International Workshop on Advanced Characterization of Li-ion Batteries by Neutrons and X-rays

20th June

Time	Speaker	Talk tittle
13:50 – 14:00	Opening by Ida Westermann, Head of Department of Materials Sciences and Engineering of NTNU	
14:00 – 14:40	Halvor Høn Hval (MORROW)	A sneak peek into Morrow and the technology of tomorrow
14:40 – 15:20	Laura Simonelli (ALBA)	Synchrotron based techniques applied to the battery investigation: ALBA capabilities and current investments
15:20 – 15:25	group picture	
15:25 – 15:40	coffee break	
15:40 – 16:20	Silje Nornes Bryntesen (FREYR)	The outline of FREYR battery and challenges behind Semi solid battery technology
16:20 – 17:00	Dmitry Chernyshov (SNBL-ESRF)	Operando crystallography of electrochemical batteries with synchrotron light

21st June

09:00 – 09:40	Obinna Eleri (BEYONDER)	Revealing capacity fade mechanisms of activated carbon lithium-ion capacitors
09:40– 10:20	Andrea Sorrentino (ALBA)	Visualizing electrodes chemical states at the nanometer scale using Soft X-ray Transmission Microscopy
10:20 – 10:40	coffee break	
10:40 – 11:20	Per Erik Vullum (SINTEF)	Microscopy characterization of Li-ion battery materials, including degradation mechanisms as a function of cycling
11:20 – 12:00	Stefano Checchia (ID15A-ESRF)	Understanding battery performance and degradation by means of spatially-resolved operando diffraction
12:00 – 12:40	Ana García Prieto (SpLine-ESRF)	Spanish CRG BM25-SpLine beamline at the ESRF, The European Synchrotron: Present opportunities for the characterization of Li-ion batteries
12:40 – 14:00	lunch	
14:00 – 14:40	Javier Campo (CSIC))	Neutron scattering: basics concepts and examples related with Li ion batteries
14:40 – 15:20	Markus Strobl (PSI)	Neutron Imaging applications in battery research at the Swiss Spallation Neutron Source (SINQ)
15:20 - 16:00	Jacob LaManna (NIST)	Using neutrons to understand electrochemical system performance and degradation.

22nd June

09:00 – 09:40	Marit Kjarvik (VIANODE)	From scrap to battery grade graphite- Using X-ray based techniques as a characterization tool in the recycling process
09:40 – 10:20	Angel Larrea (CSIC)	Operando analysis with spatial and time resolution of lead cells using gauge-volume neutron diffraction
10:20 – 11:00	Kenneth Marshall (SNBL-ESRF)	BM31: the presentation
11:00 – 12:00	DISCUSSION & CLOSURE	

Halvor Høn Hval, Morrow Batteries, Norway

A sneak peek into Morrow and the technology of tomorrow

The Norwegian battery industry is growing at a high pace, and this presentation will show this from Morrow Batteries' point of view. What is Morrow Batteries? Who are we, and what are our plans? Most of our R&D activities are aimed towards the commercialization of the next generation cathode material LNMO (LiNi0.5Mn1.5O4), which is the basis of two of our products. The first part of the talk will focus on one of these, namely the LNMO-XNO cell. Neither of these electrode materials have previously been integrated into commercial cells, so the need for advanced characterization from Morrow and our research partners is necessary for this to succeed. This technology could potentially revolutionize the industry, and it exemplifies how Morrow wants to develop and manufacture the most cost-effective and sustainable battery cells in the world. The second part of the talk will touch upon some of the key usages of X-rays into industrial aspects such as prototyping, production and quality control.

Laura Simonelli, Beamline Responsible BL22 CLAESS Core Level Absorption and Emission Spectroscopies Beamline, ALBA, Spain

Synchrotron based techniques applied to the battery investigation: ALBA capabilities and current investments

The 3rd generation ALBA synchrotron in Spain has currently 10 operational state-of-the-art beamlines, comprising soft and hard X-rays, which are devoted mainly to materials science, condensed matter, and biosciences. Four additional beamlines are currently in construction at different level of progress. 7 over the 10 running beamlines are investing in the study of battery materials (below an overview of the offered techniques), while a new laboratory devoted to the battery investigation has been recently opened to support the operando experiments in this field. ALBA offers access to XAS spectroscopy in the soft, tender, and hard X-ray ranges, XES in the hard x-ray range, XPS, XRD, SAXS/WAXS, and FTIR, and, in most of the cases, in operando conditions. Moreover, STEM microscopes are operational or under commissioning, with a third one under development, which will be dedicated to environmental conditions and coupled to STM/AFM or other techniques. The provided multi-scale and multi-modal approach allows to access complementary information, fundamental to answer scientific questions when investigating complex systems. Moreover, the biggest advantage of such a multi-technique approach lies in the observation of dynamic processes, as during operando or in-situ experiments, going beyond the sum which could be obtained by individual experimental methods.

