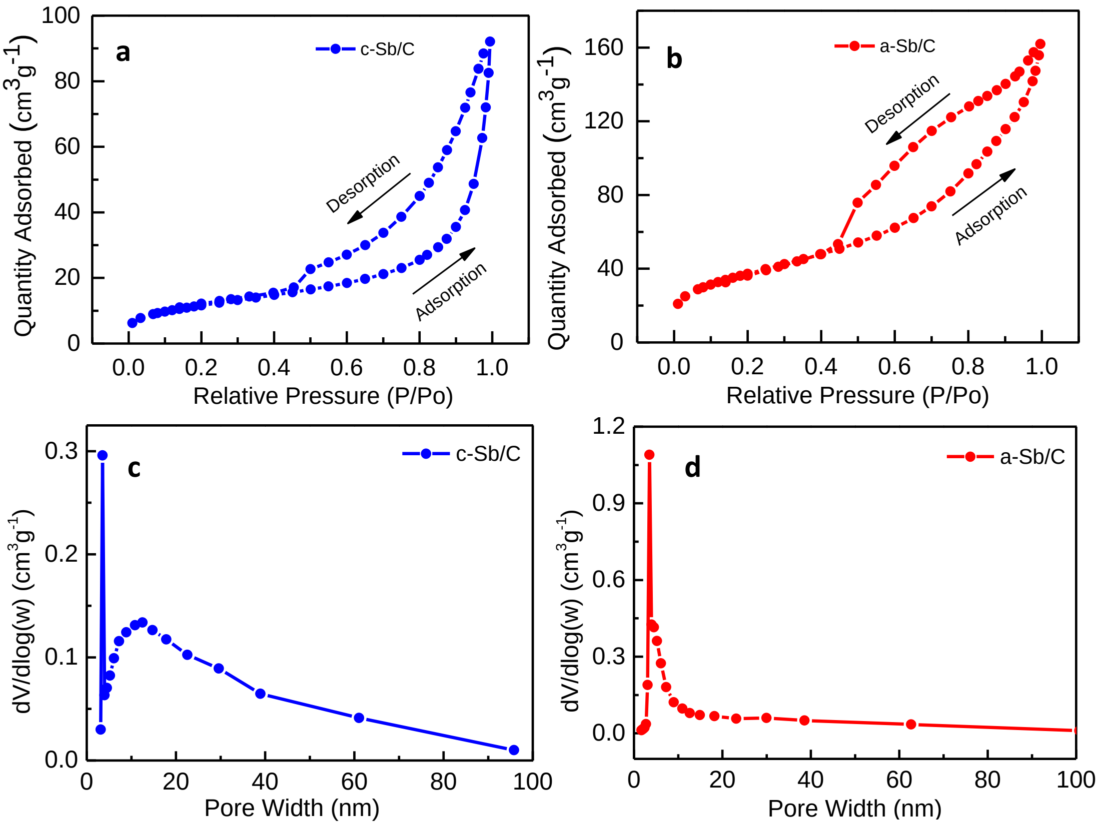

Fig. S3 Nitrogen adsorption–desorption isotherms of c-Sb/C (a) and a-Sb/C (b). Pore size distribution of c-Sb/C (c) and a-Sb/C (d).

