Electronic   Supplementary Information for

NiSb Alloy Hollow Nanospheres as Anode Materials for Rechargeable Lithium Ion Batteries

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Experimental Sections

1 Preparation of Ni nanospheres

Ni nanospheres: 2.85 g $NiCl_2 \cdot 6H_2O$ was dissolved in 60 mL ethanol, and the desired pH of the solution was adjusted from 10 to 12 using 1 M ethanol solution of sodium hydroxide with vigorous stirring at 70 ºC, then 20 mL hydrazine monohydrate (80 wt.%) was added. After 30 min, the product was collected by centrifugation and washed several times with deionized water and ethanol.

2 Preparation of NiSb alloy hollow nanospheres

0.1 g of the as-prepared Ni nanospheres was added to 50 mL N-methyl pyrrolidone (NMP), and the suspension was sonicated for 30 min to ensure that the nanospheres were fully dispersed. Then, $SbCl_3$ solution（0.3 g $SbCl_3$ in 50 mL NMP）was subsequently added with magnetic stirring at 80 ºC, vigorous stirring at 80 ºC was maintained throughout all syntheses under an N_2 atmosphere. All the resultant products were centrifuged and washed with deionized water, followed by washing with ethanol.

3 Characterization

Scanning electron microscope (SEM, FEI Quanta 200), transmission electron microscopy (TEM, JEM-2100F) and X-ray diffraction (XRD, Rigaku D/max 2550 VB+ 18kW, Cu Kα radiation) were used to characterize the morphology and composition of the hollow nanospheres.

For electrochemical performance evaluation, half-cells were assembled in an argon filled glove box. The as-prepared NiSb alloy hollow nanospheres powder was mixed with acetylene black and a carboxymethyl cellulose (CMC) binder (8:1:1 in weight) in deionized water to form a homogenous slurry, which was painted on a copper foil. After solvent evaporation, the electrode was pressed and dried at 100 ºC under vacuum for 12 h. Li metal foil was used as the counter electrode, and Celgard 2400 was used as the separator. The electrolyte was a solution of 1 M $LiPF_6$ in ethylene carbonate/dimethyl carbonate (1:1 in volume) electrolyte with 5% fluoroethylene carbonate (FEC) additive. The electrochemical impedance spectroscopy (EIS) measurement impedance measurements were performed on a Solartron Analytical at an AC voltage of 5 mV amplitude in the 100 kHz to 0.01 Hz during the 50th discharge-charge cycle. Cyclic voltammetric measurements were performed on CHI 660C electrochemical station to examine the cathode and anode reaction using the above-mentioned cell in the voltage range of 2 - 0.1 V (vs. Li/Li^+) at a scan rate of 0.1 mV s^{-1}. Galvanostatic charge-discharge tests were performed on LAND battery system (CT2001A) at a suitable current density between 0.01V and 2 V (vs. Li/Li^+) for both charge (Li extraction) and discharge (Li insertion) at room temperature.

Table S1 Electrochemical performances of NiSb alloy based anode materials reported recently.

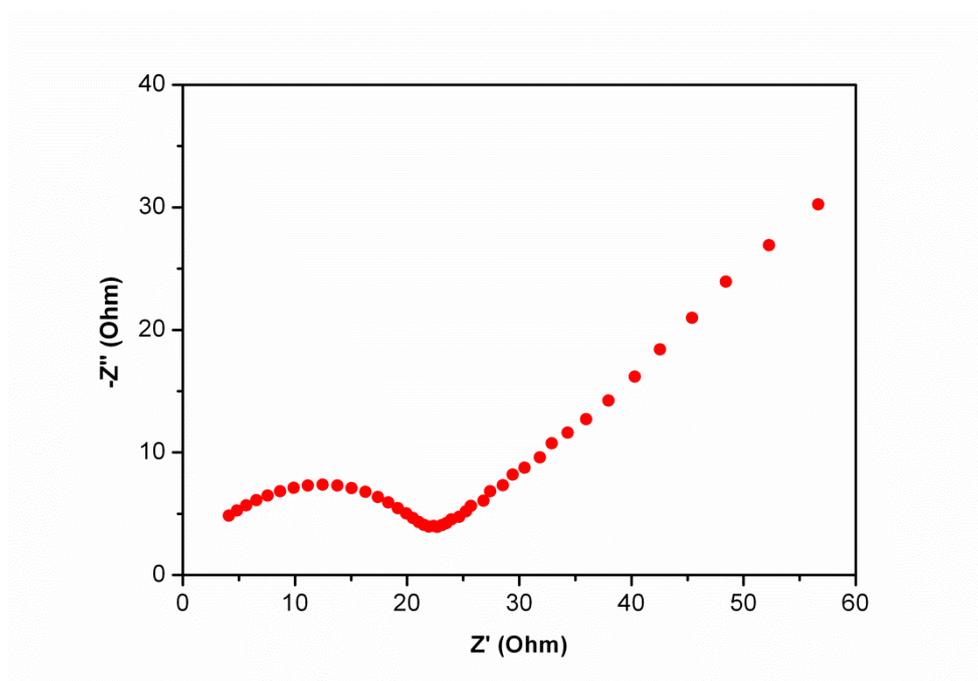| Electrode material	Reversible capactiy in the first cycle (mAh g^{-1})	Initial coulombic efficiency (%)	Reversible Capacity after 50 cycles (mAh g^{-1})	Current density (mA g^{-1})
NiSb-Al$_2$O$_3$-C[12]	~430	55.5	~300	100
Nanosized NiSb[17]	~500	62.5	~210 (20 cycles)	20
NiSb nanocrystalline[19]	223	22.7	/	0.2 mA cm^{-2}
NiSb/graphene[20]	414	48.4	~280 (30 cycles)	40
NiSb HNSs (this work)	444.8	77.1	420	100

Fig. S1 Nyquist plot of NiSb alloy HNSs.

Electrochemical impedance spectroscopy (EIS) measurement of NiSb alloy HNSs electrode was conducted to evaluate the resistance against electron transfer during cycles (Fig. S1). Generally, the high-frequency semicircle and the semicircle in the medium-frequency region are attributed to the SEI layer and/or contact resistance, and the Li^+ charge transfer impedance on the electrode-electrolyte interface, respectively. The slope in the low-frequency region of the impedance curve is related to the bulk diffusivity of the active electrode material[12, 20].