doi:10.1017/S1431927613007502
Microsc. Microanal. 19 (Suppl 2), 2013

In-Situ Analytical Electron Microscopy Study of the Lithiation of TiO_2 Nanowires used in Li-ion Batteries

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Li-ion batteries have been widely applied in almost every aspect of daily life-portable electronics, mobile devices, and electronic vehicles. In order to satisfy the demand of high capacity and high power output of the electric vehicles, a better design of the electrode materials are needed.[1-5] Most importantly, the structural and chemistry changes upon lithiation and delithiation are critical during the consideration of new designs of the electrode. Recently, in-situ TEM has been applied in the study of the structural changes of anode materials used in Li-ion batteries and significant insights have been obtained based on these in-situ studies. [1-3] Here, we report an in-situ analysis of the structural and chemical changes of one important oxide anode-anatase TiO_2 nanowires using scanning transmission electron microscopy and electron energy loss spectroscopy(EELS). The TiO_2 nanowires are synthesized using electrospinning method. The as-synthesized TiO_2 nanowires have a diameter of ~50-100nm and a length of tens of microns as shown by the SEM images in Fig 1. The TiO_2 nanowires are loaded on a gold rod which is connected into a close circuit as shown by the schematic drawing in Fig 2. The other end oppose to the TiO_2 nanowires is Li metal loaded on a tungsten tip. Due to a very short exposure to air during loading the holder into TEM column, a thin layer of Li_2O forms on the surface of Li metal, which can acts as solid electrolyte during the in-situ lithiation process. Due to the extra stability of this all-solid setup, atomic resolution images can be obtained during in-situ lithiation and delithiation. Therefore, the crystal structure changes can be directly monitored during lithium insertion/desertion. The volume changes can be directly measured based on the diameter changes before and after lithiation. Besides, the valence state changes of the Ti ions can be monitored in-situ using EELS. The Ti *L* edges can reflect the vacant density of states of the Ti 3d orbital, which is closely related to valence of Ti ions. The amount of Li ions inserted into the TiO_2 nanowires can be quantitatively compared using Li *K* edges. [6]

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[6] The research is part of the Chemical Imaging Initiative at Pacific Northwest National Laboratory (PNNL). It was conducted under the Laboratory Directed Research and Development Program at PNNL, a multiprogram national laboratory operated by Battelle under Contract DE-AC05-76RLO1830 for the U.S. Department of Energy (DOE). The work was conducted in the William R. Wiley Environmental Molecular Sciences Laboratory (EMSL), a national scientific user facility sponsored by DOE's Office of Biological and Environmental Research and located at PNNL.

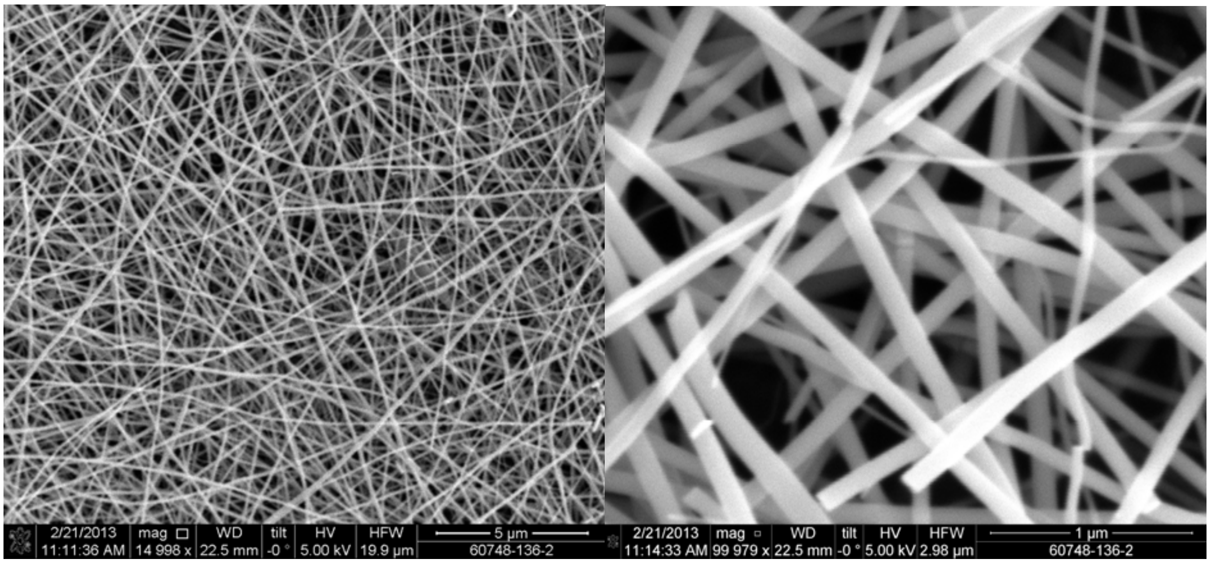

FIG. 1. (left) Overall view and (right) higher magnification SEM images of the as-synthesized TiO_2 nanowires

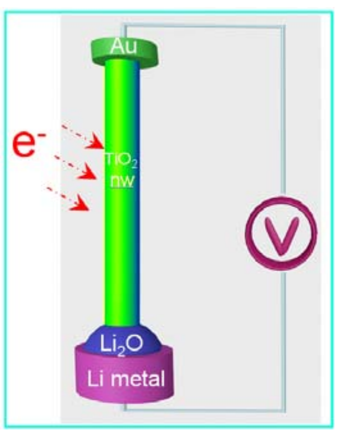

FIG. 2. Schematic drawing showing the setup of the in-situ experiment.

https://doi.org/10.1017/S1431927613007502 Published online by Cambridge University Press