Achieving electrochemical capacitor functionality from a traditional battery material: conformal, nanoscale LiMn$_2$O$_4$ coatings on 3-D, device-ready carbon nanoarchitectures

Megan B. Sassin,* Steve G. Greenbaum, Phillip E. Stallworth, Azzam N. Mansour, Benjamin P. Hahn, Katherine A. Pettigrew, and Debra R. Rolison, Jeffrey W. Long,*

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XAS experimental details
The K-edge absorption was isolated by fitting the pre-edge region (6239–6439 eV) with a quadratic polynomial, extrapolating over the entire range of the spectrum, and subtracting the pre-edge background from the entire spectrum. Step normalization was applied using the atomic absorption, which was determined by fitting the post edge region (6639–8040 eV) with a cubic polynomial. The photoelectron wave number was derived by setting the inner potential to the inflection point energy. The extended X-ray absorption fine structure (EXAFS) data, $\chi(k)$, were extracted using multi-node cubic spline procedures applied to $k^3$-weighted spectra over the $k$-range of 2.0–15.0 Å$^{-1}$. The post-edge background was optimized by minimizing the amplitude of non-physical peaks in the 0–0.9 Å region of the Fourier transform. The data analysis up to this point was carried out using the WinXAS software package (version 3.1). The Fourier transforms presented here were generated with $k^3$-weighted EXAFS spectra over the range 3.0–15 Å$^{-1}$ and a Hann window of 1.0 Å$^{-1}$ using the curve fitting code FEFFIT (version 2.984) of the University of Washington XAFS (UWXAFS) software package.

Fig. S1. Solid-state $^{23}$Na nuclear magnetic resonance spectra of Na$^+$-MnO$_x$ (—) and Li$^+$-MnO$_x$ (—).
Fig. S2. Thermal gravimetric analysis (---) and differential scanning calorimetry (––) of (A) Li\(^+\)-MnO\(_x\) in an argon atmosphere and (B) Li\(^+\)-MnO\(_x\)(300-Ar) in an oxygen atmosphere.

Fig. S3. Plot of # of Li atoms per gram of sample versus fraction of Li atoms in tetrahedral site derived from solid-state \(^7\)Li NMR for LiMn\(_2\)O\(_4\) standard (▼), Li\(^+\)-MnO\(_x\) (●), Li\(^+\)-MnO\(_x\)(300-Ar) (●), and Li\(^+\)-MnO\(_x\)(300-Ar/200-Air) (●).
Fig. S4. Scanning electron micrographs of the exterior surface of (A) Li\textsuperscript{+}-MnO\textsubscript{x}(300-Ar/200-Air), (B) Li\textsuperscript{+}-MnO\textsubscript{x}(300-Ar/200-Air) after 200 voltammetric cycles in 2.5 M Li\textsubscript{2}SO\textsubscript{4} and (C) Li\textsuperscript{+}-MnO\textsubscript{x}(300-Ar/200-Air) after 200 cycles in 2.5 M Li\textsubscript{2}SO\textsubscript{4} + 20 mM NaHCO\textsubscript{3}.

Fig. S5. X-ray photoelectron C1s spectra of Li\textsuperscript{+}-MnO\textsubscript{x}(300-Ar/200-Air) (—) and Li\textsuperscript{+}-MnO\textsubscript{x}(300-Ar/200-Air) after 200 voltammetric cycles in 2.5 M Li\textsubscript{2}SO\textsubscript{4} + 20 mM NaHCO\textsubscript{3} (—).

Notes and references


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