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Using operando X-ray Absorption Imaging for Quantification of Salt Concentration Profiles in Battery Electrolytes

For the optimization of electrochemical systems, e.g. lithium-ion batteries (LIBs), precise and accurate knowledge of electrolyte transport parameters –transference number, diffusion coefficient, thermodynamic factor, and conductivity –is required. Quantitatively determining these concentration-dependent transport parameters is non-trivial because LIB electrolytes are non-dilute and conventional electrochemical polarization methods are thus not straightforwardly applicable [1].
Recently, a variety of *operando* imaging and spectroscopic methods have been developed, which allow direct measurement of salt concentration profiles in electrolytes upon cell polarization and subsequent relaxation providing new insight [1-3]. Using dedicated electrochemical cells, we have developed a novel *operando* X-ray radiography setup to directly measure concentration profiles in polarized electrochemical cells using the relationship between salt concentration and X-ray absorption [4].
First results on the LiTFSI in PEO polymer electrolyte system will be presented. Although LiTFSI/PEO is a generally well-studied and promising electrolyte candidate for LIBs, recent results show drastically different concentration profiles (and therefore transport parameters) measured by different methods [5, 6]. We expect to shed light on this discrepancy using our X-ray radiography approach and anticipate that our novel methodology can contribute to the efficient determination of electrolyte transport parameters and thereby support knowledge-based optimization of electrolytes.
The corresponding synchrotron experiments were carried out on the imaging beamline P05 at PETRA III at DESY, operated by Helmholtz-Zentrum Hereon. The local transmission change of symmetrical Li/LiTFSI-PEO/Li electrochemical cells with different salt concentrations and molecular weights of PEO was measured during potentiostatic polarization. The distance between the electrodes was 3.5 mm and the imaging resolution was 1.25 µm.

First results for the for the 1.9 M LiTFSI in PEO system are shown in Figure 1.
It illustrates the evolution of the concentration calculated from the change in transmission over time from the center of the electrolyte channel to the electrode during the polarization with 0.3 V. A preliminary evaluation indicates that the profiles are symmetrical with respect to the center of the channel (position = 0), and we will discuss the implications for the concentration dependence of the transport numbers.
In addition, the adaptation of this method for the measurements with a laboratory-based X-ray source will be discussed.

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Primary author: Mr SKRENTNY, Emil (1. Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW) ; 2. RWTH Aachen University, Institute of Physical Chemistry)

Co-authors: Mr HUCK, Marten (1. Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW) ; 2. RWTH Aachen University, Institute of Physical Chemistry); Mr KUHLMANN, Andreas (1. Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW) ; 2. RWTH Aachen University, Institute of Physical Chemistry); Dr SALEEV, Artem (Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW)); Mr SIT, Ferit (1. Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW) ; 2. RWTH Aachen University, Institute of Physical Chemistry); Dr TOBER, Steffen (Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW)); Dr WILDE, Fabian (Helmholtz-Zentrum Hereon); Prof. STEINRÜCK, Hans-Georg (1. Forschungszentrum Jülich GmbH, Institute for a Sustainable Hydrogen Economy (INW) ; 2. RWTH Aachen University, Institute of Physical Chemistry)

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