Laboratory / Team : CEA-IRIG. SyMMES / STEP
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Title
Operando characterization of batteries using neutron and synchrotron imaging techniques

Keywords : Li-ion batteries ; fast charging ; lithiation mechanism ; synchrotron ; diffraction ; neutron imaging
Starting date : February - March 2023
Duration : 5- 6 months

Background: Operando characterization to understand and improve Li-ion batteries

Efficient, safe, and environmentally-friendly energy storage is a key requirement to successfully transitioning from depleting fossil fuels to renewable energy sources. In this regard, Li-ion batteries are playing a decisive role, particularly in meeting the demands of a highly mobile society. Nevertheless, there are great challenges remaining towards the worldwide success of battery-powered electric vehicles, which mostly revolve around increasing the energy density and improving fast charging. Fast charging typically leads to detrimental reactions such as (1) Li dendrite growth, being a microfilament of Li metal growing for the anode to the cathode, potentially leading to short circuit, (2) heterogeneous lithiation at the micro and nanoscale which might lead to underutilization of active material (some part of the anode or cathode don't store lithium ions, hence reducing the capacity).
Designing effective strategies to mitigate these harmful processes require a better understanding, and hence the development of *operando* characterization techniques: being none destructive tools to image Li concentration in the battery during fast operation. Imaging down to the microscale, in a few minutes, inside a battery is extremely challenging and can't be performed using standard characterizations.

Large Scale facilities operando tools: neutron imaging and microbeam X-ray diffraction.

ESRF (synchrotron) and ILL (nuclear reactor) are European Large Scale Facilities providing high energy and high flux X-ray and neutrons beams to perform unique cutting edge characterizations experiments. They are particularly useful for operando battery characterization because synchrotron X-ray and neutron beams can i) penetrate deeply into matter and hence probe inside a working battery, ii) can give information from the micron to nanoscale with short acquisition time. In particular, our team uses two techniques detailed on the figure and in the text below:

Operando Synchrotron microbeam X-ray Diffraction (ESRF). In this experiment, a micro X-ray beam is scanned over the battery, and is scattered by crystalline electrode material inside the battery. Alike lab X-ray diffraction, the scattered beam is collected and the data used to determine the atomic structure evolution of the electrode material during Li insertion/extraction.[1,2]

Operando neutron imaging (ILL): Schematically, this experiment is similar to medical radiography, but with neutrons instead of X-rays and a battery instead of the human body. A beam of neutron passes through the cell, and since neutrons are absorbed by Li, the transmitted neutron flux and proportional to Li concentration. This techniques can image Li concentration in liquid and solid phases.