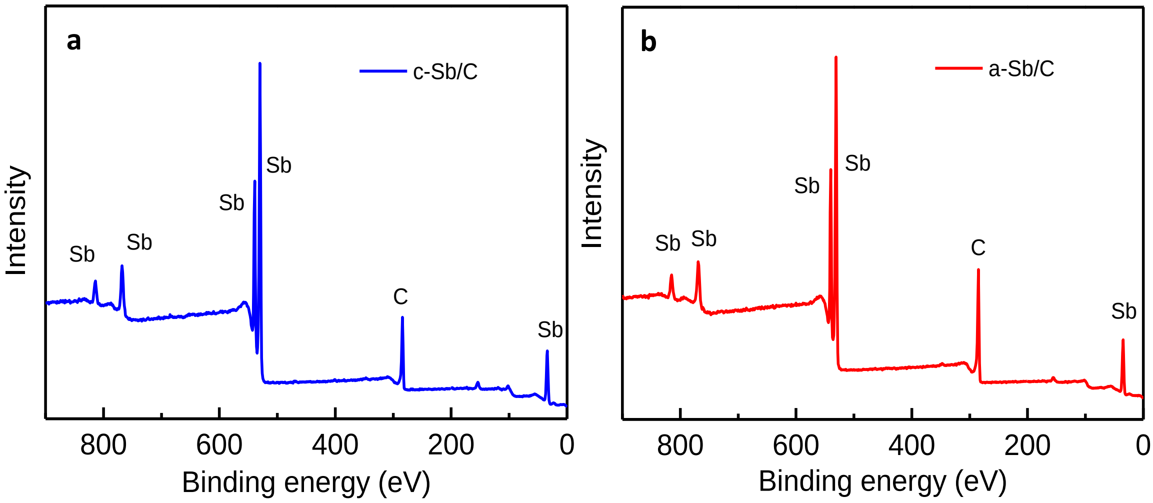

Fig.s4 XPS survey spectra of c-Sb/C (a) and a-Sb/C (b).

