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Direct Atomic-Scale Imaging of Multistep Phase Transition during the Lithiation of Nanowires by In-Situ (S)TEM

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Sb-based alloy, such as Zn-Sb[1-3], are promising anode materials because of their high theoretical capacities and suitable operating voltages. However, there have been very few studies on the detailed dynamical process of the phase transition during lithiation in the Sb-based intermetallic compounds, especially at atomic scale. By employing *in-situ* (scanning) transmission electron microscopy[4], we studied the lithium-ion diffusion and multistep phase transition during the electrochemical lithiation of individual single-crystal Zn_4Sb_3 nanowires with atomic-resolution. Continuous phase transition from crystalline rhombohedral Zn_4Sb_3 to hexagonal LiZnSb and then to cubic Li_2ZnSb phases has been directly monitored upon successive lithium-ion intercalation, which is mediated by the formation of amorphous Li_x Zn_4Sb_3 at the early stage of lithiation. The kinetics of lithiation has been found to be highly anisotropic and relevant to the dynamics of the interfacial structures of the reaction front at different stages of lithiation.

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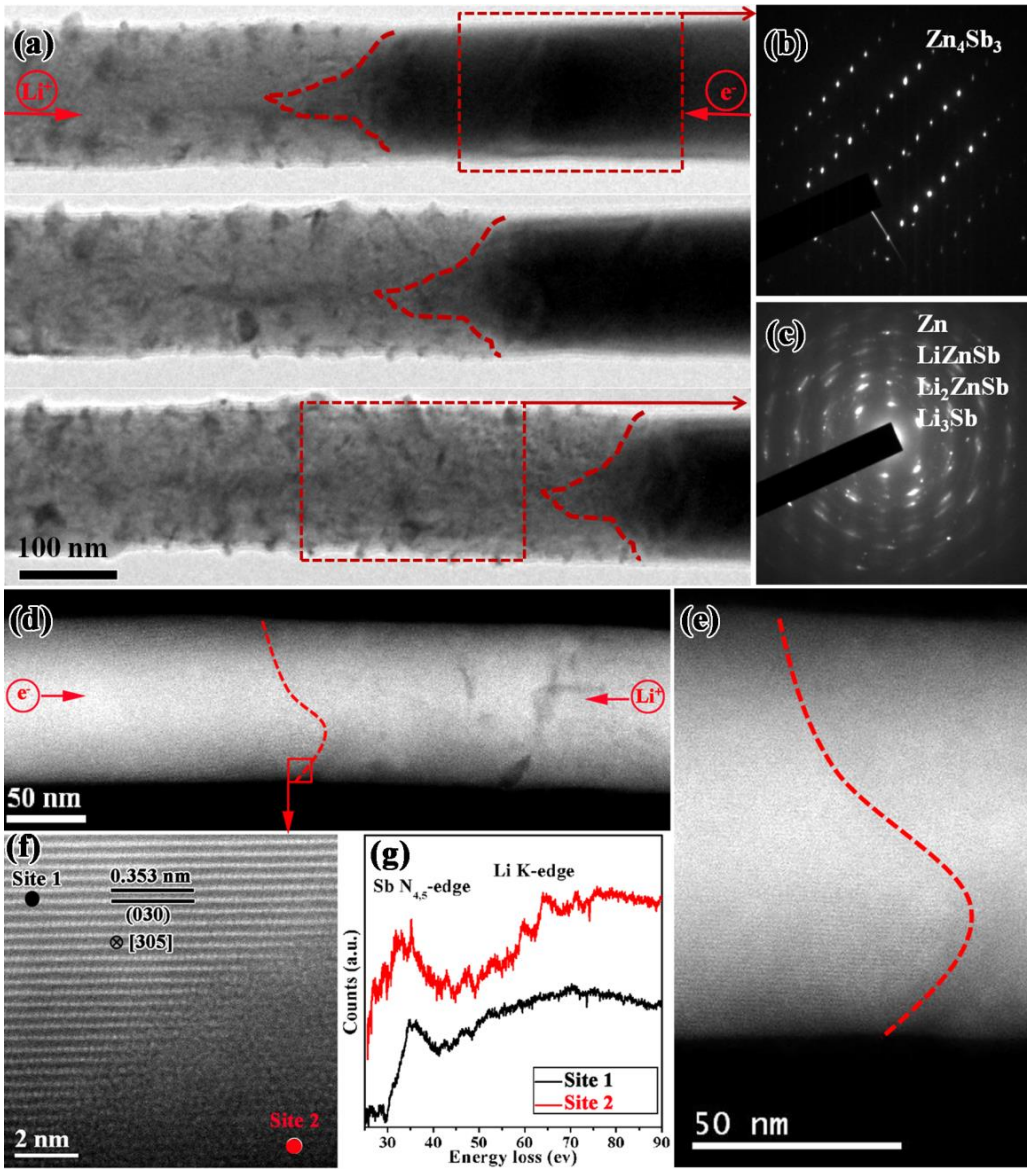

Figure 1 (a) TEM images recording the reaction front migration of the individual Zn_4Sb_3 nanowire during charging -3.0 V against lithium metal coating with Li_2O layer. (b) Electron diffraction pattern of the un-lithiated part of the nanowire indicated by the box, which is determined to be single crystal Zn_4Sb_3. (c) Electron diffraction pattern of the lithiated part of the nanowire, which shows that Zn, LiZnSb, Li_2ZnSb, and Li3Sb are formed after lithiation. (d) Low-mag HAADF image of a partial lithiated nanowire, which clear show a wedge interface between the lithiated and un-lithiated part in the nanowire. (e) High-mag HAADF image of the interface area. (f) Atomic scale resolution HAADF image of the interface, which indicates a lithiation-induced amorphous-crystalline interface at the early stage during charging. (g) EELS spectra taken from crystal and amorphous parts (Site 1 and Site t2 in panel f) of the nanowire indicating the existence of the lithium element in the lithiated part.

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