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

Selected Research Topic number: **SRT-g19**
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# Title: Ageing and Fatigue of Energy Materials

## Abstract

Ageing and fatigue of solar cells, batteries, and power electronics is energetically, environmentally and financially relevant. These devices are prone to ageing mechanisms that can be addressed with a set of complementary tools based on: X-ray and optical spectroscopy, electrical and near-field optical scanning probe microscopy (SPM), transmission electron microscopy (TEM), and electrical characterisation. Due to the complexity of most energy materials, it is often not possible to unambiguously interpret the experimental results. Proposals addressing this SRT should contribute to better understanding and clear interpretation of the measurement results by applying multiphysics modelling, as well as *in situ* and *in operando* ageing and fatigue tests.

## Keywords

Ageing, fatigue, solar cell, battery, X-ray spectroscopy, X-ray diffraction, coherent/confocal Raman spectroscopy, spectroscopic signatures, scanning probe microscopy (SPM), scanning electron microscope (SEM), focused ion beam (FIB), transmission electron microscopy (TEM)

## Background to the Metrological Challenges

The key prerequisite for understanding and potentially preventing degradation of device performance (such as efficiencies of solar cells) is to understand the causes of degradation on different scales. Very small structural changes caused by aging may already have significant negative influence on the electronic or optical properties of the device and hence on the device efficiency. Therefore, firstly, it is essential to characterise and understand correlations of structural and chemical changes with changes of opto-electronic properties by combining a suitable set of measurements. Optical methods are able to provide information about what device properties are deteriorating, whereas structural or chemical characterisation methods provide information about the causes of this deterioration. Secondly, an absolute quantification with known uncertainties is needed to reliably determine these correlations experimentally.

X-ray spectroscopy and diffraction provide a possible means for probing and identifying irreversible structural, coordination or speciation changes in a system thus allowing for the identification of potential lifetime limiting factors. Furthermore, the measurements can be performed with a spatial resolution on a micro- or nanometre scale. Factors detrimental to a stable and long-term device operation can be identified beforehand on the material level such that aging factors can be predicted and possibly prevented.

Coherent Raman spectroscopy, and in particular the coherent Raman microscopy allows the accurate characterisation of photosensitive material because Raman spectra of crystalline and microcrystalline structures have well-defined bands (frequency and width), while the presence of defects/impurities drastically changes the Raman spectrum. In general, optical methods (reflectometry, ellipsometry, Raman, UV to IR spectroscopy) have the advantage of being fast and easy to implement, and are therefore scalable into production and on-site device monitoring. The challenges with these methods are their complex data analysis schemes. Therefore, complementary methods, such as hyperspectral measurements, imaging, and multi-method data analysis, can improve the measurement capabilities in future ageing detection.

Even though the combination of measurement methods will provide comprehensive insights on the aging mechanisms, due to their complexity a detailed understanding of these mechanisms based on experimental data alone is often not possible. While measurements may reveal structural or chemical changes in functional layers or at their interfaces and find a correlation with a simultaneous deterioration of the optical properties and device performance, modelling has the potential to provide an explanation as to why these changes take place and why they are detrimental for the device performance. Modelling of ageing and fatigue in batteries, solar cells and power electronic components has already been performed and was partly linked to *in operando* data. The approach to use *in operando* data for modelling and for finding fundamental mechanisms of ageing and fatigue is well established. The next step, to quantitatively correlate *in operando* data with modelling data, is yet to be done. Developing tools able to follow degradation processes in energy materials and devices is essential for improving the quantification and understanding of their ageing and fatigue.

## 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 traceable measurement and characterisation of degradation processes in energy materials.

The specific objectives are

1. To develop X-ray spectroscopy and X-ray diffraction techniques for traceable and time resolved characterisation of materials including applying of different stimuli without the necessity of calibration samples, conducting integral characterisations (e.g. DC-IV curves), modelling physical and chemical changes and correlating these with integral characteristics. In addition, to apply these techniques to various applications (e.g. power electronic components, solar cell absorber layers and battery electrodes).
2. To develop hyperspectral methods for optical spectroscopic ageing detection with spatial resolution better than 1 µm. In addition, to characterise defects propagation using various methods (e.g. optical ageing, coherent and confocal Raman spectroscopy) and to calculate the spectroscopic signatures of defects with *ab-initio* and semi-empirical models. In addition, to apply these methods to various applications (e.g. solar cells and battery electrodes).
3. To develop scanning probe microscopy methods to measure local physical properties (elemental concentration $10^{15}$-$10^{19}$ atoms/cm$^3$, thermal conductivity, resistivity $10^{-2}$-$10^{5}$ Ωcm, capacity 10 aF-10 fF, relative dielectric constant 3-300) at frequencies from DC to infrared light up to millimetre sized scan ranges with nanometre resolution. In addition, to apply these methods to various applications (e.g. battery electrodes, power electronic components and solar cells).
4. To develop a methodology for microscopic and sub-micron characterisation of temperature change induced or piezo-electrically induced stresses and crack formation. This should include designing advanced sample preparation by focused ion beam (FIB) technique in the scanning electron microscope (SEM) for *in situ* experiments and *in operando* experiments coupled with *in situ* fatigue straining tests (force measurement up to 360 µN) in the SEM (resolution 100 µm-100 nm). In addition, to study undamaged and aged/fatigued specimens by mesoscale TEM (resolution 10 µm-10 nm) and calculate defect formation energies and its precise geometric deformation, and to apply this methodology to various applications (e.g. solar cells, power electronic components and battery electrodes).
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) and end-users (e.g. power electronics industry).

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. In particular, proposers should outline the achievements of the EMPIR project 16ENG06 ADVENT and how their proposal will build on those.

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 energy materials 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.