Silje Nornes Bryntesen, Senior Researcher, FREYR, Norway

The outline of FREYR battery and challenges behind Semi solid battery technology

FREYR Battery is a lithium-ion battery manufacturer founded in Norway in 2018, listed on the New York Stock Exchange in 2021, and planning to start mass production in 2024. FREYR will manufacture batteries in Norway, an advanced decarbonization country, exclusively using clean renewable hydroelectric power locally available. Consequently, FREYR's production of battery cell is environmentally friendly, and it gains competitive advantage in terms of abundant energy supply and lower cost, coupled with lower CO_2 emissions. FREYR is at the forefront in revolutionizing the inefficient conventional battery manufacturing procedure by industrializing the Semi-solid lithium-ion battery manufacturing technology developed by 24m Technologies (US). The Semi-solid technology is unique from a production and technological perspective as it reduces the manufacturing steps, inactive battery components, and enables production of thicker electrodes. High electrode thicknesses allow for higher battery energy density; however, the internal cell resistance increases. To fully exploit this groundbreaking technology and effectively optimize our cells, it is vital for Freyr and our partners to gain an in-depth understanding on the cell behavior. Such understanding can only be gained through advanced characterization techniques.

Dmitry Chernyshov, Swiss-Norwegian BeamLines at the European Synchrotron Radiation Facility (SNBL at ESRF), Grenoble, France

Operando crystallography of electrochemical batteries with synchrotron light

Operando synchrotron experiments with electrochemical batteries is a very popular playground to uncover microscopic mechanism underlaying kinetics of change and discharge, capacity, and cyclability. Here we discuss synchrotron diffraction experiments with operando battery cells at BM01 station of Swiss-Norwegian BeamLines at ESRF. Representative examples include a structural evaluation of Li diffusion pathway in ionic conductors, characterisation of phase transformations and phase co-existence during sodiation and lithiation of cathode materials, structural aspects of partially ordered anodes, and structure of new materials for solid state electrolytes. Practical aspects of operando experimentation at synchrotron beamline will also be considered, as well as battery-specific data analysis of big volumes of diffraction data.

Obinna Eleri, Beyonder AS, Norway

Revealing capacity fade mechanisms of activated carbon lithium-ion capacitors

Li-ion capacitors (LiC) are a unique type of hybrid energy storage solution that fills the gap between Supercapacitors (SC) and Li-ion batteries (LiB). Thanks to their innovative device architecture, they offer higher energy and power densities than SC and LiB. This structure consists of integrated SC cathodes (Activated carbon), LiB anodes (Hard carbon), and Li-ion electrolytes. However, the charge storage mechanisms of these electrodes are different, which makes interfacial reactions with the electrolyte more complex. In particular, the activated carbon (AC) has surface oxygen functionalities and defects on its high surface area, which can serve as active sites for accelerated electrolyte decomposition. Moreover, the performance of the LiC containing the AC can be influenced by confined moisture in the AC pores, the sensitivity of the commonly used $LiPF_6$ salt, and the film-forming abilities of the ethylene carbonate solvent. During my presentation, I will share the results of a spectroscopic analysis of the changes at the electrode/electrolyte interface before and after different stages of electrochemical cycling. I will also explain the mechanism behind the capacity fade. Understanding this mechanism is essential for developing high-performance LiC that can match the cycle life of SC.

Andrea Sorrentino, Beamline Scientist BL09 MISTRAL Soft X-Ray Microscopy Beamline, ALBA, Spain

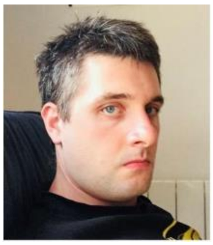
Visualizing electrodes chemical states at the nanometer scale using Soft X-ray Transmission Microscopy

In many applications the precise knowledge of composition and morphology of materials is a key information to control performance and reliability. This is especially true for battery materials: detailed information of elements distribution and particles morphology of the discharge products on the electrode as a function of the charging state are crucial for the identification and the understanding of the involved processes. Energy resolved full field transmission soft X-ray microscopy (TXM), is able to give a full picture at the nanometer scale of the chemical state and spatial distribution of many interesting elements, providing pixel-by-pixel absorption spectrum. This technique can be successfully performed at the Mistral beamline of the ALBA light source where tomography may complement the 2D chemical information with a 3D morphological description of the discharged products. In this work, after a brief description of the beamline and the available techniques, we will present some example of application from studies we have performed on Li/O_2 and Na/O_2 batteries and Lithium rich cathodes.