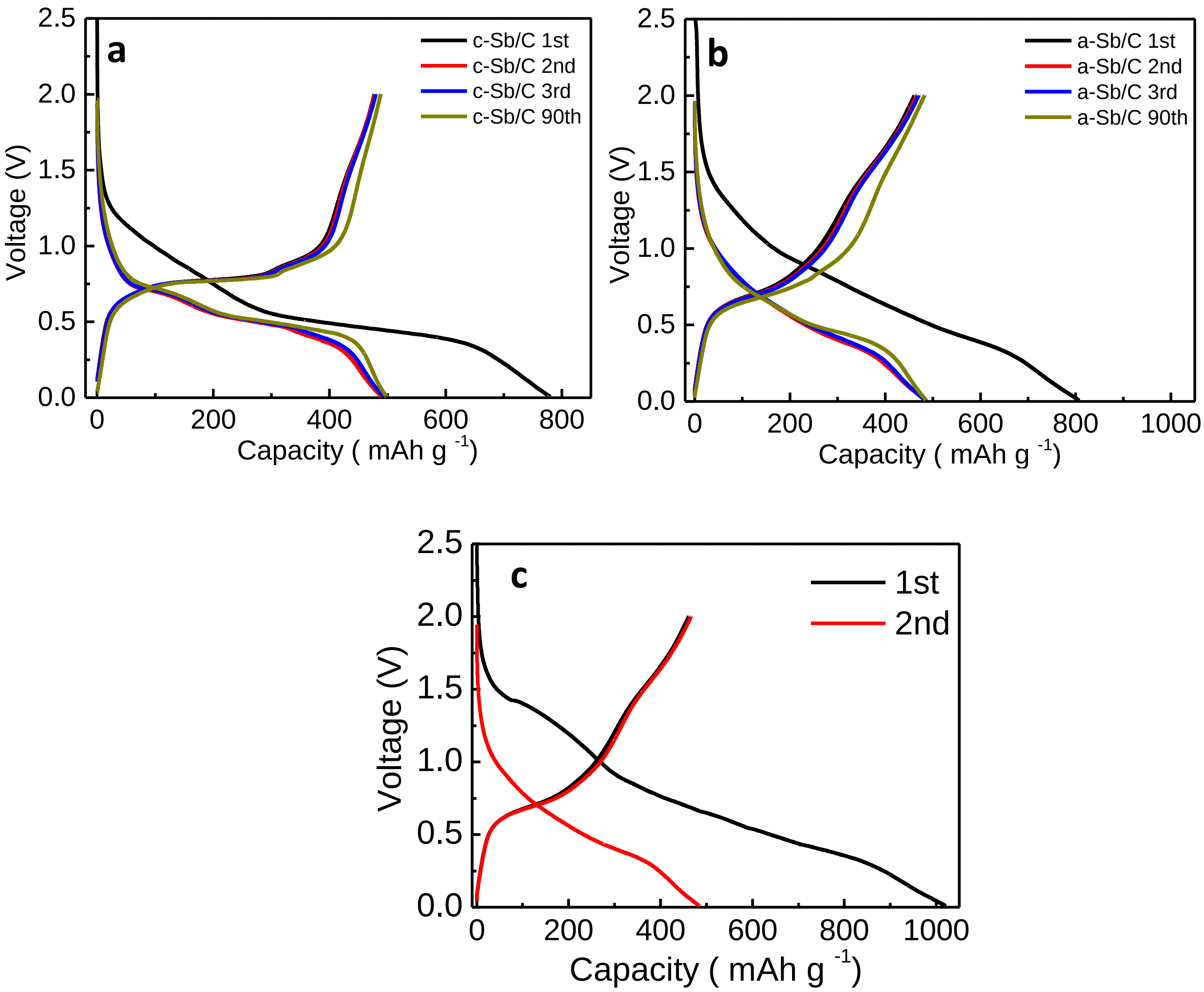

Fig. s5 Charge/discharge voltage profiles of c-Sb/C (a) and a-Sb/C (b and c) for the first three and 90th cycles at a current density of 0.1 A g^{-1}. The electrode membranes in (a) and (b) were prepared by drying the slurry coating in argon followed by drying in vacuum; the electrode membrane was prepared by drying the slurry coating in air followed by drying in vacuum. In (c) the a-Sb/C shows a charging plateau at about 1.4 V but no corresponding discharging plateau, which could be attributed to the irreversible reduction of antimony oxides formed in air.

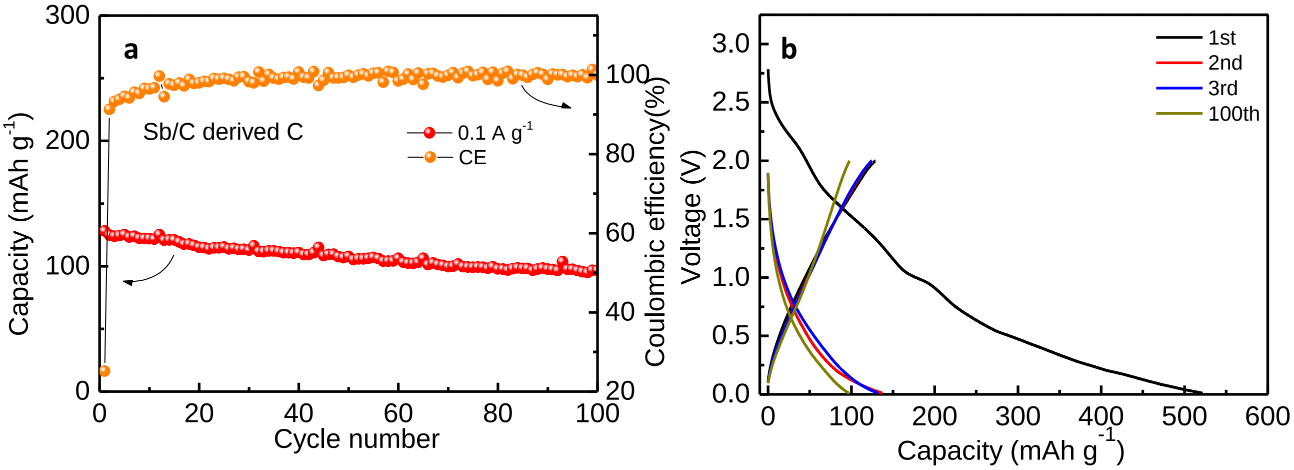

Fig. s6 (a) cyclic performance of carbon of the Sb/C after removing the Sb with aqua regia and (b) displays the charge/discharge voltage profiles for the first three and 100th cycles. The charge/discharge current density was 0.1 A g^{-1}.

