In situ and Operando XAS characterization of Functional Materials.

V. Briois
Synchrotron SOLEIL, ROCK beamline, L'Orme des Merisiers, Départementale 128, 91190 Saint-Aubin

X-ray Absorption Spectroscopy (XAS) is a well-known “workhorse” technique for *in situ* and *operando* characterization of functional materials, such as batteries or catalysts. *In situ* and *Operando* investigations both require the use of sample environments (an electrochemical cell, a furnace, a cryostat…) for measuring the materials under relevant reaction conditions. The high penetration depth of hard X-rays, such as those available at the BM08 beamline at SESAME synchrotron, is clearly an opportunity for the design of sample environments enabling the controlled exposure of the sample to the desired reaction conditions. However, strictly speaking, *in situ* and *operando* terms are not interchangeable. *Operando* experiments will simultaneously monitor the evolution of the structure and chemical state of the materials by XAS and measure its relevant activity, this one being the charge capacity or the catalytic activity if we still refer to electrode materials or to catalysts. *In situ* experiments will measure the material under reaction conditions without measurement of activity in the same experiment. The former will often require the recording of XAS data with a time resolution matching the dynamic change of the material under reaction conditions whereas the latter is often performed at steady-state conditions.

In this talk, typical sample environments for *in situ* and *operando* characterizations of battery electrode materials and catalysts will be presented, together with specific sample preparation required for good XAS measurements. With the aim of clarifying the difference between *in situ* and *operando* XAS characterizations, some examples will be discussed.