## MS42-O5 Stroboscopic neutron powder diffraction at HRPT, SINQ

Denis Sheptyakov<sup>1</sup>, Vladimir Pomjakushin<sup>1</sup>, Lucien Boulet-Roblin<sup>2</sup>, Claire Villevieille<sup>2</sup>, Gerd Theidel<sup>3</sup>, Mark Könnecke<sup>4</sup>, Roman Bürge<sup>4</sup>

1. Laboratory for Neutron Scattering and Imaging, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland

2. Electrochemistry Laboratory, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland

3. Laboratory for Particle Physics, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland

4. Laboratory for Scientific Developments and Novel Materials, Paul Scherrer Institute, 5232 Villigen PSI, Switzerland

## email: denis.cheptiakov@psi.ch

At HRPT, the high-resolution powder neutron diffractometer at SINQ\*, a new mode of operation has been realized and tested. This stroboscopic measurements mode is intended for studies of crystal or magnetic structure transformations occurring in short (available time binning down to 10 milliseconds), but multiply repeating processes triggered by some external effect, e.g. voltage, magnetic field, thermal heat pulse, etc. The diffraction data corresponding to different time slices after a certain repeating strobo signal are stored in the different histogram memory blocks (up to about 70000 independent diffraction patterns). In our tests, we could successfully slice time down to 10 milliseconds with overall duration of experiment of several hours, so the processes, or existence ranges of phases on the order of tens of milliseconds, seconds, minutes etc., - can now be addressed efficiently by neutron diffraction at HRPT. The first real studies, including the operando investigation of ultrafast charging-discharging cycles of a commercial Li-ion battery (LiFePO $_4$  vs. graphite) will be presented.

\* https://www.psi.ch/sinq/hrpt/hrpt

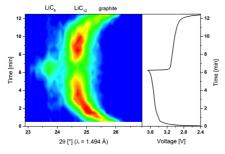


Figure 1. Selected sections of the stroboscopic neutron powder diffraction patterns corresponding to 120 charge-discharge cycles of a battery, showing transition between graphite, LiC<sub>12</sub> and LiC<sub>6</sub> phases and back. Overall charge-discharge time is approximately 12 min. Data binned with 30 seconds per pattern.

Keywords: stroboscopic, neutron diffraction

MS43 Combining x-ray diffraction and other techniques for in situ and operando studies

Chairs: Helmut Ehrenberg, François Fauth

## MS43-O1 Revealing Secrets of Lithium-Ion Battery Operation by Neutron Scattering

Martin J. Muehlbauer<sup>1,2,3</sup>, Anatoliy Senyshyn<sup>3</sup>, Michael Knapp<sup>1,2</sup>, Helmut Ehrenberg<sup>1,2</sup>

1. Helmholtz-Institute Ulm for Electrochemical Energy Storage (HIU), P.O. Box 3640, D-76021 Karlsruhe, Germany

 Institute for Applied Materials (IAM), Karlsruhe Institute of Technology (KIT), Hermann-von-Helmholtz- Platz 1, D-76344 Eggenstein-Leopoldshafen, Germany

3. Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Lichtenbergstr. 1, 85748 Garching, Germany

## email: martin.muehlbauer@kit.edu

Powering innumerable portable devices lithium-ion batteries are part of our everyday life for decades already. Although lithium-ion chemistry does a good job powering consumer electronics and tools, there are still some processes inside lithium-ion batteries that are not understood completely. Especially an increasing number of applications related to electromobility and energy storage calls for further improvements of their life span, energy/power density and rate capability. Therefore, single cells or even integrated batteries have to be investigated under real operating conditions to unravel details occurring in the millimetre to micrometre domain and reaching down to a nanometre or even atomic length scale. Neutrons offer a capability to conduct in operando investigations on standard size Li-ion cells even in combination with additional sample environment, e.g. to study temperature effects. For low temperatures an intercalation behaviour differing from the one at room temperature has been observed inside the graphitic anode [1]. Neutron radiation is not only sensitive for light elements but also enables to distinguish neighbouring elements. Therefore, structural changes, phase transitions and cation exchange reactions, e.g. in cathode materials, may be traced during intercalation and deintercalation of lithium providing detailed information about battery operation [2]. Inhomogeneities of the state of charge inside 18650-type Li-Ion cells were pointed out by neutron imaging and neutron diffraction experiments. They could be quantified by spatially resolved neutron diffraction on a macroscopic length scale as inhomogeneities of the lithium concentration inside the anode for fully charged cells [3]. Here a correlation between the cell design and the lithiation state of the graphitic anode has been found.

Results based on electrochemical cell characterization, neutron imaging and scattering techniques will be presented. They will be discussed in terms of their