# Supporting Information

## Electrochemical properties of ultrafine Sb nanocrystals embedded in carbon microspheres for use as Na-ion battery anode materials

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## Experimental

### Synthesis of Sb-carbon composite microspheres

Sb-carbon composite microspheres were prepared by the one-pot spray pyrolysis process which consisted with a droplet generator, a high-temperature tubular quartz reactor, and a Teflon bag filter (powder collector), as described in one of our previous reports.[33] The length and diameter of the quartz reactor were 1200 and 50 mm, respectively. A 1.7 MHz ultrasonic spray generator consisting of six vibrators was used to generate a large number of droplets from the spray solution, which were then carried to the quartz reactor via carrier gas. An aqueous spray solution containing 0.1 M of Sb chloride and 0.1 M of sucrose was prepared in a mixture of hydrochloric acid and distilled water. Reactor temperature and $N_2$ flow rate were fixed at 700 °C and 10 L min$^{-1}$, respectively.

### Characterization

The morphologies of the prepared Sb-carbon composite microspheres were investigated by scanning electron microscopy (SEM; JSM-6060, JEOL) and transmission electron microscopy (FE-TEM; JEM-2100F, JEOL). The crystal structures of the powders were investigated by X-ray diffractometry (XRD; X'Pert PRO MPD), using Cu K$\alpha$ radiation ($\lambda$ = 1.5418 Å), at the Korea Basic Science Institute (Daegu).

### Electrochemical Measurements

The electrochemical properties of the Sb-carbon composite microspheres and coarse Sb powder (Samchun, 99.9 % pure) samples were analyzed in a 2032-type coin cell. The anode was prepared from a mixture of the active material, carbon black, and sodium carboxymethyl cellulose (CMC) in a weight ratio of 7:2:1. Na metal and a microporous polypropylene film were used as the counter electrode and separator, respectively. The electrolyte was a 1 M solution of $NaClO_4$ (Aldrich) in a 1:1, by volume, mixture of ethylene carbonate/dimethyl carbonate (EC/DMC) and 5 wt% fluoroethylene carbonate. The discharge and charge characteristics of the samples were investigated by cycling over a voltage range of 0.001–2 V using various current densities. Cyclic voltammograms (CVs) were recorded at a scan rate of 0.1 mV s$^{-1}$. Electrochemical impedance spectra were obtained by AC electrochemical impedance spectroscopy (EIS), using a ZIVE SP1, over a frequency range of 0.01 Hz–100 kHz, using a potential of 10 mV.

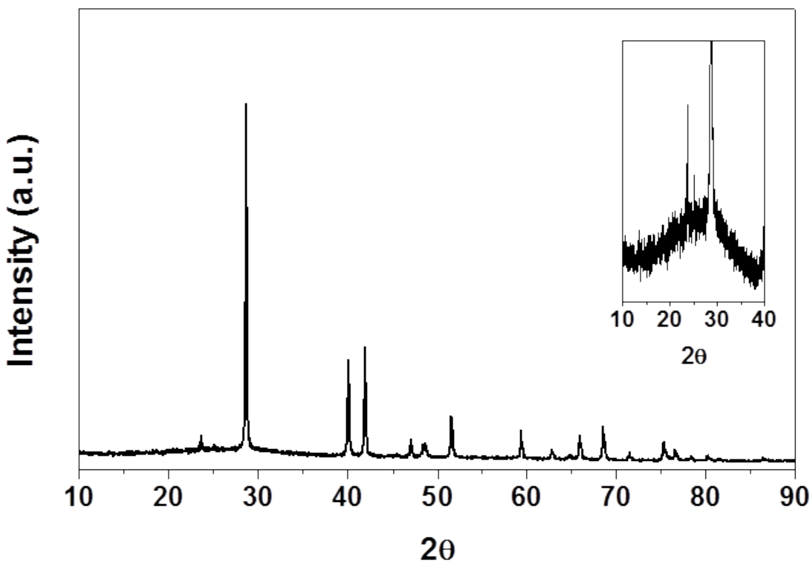
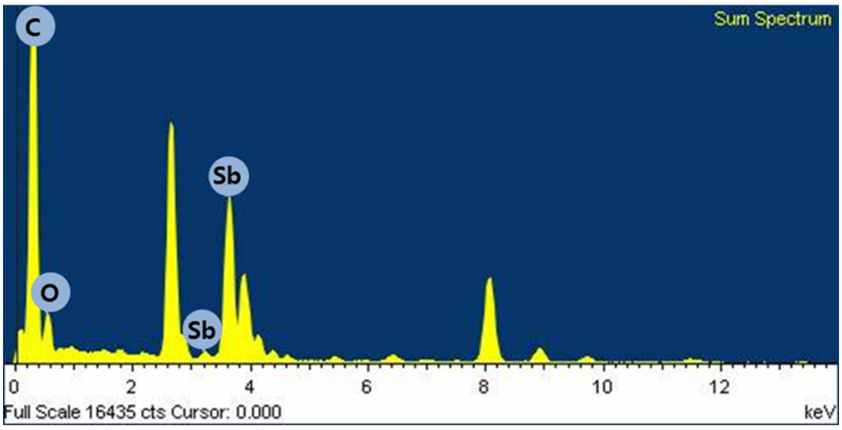



