Towards in situ TEM for rechargeable ion batteries

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Our society urgently need rechargeable ion batteries with both higher energy and power density. The battery communities of researchers from the academia, industry and government are driven to develop new electrode materials with higher energy and power, to meet the most stringent requirement for hybrid and electric vehicles, stationary and industrial applications.

The widespread applications of rechargeable ion batteries are currently hindered from high power and high-performance applications because of several issues. These problems include the evolution of solid electrolyte interphases (SEIs) during battery operation, which cannot be easily directly investigated under the working environment. The understanding of new material behavior under complex electrochemical environment is critical for the development of advanced materials for the next-generation rechargeable ion batteries. The dynamic conditions inside a working battery needs to be investigate deeply.

Various methods such as transmission electron microscopy (TEM), x-ray diffraction, neutron scattering, Raman spectroscopy, and electrochemical impedance spectroscopy (EIS) have been developed to characterize the material properties in batteries. Among those techniques in situ TEM techniques are emerging as a powerful tool to directly investigate the mechanisms responsible for the gradual capacity fading during cycling, poor power supply at low temperatures, thermal runaway and overcharge instability in rechargeable batteries. Examples of battery challenges which are possible to be observed directly by in situ TEM techniques are dendrite growth, cathode oxygen release, anode phase transformation, crystal expansion, interfacial delamination, and SEI formation.

For the in situ TEM, the sample preparation is usually a big challenge, especially for the samples including quite reactive materials such as Li. In the present work, we tackled the fabrication of Li-included carbon nanofibers (Li-CNFs) as an electrode used for in situ TEM of charge-discharge cycles. \textsuperscript{1} Ar\textsuperscript{+} ion irradiation onto carbon and carbon coated substrates induces the formation of CNF tipped cones (conical structures) without any catalyst even at room temperature \cite{1}. In this ion-induced CNF growth, a variety of elements can be included into CNFs during the CNF growth by a simultaneous supply of non-carbon elements during ion irradiation \cite{2}. The metal-included and also metal-coated CNFs fabricated on edges of carbon and metal foils can be used for the in situ TEM of transformation from amorphous carbon to carbon nanotubes and to graphene depending on the included/coated metals \cite{1,2,3,4,5}. In the present work, we will deal with the fabrication of Li-CNFs by Ar\textsuperscript{+} ion irradiation onto a carbon foil with a simultaneous Li supply for the further in situ TEM of charge-discharge cycles using Li-CNFs as an electrode.

\begin{thebibliography}{9}
\item M. I. Araby, et al., RSC Advances 7 (2017) 47353.
\item C. Takahashi, et al., Carbon 74 (2014) 277.
\item M. S. Rosmi, et al., RSC Advances 6 (2016) 8245
\end{thebibliography}