

Experimental report

16/04/2024

Proposal: 5-24-690

Council: 10/2022

Title: Temperature dependent aging behavior of commercial 18650-type Li-Ion batteries

Research area: Physics

This proposal is a new proposal

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Samples: Commercial Li-Ion battery LG INR18650HG2

Instrument	Requested days	Allocated days	From	To
D2B	3	1	24/05/2023	25/05/2023

Abstract:

Facing climate change and global warming, replacing internal combustion engine vehicles with electric vehicles (EV) is one of many important steps to reduce the emissions of greenhouse gases. However, the increasing needs of the market calls for the development of high capacity, high power and high energy density batteries at lower costs, like state-of-the art lithium-ion batteries consisting of high performance graphite anodes and high nickel content $\text{Li}_x\text{NiMnCoO}_2$ (NMC) cathodes, used in EVs, such as the BMW i3 and the Nissan Leaf. Nevertheless, the performance of the batteries is still limited by the capability of the negative electrode to store lithium ions in its structure and the performance of high nickel content NMC cathodes. In addition, there is a clear lack of information on the effect of elevated temperatures on the structural performance of the cell during electrochemical cycling, especially for commercial Li-ion batteries, such as 18650-type. Thus, the obtained data will clarify the Li_xC_6 phase formation at elevated temperatures and the aging behavior of the cell.

<h1 style="margin: 0;">ILL</h1> <h2 style="margin: 0;">Experimental Report</h2>	Experiment title: Temperature dependent aging behavior of commercial 18650-type Li-Ion batteries	
	Principal proposer: Tobias Hölderle Experimental team: Dr. Anatoliy Senyshyn, Tobias Hölderle	<i>Experiment Number:</i> 5-24-690 <i>Date:</i> 24.05.23 – 25.05.23

Experimental set up

The experiment was carried out at the high-resolution diffractometer D2B used in (335) reflection mode of the focusing Ge monochromator with an take-off angle of 135° maintaining a wavelength of $\lambda = 1.594 \text{ \AA}$, using the full flux option. The experiment was carried out on a batch of fresh and aged (at RT, 10°C , 50°C) Li-ion batteries of type INR18650HG2 by LG with a nominal capacity of 3000mAh. Each aged batch was electrochemically cycled 500 times at the home institution at a desired temperature in a climate chamber to reduce the state of health. For each cycle protocol two cells were used and prepared for the experiment in a fully charged (100%) and a fully discharged (0%) state. The cells were mounted to the automatic sample changer of the beamline, combining neutron powder diffraction with a frozen electrochemical state of charge of the cell. The exposure time for one cell was set to roughly 2 hours.

Results

The collected data of the cells show important structural changes within the aged battery cells related to the degradation during the electrochemical cycling. In Fig. 1a) reflections of the LiC_6 (001, 25°) and LiC_{12} phase (002, 26.25°), representing a fully charged state of the cells are shown, which revealed significant changes depending on the electrochemical cycling. While for a fresh cell mostly the LiC_6 phase is present, the aged cells show reduced amounts of LiC_6 with increasing LiC_{12} reflection intensities. Such a change represents the consumption of Li and the decrease of the cell capacity during the electrochemical cycle. Furthermore, small reflections at a diffraction angle of 27.5° revealed amounts of pure graphite structure, showing parts of inactive material within the cell. In addition, for the low temperature cell (10°C) a shoulder was observed close to the LiC_{12} reflection revealing parts of low lithiated graphite (LiC_{30}). Contrary, in a fully discharged state, only the cell cycled at high temperatures (50°C) shows a different behavior marked by an arising reflection observed at 26.5° , which is dedicated to low lithiated graphites. The remaining cells only show reflections at 27.5° , dedicated to a pure graphite phase (002). Such difference indicates inactive lithiated graphite parts during the discharge of the cell.

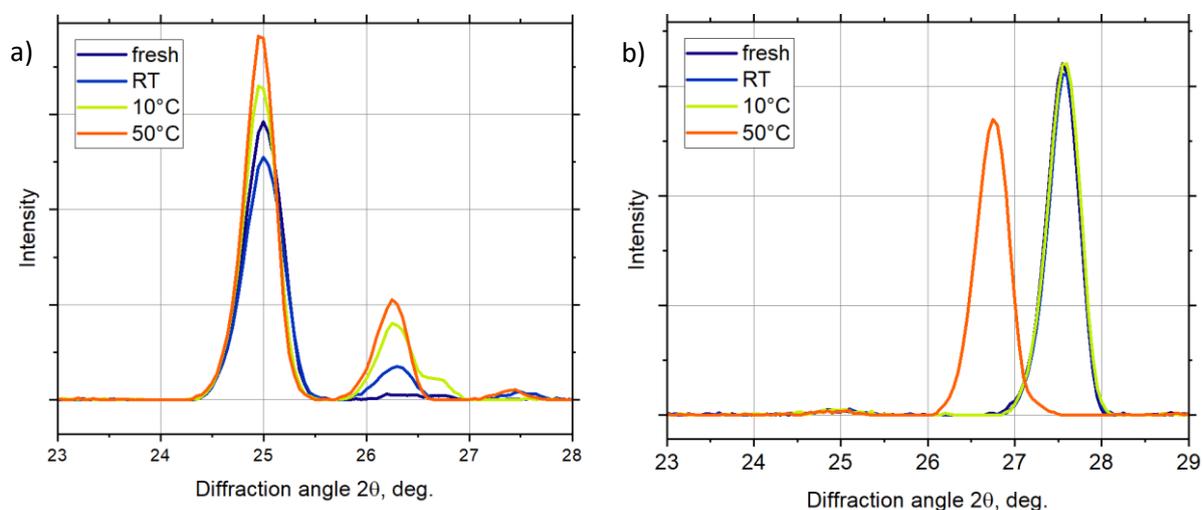


Fig. 1 Observed reflections dedicated to the lithiation of the graphite structure for (a) a fully charged state and (b) a fully discharged state of battery cells cycled within different temperatures.