**Fig. S1** XRD pattern of the Sb-carbon composite microspheres.




| Element | Weight (%) |
| --- | --- |
| C K | 52.0 |
| O K | 4.6 |
| Sb L | 43.4 |

**Fig. S2** Result of energy dispersive X-ray spectroscopy (EDS) analysis of the Sb-C composite microspheres.

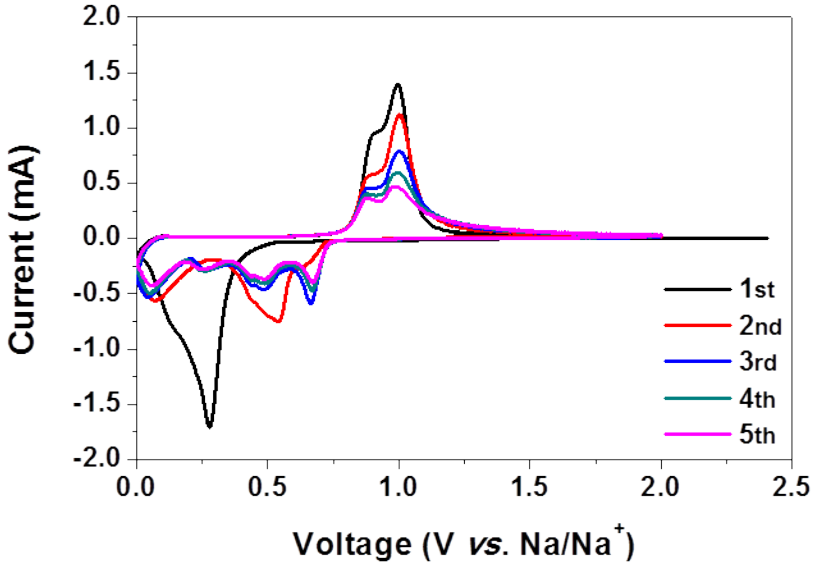



**Fig. S3** Cyclic voltammograms of the coarse Sb powders.

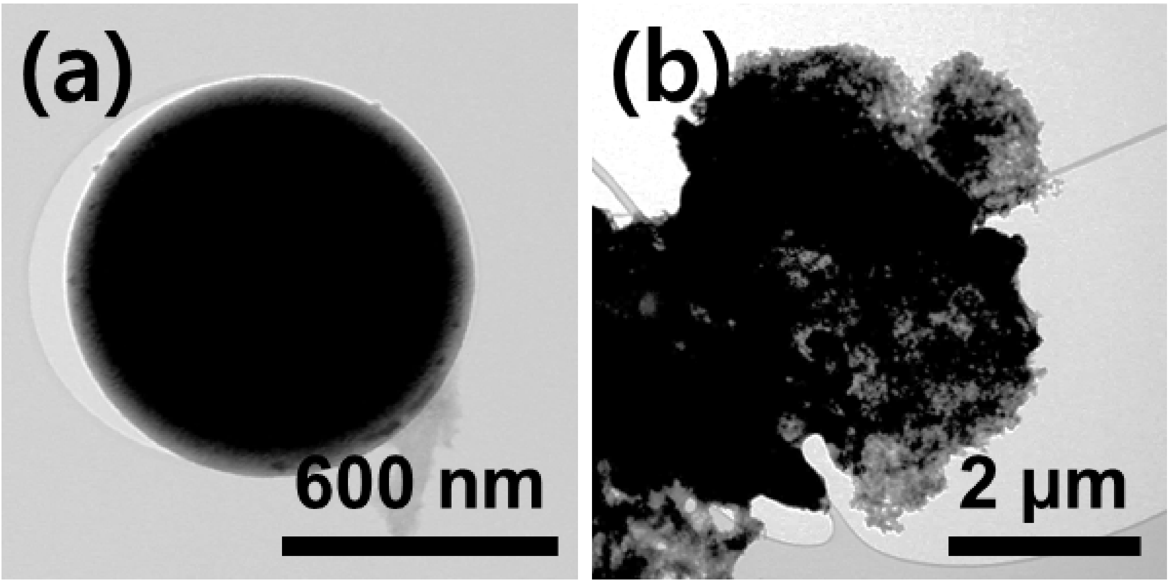



**Fig. S4.** TEM images of the electrode materials after 100 cycles: (a) Sb-carbon composite microspheres and (b) coarse Sb powders.

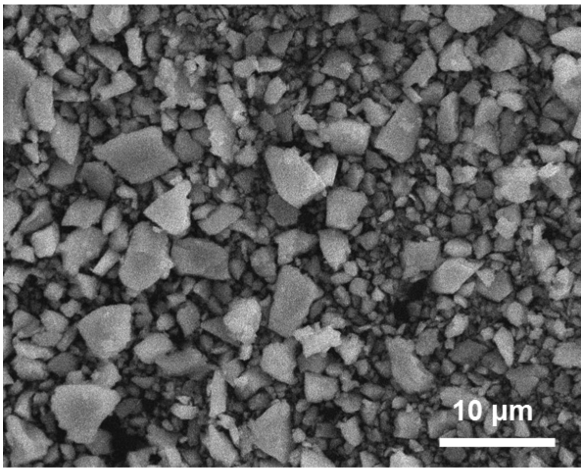



**Fig. S5** SEM image of the coarse Sb powders.