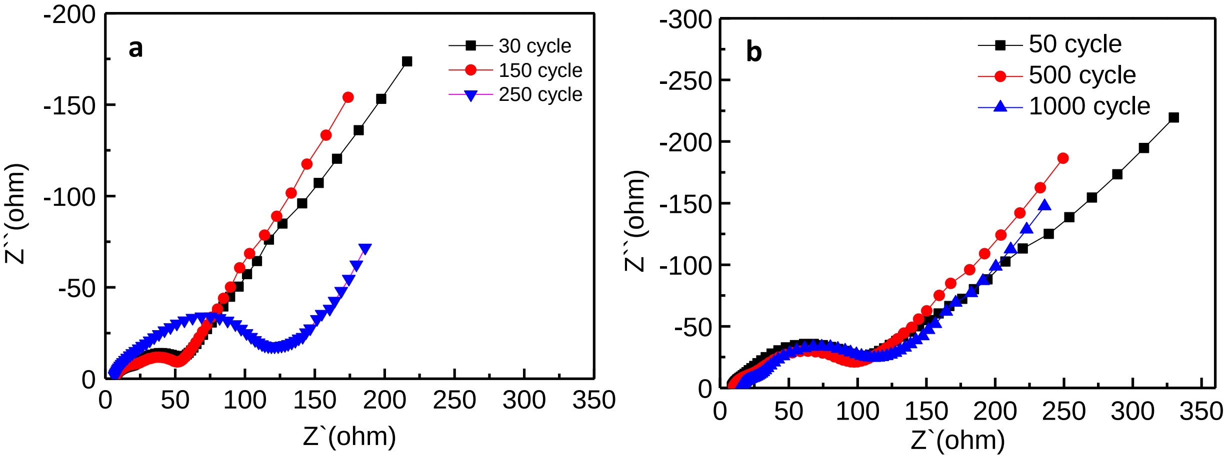

Fig. s7 Electrochemical impedance spectra (EIS) of the c-Sb/C electrode (a) after 30, 150 and 250 and the a-Sb/C electrode (b) after 50, 500 and 1000 charge/discharge cycles at 1 A g^{-1}. The spectra were recorded at open circuit potential.

