

Supporting Information:

Porous structured SnSb/C nanocomposites for Li-ion battery anodes

Cheol-Min Park,^{*a,b} and Ki-Joon Jeon^{*b}

^a School of Advanced Materials and System Engineering, Kumoh National Institute of Technology, Gumi, Gyeongbuk 730-701, Korea.

^b Environmental Energy Technologies Division, Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA.

Experimental

Materials Synthesis: The nanoporous SnSb/C composite powder was prepared as follows. SnCl₂ powder (Aldrich, ≥ 99.99 %), SbCl₃ powder (Aldrich, ≥ 99.999 %), Mg powder (Aldrich, average size: 100 μm), nano-sized amorphous carbon (Super P), and stainless steel balls (diameter: 3/8" and 3/16") with a ball-to-powder ratio of 20:1 by weight were put into a hardened steel vial having a capacity of 80 cm³. The molar ratio of SnCl₂, SbCl₃, and Mg powders used here is 1:1:2.5, respectively, and the contents of carbon are determined according to the weight percent of SnSb produced. The HEMM process (Spex-8000) was conducted under an Ar atmosphere for 2 hours. To construct the pore structured materials, MgCl₂ in the SnSb/MgCl₂/C composite was washed by solution mixed with ethanol and distilled water (1:1 by volume). These black powders were dried under vacuum at 120 °C for 24 hours. Preliminary studies showed that the

optimum amounts of SnSb and C were 60 % and 40 % by weight in terms of the first cycle capacity, initial coulombic efficiency, and cycle performance.

Materials Characterization: The nanoporous SnSn/C composite sample was characterized by X-ray diffraction (XRD, Rigaku, D-MAX2500-PC) and high-resolution transmission electron microscopy (HRTEM, JEOL 3010, operating at 300 kV). In order to investigate the pore characteristics, nitrogen adsorption-desorption isotherms were recorded at 77 K on a volumetric adsorption instrument (ASAP 2010, Micromeritics, USA) in the relative pressure range from 10^{-6} to 1. Prior to the measurement, all the samples were degassed at 250 °C under nitrogen flow for at least 3h.

Electrochemical Measurements: For the electrochemical evaluation of the SnSb and porous SnSb/C nanocomposite electrodes were prepared by coating copper foil substrates with a slurry containing the active material (70 wt%), carbon black (Denka, 15 wt%) as a conductor, and polyvinylidene fluoride (PVDF) dissolved in N-methyl pyrrolidinone (NMP) as a binder (15 wt%). These samples were pressed and dried under vacuum at 120 °C for 4 hours. Coin-type electrochemical cells were assembled in an Ar-filled glove box using Celgard 2400 as the separator, Li foil as the counter and reference electrodes, and 1 M LiPF₆ in ethylene carbonate (EC)/diethyl carbonate

(DEC) (1:1 by volume, Samsung) as the electrolyte. All of the cells were tested galvanostatically between 0.0 and 2.0 V (vs. Li / Li⁺) at a current density of 100 mA g⁻¹ using a Maccor automated tester. The gravimetric capacity was calculated with all active elements, such as SnSb and C. During discharging, Li was inserted into the working electrode, while, during charging, Li was extracted from the working electrode.