Per Erik Vullum, Senior Research Scientist in SINTEF & Associate Adjunct Professor at the Dept. of Physics, NTNU.

Microscopy characterization of Li-ion battery materials, including degradation mechanisms as a function of cycling

The functional properties of Li-ion batteries and the active materials in each of the electrodes heavily depend on the size, morphology, chemical composition, and crystal structures of the components. In the present talk it will be shown how transmission electron microscopy can be used to characterize and unveil the properties and some of the degradation mechanisms that might occur during battery cycling. Examples will be given from LNMO and NMC811 cathode materials, and from anodes based on a Si-graphite composite.



Stefano Checchia, Beamline Scientist ID15A- Materials Chemistry and Materials Engineering, ESRF, France

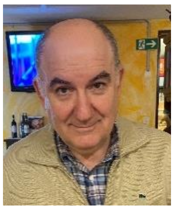
Understanding battery performance and degradation by means of spatially-resolved operando diffraction

With increasing energy demands and the planned electrification of many industrial sectors, improving the performance and safety of Li-ion batteries during high-rate operation has become a necessity. Here we show the contribution of high-energy X-ray diffraction to understanding battery degradation issues induced by fast charging/discharging and repeated cycling. In particular, we present the high-speed, high-resolution methods used at beamline ID15A at ESRF as well as technical developments for improved future capabilities. Structural information obtained with high spatial and temporal resolution during charge/discharge cycling can detect dynamic chemical and morphological inhomogeneities within batteries, and help explaining performance loss and, potentially, failure. Loss of capacity, for example, can originate from Li concentration gradients through the depth of anodes, leading to irreversible Li plating/stripping, but also from dissolution of d-metal ions from defective cathode particles upon repeated cycling. Determination of state of charge, strain and temperature inside 18650 cells through X-ray diffraction-tomography (XRD-CT) can reveal the pathways of heat accumulation for fast-charging protocols and in damaged batteries. The real-time, microscopic quantifications within these studies not only highlight the challenges faced by high energy density cells, but can also inform more accurate computational models and help guide thermal management strategies for high-rate applications.

Ana García Prieto, Beamline Scientist BM25-SpLine X-Ray Scattering and X-ray Photoelectron Spectroscopy Beamline, ESRF, France

Spanish CRG BM25-SpLine beamline at the ESRF, The European Synchrotron: Present opportunities for the characterization of Li-ion batteries

The Spanish CRG beamline (BM25-SpLine) at the European Synchrotron (ESRF) enables the characterization of different materials especially in the area of X-ray scattering and X-ray spectroscopy. BM25-SpLine accounts with three different end-stations. The first end-station is equipped with a six-circle diffractometer and a two-dimensional detector which can collect high-resolution diffraction data at relatively short acquisition time ranges. The available sample environment has been designed to characterize materials constituted by different elements such as Li-ion batteries (electrodes, electrolytes and interfaces) in multiple conditions (heating, cooling, gas atmosphere) and under working conditions. XRD can be combined with X-ray Absorption Spectroscopy to achieve a complete study of the complicated chemical and physical mechanisms occurring in the Li-ion batteries. At the second focal point, a unique end-station which combines Grazing Incidence X-ray diffraction (GI-XRD) with hard X-ray photoelectron spectroscopy (HAXPES) is present. This special instrumental configuration offers the possibility to correlate the atomic structure (crystallinity) with its compositional, chemical and electronic properties under the same experimental conditions. Variation of the analysis depth by photon energy tuning allows to access from surface electrode to buried interfaces present in Li-ion batteries. The third end-station, which is in commissioning phase, is devoted to Single Crystal Diffraction.