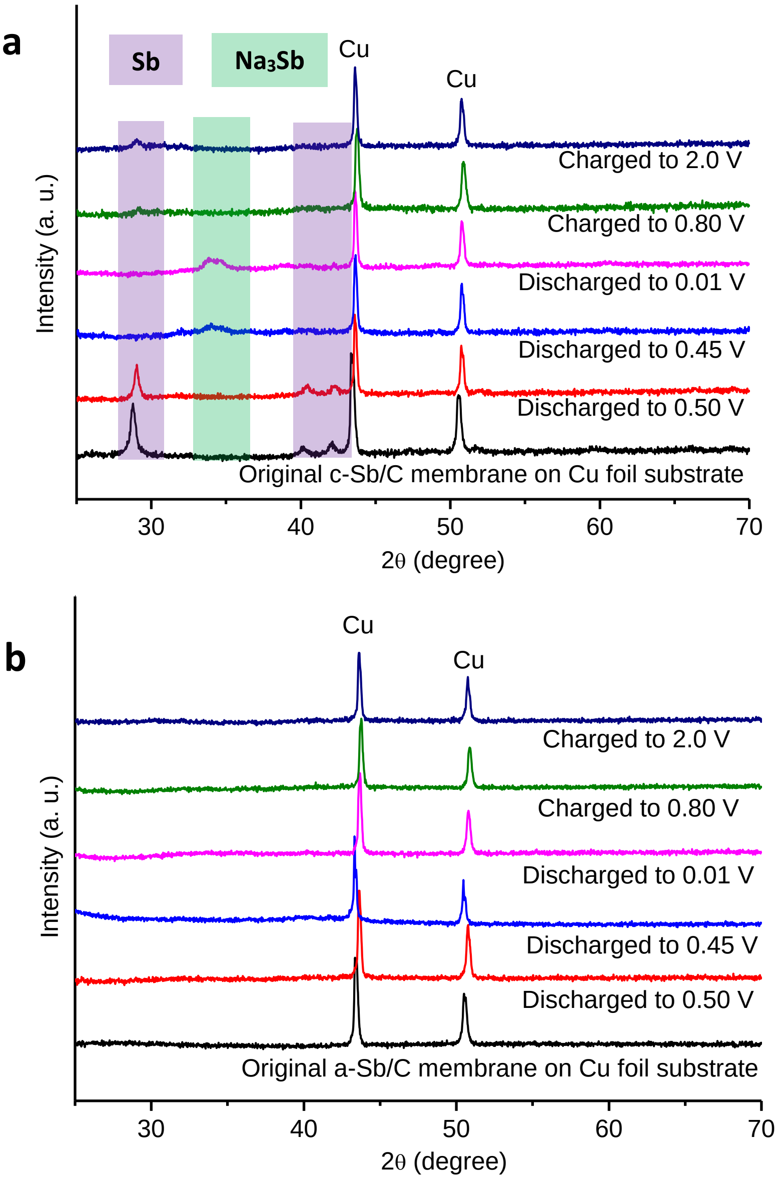

Figure s8. Ex situ XRD patterns of c-Sb/C (a) and a-Sb/C (b) at selected electrode states during the first discharging/charging process.

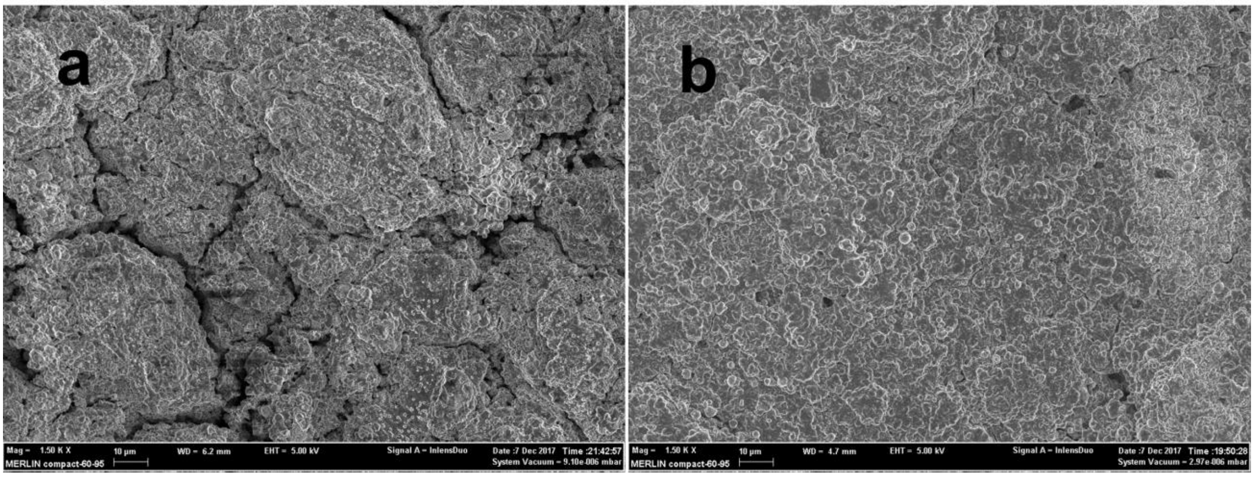

Figure S9. SEM images of the Sb/C electrodes membranes after cycling. (a) c-Sb/C after 250 charge/discharge cycles at 1 A/g; (b) a-Sb/C after 1000 charge/discharge cycles at 1 A/g.

