

Singularities in Testing the Activity and Stability of Fuel Cell Nanocatalysts Revealed by *In Situ* and *Operando* X-ray Diffraction

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Preliminary testing of fuel cell catalysts activity and stability are essential steps in the technology readiness level progression of material candidates toward the commercial device. However, in the case of platinum-based catalysts for the oxygen reduction reaction (ORR) in proton exchange membrane fuel cells (PEMFCs), there is no strict consensus on a testing protocol, as numerous parameters such as electrode potential window, potential sweep rate and the presence/frequency of intermediate cyclic voltammogram characterization which define the so-called electrochemical activation (or break-in) and the accelerated stress tests (ASTs) remain mostly arbitrarily chosen in the literature.

This presentation will highlight the importance of such parameters on the conclusions one can obtain while testing the activity and stability of PEMFC nanocatalysts. The discussion will be notably based on the interpretation of both *in situ* and *operando* X-ray diffraction data collected on Pt-based catalysts both in simulated or real PEMFC cathode environment, which reveal the sensitivity of Pt oxidation dynamics, strain relaxation and coherent domain size increase to the testing procedure^{1,2}.

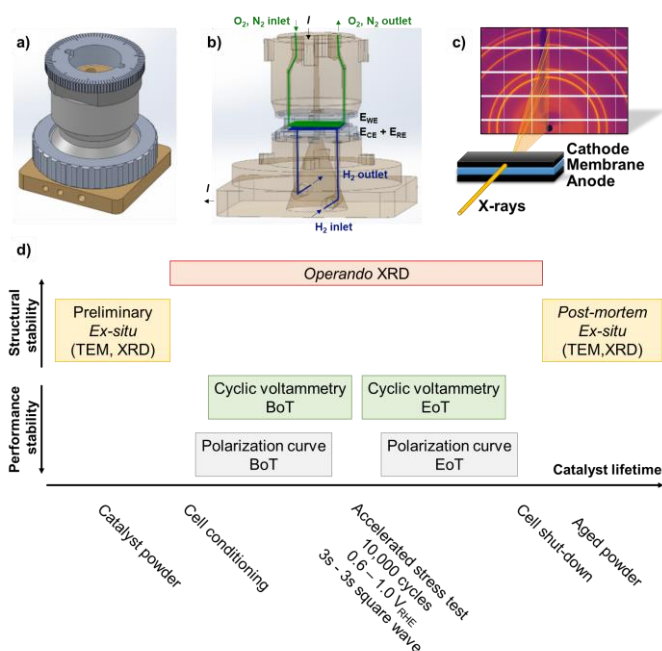


Figure: Experimental setup and protocol. a-c) X-ray transparent PEMFC for *operando* X-ray diffraction; d) timeline of the different experimental steps and characterization tools used during the catalyst lifetime.

- (1) Chattot, R.; Roiron, C.; Kumar, K.; Martin, V.; Campos Roldan, C. A.; Mirolo, M.; Martens, I.; Castanheira, L.; Viola, A.; Bacabe, R.; Cavaliere, S.; Blanchard, P.; Dubau, L.; Maillard, F.; Drnec, J. *ACS Catal.* **2022**, *12* (24), 15675–15685.
- (2) Chattot, R.; Mirolo, M.; Martens, I.; Kumar, K.; Martin, V.; Gasmi, A.; Dubau, L.; Maillard, F.; Castanheira, L.; Drnec, J. *J. Power Sources* **2023**, *555*, 232345.