EMPIR Call 2019 – Energy, Environment, Normative and Research Potential

Selected Research Topic number: **SRT-g10**
Version: 1.0

Title: In operando metrology for energy storage materials

Abstract

The European battery and automotive industries are facing strong competition in the growing global market for high capacity energy storage technologies for use in electric vehicles, portable devices and smart-grid stabilisation. The leading technology is Li ion batteries, the development of which has advanced significantly in recent years. However, costs are still too high and the capacity and lifetime too poor for many important applications. To accelerate innovation by materials developers and device manufacturers, new metrology is urgently required. This SRT calls for the development of *in operando* techniques, supported by standardised *ex-situ* analysis and electro-chemical measurements, to enable beyond state-of-the-art materials characterisation.

Keywords

In operando metrology, vibrational spectroscopy, x-ray spectroscopy, impedance spectroscopy, mass spectrometry, energy storage materials, energy efficiency, Li batteries, all solid-state batteries, super capacitors

Background to the Metrological Challenges

Global energy demand is constantly rising, and there is a need to balance energy security, stability and sustainability, whilst meeting international emissions targets and adapting to innovations in technology. These factors have created an urgent need for establishing energy storage development and manufacturing capability within Europe in order compete on the global scale, as indicated by various national investment initiatives.

The state-of-the-art energy storage technology for many applications is the lithium ion battery (LIB). However, the LIB has intrinsic limitations in terms of practical energy densities and cycle life, materials costs and sustainability. Despite materials science advances, some systems suffer from degradation effects leading to capacity fading. The chemistry of the solid-electrolyte interface is poorly understood, and its characterisation remains a key challenge. Moreover, the electrode materials and electrolytes themselves can undergo irreversible chemical and structural changes resulting in capacity fade, and these processes are difficult to characterise by post mortem *ex-situ* analysis due to their sensitivity to the specific electrochemical environment. Therefore, new *in operando* techniques are required to allow characterisation of battery materials *during* charge and discharge.

Current *in operando* spectroscopy methods are immature and lack the reliability to confidently correlate materials property changes to performance. Little attention has been paid to: (i) whether the *in operando* electro-chemical cell is representative of a real operating device, and what constitutes a “standard cell”; (ii) the effect of beam damage or localised heating/photochemical phenomena; (iii) the influence of discharge rate and other measurement parameters on the fidelity of the spectroscopic data; (iv) sample-to-sample variability. These challenges will be addressed by establishing best practice in such techniques.

EIS is a powerful method for probing electrochemical processes at a range of timescales, however, it suffers the drawback of being notoriously difficult to interpret. Confident analysis requires correlation of data with known physical and chemical processes. Devising improved measurement protocols, based on galvanostatic conditions and generating new knowledge by combining EIS with *in operando* methods, is a possible solution, thus establishing an innovative framework for electrode materials discovery and optimisation.

Finally, *ex-situ* methods play an important role in characterising battery materials as well as validating the interpretation of *in operando* data. However, many of the methods currently employed have not been optimised or standardised for this application. High-resolution chemical imaging can provide valuable mechanistic understanding but preserving the battery microstructure and chemical state information is a significant challenge. This can be addressed by establishing standards of battery materials for analytical techniques such as XPS (X-Ray photoelectron spectrometry) and secondary ion mass spectrometry (SIMS), including the development of suitable reference materials and calibration samples for reliable quantification, and a cryogenic method for preserving the native state.

Objectives

Proposers should address the objectives stated below, which are based on the PRT submissions. Proposers may identify amendments to the objectives or choose to address a subset of them in order to maximise the overall impact, or address budgetary or scientific / technical constraints, but the reasons for this should be clearly stated in the protocol.

The JRP shall focus on the development of *in operando* techniques, supported by standardised *ex-situ* analysis and electro-chemical measurements, to enable beyond state-of-the-art materials characterisation for high capacity energy storage technologies.

The specific objectives are

1. To develop novel *in operando* techniques and instrumentation for multi-parameter characterisation of high capacity energy storage materials and components.
2. To establish Best Practice Guides for emerging *in operando* spectroscopy methods including x-ray spectrometry and vibrational spectroscopy, in order to improve experimental accuracy and repeatability in correlating material property changes to battery performance.
3. To develop a novel experimental approach based on electrochemical impedance spectroscopy (EIS) combined with *in operando* spectroscopy for the correlative assessment of material structure-performance relationships.
4. To develop traceable chemical and structural analysis methods for *ex-situ* characterisation of high capacity energy storage materials in order to validate the findings of *in operando* characterisation techniques. This includes the development of measurement standards, fabrication of calibration samples and reference materials and their verification by interlaboratory studies.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (NMIs, calibration laboratories), standards developing organisations (e.g. ISO/TC 201) and end users (material suppliers, battery manufacturers).

These objectives will require large-scale approaches that are beyond the capabilities of single National Metrology Institutes and Designated Institutes. To enhance the impact of the research, the involvement of the appropriate user community such as industry, standardisation and regulatory bodies is strongly recommended, both prior to and during methodology development.

Proposers should establish the current state of the art and explain how their proposed project goes beyond this.

EURAMET expects the average EU Contribution for the selected JRPs in this TP to be 2.0 M€ and has defined an upper limit of 2.3 M€ for this project.

EURAMET also expects the EU Contribution to the external funded partners to not exceed 35 % of the total EU Contribution across all selected projects in this TP.

Potential Impact

Proposals must demonstrate adequate and appropriate participation/links to the “end user” community, describing how the project partners will engage with relevant communities during the project to facilitate knowledge transfer and accelerate the uptake of project outputs. Evidence of support from the “end user” community (e.g. letters of support) is also encouraged.

You should detail how your JRP results are going to:

- Address the SRT objectives and deliver solutions to the documented needs,
- Feed into the development of urgent documentary standards through appropriate standards bodies,
- Transfer knowledge to the materials developers and device manufacturing sector.

You should detail other impacts of your proposed JRP as specified in the document “Guide 4: Writing Joint Research Projects (JRPs)”

You should also detail how your approach to realising the objectives will further the aim of EMPIR to develop a coherent approach at the European level in the field of metrology and include the best available contributions from across the metrology community. Specifically, the opportunities for:

- improvement of the efficiency of use of available resources to better meet metrological needs and to assure the traceability of national standards
- the metrology capacity of EURAMET Member States whose metrology programmes are at an early stage of development to be increased
- organisations other than NMIs and DIs to be involved in the work.

Time-scale

The project should be of up to 3 years duration.