Table S1. Comparison of various Sb/C composites as anode materials for SIBs. Literatures reporting a cycling test over 300 cycles were selected. All the specific capacity values shown in this Table were normalized by the total mass of the Sb/C composite. The rate performance column lists the reversible capacities at charge/discharge currents of 0.1, 1 and 5 A g^{-1} or nearby.

Sb/C materials	**Sb content**	**Rate performance**	**Voltage range**	**Cyclic performance**	**Ref.**
Sb/graphene	33%	261 mAh g^{-1} (0.125 A g^{-1}) 162 mAh g^{-1} (4.5 A g^{-1})	0.01-2 V	207 mAh g^{-1} after 300 cycles at 0.125 A g^{-1}	1
Sb-C frameworks	33.5%	451 mAh g^{-1} (0.1 A g^{-1}) 255 mAh g^{-1} (3.66 A g^{-1}) 237 mAh g^{-1} (4.68 A g^{-1})	**0-2.0 V**	245 mAh g^{-1} after 1000 cycles at 3.66 A g^{-1} 250 mAh g^{-1} after 5000 cycles at 3.66 A g^{-1}	2
Sb-C nanofibers	38%	266 mAh g^{-1} (0.2 A g^{-1}) 218 mAh g^{-1} (0.9 A g^{-1}) 177 mAh g^{-1} (3 A g^{-1})	0.01-2V	243 mAh g^{-1} after 400 cycles at 0.2 A g^{-1}	3
Sb@C microspheres	41%	255 mAh g^{-1} (0.3 A g^{-1}) 120 mAh g^{-1} (4.8 A g^{-1})	0.01-2V	240 mAh g^{-1}after 300 cycles at 0.3 A g^{-1}	4
Sb/C fibers	54%	422 mAh g^{-1} (0.1 A g^{-1}) 273 mAh g^{-1} (1 A g^{-1}) 104 mAh g^{-1} (5 A g^{-1})	**0-2.0 V**	350 mAh g^{-1} after 300 cycles at 0.1 A g^{-1}	5
Yolk-shell Sb@C	60%	350 mAh g^{-1} (0.1 A g^{-1}) 300 mAh g^{-1} (0.2 A g^{-1})	**0.02-2 V**	249 mAh g^{-1} after 300 cycles at 0.2 A g^{-1}	6
Sb@C coaxial nanotubes	28.5% 60.9%	340 mAh g^{-1} (0.1 A g^{-1}) 290 mAh g^{-1} (1 A g^{-1}) 250 mAh g^{-1} (5 A g^{-1}) 460 mAh g^{-1} (0.1 A g^{-1}) 409 mAh g^{-1} (1 A g^{-1}) 370 mAh g^{-1} (5 A g^{-1})	0.01-2V	205 mAh g^{-1} after 1000 cycles at 1 A g^{-1} 200 mAh g^{-1} after 2000 cycles at 1 A g^{-1} 260 mAh g^{-1} after 1000 cycles at 1 A g^{-1} 240 mAh g^{-1} after 2000 cycles at 1 A g^{-1}	7
Spherical nano-Sb@C	68.8%	435 mAh g^{-1} (0.1 A g^{-1}) 380 mAh g^{-1} (1 A g^{-1}) 275 mAh g^{-1} (4 A g^{-1})	0.01-2V	325 mAh g^{-1}after 500 cycles at 1 A g^{-1}	8
Nanoporous a-Sb/C	**70.7%**	**480 mAh g^{-1} (0.1 A g^{-1})** **368 mAh g^{-1} (1 A g^{-1})** **287 mAh g^{-1} (5 A g^{-1})**	**0.01-2 V**	**401 mAh g^{-1} after 1000 cycles at 1 A g^{-1}** **283 mAh g^{-1} after 3000 cycles at 5 A g^{-1}**	**This work**

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