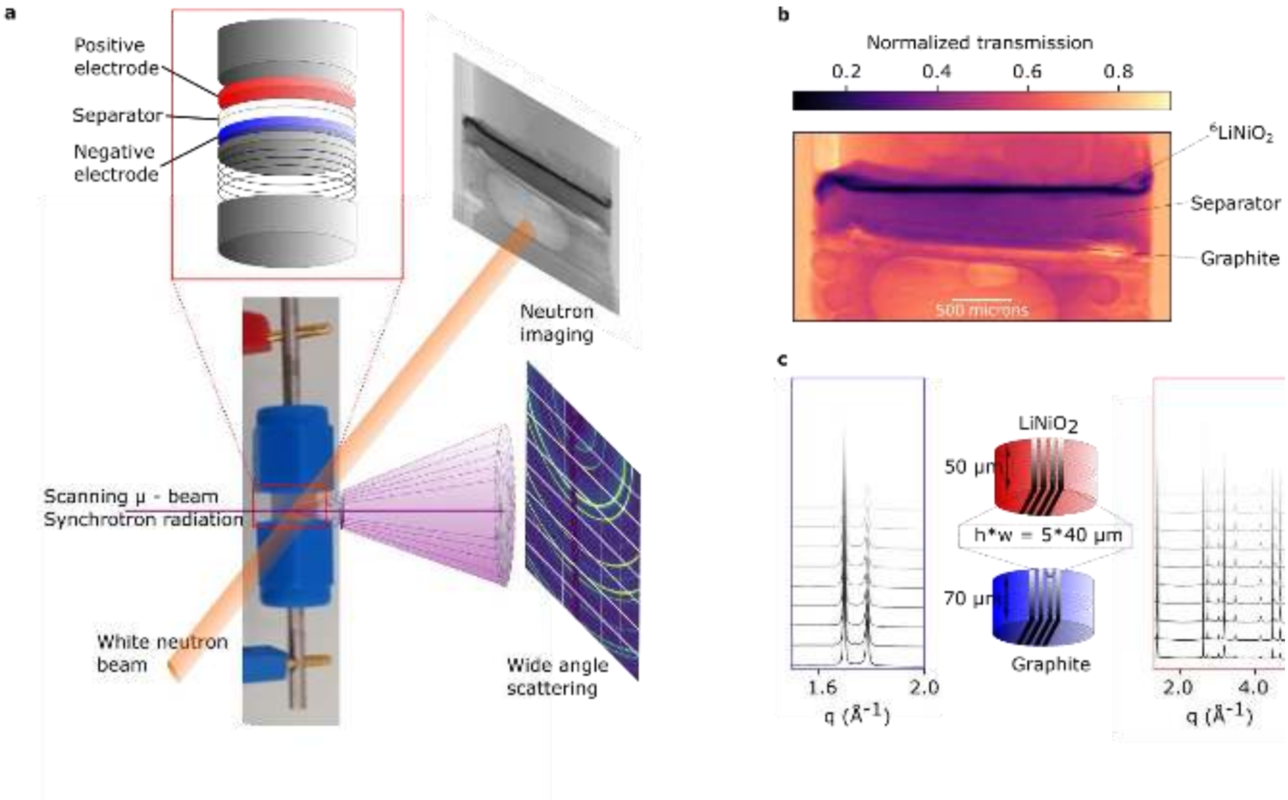

Figure 1 : a – schematic of the correlated neutron imaging and microbeam XRD experiment on a $LiNiO_2$/Graphite cell, b) typical neutron imaging for the pristine cell, c) schematic of the micro X-ray beam scanning protocol together with typical XRD patterns of LNO across the depth of the electrode.

<u>The internship</u>

The aim of the intern is to help analyzing neutron imaging and/or microbeam XRD data already obtained by the team. The analysis mainly consist in processing and handling large amount of neutron images and X-ray diffraction patterns using python codes already developed in the team. Also, the intern will have the opportunity to participate to synchrotron and neutron experiments (users are typically granted 3-4 days of continuous measurement 24h/24h – exciting experience in the heart of unique instruments). Data have been collected on $LiNiO_2$//Graphite full cell being the state of the art high energy density cell chemistry for EV-applications[3]. Materials have been provided by industries (BASF, CIDETED) through european BIG-MAP project (https://www.big-map.eu/), part of battery 2030+ initiatives (https://battery2030.eu/).The analyzed results will hence be presented and shared across a large consortium of European academic laboratory, with possible collaboration with modelling teams. The intern will be working for 6 months at CEA in Grenoble, a major French National Lab focused on the development of low-carbon energies, and more specifically at the IRIG, the fundamental science department of CEA.

<u>Requested skills:</u> M2 master student with a knowledge in data analysis (python), material characterization (diffraction, imaging) and/or energy storage devices (Li-ion batteries) will be important.

<u>Possibility to follow with a PhD</u>. Ecoles Doctorales: Ed Physique

<u>References:</u>

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(3) Xu, C.; Reeves, P. J.; Jacquet, Q.; Grey, C. P. Phase Behavior during Electrochemical Cycling of Ni-Rich Cathode Materials for Li-Ion Batteries. *Adv. Energy Mater.* **2021**, *11* (7), 2003404. https://doi.org/10.1002/aenm.202003404.