Operando small-angle x-ray scattering analysis of nanostructured materials for energy storage

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Small-Angle X-ray Scattering (SAXS) is an essential, non-destructive analytical technique utilized to investigate the nanoscale structure of materials, typically in the range of 1 to 100 nm. By measuring the X-ray scattering intensity at very small angles, SAXS provides statistically averaged information regarding the size, shape, size distribution, and internal/external arrangement (structure factor) of nano-objects.

The technique's versatility is highlighted by its broad application across diverse fields:

- Biological Materials: determining the conformation of proteins, nucleic acids, and the structure of complex biological assemblies such as membranes and vesicles.
- Self-Assembled Materials: analyzing the kinetics and mesoscopic architectures of supramolecular polymers, block copolymers, and other self-organizing systems, often in real-time.
- Energy Storage Materials: characterizing porous electrode structures, tracking electrolyte/ion interactions, and monitoring structural evolution within batteries and supercapacitors.

This PhD research activity is an integral part of the European project 24GRD09 HyMetBat ("Hybrid metrology for sustainable and low-carbon footprint battery materials"). The project focuses on developing new metrological methodologies for high-performance and sustainable battery materials. The doctoral work will concentrate on the advanced structural characterization of nanostructured materials applied to Energy Storage. This includes electrode materials such as porous silicon and self-assembled porous templates designed for use in next-generation battery systems.

The core scientific goal is to study the interfacial interactions and the structural evolution of these materials inside operational energy storage devices—specifically in operando mode. Using SAXS and related X-ray scattering techniques, the PhD candidate will aim to:

- Quantify structural changes (e.g., lattice parameter/layer spacing dilation or contraction) induced by the insertion/extraction of ions during charging and discharging cycles.
- Determine the reorganization of the electrolyte and the adsorbed ion layer within the nanoporosity, a critical factor influencing power density and long-term stability.

The primary research activity will be conducted at the INRiM laboratories. The experimental work will leverage the capabilities of a recently installed, cutting-edge in-house SAXS instrument (Anton Paar SAXS Point 5.0). This system is optimized for high-resolution measurements and to perform *operando* analysis inside an electrochemical cell.

Furthermore, the PhD candidate will have the opportunity to collaborate with European Synchrotron facilities involved as partners in the HyMetBat project (i.e. BASSY II, ESRF, SOLEIL). Access to these large-scale research infrastructures will enable high-flux X-ray SAXS measurements, which are essential for achieving the optimal time resolution required for complex and fast operando experiments. This synergy between laboratory instrumentation and large-scale facilities will ensure comprehensive training and lead to high-impact results in the field of nanostructural metrology.