Javier Campo, Scientific Researcher at the Institute of Nanoscience and Materials of Aragón (CSIC – University of Zaragoza)

Neutron scattering: basics concepts and examples related with

Neutron scattering techniques can provide fundamental insight into the different behaviors shown by matter. In this lecture, a short introduction of the properties of the neutron–matter interactions and the fundamentals of neutron scattering will be presented in order to facilitate an understanding of the peculiarities of this probe. Also, the different modalities the scientists have to access to these interesting techniques will be introduced. Then, selected examples will be presented of the use of different neutron scattering techniques on very different problems related with Li ion batteries and other materials related with the energy challenge. Li ion batteries

Markus Strobl, Leader Applied Materials, Paul Scherrer Institute, Switzerland

Neutron Imaging applications in battery research at the Swiss Spallation Neutron Source (SINQ)

Jacob LaManna, Instrument Scientist BT-2 Thermal Neutron Imaging Instrument, NIST, USA

Using neutrons to understand electrochemical system performance and degradation

The current push towards green energy and high-powered portable electronics have put a large demand on battery and fuel cell technology. To produce the most robust energy systems, it is necessary to understand the transport mechanics and degradation modes within them. Neutrons provide a nondestructive penetrating probe that can peer into commercially relevant battery and fuel cell systems while having strong sensitivity to electrochemical materials such as lithium and hydrogen. Compared to X-rays, neutrons are able to penetrate most engineering metals, such as aluminum or steel, while remaining sensitive to the light elements of lithium and hydrogen. Operando neutron imaging experiments can track electrochemical species from electrode to electrode in real time. To get a more complete understanding of a sample it is possible to combine neutron tomography with X-ray tomography as X-rays enhance information on solid phases, such as mechanical damage from battery cycling. Performing simultaneous neutron and X-ray tomography allows direct correlation of the two modalities and enhances the segmentation process of the 3D reconstructed volumes. This talk will give an overview of multiple neutron imaging techniques that are relevant to electrochemistry including Bragg-edge imaging to track material strain, far-field interferometry that probes nano-scale structure below the typical resolution limit, and simultaneous neutron and X-ray tomography for improved system characterization.

Marit Kjarvik, R&D Engineer, Vianode, Norway

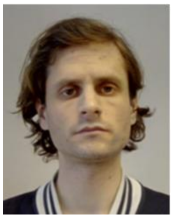
From scrap to battery grade graphite- Using X-ray based techniques as a characterization tool in the recycling process

Graphite is used as anode material in lithium-ion batteries, where it makes up 10 to 20 wt% of the total mass of a battery cell. This adds up to approximately 70 kg in an average electric vehicle. To reduce the environmental impact associated with lithium-ion batteries, recycling of end-of-life batteries and production scrap is of uttermost importance. To develop and understand the recycling process of graphite, we need reliable methods to trace metal impurities and other material characteristics- from the starting material to the final product. In this talk, I'll discuss how T-XRF and XRD are used to characterize and understand graphite, with an emphasis on recycled material- and how this relates to the performance of a lithium ion battery. This is further linked to electrochemical testing, density, surface area, degree of graphitization and particle size distribution, which are key properties to show that the scrap material has been regenerated into battery grade graphite.

Angel Larrea, Scientific Researcher at the Institute of Nanoscience and Materials of Aragón (CSIC – University of Zaragoza)

Operando analysis with spatial and time resolution of lead cells using gauge-volume neutron diffraction

Non-destructive studies of lead batteries composed of commercial thick electrodes have been carried out using neutron diffraction techniques. The analysis focused on the positive electrode, where the present limitations for stationary applications are mainly originated. The experiments were performed in the VULCAN instrument of the Spallation Neutron Source, at Oak Ridge National Laboratory (Tennessee, USA). VULCAN is a high brightness time-of- flight diffractometer equipped with a set of collimators and controlled slits that allow to carry out operando studies with both spatial and time resolutions. In this way, we have determined the absolute molar composition of the positive actives mass in different static and dynamic states, when charging and discharging the cells. The evolution of the active mass has also been correlated with the effective electric charge provided or supplied to the cell during cycling, providing information about the efficiency of the PbO_2 $PbSO_4$ conversion process.



Kenneth Marshall, Postdoctoral Researcher BM31, Swiss-Norwegian Beamline, ESRF, France

BM31: the presentation

BM31 is part of the Swiss-Norwegian beamlines specializing in combined X-ray diffraction and X-ray absorption spectroscopy for *in situ* and operando experiments. Recently we have added X-ray total scattering/ pair distribution function to our techniques which can now also be done in combined experiments. Here I will present our current capabilities and an overview of some of our selected publications in the battery field.