



## A Unitized Reversible Fuel Cell For In Operando Sxas and Xas Analysis for Fuel Cells and Electrolyzers

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### Abstract

**Introduction:** Producing efficient and cost-effective fuel cell systems and water electrolyzers is then fundamental for promoting the spread of green hydrogen generation and use. In this scenario, fuel cell technology remarkably improved in the last decades, promoting Proton Exchange Membrane Fuel Cells (PEMFCs) use in the automotive and in transportation sectors. Nonetheless, the complex architecture of fuel cells and electrolyzers, still slows down the research and development required for improving their efficiency and lifetime, and contemporary reducing their production costs, which are still strongly bounded to the cost of catalyst materials[1]. Important achievements in optimizing fuel cell architecture and/or catalyst loading, were obtained thanks to analysis carried out in-operando analysis carried out at synchrotron radiation facilities, where important insights about fuel cell operation and degradation were revealed. In particular, Small Angle X-Ray Scattering (SAXS) is suitable for revealing catalyst morphological evolution [2], while X-Ray Absorption Spectroscopy (XAS) allows to depict reaction kinetics [3] and to quantify the degree of catalyst oxidation [4].

**Objectives:** The aim of this work consisted in developing an electrochemical cell able to operate as reversible unified electrochemical cell (RUEC), optimized for studying chemical and morphological evolution of catalyst materials designed for PEMFCs, for PEM Water Electrolyzers (PEMWEs), and for RUECs to be studied by combining electrochemical characterization with SAXS and XAS.

**Material and methods:** Two different Membrane Electrode Assemblies (MEAs) for PEMFCs, loaded with commercial catalyst materials composed by Pt and Pt<sub>3</sub>Co nanoparticles were

characterized in pristine conditions at first; then catalyst evolution undergoing break-in procedure and subjected to Accelerated Stress Tests (AST) were monitored by means of in operando XAS and in operando SAXS. The analysis was then repeated on a commercial Ir/Pt catalyst for a PEMWE. All of the results were complemented with electrochemical analysis (cyclic voltammetry and impedance spectroscopy).

**Results:** Concerning the in-operando investigation of the Pt-loaded-MEA for PEMFCs, most of the reduction of the catalyst electro-activity of Pt was observed within the first 2000 cycles, in agreement with results from electrochemical data and with the recorded evolution of the particle size distribution, where an increase in particle size and agglomeration were revealed. When the Pt<sub>3</sub>Co-loaded-MEA for PEMFCs was analysed, most of the chemical evolution was detected during the break-in procedure, and a less pronounced coarsening was detected when running AST.

**Conclusions:** This work confirmed the effectiveness of properly designed electrochemical cell was demonstrated being able to reliably measure and combine electrochemical data, XAS spectra, and SAXS patterns, allowing to perform a comprehensive and deep characterization of catalyst performance undergoing the different operational regimes of PEMFCs and PEMWEs. Additionally, this work defines a benchmark for future studies of novel catalyst materials for either PEMFCs and PEMWEs.

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