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Analytical Electron Microscopy Study of SiSn/(Reduced Graphene Oxide) Nanocomposite Powder Applicable to Li-Ion Battery Anodes

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Materials such as Si and Sn have much higher theoretical capacities as electrodes than a commercial graphite material and therefore are expected to be more opted for high performance Li ion batteries [1,2]. However, the major problem to use these metal substances is a huge volume change that causes cracking and climbing on the electrode surface. Carbon could be used as a supporting material to buffer the problematic volume change. There is a new type of carbon sheet, reduced graphene oxide (rGO) that is graphene produced by reducing graphene oxide [3]. In this study, we synthesized SiSn/rGO nanocomposite powder using a solution route method and examined its structure by means of analytical electron microscopy. The electrochemical characterization of the SiSn/rGO nanocomposite was also examined.

The rGO was prepared by a modified Hummers method that oxidizes graphite powder with $KMnO_4$ in H_2SO_4, followed by a heat treatment process at 500°C for 5h in N_2 atmosphere [3]. $SnCl_2{\cdot}2(H_2O)$ (0.1980 g) was dissolved in N_2 bubbled ethylene glycol and then Si powder (0.1000 g) and rGO (0.3000 g) were added into the solution under N_2 atmosphere. The solution was sonicated for 2 h before $NaBH_4$ (0.0956 g) was slowly added and let reacted for 30 min, followed by another sonication process for 2 h. The prepared SiSn/rGO was black colored in a form of powder and had a nominal atomic ratio of Si:Sn:C=14:3.5:100. The powder samples were examined by X-ray diffraction (XRD) for phase identification. The powders were dispersed with isopropanol onto holey carbon Cu meshes, and observed with a JEM-2200FS (with an objective lens of *C*s = 2.0 mm) configured with an incorporated Omega filter that provides energy filtered transmission electron microscopy (EFTEM), an Oxford 30mm^2 SiLi detector for energy dispersive X-ray spectroscopy (EDS), and a Gatan US 1000 CCD camera. EDS analysis and high-angle annular dark-field (HAADF) imaging were performed using a 1-nm electron beam probe and a 0.5-nm probe, respectively in scanning transmission electron microscopy (STEM).

Analytical electron microscopy with STEM HAADF and EDS, electron diffraction (ED), and high-resolution transmission electron microscopy (HR-TEM) as well as XRD revealed that the SiSn/rGO powder contains crystalline particles of Sn, Si, and SiO deposited on thin rGO lamellae. The rGO was amorphous-like graphite with distorted lattices in areas of a few nm or less in size. The size of the Sn, Si, and SiO crystalline particles was less than 10 nm. Cohered Sn and Si particles were several nm and hundred nm in size, respectively. An electrochemical test revealed that the SiSn/rGO nanocomposite powder resulted in a reversible capacity of 300~450 mAh/g and the end capacity (30 cycles) of 80~90 mAh/g, with the 1st cycle insertion capacity as high as 4000 mAh/g. The present sample was synthesized for the structural analysis before applying any special treatment and process to homogenize. The results

allow us to expect SiSn/rGO nanocomposite powder to be applicable to anode materials for high energy Li-ion batteries, accordingly.

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