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Time-resolved in-operando neutron imaging of lithiation and delithiation process in custom-built rechargeable Li-ion batteries

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We are reporting an in-operando study of time-resolved neutron imaging on cycled Li-ion batteries to analyze the phase changes during lithiation and delithiation.

Rechargeable lithium-ion (Li-ion) batteries are used to power up our daily portable appliances. They are electrochemical cells consisting of a positive electrode separated from a negative electrode, in electrolyte solution, which allows only Li-ions to move between the electrodes.

The most common material used for the negative electrode in rechargeable commercial Li-ion batteries is graphite, due to its mechanical stability and good electrical conductivity. Li-ions are intercalated in between and within the graphene layers in the graphite structure, creating crystallographic phase changes. The charge-discharge process is accompanied by the challenge of (de)intercalating Li-ions into/from the crystalline structure, thus forcing the lattice to distort and create defects, which contributes to transport-related structural damage upon fast cycling, thus shortening the lifetime. [1]
Kinetic behaviour of Li-ions and phase transformation mechanisms are poorly understood due to difficulties in isolating these factors experimentally. However, these changes can be observed using neutrons [2]. Due to the large neutron cross-section of Li and due to their penetration of bulk samples they are well suited for in-operando studies of Li-ion batteries.

We present the results of in-operando time-resolved Bragg-edge transmission neutron experiments of charge-discharge cycles of a custom-built Li-ion half-cell performed at RADEN@J-PARC, Japan. The measurements were performed on a custom-made battery cell with graphite:carbon black:polyvinylidene fluoride (8:1:1) as the working electrode and metallic Li as the counter electrode. They were charged and discharged at different C-rates, the current rate normalized to the maximum battery capacity. The first cell was discharged at two C-rates: C/34 and C/68, with a short period of relaxation between the discharges, and charged at C/34. The second cell was discharged at C/20 and at C/34 until the potential reached 0.001 V, with a relaxation period in between, and charged at C/34 until 3 V.
Results of the neutron radiography experiments show phase changes in the working electrode and lithium intercalation and deintercalation during cycling. The phase changes are reflected in the variations of the graphite, LiC_{12} and LiC_6 characteristic Bragg edges.

[1] K. Persson et al., J. Phys. Chem. Lett., 2010, 1 (8), 1176-1180
[2] M. Kamata et al., J. Electrochem. Soc., 1996, 143 (6), 1866-1870

Primary author(s) : LACATUSU, Monica-Elisabeta (Technical University of Denmark); JOHNSEN, Rune E. (Technical University of Denmark); KUHN, Luise Theil (Technical University of Denmark); SALES, Morten (Technical University of Denmark); Prof. SCHMIDT, Søren (Technical University of Denmark); STROBL, Markus (PSI); TREMSIN, Anton (University of California - Berkeley); SHINOHARA, Takenao (Japan Atomic Energy Agency)

Presenter(s) : LACATUSU, Monica-Elisabeta (Technical University of Denmark)

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